Oxford Instruments NanoScience

Optistat CF

Operation Manual

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OXFORD INSTRUMENTS

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Contents

1 Introduction

This manual is designed to introduce you to the Optistat CF, 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 Optistat CF, further manuals and documentation will have been supplied with the system. These additional manuals and documents detail the components of the system, as well as important safety information and are summarised 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
MercuryiTC manual	Electronic copy on USB
Practical cryogenics	Electronic copy on USB
LLT Siphon manual	Electronic copy on USB
Transfer siphon quickstart guide	Electronic copy on USB
VCU flowmeter manual	Electronic copy on USB
Safety matters	Electronic copy on USB
Optistat CF test results	Electronic copy on USB
MercuryiTC safety sheet	Hard copy

Table 1-1: Documentation supplied with the Optistat CF.

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

1.1 Copyright

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 12 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 queries 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: Optistat CF
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)
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Email: nanoscience@oxinst.com (sales)
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Fax: +1 978 369-8287 (sales and support)
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Email: ServiceNSAsia@oxinst.com (service and support)
Web: www.oxford-instruments.cn

Japan

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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

All the materials and components used in the manufacture of the Optistat CF are in compliance without exemption with the EU Directive 2011/65/EU for Restrictions of Hazardous Substances (RoHS). This is based on information provided by Oxford Instruments suppliers and is accurate to the best of our knowledge.

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 Optistat CF 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

	COLD OBJECTS 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 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
Outer Vacuum Chamber	Relief valve to atmosphere	0.25 bar differential
Transfer Siphon	Relief valve to atmosphere	0.25 bar differential
Sample Space	Relief valve to atmosphere	0.25 bar differential

Table 2-1: Pressure relief valve information.

The system's pumping valves for the outer vacuum chamber, sample space and transfer siphon have in-built pressure relief plates, as shown in Figure 2-1. This allows the system's vacuum chambers and sample space to vent to atmosphere if it becomes over-pressurised. A restoring spring provides the force required to re-seal the cap (red) automatically when the pressure drops.

Figure 2-1: Example of the pressure relief valve on the Optistat CF.

Do not modify or tamper with these safety features in any way. Additionally, ensure that nothing can restrict the movement of any of the pressure relief valves. The relief valves should not vent during normal operation of the system.

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 cryogens 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 Temperature and voltage limits

The Optistat CF system is supplied with a MercuryiTC temperature controller. Safety features for the temperature controller are described in the MercuryiTC manual supplied with the system. You should ensure that you understand and comply with all safety warnings and cautions.

The MercuryiTC will have been set up in the factory in order 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 should 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	510 K

Table 2-2: MercuryiTC system control limit values

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 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.

3 System description

The Optistat CF is a continuous flow, top-loading static cryostat and can be held at temperatures between 3.4 K and 500 K using a MercuryiTC temperature controller. The sample is mounted on a removeable probe which is cooled via exchange gas during operation. Up to five windows can be fitted to provide optical access to the sample space. The sample space and radiation shields are thermally isolated from the room temperature surroundings by the outer vacuum chamber (OVC).

Continuous flow cryostats like the Optistat CF do not have an internal reservoir of cryogens. The liquid is supplied from a separate storage vessel through an insulated transfer siphon. It flows through a heat exchanger around the sample space and out of the cryostat to the pump. A thermometer and heater are mounted on the heat exchanger and these can be used with a temperature controller to control the temperature of the heat exchanger. The sample is mounted in a separate space, which is filled with exchange gas. This gas provides good thermal contact between the heat exchanger and the sample. The flow of liquid which cools the cryostat does not come into direct contact with the sample.

In a static exchange gas system, you can adjust the atmosphere around the sample to suit the experimental requirements. It is difficult to accidentally block the cryostat with frost while changing the sample. If the pressure in the sample space is kept low enough, it is impossible for liquid to condense around the sample, where it may interfere with optical measurements. In addition, if you are doing high voltage experiments, you may be able to reduce the risk of an electrical discharge by choosing a suitable sample space pressure.

A range of window materials are available, covering most of the electromagnetic spectrum. The inner windows are sealed with copper gasket seals. The heat exchanger fitted to the Optistat CF has a maximum operating temperature of 500 K. However, the maximum temperature the cryostat can operate at is limited by the type of window fitted.

3.1 The Optistat CF cryostat

A schematic of the Optistat CF is shown in Figure 3-1. The main features of the cryostat are:

- The outer vacuum chamber (OVC) with 4 radial windows and one axial window.
- The transfer siphon arm.
- Sample space which can optionally be filled with exchange gas.
- Sample space entry port for probe loading.
- System heat exchanger with heater and sensor.
- Two pressure relief devices, one for the OVC and another for the sample space.
- A diagnostic 10-pin sealed connector for temperature control.

