Operator's Handbook

HelioxVT

Sorption pumped ^3He Insert

Revision 3

July 06

UMC0045

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Contents

1 Important Information

1.1 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.

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

1.2 Safety

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

1.3 Important Note

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

1.4 Important Health and Safety Notice

Important Health and Safety Notice

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

1.5 Conventions used in this manual

The following conventions have been followed in this manual:

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

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

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

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

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

1.6 Disposal and recycling instructions

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

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

1.7 Other information supplied with this manual

The following information is supplied with this manual or available on request:

- Safety Matters - essential information to help you to run a system safely
- Practical Cryogenics
- Heliox insert data sheet
- Thermometry and resistor calibrations for Heliox systems
- Useful reference books

2 Introduction and description

This manual is designed to introduce you to this sorption pumped ^{3}He refrigerator, whether you are experienced in the operation of cryogenic systems or a beginner. It is a relatively simple ^{3}He system to run because the ^{3}He gas is safely stored within the Heliox insert at all times.

In you have bought a complete system from Oxford Instruments, a separate system manual will have been supplied. You should cool down the system as described in the system manual before you load the Heliox insert into the Variable Temperature Insert (VTI). This free standing manual then describes how to load the Heliox insert into a cold system.

2.1 General description of the complete system

The HelioxVT system is a low cost, short lead time ^{3}He refrigerator designed to operate in an Oxford Instruments' Variable Temperature Insert (VTI). The VTI may form part of a superconducting magnet system, or part of a stand alone cryostat (an Oxford Instruments' Variox, for example). The sample is changed by warming the entire insert to room temperature, and removing the IVC. The Heliox is cooled to < 2 K by the VTI through exchange gas. The Heliox will run in any VTI that it fits in to, but only a VTI/Heliox system supplied by Oxford Instruments will guarantee the optimum performance from the Heliox.

A leak detector or turbo pump is required to pump exchange gas from the IVC of the Heliox.

At temperatures below the base temperature of the VTI, the temperature of the sample and the sorption pump are controlled by an Oxford Instruments ITC502 or ITC503 temperature controller (if ordered). It is possible to set up a computer interface to the ITC using GPIB or RS232. This allows you to control the operation of the insert automatically using Oxford Instruments ObjectBench or Oxsoft software. Above the base temperature of the VTI, the temperature of the sample is controlled through the temperature of the VTI.

The operating temperature range of the insert is from 0.3 K to 300 K but the working range of the VTI may reduce this.

A range of calibrated thermometers is available for the Heliox insert to help you to measure the temperature of your sample accurately. The insert is fitted with uncalibrated thermometers for the range below 1 K as standard. The high temperature sensor is a cernox resistor with a 39 point calibration.

2.2 Brief description of the Heliox insert

Refer to the drawings of the Heliox in this manual.

The insert has a vacuum seal which allows it to be loaded into the VTI without allowing contamination of the neck of the VTI with ice. The inner vacuum chamber (IVC) is sealed by a greased cone seal, allowing the system to be used by relatively inexperienced personnel. There are no indium seals.

The spare port gives line of sight access through the insert from room temperature to the sample space. You can use it to install services for your experiment. However, it is important to heat sink all of these services effectively to minimise the effect on the insert's performance.

The charge of ^{3}He is sealed into a self contained storage vessel so that it is not necessary to remove the valuable gas from the insert when it is warmed to room temperature. This reduces the complexity of operation of the system. The (nominal) 2.5 litre charge is stored at a pressure of approximately 2 bar (absolute). The storage vessel is fitted with a pressure relief device.

The sample is mounted in vacuum on the underside of the ^{3}He platform, or on the end of a low eddy current sample holder at the field centre of a superconducting magnet.

2.3 Principle of operation of the Heliox insert

The sample is mounted in vacuum on the base of the ^{3}He pot, or on the sample holder as shown on the drawing inserted into this manual. When the sample has been mounted and the IVC has been sealed and evacuated, the insert may be cooled by lowering it into the VTI.

