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HelioxTL System

(Generic System Manual)

Supplied to: Dr Michael Zudov

Project Number

Please quote this number whenever you contact the factory.

Description of Low loss Dewar is only reference in the manual.

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File reference

OXFORD
INSTRUMENTS

Contents

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.

1. Introduction

1.1. How to use this manual

Each of the main sections in the manual is separated from the others by a divider card. Use the divider card index which you see when you open the front cover or the table of contents to find the section that you want to read, quickly and easily.

Some diagrams are in a separate section. A few of these diagrams are folded so that you can pull them out and see them while reading the text which refers to them.

Additional manuals may be provided with your system to describe the details of some of the component parts. In particular, most electronic equipment is supplied complete with a manual, and you may need to refer to these separate documents to find out how to carry out some of the operations if you are not familiar with the equipment.

1.1.1. Safety

Warning: **It is your responsibility to ensure your own safety, and the safety of the people working around you.**

Cryogenic fluids and high magnetic fields are potentially hazardous, and you must take precautions to ensure your own safety. The Oxford Instruments booklet *Safety Matters* has been included in this manual. It contains essential information and detailed recommendations about the precautions that you should take. Any additional information that applies specifically to your system is provided separately in the 'Safety' section of this manual.

1.1.2. Warnings

Please read this manual before assembling or commissioning the system. If you do not follow the correct procedures you might be injured or you might damage the system beyond repair. Oxford Instruments cannot accept responsibility for damage to the system caused by failure to observe the correct procedures laid down in this manual.

1.2. Stray Magnetic Field and Siting Issues

The effects of stray magnetic fields on system performance and the environment can often require complex finite element modeling. The following information is provided as a guideline only.

1.2.1. Static Steel

The presence of steel in close proximity to the magnet can cause two possible problems;

1. Excess force on the cryostat components leading to damage to the magnet support system or poor cryogenic performance.

2. Perturbations of the magnetic field leading to poor homogeneity.

In order to avoid these problems it is necessary to ensure that there is no structural steel within the 30 gauss field contour. This distance can be determined from the system stray field plot

Items such as steel beams or pillars and concrete reinforcing can cause problems, in particular any nonsymmetric steel in the structure.

It is possible that steel in the building will become magnetised and cause areas of increased field at some distance from the system, in particular steel beams may cause increased field areas in adjacent rooms that may affect VDU's.

1.2.2.   Moving Steel

The effect of moving steel can be the same as detailed above with the added problem of field disturbances that may be visible in experimental results. Objects such as vehicles or elevators should be well outside the 1 gauss contour. Large steel equipment, such as gas bottles or pallet trucks, should be kept well outside the 10 gauss contour, movement of this equipment should be controlled as even at this distance effects may be observed. These distances can be determined from the system stray field plot. The relative position and steel content of doors and windows should also be determined.

1.2.3.   Approximations to force calculation

The force between the magnet and a ferromagnetic object can be approximated if the magnetic field gradient is known. The critical limit is = 500 G/m; any iron located on this line is exerting a force on the magnet equal to its mass. Any iron within the region bounded by this line is exerting a greater force which can be approximated as force = (mass of iron object) x (field gradient at that point) / 500; e.g. A 5kg iron mass centred on the 1000 G/m line will exert a force = 5 x 1000/500 = 10 kgf = approx. 100N. However, note that this is only a guideline.

Caution: If there is any significant amount of ferromagnetic material within 2 metres of magnet centre field, contact Oxford Instruments for advice.

1.2.4.   Field Limits for Devices (Unshielded Magnet Systems)

The following can affect the system and should be positioned outside of the following limits:

1 gauss
- Motor vehicles
- Elevators

10 gauss
- Large steel equipment

30 gauss
- Typical structural steel beams

The following will be affected by the magnetic field and should be outside of the following limits:

1 gauss
- Image intensifiers
- Electron microscopes
- Accurate measuring scales
- X-ray machines
- Graphics terminals

5 gauss
- Pacemakers
- Public access without warning signs
- Cathode ray tubes

10 gauss

- Computers
- Watches and clocks
- Credit cards

20 gauss

- Magnetic storage media

50 gauss

- Magnet power supply

1.3.   Other manuals supplied with the system

The following documents (marked 🗐) are also supplied with the system.

Practical Cryogenics	🗐
Introduction to thermometry below 1K	
ILM200 family of cryogen level meters	
ITC502 temperature controller	
ITC503 temperature controller	🗐
SMC4 stepper motor controller	
Auto needle valve and Auto GFS for ITC502 and ITC503	🗐
Data sheet for a calibrated cernox thermometer	🗐
RhFe reference scale 1.5 to 800K, for 3 point calibrated sensors (1985 revised to ITS-90)	
ObjectBench system control software	
DC-SQUID	
PHI-O RF-SQUID	

The following pages describe the system and give some information about the principle of operation. If you are experienced you may not need to read the Introduction in detail but you must read the warnings contained in it.

1.4.   Heliox TL inserts

Figure 1 shows the working parts of the Heliox insert. They are all surrounded by a vacuum chamber (IVC) to provide thermal isolation from the main liquid helium reservoir. The Heliox^{TL} insert must be mounted in a bath of liquid helium in a suitable cryostat.

The sorption pump, or sorb, will absorb ^{3}He gas when cooled below 40 K and the amount of gas that can be absorbed depends on its temperature. Below about 8 K its performance does not change significantly. It is cooled by drawing liquid helium from the main bath through a heat exchanger. The rate of flow of liquid can be controlled by a room temperature needle valve.

The 1 K pot is below the sorb. It is used to condense the ^{3}He gas during the condensation phase and to reduce the heat conducted to the sample space during low temperature operation. The 1 K pot is fed from the main bath through a needle valve. This valve can be set either to keep the pot running in continuous mode or to stop the incoming flow of liquid helium to achieve the lowest possible temperature during condensation. A copper cone in the ^{3}He space is used to locate the top loading probe.

The ^{3}He capacity of the system depends on sample space diameter. The recommended charge of ^{3}He is:

- For < 15 mm sample space inserts   See specifications and test results
- For 15 - 25 mm sample space inserts   10 litres NTP
- For 26 - 38 mm sample space inserts   15 litres NTP

Some parts of the bottom of the ^{3}He tail are usually made of copper to short out any thermal gradients in the liquid, because the thermal conductivity of the liquid is so poor. If the insert is to be used with a magnet whose field sweeps very rapidly, eddy current heating in this copper pot may cause an unacceptably high heat load and the copper components may not have been fitted. If the insert takes many hours to reach base temperature it is likely that thermal gradients have not been shorted out sufficiently well. This can be improved by using a few copper wires, top loaded with the sample to conduct heat from the bottom of the liquid ^{3}He to the surface. Very little power is dissipated in the ^{3}He by using this method because this is proportional to the fourth power of the diameter of the conductor.

1.4.1. Principle of operation of the ^{3}He insert

The sample is mounted on the top loading probe and loaded directly into the liquid ^{3}He through the central access of the insert. A vacuum lock is provided to allow the sample to be inserted and withdrawn, without losing any ^{3}He or allowing any air to enter the sample space.

Figure 1 Operating principle of a sorption pumped system

1.4.1.1. Low temperature operation

When the sample has been inserted, the sorb is warmed above 30 K so that the ^{3}He gas which it has absorbed is released into the sample space (see Figure 1). It condenses on the 1 K pot assembly and runs down to cool the sample. When all the gas has been allowed into the insert the 1 K pot needle valve is closed completely so that it cools to the lowest possible temperature and the pressure in the sample space should drop to approximately 40 mbar. At this stage the sample is surrounded by liquid ^{3}He at approximately 1.2 K.

The sorb is now cooled, and it begins to reduce the vapour pressure above the liquid ^{3}He, so the sample temperature drops. The minimum temperature that can be achieved in this type of cryostat is approximately 0.25 K with no experimental heat load.

1.4.1.2. High temperature operation

Temperatures as high as 100 K may be achieved by energising a heater on the sample holder, and the temperature is monitored by a suitable sensor. The temperature can be controlled using an ITC temperature controller. The sample is surrounded by ^{3}He as exchange gas, and a small flow of ^{4}He is maintained through the 1 K pot to provide cooling. This feature is not included in a standard system, but it may be fitted to most systems. It may compromise the base temperature of the system slightly because of the heavy current leads to the heater.

1.4.2. The ITC temperature controller

We recommend you to use an ITC502 or ITC503 temperature controller to control the system. It is possible to control the system in several ways.

The simplest form of control is to use a single range card to control the temperature of the sorb. This is used to control condensation of the ^{3}He and to give coarse temperature control by setting the temperature (and thus the pumping speed) of the sorb. However, the sample temperature tends to drift slowly; typically it drifts by 10 mK/hour.

The level of control can be improved by adding another pair of sensor interfaces. One of these is used to measure the sample temperature, giving digital readout of temperature with resolution of 1 mK. This sensor can be used to control the amount of heat supplied to the sorb heater, and thus to control its temperature (and pumping speed). In this way, the sample temperature can be maintained within ± 5 mK until the ^{3}He has all evaporated. Even if the heat load from the experiment changes, the temperature controller will attempt to maintain the same sample temperature.

The other sensor interface can be used either to monitor the temperature of the 1 K pot, or to control the sample temperature in the range 1.2 to 80 K.

1.5.   Heliox top loading probes

The top loading probe is used to load the sample into the ^{3}He, and it stays in the insert while the experiment is carried out. It is fitted with a vacuum lock to prevent air entering or ^{3}He escaping from the system.

One end of the probe is at room temperature; the other may be at any temperature between 0.3 K and 100 K when the system is running. The probe is designed to give a very high degree of thermal isolation from room temperature, since a heat load of the order of 10 μW may be sufficient to affect the base temperature. The wiring and the other experimental services are mounted on the probe.

The sample is mounted on a block at the end of the probe using M3 screws. The sample block may be fitted with a temperature sensor to control the sample temperature using an ITC temperature controller. If the system is intended to run at temperatures above 1.2 K, a heater is wound on this block.

1.6.   HelioxTL external gas handling systems

The Oxford Instruments ^{3}He gas handling system includes a storage dump vessel and all the necessary valves and interconnecting pipework for handling the valuable ^{3}He gas.

The dump vessel is designed to store the gas at below atmospheric pressure so that in the event of a leak into the system the gas does not escape. Any air that enters may be removed from the gas as the system is warmed up.

The amount of gas in the dump vessel can be deuced from the value indicated by the pressure gauge. When the ^{3}He gas has been removed from the insert, most of it should be stored in the dump vessel. Then the interconnecting lines should be evacuated with the cryopump, which may then be closed off and warmed to room temperature.

Warning: **The cryopump must only be used to store the gas left in the connecting lines as described in the section about warming up the system. It may be dangerous to store larger volumes of gas in the cryopump.**

1.6.1.   Auxiliary gas handling systems

Pumping systems are required for the 1 K pot and the high vacuum spaces in the cryostat. The recommended system is shown in the drawings section of this manual.

1.7.   Low loss dewars

The dewar supplied with your system is liquid nitrogen shielded.

This means that there is a liquid nitrogen reservoir which cools a radiation shield surrounding the liquid helium reservoir. It is also linked to the neck of the helium reservoir to reduce the amount of heat conducted into the liquid helium. This can be especially useful if the neck has to be very short.

The dewar is vacuum insulated. The outer vacuum chamber (OVC) of the dewar will be fitted with a large diameter pressure relief valve at the base (or side) of the dewar. This ensures that it is not possible to build up a dangerously high pressure in the OVC.

Warning: **Do not tamper with this safety device or attempt to modify it.**

Caution: Avoid venting the vacuum in the OVC if possible. If you have to vent the OVC take the following precautions:

- Only vent the OVC when the dewar is completely warm
- Make sure that the helium reservoir has already been vented to atmospheric pressure
- Vent it very slowly to avoid any risk of collapsing the helium reservoir or moving the superinsulation.

1.8.   Fischer electrical connectors

High quality Fischer electrical connectors are used on most systems. These connectors have a self-locking mechanism to prevent the connection being accidentally broken if the cable is pulled.

Caution: Do not attempt to remove the connector by unscrewing the knurled black nut, as the wiring may be damaged. It is also likely that the nut maintains compression of a vacuum seal between the hermetic connector and the cryostat and that air will be admitted to a vacuum space.

To remove the Fischer connector from its mating part on the cryostat it is important to pull the correct piece. You will notice that part of the outside of the connector seems to be loose on the body of the connector. This is the locking mechanism. Pull this part away from the mating connector to break the connection. However, if you try to pull the connector out using the cable or another part of the body the connector and its mating part will remain locked together.

2. Safety

The following safety information is included. It is important that you read it.