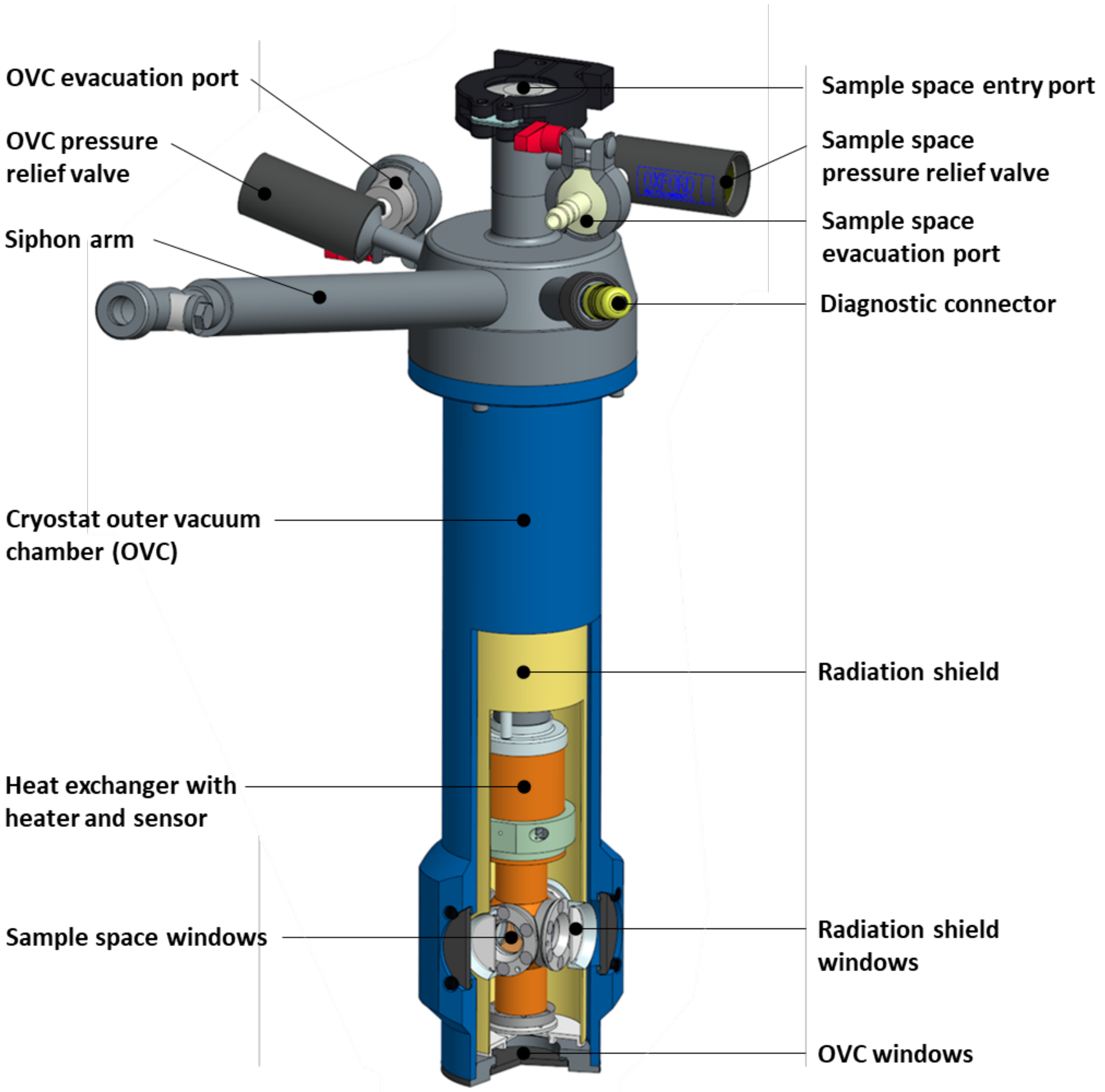

Figure 3-1 Optistat CF cryostat schematic.

3.2 The sample probe

There are three variants of Optistat CF sample probe; standard, height and rotate, and precision height and rotate. All these Optistat CF sample probes are fitted with a 10-pin seal as standard. The connector is wired to a Harwin pin ring mounted just above the sample holder. The pin configurations for the 10-pin seal and Harwin pin ring are shown in Figure 3-2 and Figure 3-3, respectively. The wiring configuration for the sample rod is set out in Table 3-1.