The insert is designed to allow the temperature of the sample to be controlled in the range from 0.28 to 300 K, and this is done using two separate modes of operation. Below the base temperature of the VTI, typically 1.5 K, the Heliox runs as a sorption pumped ^{3}He insert. Above 1.5 K, the IVC of the Heliox is flooded with exchange gas and it acts as a sample rod in the VTI. Please refer to the manual for the VTI for operation above 1.5 K.

2.3.1 Operation as a sorption pumped ^3He system

The Heliox consists of three main components, connected by a tube (see Figure 1):

1. The sorption pump (or 'sorb'). This is a volume containing a quantity of charcoal that has a very high surface area. When cold, this charcoal is capable of pumping, at high vacuum, more than 0.1 moles of ^3He gas before it becomes 'saturated'. Once the surface area is saturated with ^3He, it no longer pumps, and it must be heated ('out-gassed') in order to liberate the ^3He gas that it has adsorbed. During condensation, the sorb will be warmed to > 30 K for ~1/2 hour, which is enough time for all the ^3He to condense.

2. The 1 K plate. This plate is designed to remain at the temperature of the VTI (~1.5 K) even when the sorb is being out-gassed. It is here that the 3He gas condenses, and then drops down into the 3He pot.

3. The 3He pot. This is a reservoir for the 3He liquid. Once all the 3He has condensed into the 3He pot, the sorb is allowed to cool, and it starts pumping the gas away. As the vapour pressure drops, so does the temperature of the liquid 3He, and the sample attached to the base of the 3He pot cools too. The ultimate base temperature achieved is determined by the heat load on the 3He pot from its surroundings, and the pumping speed of the sorb.

To regulate the temperature above the natural base temperature of the Heliox, the pumping speed of the sorb is reduced by warming it. By heating the sorb and not the ^3He pot, the hold time at the desired temperature is not decreased.

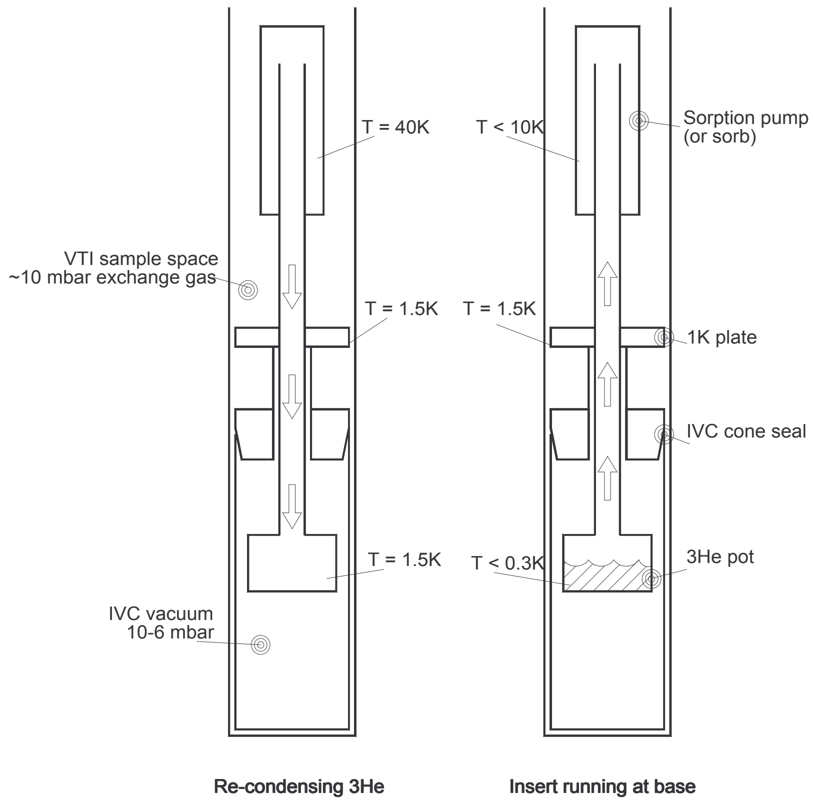

Figure 1 Operation principle of a sorption pumped ^3He insert

3 Commissioning and routine maintenance

3.1 Commissioning requirements

The Heliox Insert data booklet lists the typical commissioning requirements and indicates which parts have been supplied with the insert.