Safety Matters   Safety2.DOC

Special safety information for your system.   Included in 'Safety Matters'

3.   Assembly and thermometry

3.1.   Unpacking the system

The system should be unpacked carefully and inspected for any damage that may have been caused during shipment from Oxford Instruments. It should also be checked to ensure that none of the components are missing. If any problems are encountered you should contact Oxford Instruments (through our agent or subsidiary if appropriate).

The dewar and other parts may be fitted with internal packing to prevent movement of the inner parts during shipment. If so, it will have a label on the outside to warn you, and to explain what has to be done to remove it. Keep these instructions and the packing in case you need to transport the system again in future.

Warning: **Inspect any safety critical equipment (such as the relief valves and lifting eyes) prior to assembly. If any of this equipment shows sign of damage please contact Oxford Instruments, Superconductivity, Customer Support before assembling the system.**

3.2.   Commissioning requirements for cryogenic systems

If you are planning to install a laboratory scale cryogenic system you are likely to need most of the following equipment. Some of it may be supplied with the system; other items may only be needed occasionally. If your system contains a superconducting magnet, ^{3}He refrigerator or dilution refrigerator there are additional requirements, and these are listed separately.

3.2.1.   Safety equipment

- Personnel protection equipment including gloves and goggles
- Hazard warning signs to make sure that anyone approaching the system is aware of the potential hazards

3.2.2.   Tools

- Spanners or wrenches (open ended metric set). 5 to 19 mm
- Allen keys (metric set) 1.5 to 12 mm
- Screw drivers, pliers, side cutters etc.
- Hot air gun
- Electrical soldering iron
- Digital multimeter (with low current ohms range).

3.2.3.   Lifting equipment

- Suitable method of lifting the system from the delivery vehicle
- Suitable hoist or crane for use in the laboratory
- Lifting sling and shackles to suit the lifting points on the system

If you do not have access to lifting equipment above the position where you run the system you can use a trolley to transport the system to the hoist. It may be necessary to remove the system from the trolley when you are running it.

3.2.4. Vacuum equipment

- High vacuum pumping system to evacuate the insulating vacuum spaces, including a diffusion or turbomolecular pump and a liquid nitrogen cooled trap, flexible metal pumping lines for connection to the cryostat and a two stage backing pump. It should be capable of reaching a pressure of 10^{-6} mbar.
- A mass spectrometer leak detector system is required sometimes, especially when the system is commissioned, for routine leak testing operations.
- Oil mist filters fitted to all rotary pump exhausts.
- A range of vacuum fittings (ISO KF fittings (also known as NW or DN) are used as standard)

Caution: It is important to remember that turbo-molecular pumps have a low compression ratio for helium gas. Therefore you should always use a two stage rotary pump as a backing pump.

3.2.5. Cryogens and gas supplies

- Liquid nitrogen in a self pressurising dewar
- Liquid helium
- A supply of recovery grade helium gas with a regulator, at a pressure variable between 0 and approximately 1 bar gauge.

3.2.6. Consumables

- Roll of mylar adhesive tape
- Roll of aluminium adhesive tape
- Tube of vacuum grease
- Pair of cotton gloves for handling clean items
- 'Scotchbrite' or equivalent mild abrasive for polishing or removing old indium wire from joint faces.
- Metal polish and degreasing agent or solvent for general cleaning.
- Indium wire (1mm diameter)
- Rubber soccer ball bladders (2 needed).
- Assorted latex rubber and polythene tubing
- Fishing line or dental floss

3.2.7. Other equipment

- Helium transfer tube (or 'siphon')
- Level meters for cryogen reservoirs (if required) or a suitable 'dipstick'
- Suitable gas flow meters may be useful sometimes

3.3. Additional requirements for Heliox TL

In addition to the items listed above, HelioxTL systems typically have the following requirements.

3.3.1. Pumping system requirements

- ^{3}He gas handling system to store the gas while the cryostat is not in use, with valves to control the gas, and a cryopump to remove the gas from the insert when the system is warming up.
- Pumping system for the 1 K pot, (including a single or double stage rotary pump with a displacement of 16 m^3/hour or greater, and the valves and interconnecting lines).

Caution: It is important to note that a single stage rotary pump is not adequate to pump exchange gas out of the system effectively.

3.3.2. Electronic accessories.

- Oxford Instruments ITC temperature controller, (or an equivalent instrument) for monitoring the temperature of the sorb, supplying power to the sorb heater, and (if required), controlling the sorb temperature to maintain a steady sample temperature.
- ITC temperature controller, (or an equivalent instrument) to monitor the temperature of the sample, and control it, (if required).
- ITC temperature controller, (or an equivalent instrument) to monitor the temperature of the 1 K pot, using the Ruthenium Oxide resistor that is fitted.
- If very accurate thermometry is required it is necessary to obtain the relevant equipment to monitor the thermometer.

3.3.3. Other materials

- A charge of ^{3}He gas. (Note that it is not consumed during operation. It is recovered when the system is warmed up).

3.4. Assembling the HelioxTL

3.4.1. Unpacking

The insert has no packing bungs, and it is likely that there will be no indium in the IVC vacuum seal. Remove the IVC so that you can see the internal parts.

3.4.2. Assembling the insert

Fit the gate valve to the top of the sample space as shown on the general assembly drawing of the system.

Pump out the sample space to remove air and water from the sorb. You can warm the sorb carefully with a hot air gun if necessary but take care to avoid damaging the delicate wiring. Fit the IVC - some indium wire is supplied in the spares kit and full instructions are given in the *Background Information* section. Tape the pick up tubes to the outside of the IVC to ensure that they are not damaged as the insert is lowered into the dewar.

Warning **The eye bolts and lifting rods on the insert are not intended to support the weight of a magnet or cryostat. If these other items are to be lifted, the other eye bolts on the magnet support system or the cryostat should be used.**

The insert should now be loaded into the magnet support system (or main bath baffle set), and the alignment should be checked before loading this sub-assembly into the cryostat. When the fit has been checked, the assembly may be loaded into the cryostat without the insert if preferred. The insert is guided safely through the magnet support system baffles if it is loaded later.

Connect the ^{3}He gas handling system and the auxiliary pumping system as shown in the Drawings section of this manual. Pump the air out of the connecting lines through the 'Vent' port, but remember that the dump vessel and the cryopump may contain ^{3}He gas. Make sure that you do not pump the valuable gas away. Thoroughly check all the pumps, lines and fittings for air leaks.

Check the electrical wiring for continuity and isolation from ground. Details of the insert wiring are given later in this section. Pull the sample probe into the vacuum lock and mount it on the insert.

3.5. Assembling the dewar

3.5.1. Inspecting the cryostat

The cryostat is delivered in a packing crate. Examine the Tilt Watch and Shock Watch Indicators on the inside and outside of the cryostat box to determine whether the cryostat has been handled properly. If any are coloured, contact Oxford Instruments as internal damage may have occurred. All other components are in a separate packing case.

3.5.2. Removing the cryostat from the pallet

Caution: Take care to ensure the cryostat is not fixed to the packing before attempting to lift it.

Connect suitable lifting slings to the lifting points on top of the cryostat OVC. Use a winch to lift the cryostat from the packing crate, and support it so that it is safe for you to work underneath the cryostat. Remove the transit bung from the cryostat and replace it with the OVC drop off plate.

Caution: The packing bung must be retained and must be fitted if you need to move the equipment or return it to Oxford Instruments.

3.5.3. Loading the magnet into the cryostat

This cryostat has been supplied with the magnet in place and while it is possible to remove and replace the magnet we do not recommend that you do so.

If you want to remove the magnet from the cryostat please contact Oxford Instruments Customer Support before attempting this procedure.

3.5.4. Assembling lambda point refrigerator and its pumping system

The lambda point refrigerator is built into the cryostat and cannot be removed from it.

3.5.4.1. Pumping system

The pumping system for a lambda point refrigerator is shown on the diagram in the drawings section of this manual.

Caution: On B-T environment systems the pressure gauge shown on the diagram is a transducer. It is important that the transducer is positioned near the pump inlet, not near the cryostat, so that there is no risk of it getting too cold.

3.5.5. Assembling the other fittings onto the cryostat

3.5.5.1. Assembling the helium manifold

The manifold and other fittings are shown on the general assembly drawing. Clean and lightly grease the O-rings and carriers that will seal the manifold to the cryostat and assemble the manifold as shown on the general assembly drawing.

3.5.5.2. Fitting the non-return valve to one of the nitrogen ports

At least one of the liquid nitrogen vessel ports must be fitted with a non-return valve while the dewar is cold. This ensures that it is not possible for all three ports to become blocked by moisture condensed from the air. Even if two of the ports are accidentally blocked the nitrogen gas is free to escape through the non-return valve. One of these valves is normally fitted in the factory, using a tamper-proof screw.

3.5.6. Evacuating the OVC

Check the vacuum in the OVC before you fill the cryostat with cryogens.

Caution: Always use a liquid nitrogen cold trap between a diffusion pump and the cryostat. Keep it full of liquid nitrogen during operation of the pump. The OVC could be seriously contaminated by back streaming diffusion fluid is this is not done. If a turbo-molecular pump is used instead of a diffusion pump, a liquid nitrogen cold trap is not essential, but it does help you to remove condensable vapours.

3.5.6.1.   Checking the vacuum

If the cryostat is already under vacuum and you want to check the pressure in the outer vacuum chamber you can do so as follows. Pump the line up to the OVC valve with a diffusion pump (or turbo-molecular pump). Close off the diffusion pump and open the OVC valve. Read the pressure on a pressure gauge connected to the pumping line. If the OVC needs to be pumped you can now decide whether or not the pressure is too high for the diffusion pump. If so pump it with the rotary pump until the pressure is low enough.

Before you use the dewar you should pump the OVC to high vacuum using a diffusion or turbomolecular pump system (fitted with a cold trap to collect condensable vapours - see Caution above). Even if the OVC has been left under vacuum since the last run the surfaces inside the OVC are likely to outgas, and the vacuum will not be sufficiently high. There may be a sorb in the OVC to help to maintain the vacuum while the system is cold, and pumping the OVC whenever the system is warm helps to keep it clean. If possible you should pump the OVC overnight (or longer), until the pressure at the pump drops to $< 5 \times 10^{-5}$ mbar.

3.5.6.2.   Evacuating the dewar from atmospheric pressure

If the OVC has been vented with air or nitrogen gas use a rotary pump to evacuate the vacuum line, then slowly open the vacuum valve. (Tip: if the spring on the pressure relief valve on the base of the OVC has been stretched you may need to hold into in place until the vacuum holds it for you.) When the pressure is low enough start to pump the OVC with a diffusion pump or turbomolecular pump. It will typically take at least 24 hours to reduce the pressure in the OVC to $< 5 \times 10^{-5}$ mbar, as measured by a Penning gauge. Initial leak tests can then be carried out. If all is well, we recommend that you continue pumping for a further 48 hours and then conduct the leak tests for a second time.

3.6.   Thermometry for Heliox systems

3.6.1.   Description of common thermometers

The most common types of thermometry used on Oxford Instruments ^{3}He inserts and their relevant characteristics are described below.

a. Germanium resistors

This type of sensor is especially suitable for accurate measurement of temperature and is often fitted close to the sample. Choose a sensor which has a resistance of between 300 Ω and 10 kΩ at 0.3 K, giving good sensitivity but low susceptibility to RF pick up, which may affect the bridge circuit when measuring very high resistances. The sensor may be mounted in the ^{3}He or in vacuum. A four wire measurement technique is usually used.