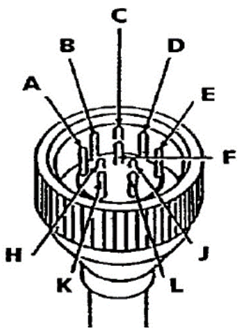

Figure 3-2: Pin configuration for the 10-pin connector.

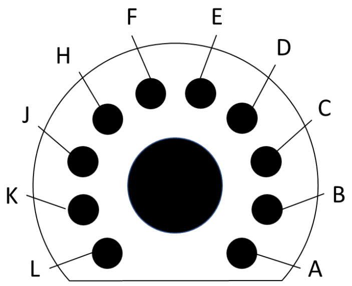

Figure 3-3: Harwin pin ring configuration, as viewed from above.

Connector Pin	Harwin Ring Pin	Wire	Function
A	A	34 SWG Cu	Not Used
B	B	34 SWG Cu	Not Used
C	C	40 SWG Cu	Not Used
D	D	40 SWG Cu	Not Used
E	E	40 SWG Cu	Not Used
F	F	40 SWG Cu	Not Used
H	H	40 SWG Cu	Not Used
J	J	40 SWG Cu	Not Used
K	K	40 SWG Cu	Not Used
L	L	40 SWG Cu	Not Used

Table 3-1: Pin configuration for the sample probe 10-pin seal to harwin pin ring.

3.2.1 Standard sample probe

The standard Optistat CF sample probe is shown in Figure 3-4. The sample rod has fittings to attach a sample holder to its base. When the sample rod is inserted into the Optistat CF, the sample holder then sits between the system's radial windows

Figure 3-4: Standard Optistat CF sample probe.

3.2.2 Height and rotate sample probe

The Optistat CF height and rotate sample probe is shown in Figure 3-5. The sample rod has fittings to attach a sample holder to its base. When the sample rod is inserted into the Optistat CF, the sample holder then sits between the system's radial windows. Additionally, the height and rotate sample probe is fitted with a combined height and rotation adjustor, this allows for sample position adjustments after the probe has been inserted and cooled within the Optistat CF.

Figure 3-5: Optistat CF height and rotate sample probe.

3.2.3 Precision height and rotate sample probe

The Optistat CF precision height and rotate sample probe is shown in Figure 3-6. The sample rod has fittings to attach a sample holder to its base. When the sample rod is inserted into the Optistat CF, the sample holder then sits between the system's radial windows. Additionally, the precision height and rotate sample probe is fitted with a high precision rotation adjustor and micrometer. These components allow for the precise positioning of samples after the probe has been inserted and cooled within the Optistat CF.

Figure 3-6: Optistat CF precision height and rotate sample probe.

3.2.4 Sample holders

There are two sample holder options for the standard sample probe supplied with the Optistat CF. The first is a plain reflectance sample holder, as shown in Figure 3-7 (Left). Several small samples may be mounted on this sample holder at once. The second is a transmission sample holder which has a 15mm diameter aperture and includes a sample retaining clamp, as shown in Figure 3-7 (Right).

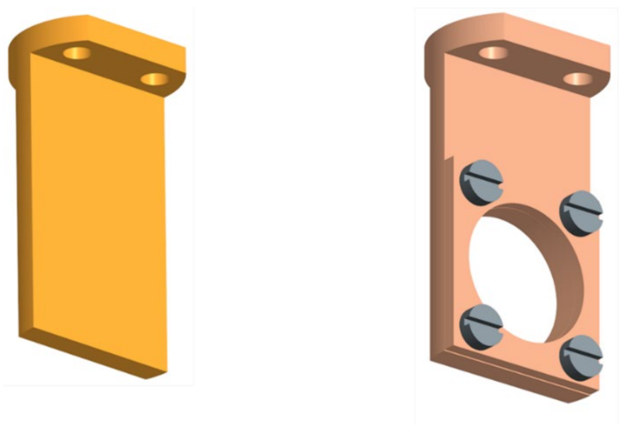

Figure 3-7: Reflectance sample holder (Left) and transmission sample holder (Right).

3.3 The cryogen transfer siphon

The LLT transfer siphon is designed for ultra-low loss performance. The cold exhaust gas from the cryostat flows along the tube, and the enthalpy of the gas is used to shield the flow of liquid from the room temperature surroundings.

The LLT 600 is the standard transfer siphon option and is manually controlled. The LLT 650 is the automated version of the LLT 600 which allows the gas flow rate to be optimised automatically using the MercuryiTC temperature controller. For more information, please refer to the LLT Siphon manual.

