

Operator's Handbook

HelioxVL

Insert Manual

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Warnings

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

A Quick Start guide is included as the last page of this manual.

Other information supplied with this manual

The following sections are supplied with this manual or available on request:

- HelioxVL Insert Manual (this document)
- HelioxVL Insert Data
- System drawings
- Auto Needle Valve for ITC502 and ITC503
- Heliox Heater Controller manual
- 2200 Ω RuO₂ Resistance Thermometer - Generic Calibration
- Safety Matters (English)
- Practical Cryogenics
- Cryospares

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

If you have bought a complete system from Oxford Instruments, a separate system manual will have been supplied. You can cool down the system as described in the system manual before you load the Heliox insert in the liquid helium reservoir. This free standing manual then describes how to load the Heliox insert into a cold system. However, if you prefer, you can load the Heliox into the system and cool it down at the same time. The manuals are structured in this way because you may be able to use the Heliox insert in several different systems in your laboratory.

2.1 General description of the complete system

The Heliox system is a low cost, short lead time ^3He refrigerator which can be used in a liquid helium storage dewar, in a typical Oxford Instruments magnet system, or in a large sample space dynamic variable-temperature insert. It has an outer diameter of less than 50 mm so that it will fit into a wide range of cryogenic systems and it is simpler to operate than a conventional ^3He system. The sample is changed by warming the entire insert to room temperature, but because the insert is so small the sample change time is not much longer than that of a top loading system.

The Heliox insert must be mounted in a bath of liquid helium in a suitable cryostat. There are many variations in the design of such liquid helium cryostats, and the precise details are not usually important. The insert will fit into any dewar which is sufficiently long and has a clear neck diameter greater than 50 mm. The minimum liquid helium level is the end of the pick up tube which supplies liquid to the 1 K plate and to the sorb. If the dewar is very short, the Heliox insert may be affected slightly. In particular, the current leads for an integral superconducting magnet may not be able to carry current continuously if a large part of their length is near to room temperature.

A simple low cost external pumping system is required for operation of the insert. You may be able to make up this system from components already available in your laboratory, but Oxford Instruments can supply a complete pumping system to suit. The temperature of the sample and the sorption pump are controlled by an Oxford Instruments ITC503 temperature controller (if ordered). The ITC can be configured to give full control of the system, and it is possible to set up a computer interface using IEEE or RS232. This allows you to control the operation of the insert automatically by software. Using one ITC you can achieve good control at high and low temperatures.

The operating temperature range of the insert is from base temperature to 300 K, but it should be noted that the maximum temperature is limited if the insert is to remain immersed in liquid helium. The radiated heat load from the sample to the liquid helium reservoir increases rapidly with temperature. In this case the upper limit is between 100 and 200 K depending upon the helium boil off that can be tolerated. This may be especially important if the insert is used with a superconducting magnet (which has to be immersed in liquid helium when it is operating).

A range of calibrated thermometers is available for the Heliox insert to help you to measure the temperature of your sample more accurately. The insert is fitted with uncalibrated thermometers for the range below 1.5 K as standard. The high temperature sensor is a Cernox.

2.2 Brief description of the HelioxVL insert and Principle of operation

Refer to the drawings of the Heliox insert overleaf.

The insert has a sliding seal, which allows it to be loaded slowly into the liquid helium without losing the gas or allowing gross contamination to enter the neck of the vessel and collect as ice. Full and efficient use is made of the enthalpy of the cold gas, and the amount of liquid boiled during the loading process is minimised. The inner vacuum chamber (IVC) is sealed by a greased cone seal, allowing the system to be used by relatively inexperienced personnel.

Caution: Anyone working with the Heliox Insert must be familiar with the precautions they must take to ensure their own safety and the safety of those people working around them.

There is no need to make indium seals during the sample changing procedure. There are no indium seals on the insert at all. An exchange gas sorb is mounted on the 1 K plate; it is used to pump the exchange gas from the IVC automatically during the cooling procedure.

The two experimental ports give line-of-sight access through the insert from room temperature to the sample space. They will either be left empty for you to install services for your experiment or they will contain one or more of the configured options you may have selected for your system. It is important, if you are fitting your own services, to heat sink them effectively to minimise the effect on the insert's performance.

The charge of ^3He 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 base of the ^3He pot see Figure 1. When the sample has been mounted, the IVC should be sealed and evacuated, and then a small amount of exchange gas added to the IVC. The insert may then be cooled by lowering it slowly into the liquid helium reservoir.