Germanium resistors are especially suitable for use as calibrated sensors because their characteristics are very repeatable after many thermal cycles. However, they are not suitable for use in magnetic fields. They are often used to calibrate other sensors which are less repeatable, but which have better properties in high magnetic fields, for example, unmounted RuO_2 resistors.

b. Allen Bradley resistors

110 Ω Allen Bradley resistors are fitted to the sorb. These resistors are suitable for the temperature range at which the sorb operates. Their accuracy is sufficiently high to enable you to condense the ^{3}He and cool to base temperature. Typical calibration curves are supplied in the following pages.

c. Rhodium Iron resistors

These resistance sensors are available for use as a high temperature sensor in conjunction with an Oxford Instruments ITC temperature controller. They are repeatable even after many thermal cycles.

d. Carbon glass resistors

Carbon glass resistors are often used if the system is designed to operate in the range from 1.5 K to 80 K. They are accurate and repeatable even after many thermal cycles. They have high sensitivity at low temperatures, but low sensitivity at high temperatures. This type of sensor is almost completely unaffected by a magnetic field, and so it is ideal for a high temperature ^{3}He/magnet system. The sensor is wired using four wires. At temperatures below approximately 1.5 K their resistance rises very rapidly, so they are not suitable for use in this range.

e. Cernox resistors

These resistance sensors are designed to operate in the temperature range 1.4 K to 300 K. They are ideal for use in magnetic fields because their resistance changes very little in an applied magnetic field and in a predictable way.

f. RuO$_2$ thick film resistors

Thick film ruthenium oxide resistors have been used increasingly as low temperature sensors in recent years. If they are properly mounted and prepared they can give highly reproducible results for many thermal cycles. In addition, by carefully specifying the tolerances of a batch of resistors the calibration of one sensor can be quite accurately predicted from measurement of another. Thus 'generic' and fully calibrated sensors are available. RuO$_2$ sensors exhibit low magneto-resistance. This can be investigated on a ^{3}He system by controlling the sorb temperature to keep the sample temperature constant over a short time period. During this time the field can be swept slowly while the resistance or indicated temperature of the sensor is measured.
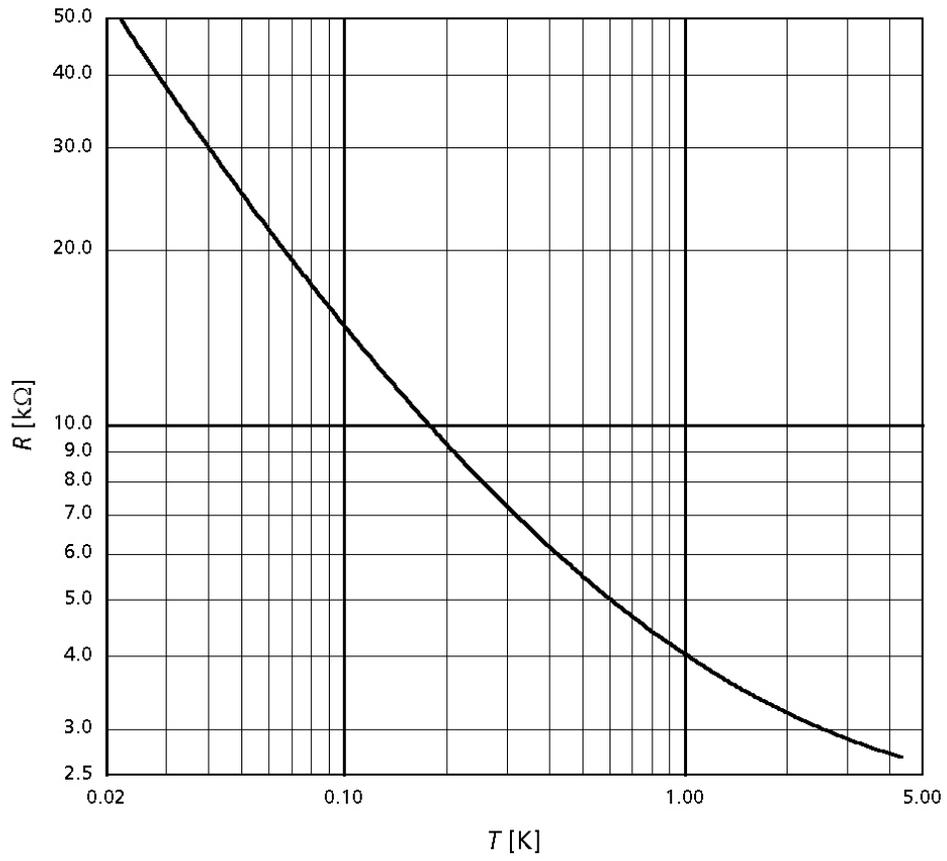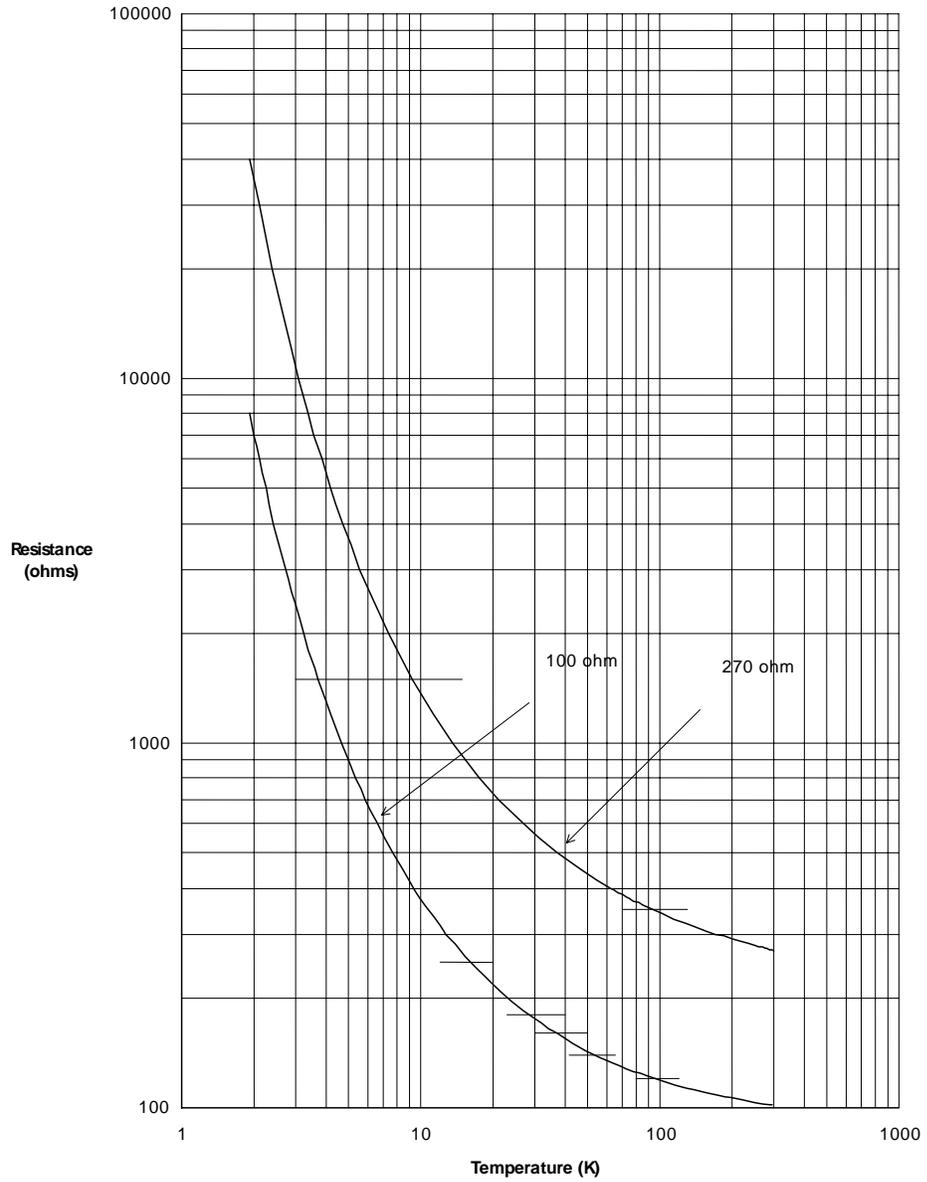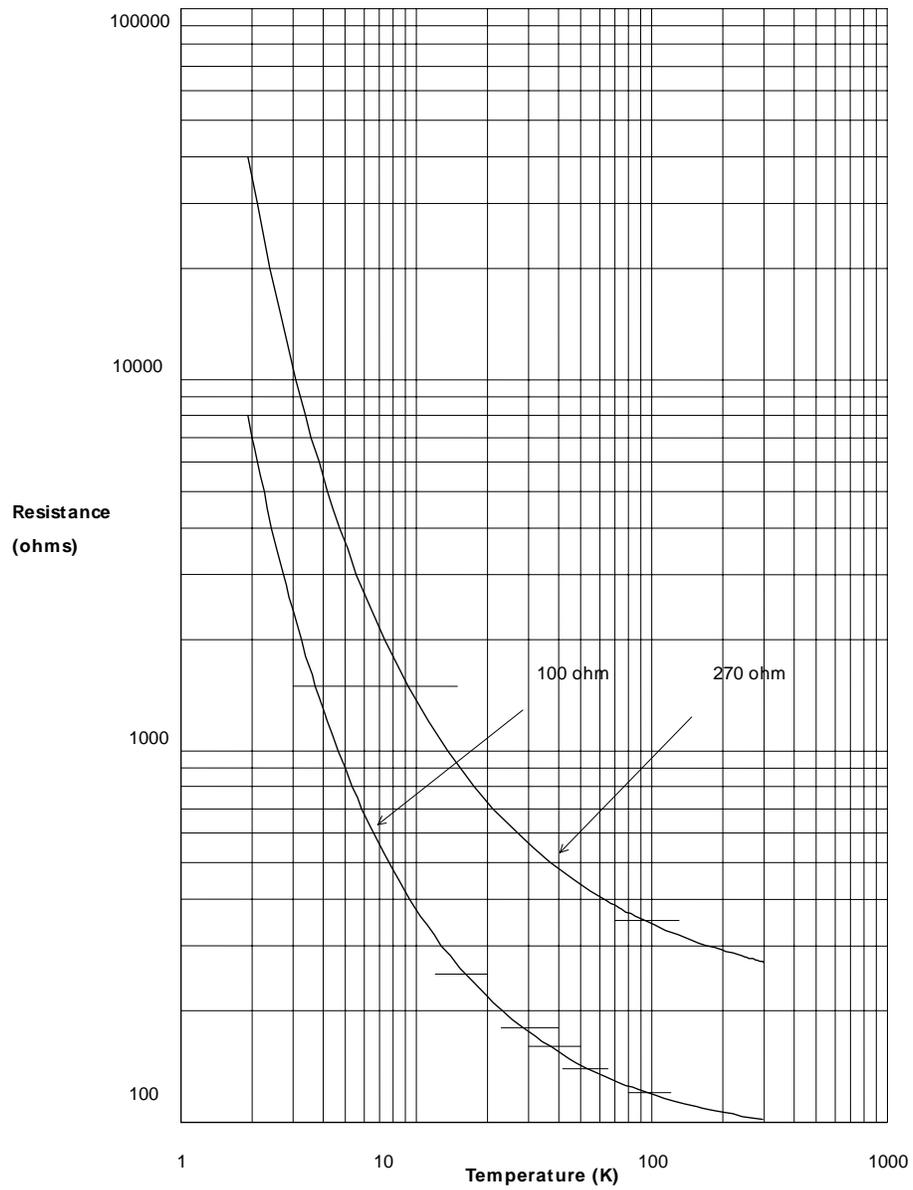
Figure 2 The generic R vs. T curve for the L series RuO_2. The nominal room temperature resistance is 2.21 k

Typical calibration curves of 100 and 270 ohm Allen Bradley resistors

3.7. Allen Bradley resistors as thermometers

Typical calibration curves of
100 and 270 ohm Allen Bradley resistors

Resistance
(ohms)

100 ohm   270 ohm

Temperature (K)

4.   Pre-cooling the system

4.1.   Preparing the HelioxTL insert for precooling

After the system has been proved to be reliable, some of the leak testing procedures described in this manual may be omitted to simplify operation of the system.

The main text in the manual also assumes that you are familiar with general cryogenic and vacuum procedures. Some background information is given in the booklet "*Practical Cryogenics"* to help beginners to understand the processes involved.

Load the insert into the dewar and assemble its pumping systems as described in the assembly section of this manual.

Push the sample probe down until the copper cone is approximately 50 cm above the contact on the 1 K pot, and use the clamp to hold it in this position while you cool down the system. (Only when the insert is cold is any ^{3}He exchange gas allowed into the sample space. Until that time, the probe cools by radiation alone.) This position is chosen for the following reasons.

a)   If the probe is not inserted, the thermal radiation down the central access will prevent the ^{3}He pot and 1 K pot from cooling properly.

b)   If it is pushed right into the system, the probe itself will hold the lower parts of the insert at a high temperature (preventing condensation of the ^{3}He gas at the later stages), because it does not cool as quickly as the insert. This effect is especially noticeable on the inserts with larger sample space diameters, because the displacers on the probe do not cool very quickly.

4.1.1.   Room temperature leak tests

We recommend that you always check the system for leaks before pre-cooling. If you have little experience of using a mass spectrometer leak detector you may find the advice in the booklet "*Practical Cryogenics*" helpful.

Evacuate the IVC and vacuum lock. The sample space should already be under vacuum. Open the gate valve. Check the visible parts for air leaks with a leak detector.

Connect the leak detector to the IVC and sample space.

Pump them until the helium signal drops to the 10^{-8} mbar l/s range on the leak detector (or a range with higher sensitivity).

Check that the needle valve is closed. To prepare the dewar for precooling follow the procedure which is described later. This will help to remove any water that may have condensed in the needle valve tube. Take care in all stages of the cool-down to use clean ^{4}He gas for all purging operations. These procedures help to avoid seizing and blocking of the needle valve due to frozen contamination in the thread or the tube. Monitor the signal on the leak detector and check for any signal rise while filling the main bath with recovery grade helium gas. Now test the 1 K pot for leaks to the IVC. Use the auxiliary pump to evacuate the pot. Close the valve and open the needle valve to fill the 1 K pot to atmospheric pressure with helium gas (from the main bath). Check that the needle valve is not blocked by observing the pressure rise when it is opened. Check that no helium signal is observed on the leak detector during these procedures.