3.4 MercuryiTC temperature controller

A MercuryiTC is used as the temperature controller for the system. The MercuryiTC monitors and controls the thermometry of the system and adjusts the system heater voltage to hold the sample holder at a defined temperature.

The MercuryiTC is configured with measurement cards in specific locations. This configuration is detailed, along with the interconnecting cables, in Table 3-2.

Slot	Card Type	Function	Connection
Main Board	Sensor & Heater	Temperature Control	Diagnostic 10-Pin
1-3	Not used	n/a	n/a

Table 3-2: MercuryiTC configuration for the Optistat CF.

If the Optistat CF Dynamic has been supplied with an automatic LLT Siphon it will be fitted with an additional ‘auxiliary’ card in MercuryiTC’s fourth slot. This auxiliary card controls the needle valve of the LLT Siphon and connects to the siphons 7-Pin connector. For more information, please refer to the LLT Siphon and MercuryiTC manuals.

3.5 System wiring

The Optistat CF cryostat is fitted with a 10-pin connector on the OVC top plate. This is used for the connection to the sample space heater and sensor. The pin configuration for the 10-pin connector is shown in Figure 3-8. The seal is held in place by a black nut, this nut should not be removed, unless access to the wiring is required.

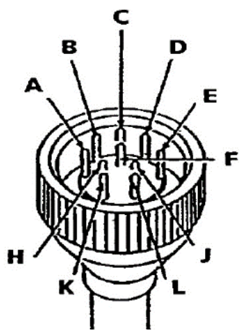

Figure 3-8: Pin configuration for the 10-pin connector.

The wiring configuration for the temperature control connector is set out in Table 3-3, as per the pin configuration shown in Figure 3-8.

Pin	Function	Polarity	Type
A	Heat Exchanger Heater	H-	Firerod (nominal 20 ohm)
B		H+	
C	Heat Exchanger Sensor	V+	Rhodium-iron resistance thermometer
D		V-	
E		I+	
F		I-	
H – L	Not Used	n/a	n/a

Table 3-3: Pin configuration for the temperature control 10-pin seal

3.6 Sensor calibrations

The calibrations for the calibrated sensor on the system, a rhodium-iron resistance thermometer, would have been loaded into the MercuryiTC at the factory. The raw calibration data from the sensor's manufacturer would have also been supplied separately.

3.7 Gas flow pump and flow controller

The Oxford Instruments GF4 gas flow pump is used to promote the flow through the cryostat. It is an oil-free, twin piston pump with a nominal displacement of 70 litres per minute. The air leak rate is guaranteed to be less than 10 cm^3min^{-1}. This pump is described fully in a separate manual.

The VC-U gas flow controller is used to control the flow of gas through the cryostat. It includes a flow meter (calibrated for helium gas) and a pressure gauge, so that the flow can be monitored. The VC-U gas flow controller is described fully in a separate manual.

3.8 System dewar

The dewar that is being used with the system must have a suitable interface so that it can link to the system's transfer siphon.

3.9 Weights and dimensions

The dimensions and weights for the main system components are given in Table 3-4.

Component	Length (mm)	Width (mm)	Height (mm)	Weight (kg)
Optistat CF Cryostat	230	480	480	3.7
MercuryiTC	310	485	90	8
LLT600 (Siphon)	1950	1570	55	5
GF4 Pump	245	130	180	9
VC-U Flowmeter	360	260	150	4

Table 3-4: Weights and dimensions of system components.

4 System installation

The setup of an Optistat CF is a straightforward procedure requiring no specialist training.

4.1 Unpacking the system

Carefully remove the cryostat and all the accessories from the packing case and check the packing list to make sure that you have all the components. Examine the system to make sure that it has not been damaged since it left the factory. If you find any signs of damage, please contact Oxford Instruments immediately.

> LIFTING THE CRYOSTAT
> The system should only be lifted by holding the main cryostat body or the system baseplate. Do not under any circumstance attempt to lift the cryostat by the siphon arm, as this will cause mechanical damage to the cryostat.

To run this system, the following components are required:

- The Optistat CF cryostat
- Cryogen transfer tube (LLT), with suitable storage dewar adaptor
- MercuryiTC temperature controller
- VC-U gas flow controller
- Oil free diaphragm pump (GF4)
- Polythene tube (10 mm outer dia.)
- Sensor & heater cable - CQB0090
- Ball bladder with an DN16NW fitting adaptor and SV12 dewar fitting
- Automatic needle valve cable - CWA0112 (option)
- High vacuum pumping system and lines (customer provided)
- Liquid helium or liquid nitrogen storage dewar (customer provided)
- Rotary pump for enhanced low temperature operation (optional, customer provided)

4.2 System configurations

Depending on the desired temperature range, the system can be operated in different configurations with different pumps. A schematic showing the operating configuration of the standard system components is shown in Figure 4-1. In this configuration, temperature down to 3.4 K can be achieved. The dashed red line is the CWA0112 cable, which is only required if the transfer siphon has been upgraded with an automatic needle valve.

