# Oxford Instruments NanoScience HelioxVT - Sorption Pumped <sup>3</sup>He Refrigerator

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The Business of Science\*

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# **1** Introduction

This manual is designed to introduce you to the HelioxVT manufactured by Oxford Instruments. This manual contains important information for the safe operation of your system. We recommend that you read this manual carefully before operating the system for the first time.

In addition to this manual for the HelioxVT, further manuals and documentation will have been supplied with the system. These further manuals and documents detail the other components of the system as well as important safety information, as shown in Table 1-1.

Please ensure you have reviewed the information supplied in all the manuals before you attempt to operate your system.

Documentation	Format
Factory test results	Electronic copy on USB & hard copy
MercuryiTC manual	Electronic copy on USB
Thermometry calibration data	Electronic copy on USB
Practical Cryogenics	Electronic copy on USB
Safety Matters	Electronic copy on USB
General Assembly (GA) drawing	Electronic copy on USB
MercuryiTC Safety Sheet	Hard copy
HelioxVL Safety Sheet	Hard copy
Third-Party Product Manuals	Hard copies

Table 1-1: Documentation supplied with the HelioxVT.

Please keep all the manuals supplied with your system and make sure that you check for updated information and incorporate any amendments. If you sell or give away the product to someone else, please give them the manuals too.

#### These are the Original Instructions.

#### 1.1 Copyright

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Oxford Instruments will not be responsible for the accuracy of the information contained in this document, which is used at your own risk and should not be relied upon. The information could include technical inaccuracies or typographical errors. Changes are periodically made to the information contained herein; these changes will be incorporated in new editions of the document.

## **1.2** Statement of intended use

The equipment has been designed to operate within the process parameter limits that are outlined in the user manual. The equipment is intended to be installed, used, and operated only for the purpose for which the equipment was designed, and only in accordance with the instructions given in the manual and other accompanying documents. Nothing stated in the manual reduces the responsibility of users to exercise sound judgement and best practice. It is the user's responsibility to ensure the system is operated in a safe manner. Consideration must be made for all aspects of the system's life-cycle including, handling, installation, normal operation, maintenance, dismantling, decontamination and disposal. It is the user's responsibility to complete suitable risk assessments to determine the magnitude of hazards.

The installation, usage and operation of the equipment are subject to laws in the jurisdictions in which the equipment is installed and in use. Users must install, use, and operate the equipment only in such ways that do not conflict with said applicable laws and regulations. If the equipment is not installed, used, maintained, refurbished, modified, and upgraded as specified by the manufacturer, then the protection it provides could be impaired. Any resultant non-compliance damage, or personal injury would be the fault of the owner or user.

Use of the equipment for purposes other than those intended and expressly stated by Oxford Instruments, as well as incorrect use or operation of the equipment, may relieve Oxford Instruments or its agent of the responsibility for any resultant non-compliance damage or injury. The system must only be used with all external covers fitted.

#### **1.3** Restrictions on use

The equipment is not suitable for use in explosive, flammable or hazardous environments. The equipment does not provide protection against the ingress of water. The equipment must be positioned so that it will not be exposed to water contact.

#### **1.4** Maintenance and adjustment

Only qualified and authorised persons should service or repair this equipment. Under no circumstances should the user attempt to repair this equipment while the electrical power supply is connected.

#### 1.5 Warranty

The Oxford Instruments customer support warranty is available to all our customers during the first twelve months of ownership from date of delivery. This warranty provides repair to faults that are a result of manufacturing defects at Oxford Instruments NanoScience.

#### **1.6** Acknowledgements

All trade names and trademarks that appear in this manual are hereby acknowledged.

### **1.7** Technical support

If you have any questions, please direct all enquiries through your nearest support facility (see below) with the following details available. Please contact Oxford Instruments first before attempting to service, repair or return components.

#### System type: HelioxVT

Serial number: The Sales Order (SO) number and/or other identifiers of your system.Contact information: How we can contact you, email/telephone details.Details of your query: The nature of your problem, part numbers of spares required, etc.

#### Europe, Middle East, Africa and India (EMEAI)

OINS, Tubney Woods, Abingdon, Oxon, OX13 5QX, UK Tel: +44(0)1865 393200 (sales) Tel: +44(0)1865 393311 (support) Fax: +44(0)1865 393333 (sales and support) Email: nanoscience@oxinst.com (sales) Email: ServiceNSUK@oxinst.com (service and support) Web: www.oxford-instruments.com

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#### Japan

OINS, IS Building, 3-32-42, Higashi-Shinagawa, Shinagawa-ku, Tokyo, 140-0002, Japan Tel: +81 3 6732 8966 (sales) Tel: +81 3 6732 8966 (support) Fax: +81 3 6732 8939 (sales and support) Email: nanoscience.jp@oxinst.com (sales, service and support) Web: www.oxford-instruments.jp

# 2 Health and safety

Before you attempt to install or operate your system, please make sure that you are aware of all safety precautions listed in this manual together with the warnings and cautions set out in other documents supplied with the system.

All cryogenic systems are potentially hazardous, and you must take precautions to ensure your own safety. The general safety precautions required when working with cryogenic systems are given in Oxford Instruments' Safety Matters document. We recommend that all users should read this document, become thoroughly familiar with the safety information provided and be aware of the potential hazards.

It is the responsibility of customers to ensure that the system is installed and operated in a safe manner. It is the responsibility of customers to conduct suitable risk assessments to determine the nature and magnitude of hazards.

#### 2.1 Disclaimer

Oxford Instruments assumes no liability for use of any document supplied with the system if any unauthorised changes to the content or format have been made.

Oxford Instruments' policy is one of continued improvement. The company reserves the right to alter without notice the specification, design, or conditions of supply of any of its products or services. Although every effort has been made to ensure that the information in this document and all accompanying documents is accurate and up to date, errors may occur. Oxford Instruments shall have no liability arising from the use of or reliance by any party on the contents of this these documents (including this document) and, to the fullest extent permitted by law, excludes all liability for loss or damages howsoever caused.

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

#### **2.2** Disposal and recycling instructions

You must contact Oxford Instruments (giving full product details) before any disposal begins. It is also important to check with the appropriate local organisations to obtain advice on local rules and regulations about disposal and recycling.

#### 2.2.1 WEEE

Oxford Instruments Nanotechnology Tools Ltd is a scheme member for end of product life disposal.

The scheme is operated by:

B2B Compliance, Emerald House, Cabin Lane, Oswestry, Shropshire, SY11 2DZ Tel: 01691 676124 Fax: 0808 280 0468 E-Mail: info@b2bcompliance.org.uk Web: www.b2bcompliance.org.uk

#### 2.2.2 RoHS compliance

This product is exempt from RoHS 2 (DIRECTIVE 2011/65/EU and DELEGATED DIRECTIVE (EU) 2015/863) as per article 2 section 4j.

#### 2.3 Maintenance

Observe the necessary maintenance schedule for the system. Consult Oxford Instruments if you are unsure about the required procedures. Only qualified and authorised persons must service or repair this equipment.

#### 2.4 General hazards

The following general hazards must be considered when planning the site for installation and operating the equipment. Please take notice of the following relevant warnings.

#### 2.4.1 Warning notices

Warning notices draw attention to hazards to health. Failures to obey a warning notice may result in exposure to the hazard and may cause serious injury or death. A typical warning notice is shown below.