3.2 Unpacking and initial assembly of the system

The system should be unpacked carefully and inspected for any damage caused during shipment from Oxford Instruments. The IVC should be removed as described in section 4.1, and the packing piece should be removed from the underside of the ^{3}He pot before attempting to run the system.

Check to ensure that none of the components are missing. If any problems are encountered you should contact Oxford Instruments (through the agent if appropriate).

3.3 Control wiring details

3.3.1 Insert control wiring

Ten Pin Fischer connector 1

Pin		Function
1	Start	Sorb heater (1 x 34 ohm heater)
2	End	
3	V-	Sorb thermometer (100 ohm Allen Bradley)
4	V+	
5	I+	
6	I-	
7	V-	1 K plate thermometer (2200 ohm RuO_2)
8	V+	
9	I+	
10	I-	

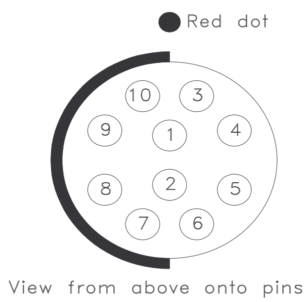

Figure 2 Pin numbers on a hermetically sealed Fischer connector 1031 Z010 (DBEE), viewed from the outside of the cryostat

Ten Pin Fischer connector 2

Pin		Function
1	Start	^{3}He pot heater (200 ohm wire wound)
2	End	
3	V-	High temperature sensor on ^{3}He pot
4	V+	
5	I+	
6	I-	
7	V-	Low temperature thermometer on ^{3}He pot (2200 ohm RuO_2)
8	V+	
9	I+	
10	I-	

3.3.2 Connections to ITC temperature controller

The ITC is configured to read the following sensors:

	Calibration	Range	Sensor location
Channel 1	100 Ω Allen Bradley	1.4 - 250 K	Sorb
Channel 2	2200 Ω RuO_2	200 mK - 7 K	^{3}He pot or 1 K plate
Channel 3	Cernox	1.5 - 300 K	^{3}He pot

Cables are supplied which allow connection from the 9-way 'D' connectors on the ITC to the 10 way Fischer connectors on the Heliox (please refer to the ITC manual to find more details of the pin connections). These cables also make electrical connection to the heaters associated with each sensor. These heaters are the sorb heater (34 Ω), and the ^{3}He pot heater (200 Ω).

For operation at the lowest temperatures, it is necessary to un-plug the high temperature sensor from the back of the ITC (channel 3), since the excitation current used on this channel dissipates enough heat in the sensor to represent a significant heat load on the ^{3}He pot.

The same type of sensor is used on the ^{3}He pot and the 1 K plate, and the temperature of the 1 K plate can be monitored by the ITC if required. Disconnect the lead from Fischer connector 1, and swap the lead from Fischer connector 2 to connector 1. The approximate temperature of the 1 K plate will now be displayed on channel 2. In normal operation, the 1 K plate will be at the same temperature as the VTI (or slightly warmer), and using the ITC to measure the 1 K plate in this way need only be used to diagnose problems.

3.4 Routine maintenance

It is unlikely that any routine maintenance will be required on the Heliox insert, but some of the accessories may require maintenance at intervals recommended by the manufacturer.

3.4.1 Pumping system

The recommendations of the manufacturer of any vacuum pumps should be followed.

Note: Most vacuum pumps require regular servicing, and the oil must be changed at the intervals specified by the manufacturer.