The insert is designed to allow the temperature of the sample to be controlled from base temperature to 300 K, using two separate modes of operation. Below the temperature of the 1 K plate it runs as a conventional sorption pumped ^3He insert. Above this temperature the ^3He pot is heated directly. Both methods are described below. It is important to note that the available temperature range may be restricted if the Heliox is used with a superconducting magnet. Normally the insert will be withdrawn into the upper part of the neck of the cryostat for operation above 100 K (so that the liquid helium boil off is not affected significantly), but clearly the magnet cannot be used unless it is immersed in liquid helium. The upper temperature limit is then set by the helium boil off that can be accepted, and the limit will typically be found to be between 100 and 200 K.

If a magnet is supplied the sample can be mounted on a low eddy current sample holder at the field centre of the superconducting magnet. The insert can be used either in an existing 52 mm bore magnet or in conjunction with an integral magnet.

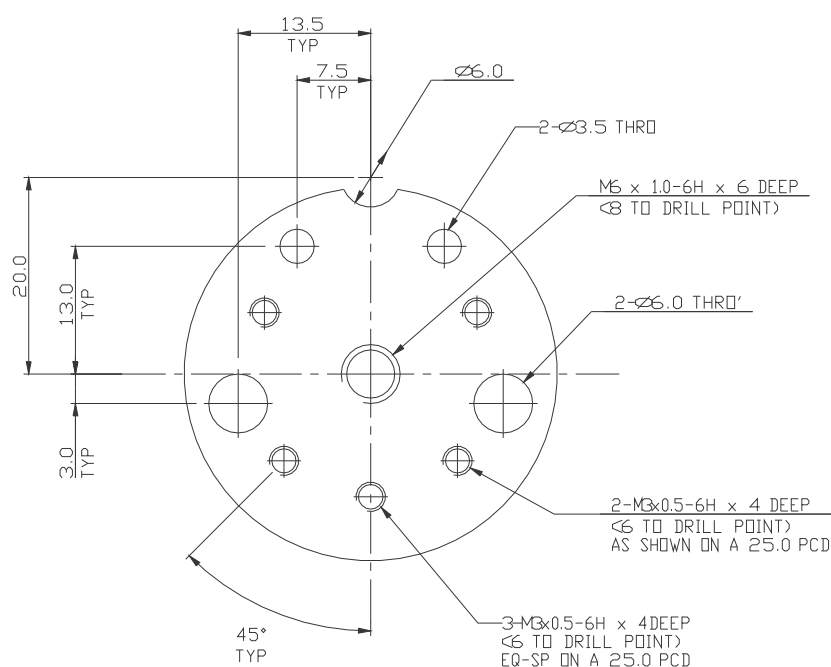


Figure 1 ^3He pot details for sample mounting

2.2.1 High temperature operation

Temperatures above the 1 K plate temperature (approx. 1.2 to 1.5 K) may be achieved by using a heater on the ^3He pot, while the temperature is monitored by a Cernox sensor. The temperature can be controlled using an ITC temperature controller, which automatically balances the heater power against the cooling from the 1 K plate. A small flow of ^4He is maintained through the 1 K plate to provide cooling.

At the highest temperatures it is most efficient to lift the Heliox insert up such that the IVC is located in the neck of the liquid helium cryostat. The available enthalpy of the cold ^4He gas evaporating from the main bath can then be used to provide the required cooling power. The IVC sorb will outgas and provide a weak thermal link to the ^3He pot. If the ^3He pot cools too quickly it may be necessary to evacuate the helium exchange gas from the IVC using the 1 K plate pump.

2.2.2 Operation as a sorption pumped ^3He system

When the sample has been mounted and the insert has been cooled to approximately 4.2 K, the 1 K plate is pumped and the needle valve is opened slightly to allow liquid helium to flow. The plate cools to below 1.5 K. The sorb is warmed to approximately 30 K so that it will not pump ^3He gas (it will release adsorbed gas). The ^3He gas is free to condense in the 1 K plate region of the central tube and runs down to cool the ^3He pot and sample, see Figure 2. After approximately 20 minutes most of the gas should have condensed. At this stage the ^3He pot is nearly full of liquid ^3He at approximately 1.5 K. At this point the sorb is cooled by removing the heat, and it begins to reduce the vapour pressure above the liquid ^3He and so the sample temperature drops. The 1 K plate needle valve is then set to a pre-determined flow setting the value of which can be found in the Insert Data section of this manual.