#### WARNING

A warning triangle highlights danger which may cause injury or, in extreme circumstances, death.

#### 2.4.2 Caution notices

Caution notices draw attention to events or procedures that could cause damage to the equipment, may severely affect the quality of your measurements, or may result in damage to your sample or measurement apparatus. Failure to obey a caution notice may result in damage to the equipment. A typical caution notice is shown below.



#### CAUTION

Caution notices highlight actions that you must take to prevent damage to the equipment. The action is explained in the text.

#### 2.5 Specific hazards

Safety information that applies specifically to the HelioxVT is provided in this manual. Where additional components are supplied as part of a system, please read and follow all safety information in the respective manuals and take additional precautions, as necessary.

#### 2.5.1 Hazardous voltages



#### HAZARDOUS VOLTAGE

Contact with hazardous voltage can cause death, severe injury or burns. Ensure that a local electrical earth (ground) connection is available at the installation site.



PROTECTIVE EARTH The cryostat and any other parts of the system fitted with earthing points must always be connected to protective earth during operation.

Parts of the system carry high voltages that can cause death or serious injury. Ensure that a local electrical earth (ground) connection is available.

The electrical supply to the system must include an isolation box to ensure that mains electrical power to the system can be isolated. The isolation box must allow the supply to be locked OFF but must NOT allow the supply to be locked ON.

#### 2.5.2 Low temperatures



Contact with cold objects and cryogens can cause serious injury to the skin. Skin may adhere to cold objects. Ensure that any cryogenic or coolant delivery systems are designed to prevent contact with the cold components.

Consider the hazards of low temperatures when planning the installation of the system. Proper safety equipment, including hand and eye protection, must be made available to all personnel expected to handle cryogenic liquids.

#### 2.5.3 Pressure relief



CLOSED VESSELS

COLD OBJECTS

Closed vessels in the system are protected by pressure relief valves that exhaust directly to atmosphere unless otherwise stated.

Do not tamper with any of the pressure relief devices fitted to the system or attempt to modify or remove them. Also ensure that the outlets of the relief devices are not obstructed. The correct operation of these relief valves is critical to the safety of the system. All closed vessels in the system are protected by pressure relief valves, as described in Table 2-1.

Location	Description	Setting
IVC pumping	Relief valve to atmosphere	0.1 bar
Integrated Helium-3 tank	Relief valve to atmosphere	50 psi
Table 2.1.	Descence well of velve information	

Table 2-1: Pressure relief valve information.

#### 2.5.4 Weight and lifting



#### HEAVY OBJECT

Incorrectly lifting heavy objects can cause severe injury. Use the appropriate lifting equipment, operated by fully trained personnel, when handling heavy system components.

Appropriate lifting equipment and Personal Protective Equipment (PPE) must be provided for the duration of the system installation and should always be used whilst operating or moving the system.

#### 2.5.5 Asphyxiation



#### ASPHYXIATION

Helium and nitrogen can displace the oxygen from air and cause death by asphyxiation. Ensure that adequate ventilation is provided.

Areas where these chemicals are stored or used must be well ventilated to avoid the danger of suffocation. Oxygen level detection equipment should be installed in suitable locations to warn personnel if the oxygen concentration falls below a threshold value. Take precautions to prevent spillage of liquid cryogens.

#### 2.5.6 Fire



FLAMMABLE GAS

Atmospheric oxygen can condense on cryogenically cooled objects. Oxygen can cause flammable substances to ignite in the presence of heat or arcing, risking severe injury.

Rooms where cryogenic liquids are being handled must be designated as no smoking areas. While liquid helium and nitrogen do not support combustion, their low temperature can cause oxygen from the air to condense on surfaces and may increase the oxygen concentration in these areas. Oxygen enrichment may cause spontaneous combustion.

#### 2.5.7 Trip hazards



#### TRIP HAZARDS

Poorly routed cables and pumping lines can be trip hazards and have the potential to cause accidents. Such accidents can result in both damage to the system and injury to personnel.

Where cables and lines are required their routings should be considered when planning the installation of the system. The cables and pumping lines of the system should be routed away from walkways and away from areas of common use to prevent the hazards.

#### 2.5.8 Slip hazards



#### SLIP HAZARDS

During normal operation ice may form on parts of the system. Upon warm up, this ice may melt and pool by the system. Water on the floor has the potential to cause accidents. Such accidents can result in both damage to the system and injury to personnel.

Drip trays should be placed appropriately around the system to catch any water runoff. Additionally, warning signs should be placed around the system.

#### 2.5.9 Magnetic fields



## MAGNETIC FIELDS

Superconducting magnets are powerful electromagnets which can exert forces on nearby ferromagnetic objects. This presents a hazard to personnel if these objects are free to move.

Strong magnetic fields can interfere with the operation of cardiac pacemakers in the vicinity of the system, causing death or serious injury. Where necessary, appropriate warning signs should be in place around the installation site. If your Heliox system is supplied or used with a superconducting magnet system, please read the additional safety information supplied in the relevant manual(s).

#### **2.5.10** Temperature and voltage limits

The HelioxVT is supplied with a MercuryiTC temperature controller. Safety features for the temperature controller are described in the MercuryiTC manual supplied. Additionally, safety features for the magnet power supply are described in the MercuryiPS manual (if supplied). You should ensure that you understand and comply with all safety warnings and cautions.

The MercuryiTC will have been set up in the factory to prevent you from accidentally exceeding the maximum safe operating temperature of the cryostat and to limit the heater voltage to a safe level. If you are planning to use an existing temperature controller or a controller made by another manufacturer, you must take the same precautions. The recommended values for the temperature controller limits are shown in Table 2-2.

Control Limit	Control Value
Heater voltage	40 V
System temperature	310 K

Table 2-2: MercuryiTC system control limit values.

If your Heliox system is supplied or used with a superconducting magnet, its MercuryiPS would have been set up in the factory in order to prevent you from accidentally exceeding the maximum safe operating current and ramp rate of the magnet. If you are planning to use an existing power supply or a power supply made by another manufacturer, you must take the same precautions. The recommended values for the maximum magnet current and ramp rate are provided in the systems test results.



#### TEMPERATURE & VOLTAGE LIMITS

If you do not safeguard the system with control limits, it is possible to cause serious damage to the system.

#### 2.6 Safety equipment

The following items are recommended for the safe operation of any system:

- Personal protective equipment, including thermally insulated gloves, face protection, body protection and protective footwear. Cryogens can act like water, soaking into clothing and causing severe burns.
- Hazard warning signs, barriers, or controlled entry systems to ensure that personnel approaching the system are aware of the potential hazards. This precaution is especially important if your system includes a superconducting magnet.
- Oxygen monitors should be fitted in the laboratory to warn personnel if the concentration of oxygen in the air falls below safe levels.

### 2.7 Risk assessments

It is the responsibility of customers to perform their own risk assessments before installing, operating, or maintaining the system. Risk assessments must obey regulations stipulated by the local regulatory authority.

#### 2.8 Handling and storage

The system may be stored in a storage environment as follows:

- Ambient temperature: -10 to +40°C.
- Relative humidity: 30% to 75% (non-condensing).
- Atmospheric pressure: 700 to 1060 mbar.