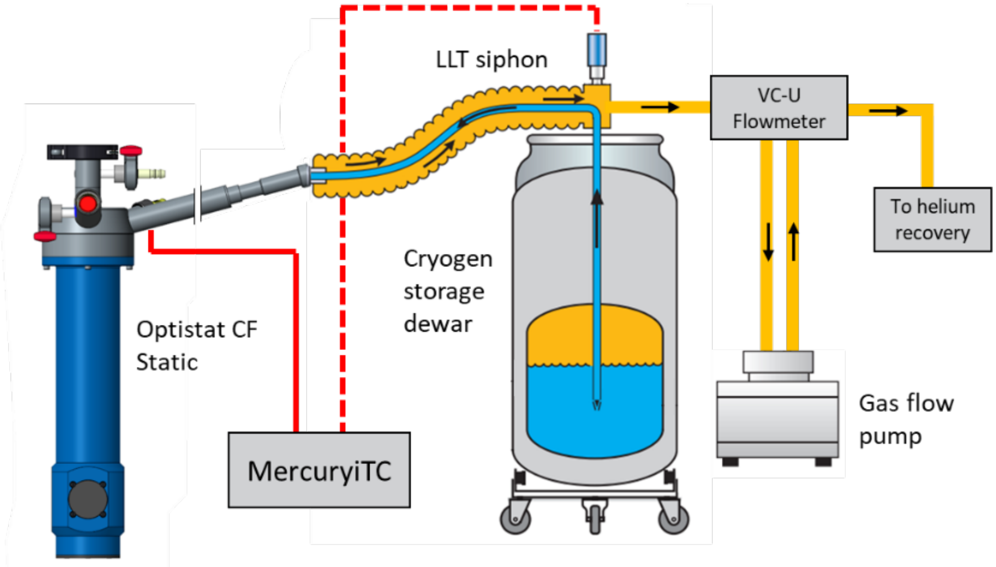

Figure 4-1: Standard system operating configuration.

A rotary pump may be used as the gas flow pump as part of a lower base temperature operating configuration, as shown in Figure 4-2. In this configuration, temperatures down to 2.3 K can be achieved. The dashed red line is the CWA0112 cable, which is only required if the transfer siphon has been upgraded with an automatic needle valve.

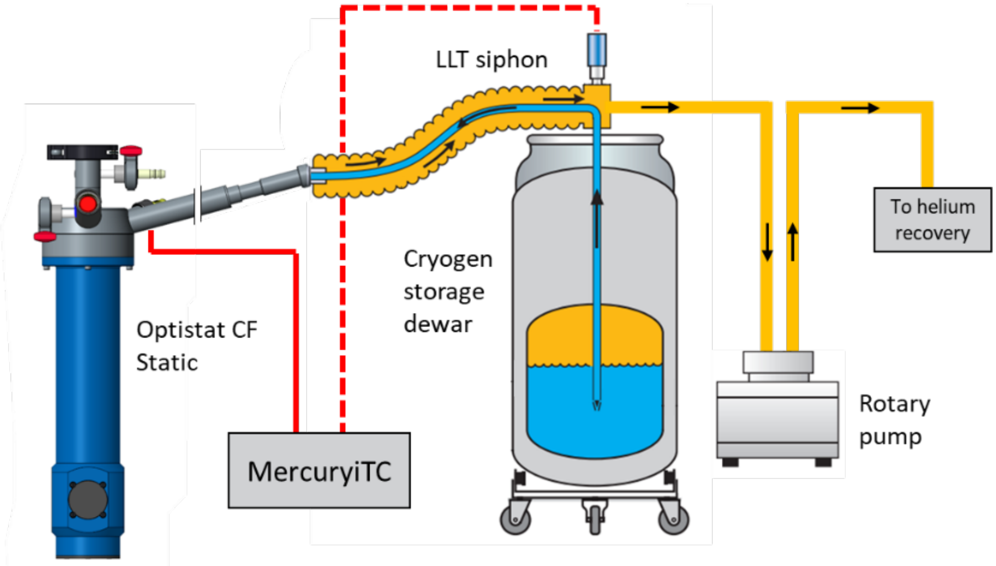

Figure 4-2: Rotary pump system operating configuration.

When setting up a chose configuration choose a suitable position to operate the cryostat safely, and if necessary, arrange for it to be supported so that it cannot fall or tip over accidentally.