Isolate the IVC from the leak detector (leaving the sample space connected to the leak detector) and let one bar of helium gas into the IVC to check that there is no leak into the sample space. Close the valve on the vacuum lock of the probe and leave it closed until the sample needs to be changed again. After this test, evacuate the helium from the IVC until the signal on the leak detector is less than 10^{-6} mbar l/s. Leave the sample space under vacuum.

4.1.2.   Choosing exchange gas

You can use nitrogen or helium as exchange gas for the Heliox system.

- Use dry air or nitrogen if you plan to test the system for leaks at 77 K (recommended)
- Use about 5 cm^3 of helium gas if you plan not to test the system for leaks at 77 K. This can be left in the IVC until the system is at 4.2 K

4.1.3.   Adding exchange gas and preparing the 1 K pot

Admit one bar of dry nitrogen gas or air to the IVC to act as exchange gas, and isolate it by closing the valve. Evacuate the 1 K pot and close the needle valve and valve V6.

4.2.   Preparing liquid nitrogen shielded dewars for pre-cooling

4.2.1.   Evacuating the OVC

Evacuate the outer vacuum chamber (OVC) of the dewar for at least 24 hours before you precool the system. It may be under vacuum already. If you want the optimum boil off performance it is important to pump the OVC with a high vacuum pump, not just a rotary pump. A 50 mm (or larger) diffusion pump fitted with a cold trap to collect condensable vapours is best because it pumps all gases well, (including helium). A turbomolecular pump with a cold trap (and backed by a two stage rotary pump) can be used but if there is any helium in the vacuum space it will take a long time to pump it away because these pumps have a low compression ratio for light molecules. Always use pumping lines which are at least 25 mm diameter and as short as possible. Do not use lines which have previously been used to carry helium gas.

It is possible to cool the system down without pumping the OVC with a diffusion pump but the system boil off is likely to be increased. We recommend that you always pump the OVC to a high vacuum before you cool down the system.

4.2.2.   Pumping and flushing the helium reservoir

This operation is typically part of the leak testing procedure for other parts of the system, so refer to the other sections describing how to prepare the system before carrying it out. Sometimes it is necessary to pump and flush the helium reservoir to purge air from other parts of the system (for example to prevent the risk of blockages caused by frozen air or moisture).

Warning;   **Do not pump on the main helium reservoir unless the OVC is already under vacuum. It may collapse if you do!**

Disconnect the helium recovery system from the cryostat. Connect a rotary pump to the exhaust of the helium reservoir and pump it to a rough vacuum (typically 1 mbar) to remove the air and moisture. Fill the helium reservoir with helium gas. If you want to check that there are no leaks from the helium reservoir to the OVC before you pre-cool the system you can do it as you vent it with helium gas.

4.2.3. Precautions to be taken before pre-cooling

- Fill the liquid helium vessel with liquid nitrogen before pre-cooling the liquid nitrogen jacket
- Make sure that at least one port on the liquid nitrogen jacket is fitted with a non-return valve

4.3. Pre-cooling the system

Make sure that you have carried out the preparations described for each part of the system before you start to pre-cool it. These are described in the other pages of this section of the manual.

Warning: *Practical Cryogenics* gives some background information about transferring liquid nitrogen. Refer to it if you are unsure of the correct procedures. It is also important to be aware of the correct safety procedures, as described in the booklet *Safety Matters* which has been included with this system.

Disconnect the main helium bath from the helium recovery line (if you have one in your laboratory). Some systems have to be pre-cooled slowly to make sure that they are not damaged by thermal shock. If so, precautions are given in the description of the preparations that you should carry out before pre-cooling the system. Insert the liquid nitrogen "blow out tube" through the siphon port. If there is a siphon cone in the system it is best to push the blow out tube into it.

Connect a suitable tube from the top of the blow out tube to a liquid nitrogen storage dewar and transfer liquid nitrogen into the main bath. Fill the helium reservoir with liquid nitrogen and leave the cryostat to pre-cool. It may take several hours to pre-cool the system, depending on the type of system.

Always wait until the liquid nitrogen has stopped boiling violently.

Caution: The OVC can be pumped during the pre-cooling procedure as long as there is a cold trap between the pump and the cryostat to prevent oil backstreaming. However, we advise that it should be isolated from the pump before the helium transfer is started.

Warning: Ensure that all the components are correctly bolted together before cooling down the system.

5.   Cooling the system to 4.2 K

Please read the whole of this section and make sure that you understand it before you proceed. This is possibly the most difficult part of the operation of the system because to do it most efficiently you have to carry out several operations at the same time. For example, you can carry out leak tests on several components together while the helium reservoir is pumped and flushed with helium gas.

5.1.   Preparing liquid nitrogen shielded systems for operations at 77 K

Insert the liquid nitrogen blow out tube into the siphon port. If there is a siphon cone on your system push the blow out tube into it, and if there is a thread on the blow out tube screw it into the siphon cone. Connect the top of this tube to one of the nitrogen jacket ports on your dewar.

You can use this liquid to fill the liquid nitrogen jacket of the dewar until it is full. If it overflows, stop blowing out the liquid nitrogen, connect the blow out tube to a separate storage dewar and blow the rest of the liquid into it.

5.2.   Blowing out the liquid nitrogen

Blow the liquid nitrogen out of the main bath using a slight overpressure of helium gas supplied through the exhaust port. 200 mbar should be sufficient. When all the liquid nitrogen has been removed withdraw the blow out tube and insert the bung in the siphon port.

You can see that liquid nitrogen is no longer being blown out of the helium reservoir by observing the following signs:

- The pressure drops in the main bath
- The flexible part of the blow out tube is no longer vibrating noticeably
- The metal part of the blow out tube nearest to the cryostat is no longer wet on the outside
- The plume of gas from the receiving vessel may change in character

If you are not planning to pump and flush the helium reservoir as described below it is wise to wait until the resistance of the Allen Bradley resistors (if fitted) drops by one or two ohms from the 77 K value measured when the reservoir was full of liquid nitrogen. This ensures that the reservoir has warmed slightly above 77 K and confirms that all the liquid nitrogen has been removed. Re-connect the main bath recovery line.

If you do not have a pressure gauge covering the range from 0 - 1000 mbar continue to blow warm helium gas through the helium reservoir for another 5 minutes to make sure that all the liquid nitrogen has been removed properly.

Warning: If the system is frozen into the cryostat with liquid nitrogen, it must be allowed to warm up naturally.

5.3.   Checking the HelioxTL insert at 77 K

It is not essential that you check the system for leaks at this stage, and after you have been using the system without problems for a few weeks you may feel confident enough to run it without further testing.

However, if the system is leaking it is better to find out before you use any liquid helium. Most cold leaks on cryogenic systems can be detected at 77 K, so if you want to ensure that there is no risk of wasting liquid helium we recommend that you carry out the following leak tests. If you do not want to test the system for leaks, proceed to the paragraph "Adding helium exchange gas to the IVC ".

Caution: Never vent the sample space to air while it is cold, because the sorb will be contaminated and it will not pump ^{3}He properly.

Wait until the diagnostic thermometers show a temperature close to 77 K. If you have used nitrogen exchange gas, evacuate the IVC. If you have used helium exchange gas it is only necessary to pump it out of the IVC if you want to carry out leak tests.

5.3.1. Leak testing the insert at 77 K

If the system is to be checked for leaks re-connect the leak detector. If you used helium exchange gas to pre-cool the insert and you want to carry out leak tests at 77 K you will have to pump the IVC for several hours with a diffusion pump to reduce the helium signal on the leak detector to an acceptable level. If you used nitrogen exchange gas it will be easy to repeat the leak tests that you carried out before you started to pre-cool the system.

Repeat the leak tests and throughput tests for the 1 K pot. Some of the tests can be done while you are blowing out the liquid nitrogen and pumping and flushing the helium reservoir. When the tests have been completed successfully, evacuate the 1 K pot, and isolate it.

Next blow out the liquid nitrogen as described below. Then complete the preparations required for the insert, as described below.

5.3.2. Sample space leak tests

Caution Do not allow any ^{4}He into the sample space when leak testing. If this is done, all traces of the ^{4}He will need to be removed from the sorb by warming it above room temperature while pumping with a diffusion pump system.

Monitor the sample space with the leak detector while you introduce the helium exchange gas to the IVC as described below. Then isolate the sample space. The helium gas should not be pumped out of the IVC until the helium transfer is complete, since it is used as exchange gas.

5.4. Checking liquid nitrogen shielded dewars at 77 K

5.4.1. Leak testing the OVC

If you want to check that there are no leaks from the liquid helium reservoir to the OVC you can do this by observing the helium signal in the OVC while the helium reservoir is filled with helium gas. Most cold leaks can be detected at 77 K, so there is little risk of a leak developing as the system is cooled to 4.2 K. However, if you have used the system without problems for a few weeks you may feel confident enough to run it without further testing. Close the OVC valve after you have completed the leak tests.

5.4.2. Filling the liquid nitrogen jacket

Fill the liquid nitrogen jacket with liquid nitrogen.

5.4.3. Preparing for the liquid helium transfer

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

5.5. Pumping and flushing the helium reservoir

It is wise to pump and flush the helium reservoir of your system (through the exhaust port) to carry out leak tests or to make sure that the liquid nitrogen has been removed completely. Complex systems with small capillary tubes could be blocked or superconducting magnets may be affected by frozen nitrogen.

If you are planning to test the system for leaks you can do many of the tests while you pump and flush the helium reservoir. Read the leak testing section for all the other parts of the system before you carry out this procedure.

Monitor the Allen Bradley resistors in the helium reservoir (if fitted) while you pump the reservoir. If you see their resistance rising as the pressure drops this is an indication that the liquid nitrogen has not been thoroughly removed, and you must try again to blow it all out.

Pump out the helium bath using the auxiliary pump to ensure that no liquid is left. The pressure should fall steadily to about 1 mbar. If this does not happen (for example, the pressure hesitates at 100 mbar) it indicates that the liquid has not all been removed. Vent the main bath to atmospheric pressure with helium gas, make sure that the blow out tube reaches the bottom of the helium reservoir, and try again to blow out any remaining liquid.

If you want to make sure that there is no liquid nitrogen in the reservoir after this process is complete, wait until the resistance of the Allen Bradley resistors drops by one or two ohms from the 77 K value measured when the reservoir was full of liquid nitrogen. This ensures that the reservoir has warmed slightly above 77 K and confirms that all the liquid nitrogen has been removed.

5.6. Preparing HelioxTL inserts for cooling to 4.2K

5.6.1. Adding helium exchange gas to the IVC

Connect the sample space to the leak detector and allow a few hundred cm^3 of ^{4}He gas into the IVC.

5.6.2. Assisting the cooldown to 4.2 K

The cooldown may be aided by drawing some of the cold gas through the 1 K pot while pumping on the pot, but the pot should not be cooled below 4.2 K until all the exchange gas has been removed.

5.7. Cooling systems to 4.2 K

Liquid helium has a very low latent heat of evaporation but the gas has high enthalpy. This means that it is very easy to evaporate the liquid but it is difficult to warm up the gas so produced. Liquid helium therefore has to be transferred very carefully. If you do not transfer it properly you may lose all the liquid from your storage dewar without collecting any in your system. Follow these instructions to get an efficient liquid helium transfer.

When you are cooling down a system to 4.2 K it is very important to transfer the liquid helium to the lowest point in the helium reservoir. If the system is warmer than 4.2 K the liquid boils almost immediately as it leaves the vacuum insulated transfer tube (or siphon). Very little cooling is obtained from this evaporation. However, this gas then has to pass over the equipment in the helium reservoir to reach the exhaust line, and this provides very useful cooling power. If you transfer the liquid helium into the system slowly you can make sure that the gas emerging from the exhaust line is not too cold. This ensures that you do not waste any cooling power. If you do transfer the liquid too quickly you may see liquid air running from the recovery line, indicating that the cooling power is being wasted.

5.7.1. Preparations for the helium transfer

Check that the leg lengths of the transfer tube are suitable. The storage dewar leg should be able to reach below the liquid level (and preferably reach the bottom of the dewar). The system leg should be able to reach the lowest point in the helium reservoir (or the siphon cone if one is fitted). Position the liquid helium storage vessel so that the transfer tube can be easily inserted to both the storage dewar and the system, and blow some helium gas through the transfer tube to remove the air.

Remove the non-return valve from the exhaust port of the helium reservoir.