4 Mounting the sample and final assembly of the system

4.1 Removing the IVC

The greased cone seal cannot easily be removed without using the slide hammer which is supplied with the insert. This is because the cone has a locking taper to ensure that the IVC does not fall off accidentally.

Caution: Do not attempt to remove the IVC by prising it away from the flange or by twisting it relative the insert. Use the slide hammer carefully as described below.

Vent the IVC by opening the valve in the pumping line. Screw the slide hammer fully onto the thread at the bottom end of the IVC, and hold the insert firmly. Slide the moving part of the hammer sharply away from the insert, taking care not to trap your fingers between it and the end flange. When the moving part hits the end flange the impact will tend to pull the IVC tube away from the flange.

Caution: It is important that you operate the hammer gently to avoid straining the vacuum tight soldered joints on the IVC. It should take between 5 and 10 gentle operations to remove the IVC.

4.2 Experimental access on the insert

The experimental wiring on the insert is described briefly in the separate Heliox insert data booklet. You can add extra wiring to the insert to suit your experiment. It is important to realise that the number of wires connected to the sample may affect the low temperature performance. In particular, it is important to consider the amount of heat conducted down the wires; in general it is preferable to use thin high resistance wires whenever possible. A heat load of 10 to 15 μW may reduce the hold time of the insert by a factor of two.

The spare port may be used to bring other services down to the sample space. Take care to ensure that additional services are heat sunk effectively, and that the lower end of the spare port tube is covered with foil to minimise thermal radiation leaks into the IVC.

4.3 Mounting the sample

Remove the IVC as described in section 4.1.

The sample is mounted in vacuum on the base of the ^{3}He pot. Several tapped holes are provided for this purpose. The sample must be thermally anchored to the ^{3}He pot if it is to be cooled efficiently. In general the best thermal contact is achieved by metal to metal contact between two clean copper surfaces firmly pressed together. If preferred, the copper surfaces may be gold plated to prevent corrosion.

If the sample is delicate and non metallic, it is often sufficient to cool it through the electrical leads, which should be thermally anchored to the ^{3}He pot.

Connect the wiring to the sample, and check all the wiring carefully before you fit the IVC.

4.4 Fitting the IVC using the greased cone seal

The IVC is sealed onto the insert using a greased cone seal. This type of seal is quick and simple to make, and it requires much less cleaning time than an indium seal when the IVC is removed again.

Caution: It is important to ensure that the surfaces of the cone seal are kept clean and free from scratches. It is good practice to remove the grease from the mating surfaces as soon as the IVC is removed so that dirt is not allowed to collect on them.

Make sure that your hands are clean so that no small particles are allowed into the seal. The seal is made using a silicon based vacuum grease[1]. Rub a thick layer of grease thoroughly onto the surface of the cone on the IVC flange; rub a thin uniform layer onto the inner surface of the IVC tube. Fit the IVC carefully over the insert, making sure that none of the grease is removed by the ^{3}He pot, 1 K plate or the sorb. Push the cone into the IVC with a slight twisting action.

Evacuate the IVC to a rough vacuum (< 1 mbar). This will pull the cone seal together very firmly, and it will not be possible to separate it again without using the slide hammer provided with the insert. The cone is designed to be a locking taper, so that the IVC will not fall off accidentally even if it is vented to atmospheric pressure.

4.5 Preparation for cool down

After you have pumped the IVC to a rough vacuum, close the IVC valve and remove the pump. Allow a small amount of (recovery grade) helium gas into the IVC. Approximately 10 cm^3 (NTP) should be sufficient. The best method for allowing a small amount of helium to enter the IVC is a bladder fitted to the IVC valve.

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[1] More than one type of grease is avialable; the most appropriate type will be supplied with the system.