Intermediate sorb temperatures can be used to set sample temperatures between base and the temperature of the 1 K plate without applying heat to the ^3He (which would affect the hold time). The Oxford Instruments ITC temperature controller is suitable for this type of control, and it is able to adjust the sorb temperature automatically to maintain a fixed sample temperature within this range for the complete hold time of the system.

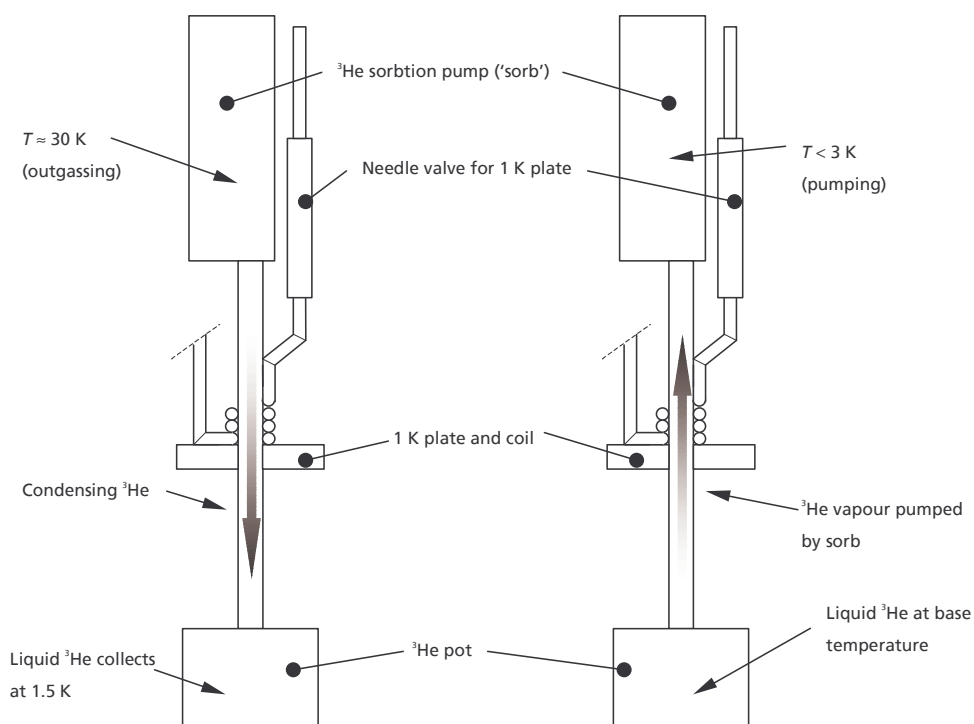


Figure 2 Operation principle of a sorption pumped ^3He insert

2.3 Auxiliary pumping systems

The recommended minimum network of valves and pumps for efficient operation of the insert is shown in Figure 3 on the following page. If the insert is used with a superconducting magnet it is preferred that the main bath is not pressurised for safety reasons.

A very simple pumping system is required for the Heliox insert. A small rotary pump is used to pump the 1 K plate, and it can also be used to evacuate the IVC to the rough vacuum required before the system is cooled down. A single-stage 8 m³/h pump is sufficient for these purposes. It may be possible to use an even smaller pump, but the condensation of ³He may not be as efficient (or quick), and the ³He hold time would therefore be affected. There is no advantage in using a two stage pump, but larger displacement pumps make the setting of the 1 K plate needle valve less critical, and so make it easier to run the system.

Access to a helium sensitive leak detector may be useful if any problems are suspected with the system, but it should not be required during normal operation. If you do use a leak detector it is important to remove the exchange gas sorb from the 1 K plate so that it does not obscure a small leak by absorbing the gas. Although for the first few operations by a new user it is useful to check the IVC even if a sorb is fitted.