Warning: **If you have a helium recovery system, connect the exhaust line of the cryostat's helium reservoir and the storage dewar to it. It is important to make sure that the impedance of the recovery line is low enough to allow an efficient helium transfer. The recovery line should be at least 25 mm diameter. Contact your recovery system administrator for advice if you need it.**

If you have a helium recovery system, connect the exhaust line of the cryostat's helium reservoir and the storage dewar to it. It is important to make sure that the impedance of the recovery line is low enough to allow an efficient helium transfer. The recovery line should be at least 25 mm diameter. Contact your recovery system administrator for advice if you need it.

If you do not have a recovery system, make sure that the exhaust is free to vent but that there is no risk that the system will be filled with air condensed from the atmosphere. You can do this by connecting a flexible line a few meters long to the exhaust port. Let the other end lie on the floor. The helium in this line is lighter than air and tends to prevent air from rising to the exhaust port. However, when the helium transfer is complete, or if the system is to be left open to air for more than a few minutes, you should put a one way valve on the cryostat exhaust port.

Figure 3 Transferring liquid helium into a typical laboratory cryostat.
Your system may not look like the one shown in the diagram.

5.7.2. Transferring liquid helium

Remove the plugs from the system's transfer tube entry port and the top of the storage vessel. 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 (or the siphon cone if fitted).

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). The transfer rate should be such that the vent pipe is frozen for not more than 2 m (6 ft.) of its length. The initial transfer rate should be equivalent to between 4 and 10 litres of liquid per hour.

Close the OVC valve on the cryostat. There is no need to continue to pump it.

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). The pressure on the storage dewar can then be increased to transfer the liquid more quickly.

When the liquid helium reservoir has been filled, stop the transfer by releasing the pressure in the storage vessel. Remove the transfer tube and replace the bungs.

The booklet *Practical Cryogenics* contains a list of solutions to the problems commonly encountered in liquid helium transfers. Refer to this booklet if you are having problems.

Caution:　　　　Remember to replace the non-return valve on the helium reservoir exhaust.

6. Running the system

6.1. Preparing HelioxTL inserts for operation

Monitor the RuO_2 resistor on the ^3He pot, the Allen Bradley resistor on the sorb and the Cernox sensor on the probe (if fitted). When they have cooled to about 10 K begin pumping the IVC exchange gas while you fill the liquid helium reservoir. Pump the IVC for at least 6 hours using a turbomolecular pump or preferably a diffusion pump. After this period, the helium signal (monitored with a leak detector) should be less than about 3 x 10^{-6} mbar l/s. This can conveniently be done overnight. Do not cool the 1 K pot below 4.2 K while you are pumping the exchange gas out as this cold surface will retain some of the gas, and may create an unacceptable heat load later.

During this procedure, the sorb temperature may rise to about 30 K.

If the leak detector is sensitive to ^3He, you can warm the sorb to about 45 K and admit a few cubic centimetres of ^3He into the ^3He space. Check that no leak signal appears in the IVC. However, this is best done when the ^3He is condensed.

Caution: Make sure that the valve on the vacuum lock of the probe is closed when ^3He is present in the vacuum lock, otherwise it is possible to pump away the valuable gas accidentally.

6.2. Condensing ^3He, and running the HelioxTL insert to base temperature

6.2.1. Preparations

The system should now be at 4.2 K and the liquid helium reservoir should have been filled. The IVC should have been pumped to a high vacuum to isolate the sorb, 1 K pot and ^3He pot from the main bath. It is important that the IVC is pumped with either a diffusion pump or an exchange gas sorb. If there is too much helium gas in the IVC the system will not work properly.

6.2.2. Condensing the ^3He

Begin pumping the 1 K pot. Open the 1 K pot needle valve and the pressure will rise slowly to about 200 mbar. Adjust the flow through the 1K pot to about 2-3 l/min of ^4He gas. Record the percentage of opening of the needle valve corresponding to this flow. (This is one of the software parameters used for auto condensation.) Open the ^3He valve on the insert, by unscrewing the valve with the red head and open the ^3He dump valve (V2) to allow ^3He gas to enter the sorb. Check that the dump pressure has dropped below 20-40 mbar and close the valve on the insert with the red head.

Note: Make sure that the valve V2 on the ^3He dump remains open at all times during operation of the ^3He refrigerator. The ^3He valve on the insert has a pressure relief function and will safely vent ^3He gas into the dump if there is any accidental heating of the system.

Heat the sorb to about 27 to 30 K Monitor the pressure in the 1K pot and ensure that it does not exceed about 20 mbar. A typical value for the flow of ^4He is 2 to 2.5 l/min.

The probe should currently be at its pre-cool height with the sensors in the 4K region. If it is not, it should now be loaded to this position following the procedure described in section 6.4.2. Check that the sensors on the probe show a temperature below 10K. Remove the clamp and lower the top loading probe slowly until the cone meets the 1K pot. Allow the ^{3}He pressure to come to equilibrium. The equilibrium pressure depends on the 1K pot temperature and therefore on the needle valve setting. The pressure should be less then 100 mbar at this stage. If it is not, close the 1K pot needle valve slightly. If the pressure still does not fall try to adjust the needle valve setting to achieve as low a temperature as possible in the 1K pot. During condensation, typical temperatures for the sorb and 1K pot are 27K and 1.45K respectively. When the ^{3}He pressure reaches its minimum value wait for about 10 minutes. This is the end of the condensation of the ^{3}He gas and the complete procedure requires about 50 minutes.

Note: It is advantageous to have the lowest possible temperature in the 1K pot region in order to maximise the amount of liquid condensed. It is possible to run the 1K pot in single shot mode by closing the needle valve completely. This will lead to a lower temperature in the 1K pot. The temperature of the 1K pot should be monitored and it should be refilled if it empties.

If a large enough pump is used on the 1K pot it should not be difficult to reduce the ^{3}He pressure to about 25 mbar, measured on the gauge fitted to the probe. This is one of the most important stages for the successful operation of the insert, (longer hold time) because the amountof ^{3}He that is condensed depends strongly on the condensing temperature.

6.2.3.   Computer controlled condensation

If the Heliox is to be computer controlled then refer to the manual on how to configure and run the software. Once the software is running you can start condensation by simply pressing the CONDENSE button on the main window displayed by the software.

6.2.4.   Cooling to base temperature

When the ^{3}He has condensed to about 45-50 mbar or below the sample can be cooled to base temperature. Turn off the sorb heater and increase the ^{4}He flow. It is most economical to cool the sorb slowly but if circumstances demand then it can be cooled quickly. In practice with a ^{4}He flow of 6-8 litres/min (needle valve setting at about 45%) it is possible to cool the sorb to <6 K in under 15 minutes.

The sorb temperature will now begin to decrease and it will start to pump ^{3}He gas when it reaches about 15 K. If there is sufficient flow through the sorb heat exchanger, the final sorb temperature will be shown as 2.2 K. At this point the ^{4}He flow needs to be reduced to 0.5 - 0.8 litres/min (set the needle valve to the appropriate value). This flow (or even a smaller flow) should be sufficient for operation a base temperature.

The ^{3}He pot and sample will cool quite rapidly to about 0.4 K and then cool to base temperature over a period of one hour or less, as long as the thermal gradients in the liquid are correctly shorted.

6.2.5.   Operating in the high temperature range

If you are running the system in the high temperature range the pot should be operated in the continuous fill mode to provide cooling for the sample. The flow should be minimised for each sample temperature to reduce the consumption of liquid helium as far as possible. In the high temperature range the pot may be run either partly full of liquid or it may run on a continuous flow of cold gas. The stability of the sample space temperature may be affected as the 1 K pot changes from one mode of operation to the other.

6.2.6. Recondensing the ^{3}He

When all the ^{3}He has evaporated the sample will begin to warm. The liquid has to be recondensed as described above except that the probe should remain in its fully loaded position. The process can be automated if you are using ObjectBench or B-T environment software.

Warning: The valve V2 on the dump vessel must be open at all times while the system is running to prevent accidental build-up of high pressure. The opening pressure ofr the safety relief function of the ^{3}He valve on the insert is 1 bar absolute, suitable to ensure that all operations can be carried out safely.

6.3.   HelioxTL temperature control

6.3.1.   Temperature control

If you use the settings suggested in the test results and these short instructions you should be able to control the temperature of the Heliox insert within the specifications. Choose the settings for the nearest temperature given in the test results.

However, if you want to understand how the system works and try to optimise the level of control further, you should read "HelioxTL temperature control - background information".

6.3.1.1.   Temperature control in the low temperature range (below about 1.2 K)

If you want to set the temperature of the system below the temperature of the 1 K pot follow these instructions.

Caution: Set the maximum temperature limit of the sorb (channel 1) to approximately 80 K. The highest ^{3}He temperature that can be achieved is the temperature of the 1 K pot. Further warming of the sorb has little effect and this prevents thermal runaway. The ITC manual describes how to set this maximum

Condense the ^{3}He as described elsewhere in this manual. Then:

- Set the ITC to control the heater from sensor 2
- Press the SET button and select the temperature that you want by pressing RAISE and LOWER
- Release SET and the display shows the temperature of the sensor indicated by the light in the DISPLAY area of the front panel (not necessarily the control sensor)
- Set the PID values given in the test results for the closest available test temperature
- Press AUTO
- Set the 1 K pot to continuous fill

You can use the SENSOR button in the DISPLAY area of the front panel to select the sensor that you want to display. This will not affect operation of the system.

6.3.1.2. Temperature control at higher temperatures (optional)

If you want to set the temperature of the system above the temperature of the 1 K pot follow these instructions. This assumes that your system is set up for this type of operation. It has to have a heater close to the sample position.

- Set the ITC to control the heater from sensor 3
- Press the SET button and select the control temperature that you want
- Release SET and the display shows the temperature of the sensor indicated by the light in the DISPLAY area of the front panel (not necessarily the control sensor)
- Set the PID values given in the test results for the closest available test temperature
- Set the 1 K pot flow rate given in the test results
- Press AUTO

The Heliox heater controller box will automatically supply most of the applied power to the ^{3}He pot heater. However, it will apply enough heat to the sorb heater to prevent the sorb from cooling too much and pumping away the ^{3}He gas in the sample space which is used as exchange gas in this mode of operation.

6.3.1.3. Autotuning temperature control with the ITC503

The software supplied with the ITC503 includes a facility to help determine the optimum temperature control settings (the PID settings). This facility is described in the software manual.

Temperature control within the specifications of the insert can usually be achieved by using the autotune facility with the full heater output voltage and reasonably coarse autotune conditions. A step size of 0.1 K and overshoot of 20% in the low temperature range, and a step size of up to 5 K and overshoot of 10% in the high temperature range should give reasonable values for P, I and D after approximately 30 minutes per temperature.

The most accurate PID settings are found when the rate of warming with full heater power is approximately equal to the rate of cooling with zero heater power. In general, this condition can be achieved by adjusting the maximum heater output voltage. Typical values for a Heliox insert are between 5 and 10 volts in the low temperature region and around 40 volts in the high temperature region, although these will vary with the experimental heat load and the flow of ^{4}He through the 1K pot.

Once PID settings have been found for a temperature range, they can be stored in a "look up" table. The ITC503 will use PID settings from the "look up" table if it is switched to the "Auto PID" mode.

6.4.   Heliox*TL* sample changing

6.4.1.   Removing the probe while the cryostat is cold

At the end of the experimental run, the ^{3}He must be pumped into the sorb or removed from the cryostat into the dump using the cryopump, so that only a negligible amount of gas is left in the vacuum lock when the gate valve is shut. The sorb is capable of reducing the pressure to below 10^{-4} mbar if it is below 8 K. A heater is provided on the ^{3}He pot to aid this procedure. When the ^{3}He pressure in the cryostat is sufficiently low the probe may be removed. (The pressure can be monitored using the gauge on the probe.) Check that the valve on the vacuum lock of the probe and the ^{3}He valve on the insert are both closed. Make sure that the gate valve is fully open. Connect a rotary pump to the NW16 flange on the probe sliding seal. This will pump away any air that leaks through the outer seal as the probe is moved, preventing it from entering the sample space. Since the ^{3}He pressure in the sample space is very low the inner seal should be sufficient to prevent a noticeable amount of gas from passing through as the probe is moved.

The sample can now be lifted slowly out of the cryostat. It will take about 10 to 15 minutes to withdraw the probe. This is most conveniently done in several stages. A clamp is provided to hold the probe at various positions during this procedure. If you see any condensation on the tube as it emerges from the seal assembly, then you are removing the sample too quickly. If you freeze the 'O' rings in the sliding seal you may let air into the sample space.

When the probe has been withdrawn completely, close the gate valve carefully (in case the probe is not fully withdrawn); it should close with a click. The sample can now be left to warm naturally to room temperature or dry nitrogen exchange gas (or air) can be introduced through the valve on the vacuum lock of the probe. Avoid using ^{4}He as exchange gas because of the risk of contaminating the charge of ^{3}He. Disconnect the rotary pump from the sliding seal port.

When the sample is at room temperature remove the probe assembly by disconnecting the Klein flange on top of the gate valve.