# 3 System description

#### 3.1 Principles of operation

At low enough temperatures gases adsorb to cold surfaces. If a material with a very large surface area can be cooled to a sufficiently low temperature, then this material can be used as a pump. HelioxVT inserts use such sorption pumps to lower the vapour pressure above the helium-3 surface and so attain low temperatures.

The sorption pump, or sorb, in the HelioxVT will absorb gas when cooled below 40 K, and the amount of gas that can be absorbed depends on its temperature. A heater is fitted to the sorb so that its temperature can be controlled. A Variable Temperature Insert (VTI) provides a temperature of <2 K that allows the helium-3 to condense and to reduce the amount of heat conducted to the sample space.

During condensation, the sorb is warmed above to ~35 K so it will not absorb any helium-3. The helium-3 condenses on the 1 K surface assembly and runs down to cool the sample and helium-3 pot to the temperature of the 1 K surface. At this stage, the helium-3 pot is full of liquid helium-3 at approximately 1.3 K. The sorb is now cooled, and it begins to reduce the vapour pressure above the liquid helium-3, so the sample temperature drops (the temperature dependence of the vapour pressure is shown in Figure 3-1). As the limiting pressure is approached, the temperature of the liquid helium-3 can be reduced to below 0.3 K. The temperature of the sample can be controlled by adjusting the temperature of the sorb. If the sorb temperature is set between 10 and 40 K it is possible to control the pressure of the helium-3 vapour, and thus the temperature of the liquid helium-3. However, if the best stability is needed, a temperature controller can be set up to measure the sample temperature and control the power supplied to the sorb heater. No heat is supplied directly to the liquid helium-3 since this would evaporate it too quickly. The temperature of the sorb is continuously adjusted by the temperature controller, and the temperature of the sample can typically be maintained for the full hold time of the system.



Figure 3-1: The vapour pressure of liquid Helium-3 as a function of temperature.

## 3.2 The HelioxVT

The HelioxVT system is a helium-3 refrigerator designed to operate in an Oxford Instruments Variable Temperature Insert (VTI) which provides a temperature of <2 K that allows the helium-3 to condense. The HelioxVT is compatible with other brands of VTI, but only a VTI and HelioxVT system supplied by Oxford Instruments will guarantee the optimum performance from the helium-3 refrigerator. The VTI may form part of a superconducting magnet system, or part of a stand-alone cryostat. Some of the important parts of the HelioxVT are shown in Figure 3-2 and Figure 3-3.



Figure 3-2: The ports around the top of the HelioxVT insert.

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 Can (IVC) is sealed using a silicon-based paste applied to a cone seal, creating a reliable seal. Normal vacuum grease is not used as this may leak in the superfluid helium-4 environment of a VTI running at its base temperature. There are no indium seals.

The charge of helium-3 is sealed into a self-contained storage vessel so that it is not necessary to remove this valuable gas from the insert when it is warmed to room temperature. This reduces the complexity of operation of the system. The (nominal) 2.7 litre (gas as NTP) charge is stored at a pressure of approximately 2 bar (absolute). The storage vessel is fitted with a pressure relief device.

A line-of-sight port gives access through the insert from room temperature to the sample space. This can be used to install services for your experiment. However, it is important to heat sink all services effectively to minimise the effect on the performance of the refrigerator.



Figure 3-3: The general layout of the HelioxVT insert. A section of the IVC has been cut out for illustration purposes.

The sample is changed by warming the helium-3 refrigerator insert to room temperature and removing the IVC. The sample is mounted in vacuum on the base of the helium-3 pot. Once the sample has been mounted, the IVC seal should be made, and this volume evacuated. A small amount (~1 cm<sup>3</sup>) of helium-4 exchange gas should then be added before loading the insert into the VTI.

### 3.3 Diagnostic wiring

There are two diagnostic wiring connectors for HelioxVT. The diagnostic connectors on the insert are hermetically sealed, 16-way 104-series, A-type Fischer connectors. The pin layout for these connectors is shown in Figure 3-4, viewed as though looking at the connector on the insert from above (as the connector on the cable mates).

**Note:** Depending on when your HelioxVT system was manufactured, it may have an older wiring configuration (typically three 10-way Fischer connectors). In such case, it is recommended to refer to the manual(s) supplied with your system at the time of installation. Should you require further information, please contact the factory, quoting the Sales Order (SO) number of your system.



Figure 3-4: Pin configurations for 104-series 16-way Fischer connectors.

The sensors and heater for the helium-3 pot are carried on connector 1:

Pin No.	Function	Polarity	Туре
1		V-	
2	$\frac{V}{V+}$ $\frac{V}{I+}$	Cornov	
3	The Pot high temp sensor	l+	Cernox
4			
5		V-	
6	$ \begin{array}{c}                                     $	DuO, generia	
7		RuO <sub>2</sub> generic	
8		I-	
9	<sup>3</sup> Uo Dat boatar	Start	2 x 100D Firered
10	The Pot Nealer	End	2 X TOOK FILELOO
11 to 16	Not used	n/a	n/a

Table 3-1: Connector 1 pin configuration.

The sensors and heaters for the helium-3 sorb and helium-4 plate are carried on connector 2:

Pin No.	Function	Polarity	Туре
1		V-	
2	Number     Number       3 He Sorb temp sensor     V-       1+     100R Allen-Bradl       1-     I-       4 He Plate temp sensor     V+       1+     V-       V+     RuO2 generic       I+     I-       V+     I+       V-     V+       I-     I+       I-     I+       I-     Start	V+	1000 Allon Bradly
3		TOOK Allen-Bradiy	
4			
5		V-	
6	<sup>3</sup> He Sorb temp sensor <sup>1</sup> He Sorb temp sensor <sup>4</sup> He Plate temp sensor <sup>4</sup> He Plate temp sensor <sup>4</sup> He Plate temp sensor <sup>1</sup> He Plate temp sensor		
7		RuO <sub>2</sub> generic	
8		I-	
9	3Us Carb baster	Start	100D wine would
10	The Sorb heater	End	TOOK WILE-WOULD
11 to 16	Not used	n/a	n/a

Table 3-2: Connector 2 pin configuration.

#### 3.4 Sensors

The HelioxVT insert is supplied with diagnostic temperature sensors at various positions to monitor the behaviour of the system and to allow the MercuryiTC Heliox Controller to automate operation.

The typical configuration is a generically calibrated carbon (Allen-Bradley) sensor on the helium-3 sorption pump, a generically calibrated ruthenium oxide ( $RuO_2$ ) sensor on both the helium-4 plate and helium-3 pot, and a fully calibrated Cernox sensor on the helium-3 pot.

Calibrations for the individually calibrated sensors on the system (usually just the Cernox sensor) will have been loaded into the MercuryiTC at the factory. The calibration data from the sensor's manufacturer will have been supplied with the system.

The Allen-Bradley and RuO<sub>2</sub> sensors supplied are typically generically calibrated, coming from a batch of similar sensors for which the mean characteristics have been determined. While individual sensors may deviate from the mean by a small amount, they are sufficiently accurate to control the system.

 $RuO_2$  sensors are useful at temperatures in the range  $0.02 < T < \sim 20$  K (suitable for the helium-3 pot and helium-4 plate on a HelioxVT), while Allen-Bradley resistors are more useful in the range  $2 < T < \sim 300$  K (suitable for the helium-3 sorb). If accurate determination of the temperature at the sample position is required, then fully calibrated sensors can be added to the sample stage.