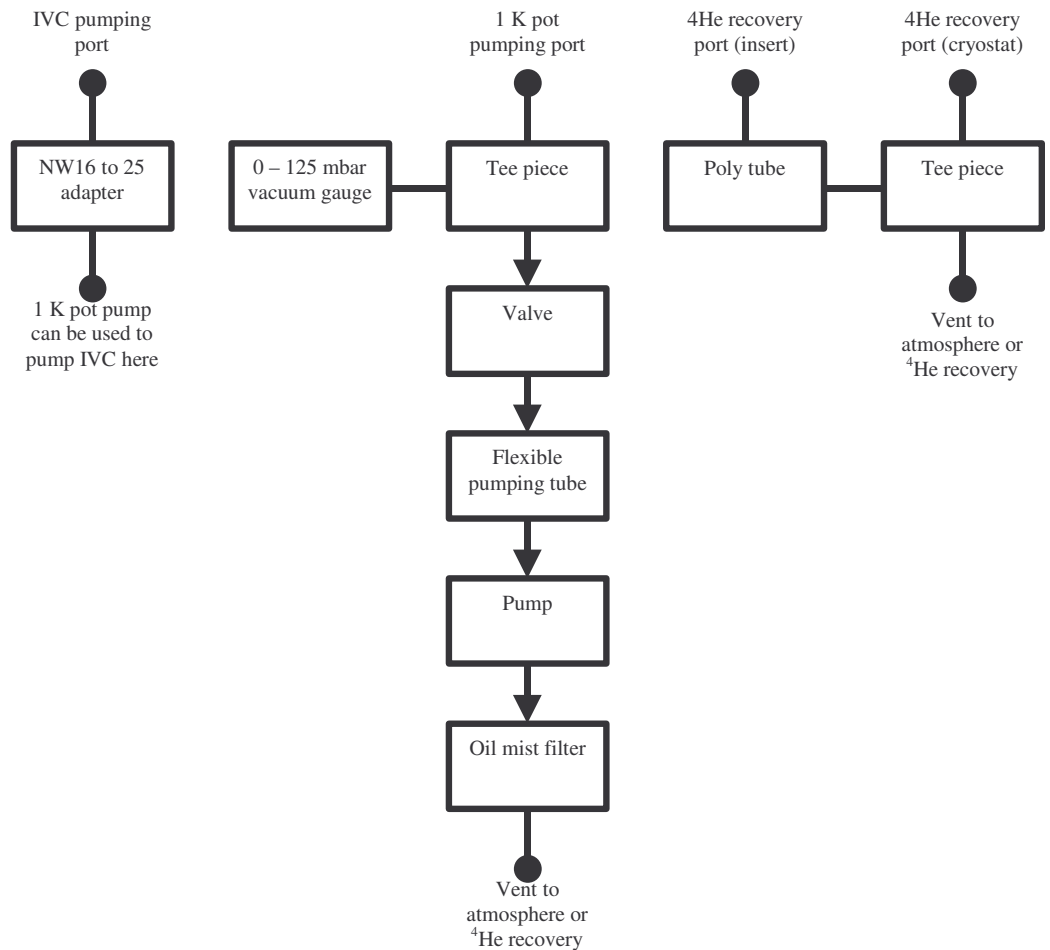


Figure 3 HelioxVL insert connections

3 Commissioning and routine maintenance

3.1 Commissioning requirements

The Heliox Insert Data section of this manual 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 ^3He pot before attempting to run the system.

Caution: The 1 K plate has three nylon 'bump stops' screwed into its cylindrical face. These are not packing pieces and must not be removed. They are there to help prevent the ^3He pot touching the inside of the IVC.

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).

Remove the IVC sorb and warm it gently with a hot air blower to remove any moisture that may have collected in it during shipment.

3.3 Control wiring details

3.3.1 Insert control wiring

The details of the insert control wiring are given in Table 1, Table 2 and Figure 4

Pin		Function	
1	Start	^3He sorb heater	
2	End	68 Ω resistor	
3	V-	^3He sorb temperature sensor	
4	V+		
5	I+		100 Ω Allen-Bradley resistor
6	I-		
7	V-	1 K plate temperature sensor	
8	V+		
9	I+		2200 Ω RuO ₂ resistor
10	I-		

Table 1 Ten pin Fischer connector No 1 for the sorb and 1 K plate

Pin		Function
1	Start	^3He pot heater
2	End	$2 \times 100 \Omega$ firerods in series
3	V-	^3He pot high temperature sensor (if fitted) Cernox
4	V+	
5	I+	
6	I-	
7	V-	^3He pot low temperature sensor 2200Ω RuO ₂ resistor
8	V+	
9	I+	
10	I-	

Table 2 Ten pin Fischer connector No 2 for the ^3He pot

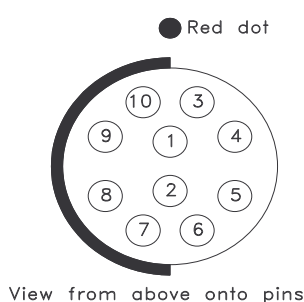


Figure 4 Pin numbers on an hermetically sealed Fischer connector (receptacle), viewed from the outside of the cryostat. (Fischer part number DBEE 1031 Z 010)

3.3.2 Connections to ITC temperature controller

A set of connecting leads ($2 \times$ dual input leads) is supplied with the ITC temperature controller. They are labelled to help you to connect them to the correct points. Please note that these leads should be plugged into the Heliox Heater Controller box, and the flying leads from this box should be plugged into the appropriate connectors on the ITC.