⚠	PRESSURE RELIEF VALVES The closed vessels in the system are protected by pressure relief devices. Ensure that the pressure relief devices are not obstructed in anyway during system setup.

4.3 Evacuating the outer vacuum chamber

Before running the system, the OVC must be pumped to high vacuum to ensure it provides the required thermal insulation. When the system is new, all the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This outgassing does not mean that the system is leaking. The OVC should be pumped thoroughly before each cooldown, especially when the cryostat is new.

The cryostat OVC is fitted with an evacuation port and safety relief valve, as previously shown in Figure 3-1. To evacuate the cryostat's vacuum space, first connect the pumping system to the DN16NW OVC evacuation port. Before you open the valve to the cryostat vacuum space, evacuate the pumping line to 10^{-4} mbar. Then open the valve by turning the relief valve cap anti-clockwise a few turns. You can then begin pumping the cryostat vacuum space to a high vacuum. If possible, leave it to pump-down overnight. Once pumping is complete, turn the relief valve cap clockwise to close the valve. Once the valve has been firmly closed, vent the pumping line and disconnect the pumping system from the cryostat. After the evacuation of the OVC, Oxford Instruments recommends fitting a DN16NW blanking flange to the evacuation port to prevent any possible leaks.

For the pumping system, Oxford Instruments recommends that you use a turbo-molecular pump, backed by a rotary pump, and fitted with a cold trap which helps the system to remove any water vapour. If the system is badly contaminated with water vapour, the gas ballast facility on the rotary pump should be used.

4.4 Preparing the transfer siphon

Like the cryostat's outer vacuum chamber, the transfer siphon has a vacuum space to isolate cryogens from the surrounding environment. The transfer tube vacuum space has its own DN16NW evacuation port separate to that of the cryostat, which can be connected directly to a high vacuum pumping system. The transfer siphon must be evacuated to a high vacuum before the system can be run. For more information, please refer to the LLT Siphon manual.

If the transfer siphon has been supplied with the cryostat, it should fit into the cryostat arm without any modification. If it was not supplied with the cryostat, check that it fits in the cryostat arm as you may need to change its length or remove a PTFE washer before beginning the cooldown process. For more information, please refer to the LLT Siphon manual.

4.5 Preparing sample probe wiring

Optistat CF sample probe connectors are wired to a Harwin pin ring, just above the sample holder mounting position. All wiring made from the sample probe's Harwin pin ring should be thermally anchored to the sample probe tube to reduce the heat load to the sample.

4.6 Exhaust gas connections

In the standard operation configuration, a polythene tube is used to connect the exhaust port on the transfer siphon to the 'FROM CRYOSTAT' connector on the VC-U gas flow controller, as shown in Figure 4-3. The other connections of the VC-U gas flow controller are also made with polythene tubing. The 'TO PUMP' should be connected to the pump inlet, and the 'FROM PUMP' to the pump outlet. The exhaust line from the VC-U can either be connected to a helium recovery system or vented directly to atmosphere. For further details, please refer to the VC-U manual.

⚠	GAS LINE CONECTIONS When setting up the exhaust gas connections for ‘continuous flow’ mode, ensure all polythene lines are securely connected.

Figure 4-3: Rear view of the VC-U gas flow controller showing the connectors.

For low temperature operation, < 3.4 K, with a rotary pump, first remove the barbed adaptor clamped to the DN16NW fitting on the transfer siphon exhaust port. Attach a pumping line between the DN16NW transfer siphon exhaust port and the rotary pump. Make sure that an oil-mist filter is attached to the exhaust of the pump. The outlet of the oil-mist filter can then either be connected to a helium recovery system or vented to the atmosphere.

4.7 Connecting to the MercuryiTC temperature controller

The MercuryiTC has been configured by Oxford Instruments to suit the ordered system. When you first switch on the MercuryiTC, you will see the instrument home screen, similar to the screen shown in Figure 4-4.

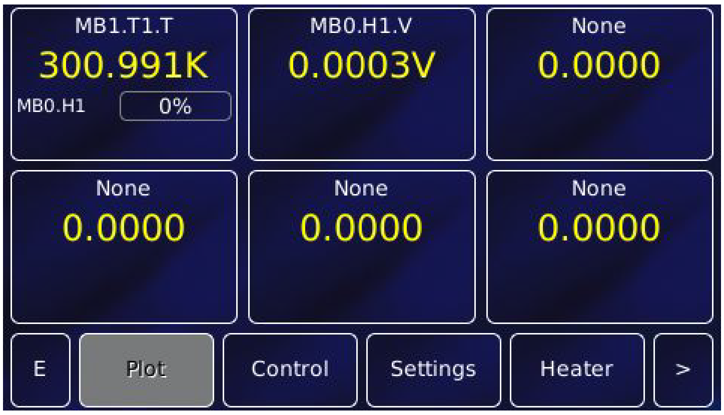

Figure 4-4: MercuryiTC home screen.