5 Running the Heliox insert

5.1 Cooling the insert down

Prepare the insert as described in section 4. The insert is now ready to be inserted into the VTI. This operation should take approximately 30 to 40 minutes once you are familiar with the procedure. Connect the insert wiring to the ITC temperature controller so that the cooldown can be monitored easily. If the ITC is connected to a computer, the temperatures can be logged to show the rate of cooling.

Warning: **Make sure that you are aware of the precautions that you must take to ensure your own safety, and the safety of other people working around you. You should receive proper training from a competent person before using cryogens.**

5.1.1 Pre-cooling using liquid nitrogen

The most rapid cooldown can be achieved using a deep bucket of liquid nitrogen to pre-cool the insert. Lower the insert slowly into the liquid nitrogen, and support it so that it will not fall over. The time required for pre-cooling will depend to some extent on the mass of the sample. If the sample is very light, the ^{3}He pot should reach 100 K after approximately 15 to 20 minutes.

Caution: Pre-cooling in liquid nitrogen should not be employed if the IVC is of bi-metallic construction (e.g. Aluminium/Beryllium, or Aluminium/Brass), since the differential thermal contraction may break the joints between the metals if they are cooled too quickly.

5.1.2 Loading the insert into the VTI

The Heliox insert can be treated like any sample rod for the VTI. A full description of the procedure for changing sample rods can be found in the manual for the VTI. A brief description is included here:

The sample space of the VTI should be pressurised to slightly more than one atmosphere of helium to avoid contamination being ‘sucked in’ during changes of the sample rod. The needle valve on the VTI should be shut. Remove the blanking cap (or the previous sample rod) from the VTI and insert the Heliox.

Start to pump the VTI. Open the needle valve on the VTI gradually, to set sufficient flow of helium through it. If a pressure gauge is available on the VTI pumping line, then the needle valve should be adjusted to maintain a pressure of 50-100 mbar. As the VTI cools, the flow of gas will increase (especially once it is below 50 K) and the needle valve will need to be closed gradually.

If the insert is to be used at a temperature below 1.5 K the procedure in section 5.2 should now be followed. If it is to be used only at higher temperatures, follow the procedure in manual for the VTI to control the sample temperature.

5.2 Pumping exchange gas, ^{3}He condensation and cooling to base temperature

When the VTI reaches ~10 K, the exchange gas should be pumped from the IVC using a leak detector or turbo pump. Connect the pump to the NW16 fitting on the insert, pump down the lines, and open the vacuum valve on the insert. If a leak detector is used, wait until the signal is < 10^{-6} mbar litres/sec. If a turbo pump is used, it is usually sufficient to pump for 1 hour.

Note: It is important that the IVC is pumped while the VTI temperature remains higher than 4 K. If the VTI cools to below 4 K during pumping, the temperature of the VTI should then be set to 4 K. Once the exchange gas has been pumped, the VTI should cool to its base temperature.

It is possible to control the Heliox insert automatically from a computer using Oxford Instruments Oxsoft or ObjectBench software. The software manuals explain how to do this.

When the insert is precooled to below 6 K as described above, it is ready for condensation of the ^{3}He gas.

Set the sorb temperature to 30-40 K to ensure that it will not pump the ^{3}He gas. Take care to ensure that the correct heater (1) is selected on the ITC so that the sample and ^{3}He pot are not accidentally warmed. As the 1 K plate cools below 3 K, the ^{3}He gas will start to condense and run down into the ^{3}He pot. The sample temperature will be seen to drop quickly to around 1.5 K. Once the ^{3}He pot and the 1K plate have reached a stable temperature, condensation is complete. The condensation process should take between 20 and 30 minutes. This method of condensation is sufficient for normal operation and it is used by the software for automatic control of the insert

When the sample temperature is steady, the insert can be cooled to base temperature. This is done by cooling the sorb, switch off the sorb heater by setting the sorb temperature on the ITC to 0 K. The sorb will be found to cool over a period of minutes and the sample temperature should drop with the sorb temperature. If the sample is light and has a high thermal conductivity, it should reach base temperature within 15 to 25 minutes.