Other sensors may be fitted in place of, or in addition to, those stated above. In such case, details relating to these sensors may be found in the factory test results supplied with the system. Calibrations for these sensors will also be loaded into the MercuryiTC.

# 4 System installation

#### 4.1 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.

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 must be done to remove it. Keep these instructions and the packing components in case you need to transport the system again in future.



SAFETY

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 before assembling the system.

#### 4.2 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 there are additional requirements; please refer to the relevant system manuals for a full description. Please refer to the site preparation guide.

#### 4.3 Equipment to be provided

To complete the installation safely and successfully, you are required to provide the following equipment for use by the installation engineer and to ensure your own safety and that of others working nearby throughout the installation procedure.

#### 4.3.1 Personal safety equipment

Perform a risk assessment and decide whether any personal safety equipment is required for the installation. Safety equipment to be considered includes:

- Hazard warning signs, barriers, or controlled entry systems to ensure that anyone approaching the system is aware of the potential hazards.
- First aid kit.
- A fire extinguisher suitable for use on electrical equipment.
- Eye protection.

The above list is not exhaustive, and it is the responsibility of customers to ensure that all necessary personal safety equipment is provided.

#### 4.3.2 Lifting and support equipment

You must provide the following lifting and support equipment for the installation. The equipment must be properly certified and have a current test certificate. You must also provide trained

personnel to operate the equipment. Be sure to consider factors such as ceiling height along with the system dimensions when determining appropriate lifting equipment.

- A suitable method of lifting the system from the delivery vehicle (forklift or pallet truck).
- A suitable overhead hoist or crane for use in the laboratory.
- A lifting sling and shackles to suit the lifting points on the system.
- A pair of heavy-duty adjustable support trestles or a suitable frame assembly to support the cryostat top plate when working underneath the cryostat.

#### 4.3.3 Vacuum equipment

You must provide the following vacuum equipment:

- A vacuum leak detector to verify the integrity of the vacuum chamber and gas handling system connections. A mass spectrometer type is recommended.
- A selection of KF/NW vacuum fittings, centering O-rings and clamps.
- A turbo-molecular pump system. It is recommended that the pumping system should include a dry (oil-free) backing pump to avoid the risk of oil-vapour back-streaming.



#### CAUTION

All pumps should be dry (oil free). If wet pumps are used, appropriate foreline traps must be installed.

#### 4.3.4 Tools and accessories

OINS installation engineers will come equipped with their own toolkit. However, it is useful to have the following items made available during the installation and operation of the system:

- Spanners (wrenches), open ended metric set, 5.5 mm to 19 mm.
- Allen keys (hex wrenches), metric set 1.5 mm to 12 mm.
- Screw drivers (cross- and flat-head), pliers, side cutters etc.
- Digital multimeter (with a low-current Ω range).
- A de-greasing agent or solvent for general cleaning (e.g. isopropanol).
- A heat gun or fan heater.
- A soldering iron for electrical work.

#### 4.3.5 Laboratory services

The HelioxVT system needs to be placed within another cryogenic system such as an Oxford Instruments Variable Temperature Insert (VTI).

Depending on which one of these is available, the list of additional requirements will change. Consult the manual of the system used for details and required laboratory services.

#### 4.4 Consumables

- Vacuum grease.
- CAF paste.
- Latex, nitrile and/or cotton gloves for handling clean items.
- Metal polish and degreasing agent or solvent for general cleaning.
- Fishing line or dental floss.

- Rubber soccer ball bladders.
- Rubber tubing.
- Aluminium foil tape.
- NRC2 foil.

# 5 System operation

#### 5.1 Preparations

Ensure that the system has been properly prepared for operation, as described below.

#### 5.1.1 Experimental services

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 end of the spare port tube is covered with foil to minimise thermal radiation leaks into the IVC. Make a few small holes in the tape to prevent the trapping of air in the line-of-sight port. It can also be useful to fill the lower end of the port with a small amount of insulating foil, such as NRC2. See Figure 5-1 for an example.



Figure 5-1: Aluminium foil tape (with pierced holes) covering the line-of-sight port. NRC2 foil (shown underneath) is added inside the port before it is taped over.

When deciding on the quantity and type of additional services to be installed the conducted heat load should be considered. An additional 10  $\mu$ W to the helium-3 pot may reduce the hold time by a factor of 2 or 3. Use thin, high-resistance wiring wherever possible.

#### 5.1.2 Mounting the sample

The sample is mounted in vacuum on the base of the helium-3 pot. Several tapped holes are provided for this purpose. Ensure any screws used to mount samples do not bottom out, as this may cause damage to the helium-3 pot.

The sample must be thermally anchored to the helium-3 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. The copper surfaces may be gold plated to prevent corrosion.

Delicate, non-metallic samples may be cooled through the electrical leads, which should be thermally anchored to the helium-3 pot. Connect the wiring to the sample and check all the wiring carefully before fitting the IVC.

#### 5.1.3 Outgassing or replacing the charcoal

If the IVC sorb has not been used for some time and if it has been open to the air, warm it with a hot air blower before you fit it to the insert. If the body has already been fitted to the insert you can remove the charcoal so that there is no risk of burning any delicate wiring. The charcoal cloth is wrapped in copper gauze and it can be pulled out of the sorb body easily. If you suspect that the charcoal has been badly contaminated with moisture, you should replace it. Unwrap the cloth and put in a new piece of the same size. Warm this with a hot air blower to approximately 80 °C before you put it into the sorb body.

#### 5.1.4 Fitting the IVC

The IVC is sealed onto the insert using a cone seal. This type of seal is quick and simple to make and requires much less cleaning than an indium seal when the IVC is removed again. For systems operating in VTIs, a silicon-based paste (e.g. CAF1 Paste) should be used to make the seal. Check the paste is within its expiry date before use.



CAUTION It is important to ensure that the surfaces of the cone seal are kept clean and free from scratches.

It is recommended rubber gloves (nitrile or latex) are worn whilst fitting the IVC. Where necessary, solvents should be used to clean surfaces. Ensure no small particles are allowed into the seal. To make the seal, the IVC will need to be evacuated; it may be convenient to attach a pump before proceeding.

Before making the seal ensure that the groove and the mating surfaces are clean. Apply a layer of paste thoroughly and evenly onto the whole surface of the cone on the IVC flange and apply a thin uniform layer onto the inner mating surface of the IVC tube, as shown in Figure 5-2.



Figure 5-2: Typical application of CAF paste to an IVC cone flange (left) and inner mating surface of an IVC tube (right).

Fit the IVC carefully over the insert, making sure that none of the paste is removed by the helium-3 pot. 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. This will pull the cone seal together very firmly, and it will not be possible to separate it again without using the jacking plate. The cone seal is designed with a locking taper, so that the IVC will not fall off accidentally even if it is vented to atmospheric pressure. Whilst

evacuating the IVC it is advisable to connect all electronics and check that all sensors are reading OK. Disconnect when finished to facilitate the loading of the insert.

### 5.2 Loading the HelioxVT insert

Before loading the HelioxVT into the VTI, carefully inspect the O-ring seal where the insert interfaces with the VTI to ensure it is clean and undamaged. If in doubt, replace the O-ring before proceeding. Also inspect the sealing surfaces of the HelioxVT and VTI. Once the insert is loaded, it is advisable to leak test the seal using a leak detector.