The first lead is used for control of the sorb temperature, and will plug into ten pin Fischer connector 1. There are two connectors at the Heater Controller box end of this lead, one of which plugs into Sensor channel 1 and another which can be used to monitor the temperature of the 1 K plate (it can be plugged into Sensor channel 2, in place of the ^3He low temperature sensor). The same type of sensor (2200Ω RuO₂) is used on the ^3He pot and 1 K plate.

The second lead plugs into ten pin Fischer connector 2. This is for the sample temperature, which is displayed either on Sensor channel 2 (^3He low temperature sensor) or Sensor channel 3 (high temperature sensor). In normal use both plugs at the Heliox Heater Controller box end of this lead should be connected to the controller box.

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 Replacing the charcoal in the exchange gas sorb

The performance of the exchange gas sorb can often be improved by warming it with a hot air blower or under a lamp. However, occasionally the charcoal in the exchange gas sorb will need to be replaced. This will need to be done more often if the IVC is removed while the insert is still cold, as water from the atmosphere will be condensed into the charcoal, contaminating it. It is difficult to remove this water from the charcoal effectively.

Some spare charcoal cloth and copper gauze is supplied in the spares kit, and a copy of the original sorb should be made. Slide this into the copper holder which is mounted on the 1 K plate.

3.4.2 Pumping system

The recommendations of the manufacturer of the individual components should be followed.

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

4 Final assembly of the system and mounting the sample

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 section of this manual. 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 ports 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 ends of the spare port tubes are covered with foil to minimise thermal radiation leaks into the IVC. All ports into the IVC should have some radiation baffling.

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 ^3He pot. Several tapped holes are provided for this purpose. The sample must be well thermally anchored to the ^3He 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 in any case be thermally anchored to the ^3He pot thoroughly.

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

Make sure that the IVC Sorb is dry, warm and replaced in its holder at this point.

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 ordinary silicon based vacuum grease. Rub a layer of grease thoroughly and evenly onto the whole 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 ^3He pot, 1 K plate or the sorb. Push the cone into the IVC with a slight twisting action. Immediately carry on to the next step as the IVC is not safely attached to the insert until it has been pumped out.

Evacuate the IVC to a rough vacuum using the 1 K plate pump. 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.

The tufnol guide on the end of the IVC may help to protect the pick up tube (if it is fitted). If in doubt tie or tape the pick up tube to the IVC so that it will not be damaged as the insert is lowered into the dewar.

4.5 Preparation for cool down

After you have pumped the IVC to a rough vacuum (< 0.5 mbar), 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, and it is easiest to allow this into the IVC using a bladder fitted to the IVC valve.

Evacuate the 1 K coil to a rough vacuum with the needle valve closed. Attach a supply of dry Helium Gas to the pick-up via a rubber tube, then open the needle valve and pump Helium from the pick-up through the 1 K coil for approximately 5 minutes. This helps to remove traces of moisture from the 1 K plate and needle valve. The needle valve can then be closed followed by the pumping line valve.

The insert is now ready to cool down.

5 Running the Heliox\VL insert

5.1 Cooling the insert down

Prepare the insert as described in section 4. The insert is now ready to be cooled down to 4.2 K. This operation can be carried out in approximately 30 to 40 minutes with practice. Connect the insert wiring to the ITC temperature controller so that the cooldown can be monitored easily. A computer or other data logging device may be connected to the ITC so that the rate of cooling can be observed easily.

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 cryogenics.**

5.1.1 Pre-cooling using liquid nitrogen

Use 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 has a small heat capacity the ^3He pot should reach 100 K after approximately 15 to 20 minutes.

If preferred, the insert can be left in the liquid nitrogen for long enough for it to reach 80 K. However, it can be cooled more quickly by transferring it to the helium dewar when it reaches a temperature around 100 to 120 K.