6.4.2.   Inserting the sample probe when the insert is cold

Connect the assembly to the gate valve. Evacuate the vacuum lock through the valve on the probe, then close this valve. Connect the pump to the sliding seal port again, to prevent the ingress of air as the probe is moved. Open the gate valve. Use the clamp to hold the probe in this position. Check that the valve V2 to the dump is open and that the ^{3}He valve on the insert is closed. Warm the sorb slightly to introduce a few mbar of ^{3}He exchange gas into the sample space (monitored on the pressure gauge on the probe). Lower the probe slowly until the copper cone is approximately 30 to 50 cm above the contact on the 1 K pot, and allow the sample to cool to below 20 K before pushing the probe down into position.

Inserting the probe will warm the 1 K pot. It might be necessary to increase the flow of ^{4}He through the 1K pot to cool it down again. Allow the whole ^{3}He charge to condense fully into the ^{3}He pot.

With some experience, you may prefer to use a slightly different procedure to improve the efficiency of the top loading process. Provided that care is taken, there is no reason why the ^{3}He should not be condensed into the ^{3}He pot before the gate valve is opened. This allows the probe to be cooled gradually as it is pushed slowly into the insert. If you use this technique you must ensure that the valve V2 to the dump vessel is open and that there is no risk of pumping ^{3}He away if the sliding seal or one of the valves leaks. For this reason, the procedure described first will be the most appropriate for inexperienced users.

6.4.3.   Sample mounting.

The sample may be bolted to the end of the top loading probe. Several holes are provided. Since the sample is loaded directly into the ^{3}He it is likely to be in good thermal contact with the liquid and the sensor/heater block on the end of the probe even if it is not tightly fixed to the probe.

6.5. Leaving the system unattended

6.5.1. Running the system unattended

If you plan to leave the system to run unattended you must take the following precautions. Remember that it is your responsibility to make sure that no one is put into danger by the system. Read and learn the contents of the Safety section of this manual and take appropriate actions.

- Erect suitable warning signs to prevent tampering by other people
- Try to make sure that only competent people have access to the system
- Make sure that there are sufficient cryogens in the system
- Arrange for the cryogens to be re-filled if necessary
- Connect the exhaust of the helium reservoir to a recovery system or fit an appropriate one way valve to prevent air or moisture from entering
- Make sure that the system can vent safely, even if it is accidentally warmed up or pumps stop running unexpectedly
- Leave a telephone number so that you can be contacted in an emergency
- Make sure that there is sufficient ventilation in the laboratory to avoid a potential asphyxiation hazard when you return

If there are any closed volumes that are pumped during normal operation make sure that they are free to vent either into the cryostat reservoirs or through the pumping line. If there are valves in the pumping line and on the inlet to these volumes make sure that you do not leave them both closed.

6.5.2. Leaving the system static

If you are not using the system for a few days (for example over the weekend) it is often possible to close it down and leave it in a static condition. This could save liquid helium or reduce some of the potential hazards associated with the system. To leave a typical system in static mode:

- De-energise the superconducting magnet
- Close down the lambda point refrigerator (if one is fitted) and vent it safely
- Close down any variable temperature insert, Heliox insert or Kelvinox insert

6.6. Re-filling the liquid helium

When the liquid helium level drops close to the minimum working level you should carefully re-fill it. When you refill the liquid helium you should take care to pre-cool the transfer tube thoroughly before you put it into the system. Otherwise the warm gas passing through the tube will evaporate liquid in the helium reservoir. The booklet *Practical Cryogenics* contains a list of practical solutions to the problems commonly encountered in liquid helium transfers.

Important Note: This describes the easiest method of transferring liquid into a cold system for beginners. However, some laboratories have strict rules about recovering all helium gas. If you have a helium recovery system ask the administrator to show you the preferred method of transferring helium.

Caution: If your system contains a superconducting magnet:

- Make sure that the liquid helium level does not drop below the minimum level shown on the drawing while it is energised.
- Run down the magnet, if in doubt
- Beware of the stray magnetic field while you are working close to the cryostat.

Some transfer tubes are supplied with special fittings for refilling the liquid helium. These fittings are screwed onto the end of the transfer tube and divert the gas and liquid from the transfer tube up and away from the liquid surface. The gas passes out of the cryostat and the heavier liquid falls into the reservoir.

6.6.1.   Pre-cooling the transfer tube (or siphon)

Prepare the storage dewar and transfer tube as described in the section about "Cooling systems to 4.2K". Insert one leg of the transfer tube into the helium storage vessel, but leave the other leg outside the cryostat. Unscrew the cryostat 'siphon entry' fittings (the O-ring and the knurled nut) and slide it onto the leg of the transfer tube which will go into the cryostat. Put the bung loosely in the transfer tube entry port on the system to prevent gross contamination with air. Pressurise the transport dewar slightly, in the normal way. After about 20 seconds you should hear oscillations in the tube, gradually increasing in frequency and intensity. When these stop you should see white vapour and when liquid starts to emerge you may see a white cone (like a gas flame).

6.6.2.   Transferring the liquid helium

If you have a rigid transfer tube quickly release the pressure in the transport dewar, lift the transfer tube and insert the open end into the cryostat. If you have a transfer tube with a flexible section it is easy to do this without releasing the pressure or moving the leg in the storage dewar.

Push the transfer tube into the system to approximately the maximum helium level. Do not push it to the bottom of the helium reservoir or into the siphon cone (if there is one on your system).

Caution: Do not push the transfer tube below the maximum helium level if you have a superconducting magnet in the system. You may quench the magnet.

Quickly increase the pressure in the storage dewar again. It is most efficient to transfer the liquid quickly to reduce the losses in the transfer tube. However, 200 mbar is usually sufficient pressure to do this.

Note: The reading on the helium level probe may be affected when using the siphon entry on the dewar to top up with helium. For best results use the siphon entry on the insert (if fitted)

7.   Warming up the system

7.1.   Warming up HelioxTL inserts

Ensure that the ^{3}He storage vessel is connected to the cryostat and that the valves on the insert (red valve) and the dump (valve V2) are open. Close the 1K pot needle valve but wait until the pot empties before closing valve V6 to stop pumping the 1K pot. Warm the sorb to about 50 K. Apply heat to the ^{3}He pot to boil-off any remaining liquid ^{3}He. (50 mW is sufficient to remove all the liquid in a few minutes.) The pressure in the ^{3}He storage vessel should begin to rise as the ^{3}He leaves the cryostat. When an equilibrium pressure is reached, the ^{3}He remaining in the insert can be removed using a cryopump, as described below.

Remove all the remaining ^{3}He from the insert as follows . Open the valve on the cryopump. Wait until the pressure in the dump equalises with the pressure in the insert and then close valve V2. Slowly lower the cryopump into liquid helium at 4.2 K and keep the valve on the top of the cryopump open. After about 10 minutes, the remaing ^{3}He gas in the insert should be at a low pressure. Close off the ^{3}He valve (red valve) on the insert and open V2. Keep the cryopump valve open and lift the cryopump slowly to the top of dewar. Check that the ^{3}He gas in the cryopump is expanding into the dump by observing the dump pressure gauge. When the pressure in the dump stabilises close V2 and slowly lower the cryopump into the dewar again. After 5 minutes open the ^{3}He valve on the insert to adsorb another portion of the ^{3}He charge which remains in the insert. Close the ^{3}He valve on the insert, open V2 and lift the cryopump up again. This procedure must be repeated until the dump pressure no longer increases as the cryopump is warmed up. This means that all the ^{3}He gas has been pumped out of the Insert. Cool the cryopump to 4.2 K again and collect the ^{3}He gas from the pipework of the gas handling system. (Make sure that V2 and the ^{3}He valve on the Insert are now closed.) Close the valve on the top of the cryopump while the cryopump is cold and remove the cryopump from the dewar. The ^{3}He gas from the pipework will be stored in the cryopump.

Warning: **If too much ^{3}He is in the cryopump when it is warmed up to room temperature, dangerously high pressures may be produced.**

Up to 2 litres of ^{3}He may be stored in the cryopump, but for security reasons, it is recommended that all the ^{3}He charge is stored in the dump at below atmospheric pressure, except the gas that is removed during final cryopumping of the connecting lines. The ^{3}He should be removed with the cryostat between 4.2 K and 80 K, to ensure that any air or water that has entered the system is removed from the gas. The system may then be warmed up to room temperature. Ensure that any vessels that contain cold liquid or gases (including exchange gas) will not be pressurised as the temperature rises.

At the end of the first run, the ^{3}He dump pressure will be slightly less than at the start, as a small amount of gas is stored within the cryopump. This gas can be condensed back into the cryostat for the next experiment at the same time as the dump charge is condensed. On subsequent runs the final pressure should not be significantly different from the initial pressure, but the reading may be affected by the ambient temperature.

Between runs, the sample space should be kept under vacuum to keep the charcoal in the sorb as clean as possible.

7.2. Warming up the system - liquid nitrogen shielded dewars

7.2.1. Preparations

Before you start to warm up the system you must make sure that it is safe. The Safety section of this manual gives some guidelines.

Make sure that there are no trapped volumes of liquid, gas or condensed solids inside the system. You may not know that they are there if they have accidentally been condensed into the system while it has been cold. Therefore you must make sure that all closed volumes are free to vent or that they are pumped continuously as the system warms up.

Close down any other parts of the system. In particular if your system contains any of the following items prepare them properly.

- Superconducting magnets must be de-energised
- Lambda point refrigerators must be closed down and pumped out (and pumped continuously during warm-up) or vented to the main helium reservoir
- Variable temperature inserts, Heliox inserts or Kelvinox inserts must be closed down and vented (or pumped continuously during warm-up)

7.2.2. Allowing the system to warm naturally

When you have prepared the system you can leave it to warm up naturally. When the cryogens have all evaporated the system will warm slowly to room temperature. If you do not need to use it again soon this is the easiest way to warm the system up.

7.2.3. Warming the system quickly

If you want to warm up the system more quickly you have to blow out the cryogens and break the insulating vacuum in the outer vacuum chamber.

The liquid helium can be blown out of the system either into a storage vessel for use elsewhere or into a helium gas recovery system. Liquid nitrogen can be blown into a storage vessel or disposed of safely. The system will then begin to warm up.

8.   Background information

8.1.   Making indium seals

Oxford Instruments uses two main types of indium seal, as illustrated in the diagram below. They both use 1mm diameter wire, retained

- Either in a groove by a flat surface
- Or in a corner between two flanges

In both cases, the indium wire is overlapped by bending one end of the wire sharply outwards and laying the other end across the corner of the bend. The wire is so soft that the joint will be compressed into a cold weld.

Figure 4 Indium seals

8.1.1.   Preparations

Before you make the seal ensure that the groove and the mating surfaces are clean. Thoroughly remove any old indium wire from the seal faces. If necessary a solvent can be used for cleaning. Some people like to grease the metal surfaces with silicone vacuum grease to make it easier to remove the wire later, but this is not necessary.

8.1.2.   Making the seal

Lay a new piece of indium wire in the groove or round the male spigot on one of the flanges and overlap it as shown on the diagram. There are usually alignment marks on the flanges to indicate the correct orientation. Carefully bring the two flanges together and hold them loosely in place with two bolts while you put the other bolts into the flanges and tighten them by finger only. Slowly and evenly tighten all of the bolts with a small spanner (wrench) or Allen key. Do not tighten them too much. There is no need to use an extension on the tool to give extra leverage. On large seals (typically > 50mm diameter) it is then best to leave them for about an hour. The indium flows slightly during this period so it is often possible to tighten the bolts slightly more.

8.1.3.   Separating indium seal flanges

It is often difficult to separate indium seal flanges because the indium metal seems to glue them together. Most large indium seals made by Oxford Instruments have two or more threaded holes in one of the flanges for 'jacking screws'.

Remove the bolts that hold the indium seal together (leaving two of the bolts loosely in place so that the flanges do not fall apart when they separate). Use another two of these bolts to jack the flanges apart by screwing them evenly into the jacking screw holes from the same side of the flange. This will push the flanges apart.

If there are no jacking screw holes (as often happens on small diameter indium seals), the flanges can be separated by inserting a sharp blade between the flanges. Make sure that the blade does not slip and cut you as the flanges separate.

8.2. Routine maintenance for HelioxTL

8.2.1. Removing and replacing the IVC (and ^{3}He tail)

The only major item that can easily be removed from the insert is the IVC tail, which is attached with an indium seal.

Vent the IVC and remove it as described in the assembly section of this manual. Withdraw the IVC carefully to avoid damaging the delicate wiring.

The ^{3}He tail can be removed from most systems. Disconnect the wiring for the heater and thermometer on this tail and vent the sample space to atmospheric pressure before you separate the indium seal.

Before re-assembly all flanges must be cleaned thoroughly and all the old indium must be removed carefully. Make the indium seal as described in the Assembly section of this manual.