The Heliox firmware can control the cool down of the insert provided the electronics are connected as the insert is loaded. A sliding seal can be used to seal the insert at the top of the cryostat before it is fully loaded.

The HelioxVT insert can be treated like any other sample rod for a Variable Temperature Insert (VTI). A full description of the procedure for changing sample rods can be found in the relevant system manuals. A brief description is included here.



Prevention of Blockages Care must be taken to avoid the possibility of incurring a VTI blockage as a result of a leak. The most likely source of a leak is the O-ring seal where the insert interfaces with the VTI. **Ensure the advice in Safety Matters is read and followed.** 

When loading into a VTI, the sample space should be pressurised with a flow of Helium gas to slightly above atmosphere to prevent the ingress of air when loading the insert. The needle valve on the VTI should be shut. Remove the blanking cap (or the previous insert) from the VTI and insert the Heliox.

When the insert is far enough into the VTI to allow, connect cables to diagnostic wiring manifold on the top of the insert. The sliding seal should be used to seal the insert at the top of the cryostat before it is fully loaded.

Connect the auto-needle valve control cable between the Heliox Controller and the VTI auto-needle valve and open the pumping line. At this stage, the MercuryiTC can be powered on and allowed to control the cool down.

#### 5.3 System control

All Heliox sorption pumped helium-3 refrigerators only have a finite quantity of helium-3 which can be condensed into the system. Experimental heat loads are taken up by the latent heat of vaporisation of the liquid as it is transformed into vapour. Once all the liquid helium-3 is consumed in this way, the system will start to warm.

Cooling the system again requires the "regeneration" of the helium-3 sorption pump such that the absorbed gas is released from the sorb and re-condensed into the helium-3 pot. The process for cooling and temperature-controlling Heliox helium-3 refrigerators is described in the following section. The Mercury iTC firmware automates the process, only set points on the controller's front panel will require user input.

If you purchased a complete system from Oxford Instruments, then the system will arrive fully configured. If the HelioxVT is being retro-fitted into an existing system, some of the parameters may need to be updated to attain optimum performance. If this is the case, please refer to Appendix A – Configuring temperature controller.

### 5.4 <sup>3</sup>He regeneration

To run the refrigerator, the helium-3 charge must be condensed. This is achieved by warming the helium-3 sorption pump, using its in-built heater, to a temperature of ~30 K such that it cannot pump Helium gas (any gas previously adsorbed in the pump will be released). This raises the pressure of the helium-3 gas in the insert.

Simultaneously, the VTI should be run to ensure there is a cold surface for the gas to condense onto. The condensed liquid helium-3 then runs down into the helium-3 pot, filling it with liquid at a similar temperature to that of the VTI.

#### 5.5 Low-temperature operation

To control the temperature of the helium-3 pot between the lowest attainable temperature and a temperature approximately equal to that of the VTI it is most efficient to control the temperature, and therefore the pumping efficiency, of the helium-3 sorption pump.

Varying the pressure above the liquid surface (see Figure 3-1) can control the temperature of the helium-3 without supplying an additional heat load to the helium-3 pot, maximising the low-temperature hold time. Closed loop control of the helium-3 pot temperature can be achieved by servo-controlling the helium-3 sorption pump heater to control the helium-3 pot at a set point.

#### 5.6 High-temperature operation

To control the temperature of the helium-3 pot at temperatures above that of the VTI it is most efficient to apply electrical power directly to the helium-3 pot heater.

To provide cooling the helium-3 sorb is warmed to ~15 K to partially release the helium-3 charge from the sorption pump, thus providing a thermal link (through the gas) between the VTI and the helium-3 pot.

Closed-loop control of the helium-3 pot temperature can be achieved through PID control of the helium-3 pot heater.

#### 5.7 Rapid cool down

The initial cool down of the insert, or recovery from high-temperature control, to run experiments at low-temperatures is best accomplished by admitting helium-4 "exchange-gas" into the IVC.

The HelioxVT has a small sorption pump inside the IVC (attached to the IVC flange). During routine operation, the VTI heat exchanger Heater and Sensor are typically used for temperature control of the helium-4 plate.

Closed-loop control of the helium-4 sorb can be achieved through PID control of the VTI heater.

#### 5.8 The MercuryiTC Heliox Controller

The routines for regeneration, high- and low-temperature control, and rapid cool down as described above have been implemented in the firmware of the Oxford Instruments' MercuryiTC Heliox Controller. The MercuryiTC is configured with measurement cards in specific locations for use as a Heliox Controller. This configuration is detailed in Table 5-1.

Slot Number	Card Type	Function
Main Board	Temperature and Heater	<sup>3</sup> He Sorb
1	Heater	VTI Heat Exchanger Heater
2	Heater	<sup>3</sup> He Pot Heater
3	Pressure	VTI Pressure Sensor
4	Needle Valve	VTI Needle Valve
5	Temperature	Helium-4 Plate
6	Temperature	VTI Heat Exchanger Sensor
7	Temperature	<sup>3</sup> He Pot High Sensor
8	Temperature	<sup>3</sup> He Pot Low Sensor

Table 5-1: MercuryiTC Heliox Controller configuration.

The front panel display of the MercuryiTC Heliox Controller is shown in Figure 5-3, Figure 5-4 & Figure 5-5.



Figure 5-3: MercuryiTC Heliox Controller front panel page 1.

MB1_He3_Sorb.T 294.245K MB0_Sorb_H_0%	DB6_H 164.2 KPot_htr (	e4Pot.T 210K 0%	DB3_He4Pr 345.000 DB4.G1	es.p )mB 0%
DB7_He3_Pot_CRN.T 293.582K HePotHtr 0%	DB8_He3_ 45.10	Pot_Low.T 012K	HelioxX High Te	.s mp
< Piot	Control	Settings	Heater	) >

Figure 5-4: MercuryiTC Heliox Controller front panel page 2.

0.	None 0000	DB4	G1.% 503%	DB4.G1.s OFF	
не 0.0	PotHtr.P 000W	0.C	one 0000	DB4.G1.s OFF	
	Plot	Control	Settings	Heater	

Figure 5-5: MercuryiTC Heliox Controller front panel page 3.

The MercuryiTC display panel is a touch screen. Touching "setpoint" allows the user to input the required helium-3 pot set point temperature (TSET). When a new set point is entered the actions taken by the Heliox Controller are determined based on the new set point and the current state of the system.

Temperature control set points can also be sent to the Controller remotely, as described in Appendix B - Remote operation.

#### 5.9 Firmware control routine

The control routine begins with a new temperature control setpoint (TSET) being input by the user. Without making a change to TSET (from the front panel or via remote command) the state of the system will not change. The only exception to this is at the end of "Regeneration", the system will automatically go into "Low Temp" control mode. Inputting a value of 0 (zero) for TSET will put the system into "Regenerate" mode.