The cables from the MercuryiTC should be connected as per Table 4-1 with the MercuryiTC switched off. Note that cable CWA0112 is only required if you are using a transfer siphon with an automatic needle valve (LLT650). After the cable connections have been made, turn on the MercuryiTC. The temperature should read approximately 295 K (room temperature).

Cable	From (Mercury)	To	Function
CQB0090	MB1 (Sensor & heater)	OVC body (Diagnostic 10-pin)	Heat exchanger, sensor & heater
CWA0112	Auxiliary socket	LLT650 (7-pin)	Automatic needle valve

Table 4-1: System wired connections.

5 System operation

This section describes the operation of the Optistat CF in conjunction with an Oxford Instruments MercuryiTC temperature controller. The cryostat can be operated manually if a temperature controller is not available, although it may be difficult to obtain good temperature control in this configuration.

Either liquid helium or liquid nitrogen can be used as the cryogen for running the Optistat CF. Temperatures down to 77 K can be reached using liquid nitrogen, however, even at these temperatures, better temperature stability can be achieved using liquid helium. If liquid helium is used, it is possible to maintain a temperature below 4.2 K using the standard gas flow pump (GF4) and gas flow controller (VC-U). Lower temperatures (down to 2.3 K) can be achieved with a larger pump, such as a rotary pump.

5.1 Preparations

Ensure that the system has been properly prepared for operation, as described in Section 4.

SYSTEM PUMP DOWN
The system's OVC, sample space and transfer siphon must be pumped thoroughly to a high vacuum before each cooldown.

The following procedure assumes that you are using liquid helium with the system. The system can also be used with liquid nitrogen, but some of the techniques are different. Please see Section 5.10 at the end of this chapter for more details about the operation of the Optistat CF with liquid nitrogen.

WORKING WITH CRYOGENS
Before you start to use any cryogens, make sure that you are aware of the precautions that are necessary to ensure your safety. Refer to *Safety Matters* for more information.

5.2 Loading the sample probe and exchange gas

Oxford Instruments recommends that helium is used as the exchange gas within the Optistat CF sample space. The following instructions presume that helium, which is initially at atmospheric pressure, is used.

To load a sample probe in exchange gas, first remove the DN25NW clamp and blank from the sample space. Insert the desired sample probe into the sample space and replace the DN25NW clamp to secure it.

SAMPLE PROBE LOADING
Before loading the sample probe, inspect the DN25NW flanges on both the cryostat and sample probe for any damage or debris that could cause an air leak. Also inspect the O-ring to ensure it is not damaged or contaminated with any debris that could cause an air leak. If required, clean the O-ring with isopropanol and apply a light coating of vacuum grease before re-fitting the O-ring.

To begin evacuating the sample space, connect the pumping system to the DN16NW sample space evacuation port. Open the sample space valve by turning the valve cap anti-clockwise by a few turns and then turn the pump on to begin evacuating the sample space. If a high-vacuum pump is being used, it is sufficient to pump once. However, if a twin-piston pump, such as the GF4 supplied with the system, is being used, pump and flush the sample space with helium two or three times before continuing. Once pumping is complete, turn the valve cap clockwise to close the valve. After the valve has been firmly closed, vent the pumping line and disconnect the pumping system from the cryostat.

Fit the ‘SV12’ dewar fitting supplied with the system to the helium dewar in preparation to fill a bladder with helium. The SV12 has a valve to prevent the dewar depressurising suddenly as the bladder is removed. Fit an empty bladder to the free brass tube on the SV12 and ensure that the valve on the SV12 is closed. The bladder should then begin to fill, to speed up the process, squeeze and release the bladder a few times to introduce warm helium into the dewar and accelerate boil-off. Once the bladder is filled, clamp the rubber hose and open the valve on the SV12 to release any stored pressure within the dewar.

The final step is to fill the sample space with the exchange gas. Start by fitting the hose adaptor to the sample space evacuation port. Attach the helium filled bladder to the adaptor’s nozzle, allowing some helium gas to escape in order to flush air out of the connection as you make it. Slowly open the valve to let helium gas into the sample space. Leave the valve open with the bladder connected as this helps to monitor the pressure within the sample space.