If the experimental heat load is very low, the insert should stay at base temperature for longer than 30 hours. The VTI temperature must be maintained at less than 2 K while the Heliox is at base temperature. The ITC that controls the VTI (if one was ordered) will automatically control the needle valve to ensure that the VTI does not run out of liquid helium (refer to the manual for the VTI).

The base temperature and hold time of the insert may be slightly affected by the self heating of the high temperature range sensor. If the lowest possible temperature and longest possible hold time are desired, it is necessary to unplug the high temperature sensor from the ITC (channel 3). This will ensure that the excitation current in the sensor does not supply heat to the system. The base temperature may be improved by 10 to 30 mK, and the hold time may be doubled by disconnecting the sensor.

5.3 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 resistor mounted on the ^3He pot. These resistors are quite insensitive to a magnetic field and so are very useful for control of temperature, even when the field is swept. If the temperature is to be measured accurately, it is recommended that a calibrated sensor is used; for example, a germanium or RuO_2 resistor.

Two separate heater outputs from the ITC are required (for example, for sorb control and for high temperature control using the ^3He pot heater), so a switching card (part number CQB 1800) is fitted to the back of the instrument to switch the heater output automatically. The switching card should be fitted to the ITC and the link between the heater outputs from card 1 and card 2 (marked LK 1-2 on the printed circuit board) should be made by soldering a zero ohm resistor onto the board. The switching card is not needed if the insert will only be used at temperatures below 1.5 K, but the heater on the ^3He pot must then be disconnected. As an option to the CQB1800 a CQB2200 heater share box can also be supplied.

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.

Card 1	For use with the sensor fitted to the sorb (for example, Allen Bradley carbon resistor). This gives an indication of the temperature of the sorb during re-condensation of the ^3He. Note that the accuracy of this sensor is not critical for successful operation of the system.
Card 2	For measurement of the ^3He pot temperature with resolution of 1 mK. This card is configured for a RuO_2 resistor.
Card 3	For measurement of the ^3He pot temperature in the range 1.5 to 300 K. This card is configured for a cernox resistor.

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

5.3.1 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 below 8 K, so the ^3He 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 ^3He temperature will be largely dependent upon the temperature of the 1 K plate. Intermediate sorb temperatures are used to control the ^3He 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 plate. 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 range card is used, and this is described in Table 1 (see card 2).

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 ^3He temperature and because the time constant for reduction in temperature is quite long.

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.

Caution: Set the maximum temperature limit of the sorb (card 1) to approximately 80 K. The highest ^3He temperature that can be achieved is the temperature of the 1 K plate. 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.

5.3.2 Temperature control at higher temperatures

A Cernox or RhFe sensor is used as the high temperature sensor as standard; the choice depending on the magnetic field of the sample environment.

The simplest way to control at higher temperatures is to flood the IVC with exchange gas and set the temperature using the VTI. This method should work over the entire temperature range of the VTI (1.5 - 300 K), but the temperature of the ^3He pot may be slow to respond to changes in the VTI temperature.

Alternatively, temperatures above that of the 1 K plate can be achieved by supplying heat directly to the heater on the ^{3}He pot. Keep the IVC under high vacuum. In this mode of temperature control, 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 tube of the insert. In order to keep a reasonable pressure of ^{3}He gas in the central tube, it is necessary to apply some heat to the sorb. The temperature of the sorb is not critical, but ideally it should be kept within the temperature range from 30 K to 50 K. This is handled automatically if a heater share box CQB2200 is used, if this box is not available, a separate power supply needs to be found. Select channel 3 for high temperature control of the insert.

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.

5.3.3 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 sorb heat exchanger flow.

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.

5.4 Warming the system to room temperature

The procedure to be followed to warm the insert to room temperature is quite straightforward, and it can be done quickly (typically 10 to 15 minutes). However, it is important to remember several points for safety reasons.