When the insert has been reassembled, check the wiring and follow the standard leak testing procedures carefully.

8.2.1.1. Contamination in the sorb

After an extended period of operation, the pumping efficiency of the sorb may deteriorate gradually. This is a sign that the charcoal has absorbed some air, nitrogen, or water and that it will have to be outgassed.

This condition may be indicated by a failure to reach or maintain the lowest possible temperature, or by the temperature of the ^{3}He drifting up markedly with time when there is a constant experimental heat load.

Remove all the ^{3}He from the system before warming it up to room temperature as described in the section about warming up the system. Vent the IVC to allow free convection of the air around the sorb; this should protect the wiring. Pump the sample space with a diffusion pump system, and warm the sorb to slightly above room temperature. The current through the sorb heater must not exceed 1 amp for a short period, and if it is left running continuously the current should not exceed 0.25 amps. Leave the system overnight, using the heater to keep the sorb warm, and not to heat it to a very high temperature.

8.2.2. Pumping system.

Individual components of the pumping system may need regular servicing. Follow the instructions supplied by the manufacturer.

8.3. HelioxTL temperature control - background information

8.3.1. Temperature control

The following instructions are intended to help you to understand the factors affecting the temperature of the sample. The test results for the insert will allow you to control the insert to the level required by the specifications. Settings to achieve improved stability may be found with some experience.

In the low temperature range the sample is usually monitored using a RuO_2 thick film resistor mounted on the ^{3}He pot. These resistors are quite insensitive to a magnetic field and are used to control the temperature, even when the field is swept. If you want to measure the temperature accurately you should use a calibrated sensor, such as a germanium or RuO_2 resistor.

The level of temperature control can be improved over the entire range by adjusting the maximum heater output available from the ITC temperature controller. It is best to balance the maximum rate at which the temperature can rise or fall when it is outside the proportional band. For example, if the sample or sorb temperature can rise twice as quickly as it can fall, it may be helpful to reduce the maximum heater output by a factor of two. This can be optimised by plotting the temperature against time and matching the gradients.

8.3.1.1.   High temperature operation

Two separate heater outputs from the ITC are required (for example, for sorb control and for high temperature control using the ^{3}He pot heater), so a heater controller box is connected to the cables between the ITC and the insert (part number CQB 2200) to switch the heater output automatically. The heater controller box is not needed if the insert will only be used at temperatures below 1.5 K, but the heater on the ^{3}He pot must then be disconnected.

The following table gives typical set-up information for a system using one ITC temperature controller. If two controllers are used on a system the sensors may be arranged differently, and details will be given in the "Test results and specifications" section of the manual.

Channel 1	For the sorb thermometer (for example, Allen Bradley carbon resistor). This gives an indication of the temperature of the sorb during re-condensation of the ^{3}He. Note that the accuracy of this sensor is not critical for successful operation of the system.
Channel 2	For measurement of the sample temperature with resolution of 1 mK. The channel uses an RuO_2 resistor as its temperature sensor. Note that resistor does not measure the temperature accurately unless it is calibrated, but it can be used to achieve accurate control, even in a magnetic field.
Channel 3	For measurement of the sample temperature in the range 1.5 to 300 K. The channel typically uses a Cernox resistance sensor.

Table 1 ITC Range Channels (A summary of the available options)

8.3.1.2.   Temperature control in the low temperature range
a Basic Control (background information)

Coarse control can be achieved by setting the temperature of the sorb to a constant value and thereby setting its pumping speed. If the sorb is cooled to its lowest temperature, it pumps at its maximum speed, so the ^{3}He temperature will be reduced to a minimum for a given experimental heat load.

If the sorb is above 40 K, it will not pump, and the ^{3}He temperature will be largely dependent upon the temperature of the 1 K pot. Intermediate sorb temperatures are used to control the ^{3}He temperature within these limits.

When the temperature of the sorb is controlled at a constant level by an ITC temperature controller, the sample temperature tends to drift up slowly as the charcoal saturates. If there is no experimental heat load, it should be expected to rise by about 0.01 K per hour.

The upper temperature limit for this method of control is the temperature of the 1 K pot. That is, at some stage further warming of the sorb will not result in further warming of the sample because these are the conditions used during the condensing procedure.

b Temperature control with feedback from the sample temperature sensor

The level of control can be greatly improved by measuring the sample temperature with the ITC, and arranging for the controller to adjust the temperature of the sorb continuously to maintain a steady sample temperature. In this way, the temperature can be held stable within a few milli-kelvin. In order to achieve this, an extra channel is used. The procedure described below should be followed. Throughout this procedure the sorb heat exchanger flow should be set at the minimum which will supply sufficient cooling to the sorb using the needle valve on the heat exchanger exhaust line.

The settings on the three term controller of the ITC (that is, PID settings) need to be optimised to suit the operating conditions. The test results will indicate the settings used at Oxford Instruments, and should be regarded as a starting point. Improved stability may be achieved by fine adjustment. It is very difficult to optimise the settings in a region where the cooling and warming effects are not balanced. For example, it is difficult to control accurately below say 0.32 K, because warming the sorb slightly has a large effect on the ^{3}He temperature and because the time constant for reduction in temperature is quite long.

Keep the 1 K pot in continuous fill mode. The temperature may be monitored using the 1 K pot resistor to check that the pot is not empty. The highest sample temperature that can be achieved by varying the pumping speed of the sorb is the temperature of the 1 K pot (as explained above), and so it may be helpful to allow the pot to warm slightly when trying to control the temperature in the region of 1.0 K or above. This may be done by reducing the pumping speed of the 1 K pot pump by partially closing the valve in the pumping line.

If a magnetic field is swept very rapidly the temperature stability may be affected because of the eddy current heating in the metallic components in the sample region.

Warning: **Set the maximum temperature limit of the sorb (channel 1) to approximately 80 K. The highest ^{3}He temperature that can be achieved is the temperature of the 1 K pot. Further warming of the sorb has little effect.**

Therefore it is possible for the sorb to continue to warm in an attempt to warm the sample further, and thermal runaway may occur. If this temperature is reached, the heater will be turned off, and there will be no risk of damage to the wiring and heaters. The ITC manual describes how to set this maximum temperature.

8.3.1.3.   Temperature control at higher temperatures

Temperatures above that of the 1 K pot can be achieved by supplying heat directly to the heater on the ^{3}He pot. In this high temperature range, the sample temperature is controlled by balancing the heat supplied on the ^{3}He pot with the cooling power of the insert. The sample is cooled by conduction through, and convection of the ^{3}He gas in the central access of the insert. The temperature of the sorb is not critical but ideally it should be kept within the temperature range from 30 K to 50 K, so that it does not pump away the ^{3}He gas in the sample space which is acting as exchange gas.

Keep the IVC under high vacuum for the entire operating temperature range of the insert.

A Cernox resistance sensor is used as the high temperature sensor as standard.

Select channel 3 for high temperature control of the insert. The Heliox heater controller box will automatically direct most of the applied power to the heater on the ^{3}He pot. In this way the temperature is controlled by supplying heat directly to the ^{3}He pot (and not to the sorb). The heater controller box will also apply some power to the sorb heater to make sure that it does not cool too much.

The three term controller settings for the ITC will be found to be significantly different from those used in the low temperature range, and may vary over the high temperature range as indicated in the test results. The test results will indicate the settings used at Oxford Instruments, and should be regarded as a starting point to allow you to control to the specifications. Better settings may be found by trial and error.

This type of insert is always found to cool samples quite slowly in the high temperature range. If you want to take readings while the temperature sweeps rapidly make sure that you take the lowest temperature readings first.

8.4. Thermal gradients in liquid ^{3}He

Some of the physical properties of liquid ^{3}He can make it difficult to achieve good thermal contact to the liquid in ^{3}He refrigerators, especially at temperatures below about 0.5 K.

- Liquid ^{3}He has a very low thermal conductivity (lower than that of a typical unfilled epoxy resin)
- At temperatures below 1.4 K its density does not change significantly with temperature and therefore convection does not assist thermal transport
- Below 0.5 K the hydrostatic pressure below the surface of the liquid may be much higher than the vapour pressure, so boiling can only occur near the liquid surface

If a pool of liquid ^{3}He is cooled by reducing its vapour pressure (as in a Heliox system) some precautions have to be taken to make sure that the sample is cooled as well as possible. If these precautions were not taken the surface of the liquid would cool to about 0.3 K but the liquid below the surface may remain at a temperature as high as 0.5 K, and cool very slowly. It may also be difficult to measure the sample temperature accurately.

In principle it would be possible to design the insert to optimise the thermal contact between the sample and the coldest part of the liquid and reduce the thermal gradients to a negligible level. However, such a system would not perform well in a sweeping magnetic field, because of eddy currents induced in high thermal conductivity components. Design of these systems is therefore the result of a compromise between good thermal contact and good performance in magnetic fields.

Two approaches can be adopted to solve these problems:

a) The inside of the ^{3}He pot (on most systems) is lined with a thin copper sheet. A slot is cut in it to minimise induction of eddy currents. If you need to reduce thermal gradients in the liquid further, (or if your system does not have this copper lining), the simplest approach is to tie several straight 0.5 mm diameter copper wires around the sample. These should be long enough to reach from the liquid surface to the bottom of the sample space. Since their diameter is small very little power will be dissipated by a sweeping magnetic field. (Power dissipation is proportional to the fourth power of diameter.) The wires conduct heat from the warmer bulk of the liquid to the colder surface and reduce thermal gradients in the liquid.

b) For systems designed to operate in a pulsed magnetic field or a rapidly sweeping field, even small diameter copper components are not permitted in the high field region. Therefore it is difficult to make sure that the sample is as cold as possible and even more difficult to measure its temperature accurately. In this case you have to calculate the position of the sample and condense enough ^{3}He to cover it barely. 1 litre of gas at NTP condenses to form approximately 1.5 cm^3 of liquid at 1.2 K. Allow an extra 10%: this will evaporate to cool the rest of the liquid to base temperature.

To measure the total volume of liquid ^{3}He that has been condensed, a simple experiment can be performed. After obtaining a remperaure of 1K apply a power of 2mW to the ^{3}He pot and measure the time required to boil off all the ^{3}He, which is characterised by a sharp rise in temperature. This usually takes a bit more than 1 hour. The total energy dissipated in the ^{3}He pot is simply the power multiplied by the hold time. The amount of ^{3}He which was condensed is calculated by dividing this energy by the latent heat of ^{3}He (30 J/mol).

Experienced users may notice a rise in temperature of the probe a few hours after obtaining base temperaure. This effect is rather small and is simply caused by the temperature profile along the probe which changes slightly in the absence of exchange gas. At temperatures below 250 mK the vapour pressure of ^{3}He is less than 10^{-4} mbar.

8.4.1. Thermal gradients between the sample and the liquid

Thermal contact between the sample and the liquid ^{3}He is difficult to calculate. However, remember the following guidelines:

- The sample will be warmer than the liquid if any power is dissipated in it
- The higher the power dissipation the higher the sample temperature will be
- The smaller the surface area of the sample the poorer the thermal contact will be
- Measuring the liquid temperature may not give you the sample temperature with sufficient accuracy
- The best thermometer to use for your sample is some effect in the sample itself (if you already know the temperature dependence of the effect that you are measuring)

If you have a small sample which does not reach the bottom of the liquid pool you may not be able to use the full hold time of the sample because the liquid level soon drops below the sample position. The sample will warm significantly if it is not covered with liquid, even if it is close to the surface.

You can improve this situation by attaching copper wires to reach the bottom of the sample space as described above, but the sample temperature will still increase when it is above the liquid surface. Another possible approach is to put a displacer below the sample to raise the liquid level. It must be made of a suitable material (for example stainless steel), so that it does not absorb liquid and so that eddy currents are not induced in it by a sweeping magnetic field.

8.5. HelioxTL fault finding

This is a summary of common faults and a description of the most likely cause of the problem. If you cannot solve the problem and suspect that there is another fault produce a full set of test data and send it to Oxford Instruments for diagnosis, along with details of any additions or modifications that you have made to the system.