Whenever a TSET variable is set (locally or remotely), the current state of the Heliox Controller is reevaluated and based on the new TSET value the appropriate course of action is taken. There are 5 modes of operation. Please note that the mode might change, even if the new TSET value is the same as before. The modes are entered in the following preference order:

- **Rapid Cool** Initiates heating of the VTI to introduce exchange gas into the IVC.
- **He4 Fill** Opens the VTI needle valve, increasing the helium-4 flow to accelerate the cool down of the pot. The needle valve is closed again once the VTI and helium-3 sorb are cold.
- **High Temp** Begins the high-temperature control routine at the set point if the TSET value is greater than CMODE\_XOVER parameter.
- **Regenerate** If the helium-3 pot temperature is higher than REGEN\_ABOVE ("automatic regeneration") or if new TSET value is zero ("user requested regeneration"). The helium-3 charge re-condenses into the pot.
- Low Temp If none of the above conditions are fulfilled, the Heliox must be already in low temperature control mode, so it will simply attempt to control at the new TSET value.

#### 5.10 Firmware control parameters

A set of firmware parameters stored in the Controller are used in the control logic. These settings can be accessed tapping the HelioxX.s widget on the lower right of the Controller front panel. The control parameter display is shown in Figure 5-6.

Cool	inactive		ACCEPT_BASE	0.2950K	
ExtractingHe3	inactive		CMODE_XOVER	1.6000K	
He3 Stability	0.244247	1 <u>.</u>	CONDENSED_TE	1.6600K	
He3 Stable	Stable		He3_POT_BOILO	5.0000K	
He4 Stability	0.0955203		He3_SORB_COLI	1.8000K	
He4 Stable	Stable	Ŀ	HAR SORR HT (	15 0000K	Ľ

Figure 5-6: MercuryiTC Heliox Controller settings screen.

These values will have been configured in the factory for correct operation of the HelioxVT insert. Typical values, along with a brief description of each parameter, are given in Table 5-2.

Parameter	Typical Value	Function
ACCEPT_BASE	0.25 K	Accept this temperature as a valid base (e.g. when system is stable
		and TSET <= ACCEPT_BASE)
CMODE_XOVER	1.85 K	Crossover temperature between high- and low-temperature
		control (and displayed sensor) modes.
CONDENSED_TEMP	1.8 K	Temperature that both the <sup>4</sup> He plate and <sup>3</sup> He pot must be below
		(and stable) to consider the system condensed.
He3_SORB_COLD	1.8 K	Temperature the <sup>3</sup> He sorb must be below at the end of the
		regeneration routine.
He3_SORB_HT_CONTR	15 K	Temperature that the <sup>3</sup> He sorb is controlled at during high-
		temperature control.
He3_SORB_REGEN	32 K	Temperature to control the <sup>3</sup> He sorb at while regenerating.
He4_SORB_RCOOL	20 K	Temperature to control the VTI during a rapid cool.
OPT_NV_HT	10 mbar	The pressure control set point in high-temperature operation.
OPT_NV_LT	5 mbar	The pressure control set point in low-temperature operation.
OPT_NV_RCON	15 mbar	The pressure control set point during regeneration.

Parameter	Typical Value	Function
POT_EMPTY	3.5 K	Temperature above which to consider the VTI empty.
RAPID_COOL_DELTA	10 K	Temperature difference required between current temperature
		and set point to trigger a rapid cool.
RAPID_COOL_END	10 K	Stop rapid cool at this temperature (don't start if already below it).
RAPID_COOL_EXIT	5 K	Temperature difference between <sup>3</sup> He pot and <sup>4</sup> He plate above
		which the current control mode is interrupted, and an evaluation
		sequence is run.
REGEN_ABOVE	1.8 K	If the current <sup>3</sup> He pot temperature is above this, and a new low-
		temperature control set point is entered, regenerate anyway.
T_DELTA	0.005 K	Ratio of the temperature delta to the set point temperature below
		which the system is considered to be at the set point.
POT EMPTY	15 K	Temperature above which to consider the VTI empty.

Table 5-2: Typical firmware control parameters and their functions.

## 5.11 VTI flow control

The operation of the HelioxVT is sensitive to the Helium flow rate through the VTI. As such, "openloop" control based solely on the VTI needle valve setting may be unsatisfactory. A pressure gauge is supplied with the Heliox Controller for installation close to the front of the VTI pumping port.

Using this gauge, the needle valve position is controlled to maintain a constant pressure (and consequently flow) at the inlet to the pump.

The Heliox control parameters that relate to needle valve operation are target control pressures for the different operating modes.

#### 5.12 Warming up

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



Frozen-in inserts

If you find that your VTI is blocked and the Heliox is frozen in, there is risk of death or injury if the insert becomes a projectile. Special care must be taken to safely remove it. Refer to Safety Matters for more information.



#### CAUTION

When warming up, ensure the IVC is free to vent safely. Any remaining cryogens or contamination can then escape without causing damaging the insert.

Connect an empty bladder to the IVC pumping line and open the valve to allow any gas that may be in the IVC to expand freely. The bladder is unlikely to expand as the insert is warmed since the only gas present in IVC is the 1 cm<sup>3</sup> of Helium exchange gas.

The sample space of the VTI should be pressurised with a flow of Helium gas to slightly above atmosphere to prevent the ingress of air when removing the insert.

Set the VTI temperature to >100 K. Once the sorb sensor on the HelioxVT reads >100 K, it can be removed. If the HelioxVT 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 it out swiftly to prevent the collection of solidified air in the VTI.

Cover the top of the VTI as soon as possible, using a blanking flange or baffle stick. Evacuate the VTI again once the HelioxVT is safely stored.

Heat can be carefully applied to the HelioxVT IVC to speed the warming. 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 helium-3 pot has warmed above the dew point to prevent the condensation of moisture on the sample or the exchange gas sorb.

#### 5.13 Removing the Inner Vacuum Can (IVC)

The IVC is sealed onto the insert using a cone seal. The cone seal cannot be easily separated because it uses a locking taper. A jacking plate should always be used for separating the cone seal on a HelioxVT, as shown in Figure 5-7. A jacking plate is provided as part of every HelioxVT system. Should you require additional or replacement items, please contact your nearest support facility.



#### CAUTION

Do not attempt to remove the Inner Vacuum Can (IVC) by prizing it away from the flange or by twisting it relative to the insert. Use the jacking plate as described below.



#### CAUTION

Do not allow the tail to fall to the floor as it is removed as this will deform the tapered sealing face.



#### 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 paste from the mating surfaces as soon as the IVC is removed so that dirt does not collect on them. Make sure that your hands are clean so that no small particles are allowed into the seal.



Figure 5-7: The jacking plate assembled onto a Heliox IVC tail (HelioxVL model shown).

With the insert at room temperature, vent the IVC by opening the valve on the top of the insert; if a bladder is still fitted following a warm up, you will need to remove this.

Support the insert and tail horizontally on a flat surface using V-Block supports.

Fit one set of the jacking plates to the IVC cone above the tapered joint. Ensure that the counterbores for the jacking screws are facing towards the IVC tail. Note that the plate cut out is designed to clear the pickup tube.

Wind the jacking screws into the second set of jacking plates so that they protrude approximately 30 mm from the surface.

Bring the second set of plates up to the IVC tail. Loosely bolt together and check that the jacking screws will align with the counterbores on the jacking plates fitted to the IVC cone. Apply torque to the fixing bolts to clamp the second set of plates to the tail.

Ensure that the insert and the tail are supported before separating the joint.

Wind the jacking bolts by hand until they contact the IVC cone jacking plates. Then use a 4 mm Allen key to turn the bolts – note that it is important to apply the jacking screws evenly, half a turn at a time. Very little force will be required to separate the joint.