Warning: **When you warm up the insert make sure that the IVC is free to vent safely. Any remaining cryogens or contamination can then escape without causing damage to the insert.**

Connect an empty bladder to the IVC pumping line and open the valve. This ensures that any gas that has leaked into the IVC can expand freely into the bladder. It is unlikely that the bladder will expand at all as the insert is warmed since there should be no gas in the IVC.

Shut the needle valve on the VTI and set the VTI temperature to >100K. When the sorb sensor on the Heliox reads >100 K, the insert can then be pulled out of the dewar quickly. If the Heliox has not been allowed to warm above 100 K, some liquid air will run from the cold metal as it is withdrawn, so it is preferable to lift the insert out quickly to prevent the collection of solidified air in the VTI. Cover the top of the VTI as soon as possible, using a blank flange or bung.

The insert may now be warmed gently with a hot air blower. If the IVC was under high vacuum, some exchange gas should be re-introduced. It is important to ensure that the IVC is not removed before the ^{3}He pot is warmed to near room temperature to prevent the condensation of moisture on the sample.

The IVC may then be removed using the slide hammer as described in section 4.1.

5.5 Fault Finding

This section begins with a more detailed explanation of the workings of the Heliox, in order to clarify the possible causes of a fault. Particular faults are then discussed with respect to the correct functioning of the Heliox. If the problem cannot be solved with the aid of this manual and another operational fault is suspected, a full set of test data should be produced and sent to Oxford Instruments for diagnosis along with details of any additions or modifications that you have made to the system.

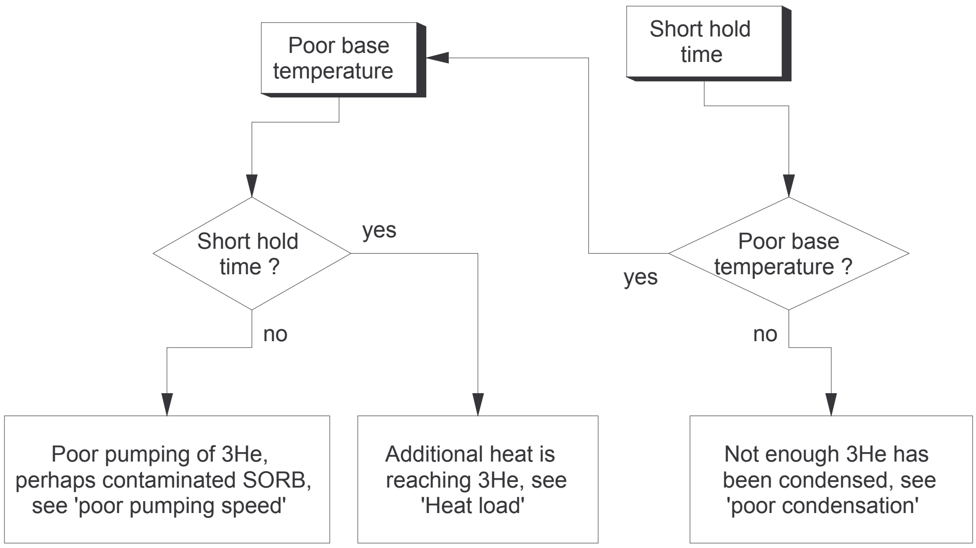
5.5.1 Normal operation

After condensation, there will be n moles of 3He liquid sitting in the 3He pot. Once the sorb has been pumping for a while, and the 3He has cooled to base temperature, a steady state is established where the 3He flows up to the SORB at a rate of $\dot{n}$ moles/sec. The hold time of the system is then given by $n/\dot{n}$. The heat load on the 3He pot is:

$$\dot{Q} = \dot{n}L$$

where L is the latent heat of 3He at base temperature (26.2 joule/mol @ 300 mK, varying slowly with temperature). $\dot{Q}$ is the total heat load on the 3He pot from all external sources, which may be through the support and wires connected to the pot, or through heat applied by the user. When the Heliox is running at base temperature, and no heat is being applied by the user, the remaining heat load is called the heat leak, $\dot{Q} = \dot{Q}_{LEAK}$.