5.1.2 Pre-cooling without liquid nitrogen

If a large dewar of liquid nitrogen is not available pre-cool the insert by clamping it in the upper position on the sliding seal, so that the IVC is held in the neck of the liquid helium dewar. Clearly this will consume a slightly larger amount of the liquid helium, but it has the advantage that it is less likely for the pick up tube to be blocked by ice as the insert is transferred to the liquid helium.

For test purposes at Oxford Instruments this is the preferred method as it is easier, faster, and is less likely to block the pick-up tube.

5.1.3 Cooling the insert to 4.2 K

Prepare the helium vessel so that the insert can be loaded very quickly. Slide the NW50 flange on the insert down to lower end of the sliding tube and clamp it there. Load the insert into the helium vessel quickly, and clamp the NW50 flange to the top of the dewar. Connect the small recovery line on the insert, so that the cold helium gas boiled from the reservoir can pass up the inside of the sliding tube and cool the insert. By this time, the ^3He pot should be seen to be cooling again (probably faster than it did in the LN_2).

Slowly lower the insert into the liquid helium. There is no point in lowering it very quickly because the liquid helium consumption will be increased but the insert will not cool much more quickly. With practice you will find that it is possible to cool the insert to 4.2 K within 15 to 20 minutes providing that the sample has a small heat capacity.

Connect the 1 K plate pumping line to the valve on the insert and evacuate the air from the 1 K plate pumping line. Check that the small capillary tube in the insert is clear by opening the 1 K plate needle valve and pumping line valve to check for gas flow. Close them again quickly to prevent some parts of the insert from cooling faster than others. The exchange gas will allow it to cool uniformly.

When the 1 K plate reaches a temperature close to 10 K the exchange gas sorb (which is attached to it) will start to pump the exchange gas automatically. Providing that the sample's heat capacity is not too large it and the ^3He pot should already be below 10 K, so that it will be easy to cool it as described in the next section. If, however, the sample does have a significant heat capacity, it may be found that it is still much warmer than 10 K. In this case, it may be possible to improve the cooling by lowering the insert into the helium more slowly. In any case, it will be possible to cool it using the ^3He condensation as described in section 5.2, but it will take longer.

If the insert is to be used at a temperature below 4.2 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 section 5.3.2 to control the sample temperature.

5.2 Condensation of ^3He and cooling to base temperature

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

When the insert is precooled to below 10 K as described in section 5.1 it is ready for condensation of the ^3He gas. Pump the 1 K plate, and open the needle valve slightly to allow the flow of liquid helium to start.

Once the 1 K plate temperature has cooled below 4.2 K (see section 3.3.2. for how to monitor the 1 K plate temperature), set the sorb temperature to 30 K to ensure that it will not pump the ^3He gas. Take care to ensure that the correct heater (1) is selected on the ITC, so that the sample and ^3He pot are not accidentally warmed. As the 1 K plate cools below 3 K, the ^3He gas will start to condense and run down into the ^3He pot. The sample and 1 K Plate temperatures will be seen to drop quickly to around 1.5 K.

If it starts to warm again this indicates that the flow is not enough to keep liquid in the 1 K region and it will be necessary to open the 1 K needle valve slightly. Over a period of time the ideal setting can be found.

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.

If the insert does not seem to condense well, and no improvement is seen even after adjustment of the 1 K plate needle valve, it is possible that;

- There is a leak on the cone seal
- There is an excessive quantity of exchange gas
- The exchange gas sorb on the 1 K plate is saturated or contaminated.

Any of the above will also be indicated by difficulty in keeping the sorb at 30 K.

If a mass spectrometer leak detector is available the IVC helium level background should be checked at this point.

Warm the insert to room temperature as described in section 5.4 with a bladder on the IVC port so that the helium gas can expand safely from the IVC without over-pressurising it. Re-make the cone seal and cool the insert to 4.2 K again as described above. If the same symptoms are observed again replace the exchange gas sorb as described in section 3.4.1.

When the sample temperature is steady, the insert can be cooled to base temperature. This is done by cooling the sorb, so switch off the sorb heater by setting the sorb temperature on the ITC to 2.0 K. The sorb will be found to cool to below 3.5 K within a few minutes and the sample temperature should drop with the sorb temperature. If the sample has a small heat capacity and a high thermal conductivity, it should reach base temperature within 15 to 25 minutes.

The temperature of the 1 K plate can be checked and if necessary set more accurately at this point by adjustment of the needle valve (the value to set can be found in the test results).