Symptom and explanation	Possible cause and cure
Poor base temperature with	Check 1 K pot temperature, ensure 1 K pot is not empty and not overfilling:

Symptom and explanation	Possible cause and cure
short hold time (indicating a high heat load). Poor base temperature with long hold time, indicating that the sorb is contaminated	close 1 K pot needle valve and monitor any change. If the liquid helium level in the cryostat is below the end of the pick up tube it will not be possible to fill the 1 K pot. OR High level of exchange gas in IVC, warm to 4 K and pump with diffusion pump. OR Too much or too heavy wiring has been fitted to the probe, and is conducting too much heat into the ^{3}He OR One of the experimental access ports on the probe is not covered sufficiently well, and heat is being radiated from the warmer regions above. OR There may be a touch from the ^{3}He pot to the IVC; if all the other possibilities have been discounted, warm up the system and check the alignment. OR The heater on the probe may not be properly disconnected from its power supply (ITC). OR Some types of high temperature sensor may cause an increased heat load on the system. Try disconnecting the high temperature sensor (if fitted). Remove all the ^{3}He gas from the insert while it is still cold. Warm the system to room temperature. Pump the sample space with a rotary pump and then with a diffusion pump to remove any contamination.
Good base temperature, short hold time - (indicating poor condensation of ^{3}He)	Check 1 K pot temperature during condensation, close 1 K pot needle valve, increase flow through sorb heat exchanger to reduce heat load from sorb to 1 K pot. Check effect of larger 1 K pot pump.
Rapid temperature rise with constant experimental heat load (indicating that the sorb is contaminated)	See *Poor base temperature* above.
Very slow cool down to base temperature (indicating that there are thermal gradients in ^{3}He)	Check copper shorting wires correctly installed. OR Poor initial base temperature, with sudden drop to lower temperature - possible contamination of ^{3}He with ^{4}He Note that you can check this by lifting the probe slightly. The top of the liquid pool will be colder that the bulk of the liquid.

8.6. Heliox systems - contamination of the ^{3}He gas

We strongly recommend that you avoid contaminating your valuable charge of ^{3}He gas with ^{4}He. It is difficult to separate the two gases completely, once they have been mixed.

Impurities of ^{4}He dissolve in liquid ^{3}He according to the phase diagram of liquid solution of ^{3}He and ^{4}He. At low temperatures solutions of ^{4}He in ^{3}He phase separate and the temperature at which this occurs depends on the relative concentrations of ^{3}He and ^{4}He. For example for 1.5% ^{4}He in ^{3}He the temperature of phase separation is about 0.28K. The ^{4}He rich phase after separation is superfluid and will give rise to superfluid film flow on the solid surfaces of the ^{3}He pot up to higher temperatures where it will evaporate. Its subsequent recondensation in the ^{3}He pot will increase the heat load to the pot, thus reducing the hold time and increasing the base temperature of the refrigerator.

It is unlikely that the ^{4}He vapour will be effectively pumped out, hence the ^{4}He concentration in the liquid will gradually increase during the fridge operation, and the temperature at which phase separation occurs will also increase. Towards the end of the hold time, when most of the ^{3}He charge has been consumed the ^{4}He concentration may be large enough for this phase separation to occur. Therefore we recommend that the initial concentration of ^{4}He in the ^{3}He charge should not exceed 0.1%.

If you run the insert using pure ^{4}He, temperatures below 1.0 K can be achieved for a short time. The hold time is short due to a similar effect, in which superfluid ^{4}He creeps up the walls of the sample space until it reaches a point that is warm enough for it to evaporate. If you use ^{4}He gas in the insert, make sure that it is pumped away thoroughly (with the sorb at room temperature) before you run it with ^{3}He again.

In summary, it is not essential to buy the purest grade of ^{3}He for use in the system. Typical ^{4}He contamination levels for ^{3}He gas are 0.1% (that is, the isotopic purity is 99.9%) which is sufficiently pure to run the system. Other impurities can be tolerated at much higher levels because they will be removed at the end of the first run.

8.7. Cryostat fault finding

Symptom	Possible cause	Solutions
Poor vacuum in OVC	Leak on pumping system	Close the cryostat OVC valve and check pumping system base pressure.
	Leak on dewar or insert	Obtain a mass spectrometer leak detector and identify the source of the leak. The booklet *Practical Cryogenics* gives advice on this subject.
	Excessive moisture in OVC	Pump and flush the OVC with dry nitrogen several times, then pump to high vacuum again.
Condensation or frost on the OVC when the system is cooled down	Poor vacuum in the OVC	Pump the OVC again. Check with a mass spectrometer leak detector for leaks including leaks from the helium reservoir.
Transfer tube gets frosty	Poor transfer tube vacuum	Pump its vacuum space to high vacuum again.

Symptom	Possible cause	Solutions
Transfer tube shows ice "spots"	Internal capillary touches outer tube	If the transfer tube is still under warranty and it has not been damaged contact us for a replacement. Otherwise consider replacing the transfer tube if the liquid helium consumption is unacceptably high.
Difficulties transferring liquid helium into the system.		See the chapter on this subject in the booklet *Practical Cryogenics*.

8.8. Helium recovery systems

Helium gas recovery systems are often used to collect the exhaust gas from cryostats. They are useful for the following reasons:

- To allow the gas to be liquefied and recycled
- To collect gas for other uses (for example vacuum leak detection)
- To prevent air from entering and contaminating the cryostat
- To conserve the Earth's helium supply.

A typical recovery system consists of a low pressure gas collector, a compressor and high pressure gas cylinders to store the gas. Many different cryostats are usually connected to a central low pressure gas collector. The recovery system typically has non-return valves at strategic points to make sure that the cryostats do not interact, and the system operates slightly above atmospheric pressure to reduce the risk of contaminating the gas if there is an air leak. The compressor should be specifically chosen for use with helium because a large amount of heat is generated when it is compressed.

Many factors affect the financial implications of building and using a helium recovery system. In particular it is important to consider:

- The cost of liquid helium in your laboratory
- The cost of installing and running a recovery system and liquefier

If you do use a recovery system you should take precautions to make sure that you recover as much gas as possible and avoid contaminating the gas with air or other substances.

8.9. Useful reference books

The following books may be found useful as background reading.

Experimental Techniques in Low Temperature Physics,
by G.K.White, Oxford University Press, ISBN 0-19-851381-X

Experimental Principles and Methods below 1 K,
by O.V.Lounasmaa, Academic Press, ISBN 0-12-455950-6

Low Temperature Laboratory Techniques,
by A.C.Rose-Innes,
London: English Universities Press, ISBN 0-34004778-X
(Probably out of print, but worth looking in the library).

Properties of Materials at Low Temperature, A Compendium.
General Editor Victor J. Johnson, National Bureau of Standards.
Pergamon Press, 1961.

Vacuum Technology its Foundations Formulae and Tables
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Oxford Instruments Ltd., Ultra Low Temperature Group, 1990.

9.   Drawings

The following drawings are included. Some of these drawings have been folded so that you can see them while you are reading text elsewhere in the manual.

General assembly drawing of the system   A100269

Auxiliary gas handling system for HelioxTL   AHA0498 rev 4

HelioxTL top loading probe (rev 1)   HLXPROB1.DOC

In-Field Failure Form (IFF) (for use if you have to return any equipment to the factory).

10.   Specifications, wiring and test results

10.1.   4 and 10 way Fischer connector pin labels

Figure 5 Pin numbers on a 4 way hermetically sealed Fischer connector viewed onto pins from the outside of the cryostat. Fischer part number 103 Z053 (DBEE).

(Mating connector Fischer part number SE103 Z053)

Tip   The connector on the cryostat is a plug and the pins are accessible.

Figure 6 Pin numbers on a 10 way hermetically sealed Fischer connector viewed onto pins from the outside of the cryostat. Fischer part number 1031 Z010 (DBEE).

(Mating connector Fischer part number SE1031 Z010)

Tip   The connector on the cryostat is a plug and the pins are accessible.

10.2.   Flying leads for Fischer connectors

Leads attached to Fischer connectors by Oxford Instruments are colour coded according to the following convention.

1	Red	11	Turquoise	21	Blue/Black
2	Blue	12	Grey	22	Orange/Blue
3	Green	13	Red/Blue	23	Yellow/Green
4	Yellow	14	Green/Red	24	White/Green
5	White	15	Yellow/Red		
6	Black	16	White/Red		
7	Brown	17	Red/Black		
8	Violet	18	Red/Brown		
9	Orange	19	Yellow/Blue		
10	Pink	20	White/Blue	Body	Screen

Note: That leads with less than 24 wires are colour coded in the same way. If you have a 10 way lead you should simply ignore the information given for pins 11 to 24.

10.3. HelioxTL insert wiring

10.3.1. Insert control wiring

10.3.1.1. Ten Pin Fischer connector 1

PIN		Function
1		Sorb Heater (2 x 68 Ω heaters in parallel)
2		
3	V-	Sorb Temperarture Sensor (100Ω
4	V+	Allen Bradley)
5	I-	
6	I+	
7	V-	1K regoin RuO2 resistance sensor
8	V+	
9	I-	
10	I+	

10.3.1.2. Ten Pin Fischer connector 2

PIN		Function
1		3He pot Heater (500Ω wire wound
2		heater)
3	V-	Spare wire Terminated at 4.2 K
4	V+	
5	I-	
6	I+	
7	V-	3He pot temperature RuO2 resistance
8	V+	sensor
9	I-	
10	I+	

10.3.2. Control wiring on the top loading probe

10.3.2.1. Ten Pin Fischer connector

PIN		Function
1		Wire wound Heater on end of probe (200Ω nornal) For High
2		Temperature option only
3	V-	Cernox Sensor (For high temperature option only)
4	V+	
5	I-	
6	I+	
7	V-	RuO2 resistance sensor (Low temperature range only)
8	V+	
9	I-	
10	I+	

10.3.3. Services on the top loading probe

10.3.3.1. 24 pin Fischer connector

The Fischer connector is wired to the 1 K pot with 0.1 mm constantan wire.

10.3.3.2. Connections to ITC temperature controller

A set of connecting leads is supplied with the ITC temperature controller. They are labelled to help you to connect them to the correct points. One of the leads is used for control of the sorb temperature, and the other for the sample. The sorb lead also has a connector to allow the temperature of the 1 K pot to be monitored by the sorb, but this is not normally used.

The sample temperature may be displayed on channel 2 or channel 3 of the ITC, depending on the temperature, so there are two connectors to plug into the ITC. In normal operation, both leads may be left connected, but if you require the lowest possible temperatures and the longest hold time, it may help to disconnect the high temperature sensor from the ITC (channel 3).

10.3.3.3. Monitoring the 1 K pot temperature

If the same type of sensor is used on the ^{3}He pot and 1 K pot, the 1 K pot temperature can be monitored by the ITC if required. Disconnect the lead from the ITC (channel 2) and connect the second connector on the sorb lead (labelled 1K pot) to channel 2 of the ITC. The ITC will give the approximate temperature of the 1 K pot on channel 2. However, it is not usually necessary to do this, as the 1 K pot can be controlled by observing the pressure in the pumping line alone.

10.4. Test results

The system test results are given after this page.

Heliox TL insert

Specifications and special features

Base temperature	0.26 K
Hold time at base temperature > 50 hours	
Maximum ^{3}He charge	18 litres
Gas handling system dump volume 30 litres (nominal)	

Temperature control	- in range 0.3 to 1.2 K	± 3 mK expected
	- in range 1.2 to 80 K	± 0.1 K

Sample space diameter	38 mm

The ITC temperature controller

It is recommended that an ITC temperature controller is used to control the Heliox^{VL} system.

The ITC502 or ITC503 may be used to monitor and control the temperature of the sorb and sample, over the entire operating range of the system. It can also be used to drive an auto needle valve. If you use two ITC temperature controllers you can achieve better control in the high temperature range and drive a second auto needle valve stepper motor.

The ITC is set up with the following range cards.

Temperature controller model: ITC503

Card No.	Type of Sensor	Temperature range (K)
1	Allen Bradley, 270 ohm	4.0 - 250
2	RuO2 resistor, 2.21 kohm	0.1 to 7
3	Cernox	1.4 to 300.0

The software provided with the ITC502 or ITC503 allows the ranges to be altered easily to suit a wide range of sensors. Internal switches may need to be altered to change the excitation current and other parameters. The ITC manual contains full details of this procedure.

Test results

Base Temperature, measured with calibrated thermometer	K
Hold time at base temperature (full charge of ^{3}He)	hours
1 K pot helium boil off (with insert at base temperature) in continuous fill mode.	cm^3/hour

Sensor calibrations

High temperature sensor

Sensor serial number

Temperature	300 K	77 K	4.2 K
Resistance (ohms)			

Diagnostic Thermometer calibrations (ohms)

	300 K (2 wire)	4.2 K (2 wire)	Base temperature
1K plate resistor			
^{3}He pot resistor			
Probe resistor			

Temperature control tests

Temperature control in high temperature range

Temperature (K)	Stability (mK)	Sorb temperature (K)	P	I	D

Heater voltage limit (high temperature range)   40 volts

Temperature control in low temperature range

Temperature (K)	Stability (mK)	P	I	D	1 K pot flow l/min	Sorb temperature (K)

Heater voltage limit (low temperature range)   40 volts

* Using card 2 to control (others using card 3).

Weight and dimensions of the system

Approximate weight of the insert   30 kg

Minimum ceiling height requirement for sample loading   * m

Minimum ceiling height requirement for helium transfer   * m

* See system GA (General Assembly) drawing for room height details. *

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