In general, poor performance of the Heliox can be attributed to one of three causes. The following flow diagram explains how to distinguish between these three general problems, then they are each explained in more detail:



5.5.2 High heat load

This means that $\dot{Q}_{LEAK}$ is too high. This causes $\dot{n}$ to be too high, and the hold time at base temperature will be too short. In addition, the base temperature is degraded because the pressure (and hence temperature) in the 3He pot must be high in order to drive the high flow rate. This is probably the most frequently encountered problem with HelioxVT inserts, possible causes are

- Temperature of 1 K plate too high - compare the temperature of the 1 K plate with that achieved during system test. If this is the case, check the temperature of the VTI, and adjust the flow so that the 1 K plate cools.

Note: If the Heliox is being used in a different VTI from the one it was tested in it may be impossible to reach the same temperature on the 1 K plate.

- High level of exchange gas in IVC. Check the quantity of 4He gas in the IVC with a leak detector, this should be give a leak rate signal <10^{-5} mbar litre/sec. The cone seal is the easiest joint to check; see section 5.2.

- Too much or too heavy wiring has been fitted to the insert and is conducting too much heat into the ^{3}He. Try to improve the heat sinking, reduce the diameter of the wiring or increase its length.

- One of the ports on the IVC is not baffled off sufficiently, and heat is being radiated from the warmer regions above. Check the line of sight port especially carefully.

- The heater on the ^{3}He pot may not be properly disconnected from its power supply (ITC). Check that the heater switching card on the back panel of the ITC is working correctly (i.e. no volts across heater pins with 200 Ω across them).

- There may be a touch from the ^{3}He pot, the sample, or the wiring to the IVC; See if the ^{3}He pot temperature is affected by small changes in the VTI temperature. If all the other possibilities have been discounted, warm up the system and check the alignment.

- The excitation current for the high temperature sensor may introduce a small heat load onto the ^{3}He pot and warm it slightly. If a RhFe resistor is used, it is best to unplug it from the ITC when operating the insert at the lowest temperatures.

5.5.3 Poor condensation

- Check the temperature of the SORB and 1 K plate during condensation. The SORB should be set to the temperature recommended in the test results section of the manual. If the 1K plate is warmer than that appearing in the test results then there will be less 3He condensed and the hold time will be lower than that measured during system test at the factory. If the 1K plate is too warm, then try adjusting the flow of gas through the VTI to see if lower temperatures can be achieved there.

- Loss of ^{3}He, check pressure of ^{3}He at room temperature using a small volume gauge, it should be ~2 bar.

If this is suspected, it is worth making some checks on the thermometry:

Try and check the base temperature with an independent thermometer - it is possible that the resistance thermometer has been damaged or its calibration has changed after a violent thermal shock.

- If the thermometry is correct, then it is possible that the SORB is contaminated, either contact Oxford Instruments for advice, or remove the ^{3}He from the insert and clean it using a cold trap, then pump the sorb using a diffusion pump while warming it to 50°C.

5.5.4 Poor pumping speed

5.5.4.1 Consistent leaks on the IVC cone seal

Check for scratches and polish them away carefully. Deep scratches in the male cone could be filled with low melting point solder (for example Wood's metal), and then polished. Check that you are using enough grease to seal the cone properly. A good fillet of grease should be visible after the seal has been made. It is helpful to rub the grease thoroughly into the metal surface.

5.6 Test results and system specific information

The test results for your system are given in a separate booklet (called "Heliox insert data") which includes all the information specific to your insert. In particular it gives details of:

- Experimental wiring
- Services fitted in the spare port
- Equipment supplied with your system and commissioning requirements

5.7 Summary flow chart for experienced users

Numbers in bold type refer to the section number in this document.