If the experimental heat load is very low, the insert should stay at base temperature for more than 60 hours.

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 (in the Insert Data section of this manual) 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 monitored using a RuO₂ sensor mounted on the ³He pot. These sensors 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 in addition to the uncalibrated one supplied.

Two separate heater outputs from the ITC are required (e.g. for sorb control and high temperature control using the ³He pot heater), so a Heliox Heater Controller box is supplied with the ITC. A separate manual is supplied for the Heliox Heater Controller.

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.

5.3.1 Temperature control in the low temperature range

5.3.1.1 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 temperature of the sorb is below 3 K, the ^3He pot temperature will be reduced to a minimum for a given experimental heat load.

If the sorb is above 30 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 pot 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.

5.3.1.2 Temperature control with feedback from the sample 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 mK. In order to achieve this, an extra input channel is used, and this is described in Table 3 (see Sensor Channel 2).

The settings on the three-term controller of the ITC (that is, the PID settings) need to be optimised to suit the operating conditions. The test results, in the Insert Data section of this manual, 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 pot 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.

Channel	Sensor/location	Notes
1	Allen-Bradley resistance sensor on the ^3He sorb.	The accuracy of this sensor is not critical for operation of the system.
2	RuO ₂ resistance sensor on the ^3He pot. (RuO ₂ resistance sensor on the 1 K plate.)	Measures the sample temperature with a resolution of 1 mK from base temperature to approx. 1.5 K. Note that an uncalibrated resistor does not measure the temperature accurately, but it can be used to achieve accurate temperature control.
3	Cernox sensor on the ^3He pot.	Measures the sample temperature in the range 1.5 to 300 K.

Table 3 ITC input channels summary

Caution: Set the maximum temperature limit of the sorb (Sensor Channel 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

Temperatures above that of the 1 K plate can be achieved by supplying heat directly to the heater on the ^3He pot. In this high temperature range, the sample temperature is controlled by balancing the heat supplied on the ^3He pot with the cooling power of the insert. The sample is cooled by conduction through, and convection of the ^3He 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 20 K to 30 K. This is handled automatically by the heater controller box.

Keep the IVC under vacuum for the entire temperature range of the insert for ease of use. The exchange gas sorb can do this for you over much of the operating temperature range. However, when the system is operating at high temperatures the exchange gas sorb (which is mounted on the 1 K plate) may warm and release the helium gas into the IVC softening the vacuum. The flow of helium through the 1 K plate can be increased slightly to keep the exchange gas sorb cold, and sample temperatures up to 100 K can easily be achieved without pumping on the IVC.

When the exchange gas sorb warms enough to release the gas, the temperature controller may not be able to supply enough power to maintain the sample temperature. It will then be necessary to pump the IVC using the 1 K plate pump if the IVC is to be kept under vacuum. It will normally be found to be best to pump the IVC thoroughly if the insert is to be operated at temperatures in excess of 100 K.

If possible, at high temperatures (>100 K) the insert should be raised into the neck of the dewar to reduce the radiative heat load into the helium liquid in the main bath.

Select Sensor Channel 3 for high temperature control of the insert.

The heater controller box manages the ITC heater supply so that power can be delivered to both the sorb heater and ^3He pot heater. The Heliox Heater Controller manual explains this feature in detail.

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. However, you can force the insert to cool more quickly and efficiently by pulling it up into the neck of the liquid helium vessel and following the pre-cooling procedure given in section 5.1.

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 V in the low temperature region and around 30 V 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.

Caution: When you warm up the insert make sure that the IVC and 1 K plate are free to vent safely. Any remaining cryogenics or contamination can then escape without causing damage to the insert.

Pump the 1 K coil (with the plate needle valve closed fully) until the temperature is seen to rise above 4 K, indicating that the coil is empty (see section 3.3.2. for sensor connections). Close off the valve in the pumping line. Connect an empty bladder to the IVC pumping line and open the valve. This ensures that any gas that has leaked into the IVC and been absorbed by the exchange gas sorb can expand freely into the bladder. It is unlikely that the bladder will expand at all as the insert is warmed since the only gas which has deliberately been introduced to the IVC is the 10 cm³ of helium exchange gas.

Disconnect all of the pumping lines and the recovery line from the insert, (leaving the valve on the insert's 1 K plate flange), disconnect the ITC leads, and remove the NW50 clamp which connects the insert to the dewar. The insert can then be pulled out of the dewar quickly. 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 ice in the bath of liquid helium. Cover the top of the dewar as soon as possible, using a blank flange, bung or dummy baffle stick.