Once the joint is separated, continue winding the bolts evenly by hand until the tail and cone are separated.

Now the components are separated, remove the jacking tools and clean up the seal faces, immediately so that dirt does not collect on them. Care is required to avoid scratching – do not use metal tools. Make sure that your hands are clean so that no small particles are allowed into the seal.



CAUTION

# Recommended torque for fixing bolts is **4 Nm**, do not damage the IVC by overtightening these bolts.

# 6 Service and maintenance

The HelioxVT will deliver repeatable and reliable performance if maintained properly during its usage. This section contains basic and essential maintenance information.

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

#### 6.1 O-rings

Oxford Instruments recommends replacing the cryostat's O-rings on a two-year cycle. Whenever a part of the cryostat is removed, or if there is a suspected leak on the system, check the relevant O-rings. Ensure that the O-rings are clean, undamaged and lightly greased. Any damaged O-rings should be replaced immediately.

#### 6.2 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 can be difficult to remove 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 IVC flange. Care should be taken when handling (wearing gloves) and storing (sealed bag/box) to protect the charcoal from contamination.

#### 6.3 Troubleshooting

Should you encounter a problem with your system, it is first important to establish the source.

Refer to the troubleshooting recommendations in Table 6-1 for problems arising from the cryostat itself, or a combination of the above.

If you are unable to resolve the problem, please direct all enquiries through your nearest support facility. Please provide a full set of test data for diagnosis, along with details of any additions or modifications that you may have made to the system.

Issue	Possible cause	Recommendation
Poor base temperature with short hold time	High heat load from VTI.	Check VTI temperature. Try opening the needle valve slightly and monitor any change.
	High level of exchange gas in IVC.	Suspect a leak into the IVC or contamination of the exchange gas sorb. The cone seal is the easiest joint to check. If the problem is observed on two successive cool downs the charcoal in

Issue	Possible cause	Recommendation
Poor base temperature		the exchange gas sorb should be
with short hold time		changed.
	Increased heat load due to additional	Try to improve the heat sinking at each
	experimental wiring installed.	stage, reduce the diameter of the wire,
		or increase its length between stages.
	Radiative heat load down the line-of-	Check the line-of-sight port especially
	sight port.	carefully.
	The heater on the <sup>3</sup> He pot may be	Unplug the heater (and sensor) from
	being energised unintentionally.	the MercuryiTC and check the base
		temperature and hold time again.
	A touch from the <sup>3</sup> He pot, sample, or	Warm up the system and check the
	the wiring to the IVC.	alignment.
	The excitation current for the high	Reduce the excitation current.
	temperature sensor may introduce a	
	small heat load onto the <sup>3</sup> He pot.	
	An air leak into the IVC.	Check the IVC valve and Fischer
		connector seals.
Poor base temperature	Check that the <sup>3</sup> He pot thermometer is	The calibration of some resistance
with long hold time	reading the correct temperature by	thermometers can change with time.
	comparing it with another known	
	thermometer.	
	If this is found to be reading the	First contact Oxford Instruments for
	correct temperature it is possible that	advice.
	the <sup>3</sup> He sorb is contaminated.	To clean the sorb, the <sup>3</sup> He needs to be
		removed from the insert using a cold
		trap. Pump the sorb using a turbo-
		molecular pump whilst warming it
		slightly. Once clean, return the <sup>3</sup> He to
		insert by cooling the sorb to <5 K.
Good base temperature	Poor condensation of <sup>3</sup> He.	Check the 1 K plate temperature
with short hold time		during condensation.
		Check that the sorb thermometer is
		reading correctly, and that the sorb
		temperature is correct during
		condensation.
		Check the effect of condensing at a
		Charlette affect of many flow through
Condensation difficult	High neat load on 1 K plate during	the VT
	Condensation.	the VII.
very slow cool down to	Poor thermal contact to the "He pot.	check the mounting of the sample and
Cancistant looks on the	Deer cooling around the N/C core is int	Sensor on the pot.
Consistent leaks on the	Poor sealing around the IVC cone joint.	check that you are using enough CAF
IVC COTTE SEdi		Check the expire date of the paste
		If there are deep scratches in the
		mating surfaces rework or new parts
		may be required Contact Oxford
		Instruments for advice
		instruments for advice.

Table 6-1: Common issues and most likely causes.

#### **HelioxVT specifications** 7

#### 7.1 Performance

Performance	Specification
Base Temperature	300 mK for 40 hrs (no applied heat load)
Cooling Power	50 $\mu$ W at $\leq$ 350 mK, with a hold time of 6 hrs
Temperature control range	300 mK to 300 K
Temperature control stability	$\pm 3$ mK for T $\leq$ 1.2 K and $\pm$ 0.1 K for T > 1.2 K

Table 7-1: Performance specifications of the HelioxVT.

#### 7.2 **Electrical power**

Component	Power Consumption	Voltage	Frequency
MercuryiTC temperature controller	450 W	100 – 240 V AC	50 – 60 Hz
Table 7-2: Electrical specifications.			

7.3 **Physical** 

Parameter	Nominal Value
Weight (kg)	<50
Dimensions W × D × H (mm)	246 × 320 × Variable. See System GA
Table 7-3: Physical specifications of Heliox//T insert	

Table 7-3: Physical specifications of HelioxVT insert.

#### 7.4 **Technical exclusions and assumptions**

Performance specifications are given for standard configurations and intended use. Siting, environment, system variations, modifications and upgrades may affect the performance.

#### 7.5 Drawings

A General Assembly (GA) drawing is included on the USB stick supplied with the system.

Please quote you Sales Order number if you require information or copies of the drawings.

#### 7.6 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

#### Properties of Materials at Low Temperature, A Compendium

Victor J. Johnson (General Editor), National Bureau of Standards.

Pergamon Press, 1961.

Vacuum Technology its Foundations Formulae and Tables Leybold Heraeus GMBH.

Superconducting Magnets by Martin N. Wilson, Clarendon Press, Oxford, 1983, ISBN 0-19-854805-2

> **Eléménts de Cryogénie** (in French) by R.R. Conte, Masson & Co, Paris, 1970.

**Experimental Techniques in Condensed Matter Physics at Low Temperatures** 

by Robert C Richardson and Eric N Smith, Addison Wesley Publishing Company Inc, 1988, ISBN 0-201-15002-6

#### Matter and Methods at Low Temperatures

by Frank Pobell, Springer Verlag, 1992, ISBN 0 540 53751 1 and 0 387 53751-1

#### **Practical Cryogenics**

An Introduction to Laboratory Cryogenics. by N.H. Balshaw, Oxford Instruments Ltd, 1996.

#### Introduction to Thermometry below 1 K

(A review of the available techniques) Oxford Instruments Ltd., Ultra Low Temperature Group, 1990.

## 7.7 Cleaning and general care

All stainless-steel surfaces may be cleaned with water or isopropanol, a mild abrasive may also be used like "scotchbrite" on matt or unpolished surfaces.

All painted surfaces and labels should be cleaned with warm soap and water, no solvents or abrasives should be used.



CLEANING SOLVENTS Never use incompatible solvents when cleaning, never clean the system when cooling, cold or evacuating, O-ring failure may result in vacuum loss which could damage the system.