The insert may now be warmed gently with a hot air blower. The exchange gas in the IVC will transfer the heat to the inner parts of the insert quite quickly. It is important to ensure that the IVC is not removed before the 1 K plate and sample are warmed to near room temperature to prevent the condensation of moisture on the sample or the exchange gas sorb.

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

5.5 Fault Finding

This is a summary of common faults and a description of the most likely cause of the problem. If the problem cannot be solved 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 Poor base temperature with short hold time

Either

High heat load. Check 1 K plate temperature. Try opening the needle valve slightly and monitor any change.

Or

High level of exchange gas in IVC. This may be confirmed if you have had difficulty warming the sorb, or cooling the 1 K plate during condensation. Suspect a leak into the IVC or contamination of the exchange gas sorb. The cone seal is the easiest joint to check; see section 5.2. If the problem is observed on two successive cooldowns the charcoal in the exchange gas sorb should be changed as described in section 3.4.1.

Or

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

Or

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 ports especially carefully.

Or

The heater on the ^3He pot may not be properly disconnected from its power supply (ITC). Check that the Heliox heater controller box at the back of the ITC is working correctly.

Or

There may be a touch from the ^3He pot, the sample, or the wiring to the IVC. If all the other possibilities have been discounted, warm up the system and check the alignment.

Or

The excitation current for the high temperature sensor may introduce a small heat load onto the ^3He pot and warm it slightly.

Or

An air leak into the IVC. Check the IVC valve and Fischer connector seals.

5.5.2 Poor base temperature with long hold time

Check that the ^3He pot thermometer is reading the correct temperature by comparing it with another known thermometer. The calibration of some resistance thermometers can change with time. If this is found to be reading the correct temperature it is possible that the ^3He sorb is contaminated.

Either

Contact Oxford Instruments for advice.

Or

Remove the ^3He from the insert and clean it using a cold trap, then pump the sorb using a diffusion pump while warming it slightly. After the sorb has stopped outgassing, return the ^3He to insert by cooling the sorb to 5 K.

5.5.3 Good base temperature, short hold time

Poor condensation of ^3He . Check 1 K plate temperature during condensation. Check that the sorb thermometer is reading the correct temperature, and that the sorb temperature is correct during condensation. Check effect of larger 1 K plate pump. Check the effect of condensing at a different temperature.

5.5.4 Condensation difficult

High heat load on 1 K plate during condensation

Check that the 1 K plate is not empty or overfilling. Check that there is enough liquid helium in the main bath to reach the end of the pick up tube.

It is possible that there is a leak into the IVC so there will be a large heat load onto the plate, and it will not cool properly. Warm up the insert as described in section 5.2, check for touches and carefully re-make the cone seal.

While the insert is warm, also check that the exchange gas sorb does not touch the body of the ^3He sorb.

5.5.5 Very slow cool down to base temperature

Poor thermal contact to the ^3He pot. Check the mounting of the sample and sensor on the pot.

5.5.6 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 the 1 K plate spacers if fitted are not pushing the IVC to one side. 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 System specific information

The Insert Data section of this manual includes all the information specific to your insert. In particular it gives details of:

- System specification and expected performance
- Experimental wiring
- Services fitted into the spare ports
- Equipment supplied with your system and commissioning requirements

5.7 Summary for experienced users

5.7.1 Preparation

- Ensure that the IVC sorb is clean and dry Section 4.3 (3.4.1)
- Check wiring - diagnostic and experimental Section 3.3 and 5.6
- Check for heat leaks (radiation and conduction) Section 4.2
- Fit IVC Section 4.4
- Pump IVC (rough vacuum) Section 4.4 and 4.5
- Add exchange gas (approx. 10 cm³ NTP) Section 4.5

5.7.2 Cooldown

- Pre-cool insert using LN₂ (if required) Section 5.1.1
- Load insert Section 5.1.2 and 5.1.3
- Check main bath level
- Allow insert to cool and connect pump and lines Section 5.1.3
- Set 1 K plate temperature using needle valve Section 5.2
- Heat sorb to 30 K Section 5.2
- Allow time for condensation Section 5.2
- Remove heat from sorb Section 5.2
- Check 1 K plate temperature (fine tune) Section 5.2
- Run to base temp. and set low temp. control Section 5.3 and 5.3.1
- Set high temperature control Section 5.3 and 5.3.2