# 8 Appendix A – Configuring temperature controller

A Heliox system is usually supplied with a MercuryiTC Temperature Controller configured for use with the system. If your system does not include its own MercuryiTC, the configuration files should still be available from the factory testing and are normally supplied as part of the customer documentation on the USB stick included.

The following instructions are provided should you need to configure or re-configure your Heliox system with a new or different MercuryiTC.

#### 8.1 Regeneration parameters

Prior to cooling the system, the parameters CONDENSED\_TEMP and ACCEPT\_BASE should be set to a lower-than-expected value (e.g. 0.1 K). This will ensure the system never completes the regeneration routine and allows time for other parameters to be optimised.

A set point of 0 K should be entered and then the system cooled. The Controller will wait for the helium-3 pot to cool and then attempt to regenerate. The temperature at which the helium-3 sorption pump will be controlled during regeneration will have been optimised in the factory and will not need to be changed, but the OPT\_NV\_RCON setting may need to be adjusted. The flow through the VTI needle valve should be adjusted to give the lowest possible helium-3 pot temperature.

**Note:** This does not necessarily correspond to the lowest possible helium-4 plate temperature, particularly on systems running in VTIs.

This will ensure that the maximum fraction of the helium-3 charge is condensed. At this stage CONDENSED\_TEMP should be set to a value 200 mK above this minimum temperature to allow for small variations in performance. The system will then cool down towards its base temperature, where the low-temperature parameters can be determined.

#### 8.2 Low-temperature operation parameters

The minimum helium-3 pot temperature will be obtained when the helium-3 sorption pump is as cold as possible. The OPT\_NV\_LT parameter should now be adjusted to minimise the helium-3 pot temperature.

Once the minimum temperature is found ACCEPT\_BASE should be set to a value 20 mK above this minimum temperature to allow for small variations in performance.

**Note:** The minimum temperate and optimum flow found in this way assume that there is no experimental heat load to be applied to the helium-3 pot.

If your experiment will generate a significant heat load (~100  $\mu$ W or more) then the additional helium-3 evaporation will apply a large heat load to the sorption pump (the heat of adsorption is much greater than the latent heat of vaporisation). In this case the OPT\_NV\_LT may need to be increased to ensure the sorption pump temperature remains as low as possible during operation.

# 8.3 High-temperature operation parameters

In high temperature operation OPT\_NV\_HT should be set such that the VTI temperature remains below ~3 K during operation. This ensures the IVC exchange gas sorption pump remains cold.

# 9 Appendix B - Remote operation

The Remote Interface (RI) interacts with the MercuryiTC Heliox Controller using the Transmission Control Protocol (TCP), part of the Internet Protocol Suite, as the transport layer. This section will describe the functionality available through the RI and give some examples of implementations in various programming languages.

### 9.1 **Prior to using the remote interface**

As stated above, the communication to the RI is via TCP. To establish a connection to the RI you also need to know the Internet Socket Port Number (ISPN) over which the communication will take place; for a MercuryiTC this is always set to 7020. Access to this port should be configured on your local network.

To open a connection to the RI you must also know the Internet Protocol (IP) address of the MercuryiTC Heliox Controller. This may be found by tapping the "Settings" button on the Controller front panel, then using the arrows to scroll across to the "Ethernet" tab, as shown in Figure 9-1.



Figure 9-1: MercuryiTC Heliox Controller Ethernet settings.

**Note:** If you see that DHCP is 'ON' then the Controller is using the Dynamic Host Configuration Protocol to be automatically assigned an IP address. It is possible that this IP address could be changed automatically by the DHCP server when the current lease expires; this may mean that you will lose the ability to communicate with your system unexpectedly. You may prefer to ask your network administrator to assign the Controller a static IP address.

The application you create to communicate with the RI can be constructed in any development environment provided access to the TCP port is possible, throughout the rest of this manual the features of the RI will be explained in detail to enable software to be constructed to access the RI in any programming language.

# 9.2 Communicating with the remote interface

All commands sent to the RI application layer are encoded as ASCII text. Commands can either be to request data (READ commands) or to define the state of the system (SET commands). The remote interface provides verification to all commands (STAT response). All commands should be terminated with a carriage return and line-feed 'CRLF'; ASCII characters 13 ('0x0D') and 10 ('0x0A').

The syntax for the communication is hierarchical and similar in concept to the SCPI protocol. Commands are constructed from keywords as follows:

```
<VERB>:
```

Where the verb is either 'READ' or 'SET'.

<VERB>:<NOUN>

Where the noun is either 'SYS' when the command is addressed to the Controller itself, or 'DEV' if the abstracted "Heliox" type or a specific system component (such as an individual temperature measurement board) is to be addressed.

The command key words should be separated with a colon; ASCII character 58 ('0x3A'). All commands will generate a response, if the command or verb or noun is impossible to interpret the command will return:

```
INVALID | <VERB>:INVALID | <VERB>:<NOUN>:INVALID
```

If the user does not have the required permission to change or read the parameter being addressed the command will return:

DENIED

Refer to the MercuryiTC manual for more information on user permissions.

#### 9.3 Remote interface commands

Hardware commands will have the form:

<VERB>:<NOUN>:<UID>

where UID is the unique identifier of the system component being addressed. If the UID does not exist in the system being addressed the command will return:

NOT\_FOUND

If the command is directed to a system sub-component, then the type of hardware is specified

```
<VERB>:<NOUN>:<UID>:<HW-TYPE>
```

where 'HW-TYPE' defines the hardware referred to by the UID. The hardware may have a status, or other parameters that could be read / set

<VERB>:<NOUN>:<UID>:<HW-TYPE>:<PARMAS>

where 'PARMS' defines the parameter of interest. Additionally, a given piece of hardware could contain many data streams - these are accessed as "signals"

<VERB>:<NOUN>:<UID>:<HW-TYPE>:SIG:<S-TYPE>

where 'S-TYPE' defines the signal to be accessed. A signal will be returned as a value, followed by a SI unit prefix (n, u, m, k, M etc.) if necessary, followed by the units (A, V, mb, K etc). Not all signals are available on all hardware types (most needle valves do not perform temperature measurements for example). If the function is not applicable to the device being addressed the command will return:

N/A | NOT\_FOUND | INVALID

The command sets for the MercuryiTC are detailed in the manual for that device. The abstracted Heliox commands are an additional set of commands over the standard MercuryiTC set. The software

control is configured such that only this superset of commands should be required for standard operation.

#### 9.4 Heliox commands

The general structure of commands will be:

<verb></verb>	[READ   SET]
<noun></noun>	[DEV]
<uid></uid>	[HelioxX]
<hw-type></hw-type>	[HEL]
<s-type></s-type>	[STAT   TEMP   TSET]

Therefore, the current helium-3 pot temperature can be read back from the Controller with:

READ:DEV:HelioxX:HEL:SIG:TEMP

And the state of the system read back with:

```
READ:DEV:HelioxX:HEL:SIG:STAT
```

## 9.5 Control

Full control of the system in provided through one settable parameter, the helium-3 pot temperature control set-point. A new set point (350 mK in this case) can be entered with:

SET:DEV:HelioxX:HEL:TSET:0.350

#### 9.6 Testing the connection

The remote interface connection can be tested using Telnet. Connection is established with the command:

telnet xxx.xxx.xxx yyyy

where xxx.xxx.xxx is the IP address of the Controller and yyyy is the port number (7020) to connect on.