5.3 Cooling the system

> ⚠ CHECKING FOR LEAKS
> The seal at the systems sample space entry port must be checked before cooldown. The test must be done using a leak detector and the leak rate must be below 10^{-8} mbarLs^{-1}. If the leak rate is not below this level the seal must be remade.

Before cooling the cryostat, the transfer siphon should be pre-cooled. To begin pre-cooling the siphon, set up the system components as shown in Figure 5-1. Note that the transfer siphon cryostat arm is still covered by its protection tube and is not in the cryostat.

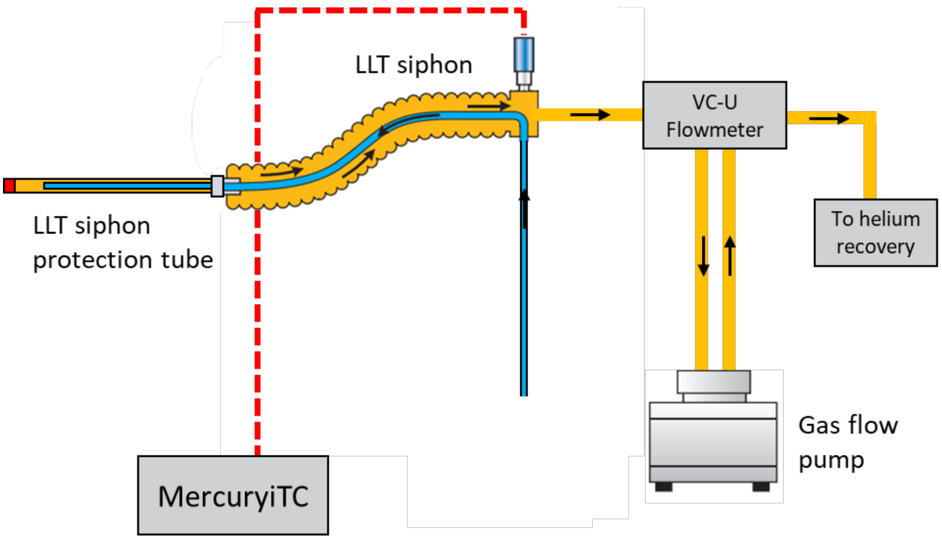

Figure 5-1: Transfer siphon pre-cooling configuration.

To start, fully close then fully open the needle valve on the transfer siphon. If a manual transfer siphon (LLT600) is being used, rotate the needle valve fully clockwise to close it, then open it by rotating six full anti-clockwise turns. If an automatic transfer siphon (LLT650) is being used, close the siphon needle valve by setting the gas flow to 0 % on the MercuryiTC. After the valve has finished moving, set the gas flow to 100 % to fully open the needle valve. If desired, it is possible to operate an automatic transfer siphon manually by setting the gas flow control on the MercuryiTC to **Manual**.

Next, fully open the needle valve on the VC-U gas flow controller. Open the exhaust valve of the liquid helium dewar to release any pressure. Remove the plug in the transfer siphon entry fitting and slowly lower the dewar leg of the siphon into the dewar. Switch on the GF4 or rotary pump. Some liquid will be used to cool the leg, and the dewar exhaust must be open to allow the boil-off to escape. If you try to cool the leg too quickly, a large amount of liquid will be wasted.

Once the siphon leg has been loaded into the helium dewar, monitor the helium flowmeter on the VC-U gas flow controller. When you see the flow of helium, turn off the pump and disconnect the protection tube from the siphon. Push the cryostat entry arm of the siphon into the siphon entry arm of the cryostat and engage the nut on the siphon with the thread on the cryostat arm. Take care not to over-tighten the nut. Set up the desired operating configuration with either the GF4 or rotary pump, as shown in Figure 4-1 & Figure 4-2, respectively.

Once set up, turn the system pump on. The cryostat heat exchanger and sample should now cool steadily, however, the transfer siphon and cryostat arm may contract by different amounts. To address this, the knurled nut on the cryostat arm should be tightened occasionally to ensure the seal with the cryostat is maintained. The system should typically cool to 4.2 K within 10 minutes. When the cryostat reaches 20 K, as read on the MercuryiTC, close the sample space valve to ensure no liquid condenses in the sample space. This will also reduce the risk of allowing air into the cryostat sample space which can happen if there is a leak in the bladder.

5.4 Operation below 4.2 K

Temperatures below 4.2 K are achieved by lowering the pressure in the heat exchanger. Since the pumping speed of any pump is limited, this can only be achieved by limiting the rate at which helium is supplied, using the needle valve in the transfer tube.

The dependence of temperature on flow rate is illustrated in Figure 5-2. It is important for continuous operation at low temperatures that the cryostat is not running in single-shot mode, i.e. with a pool of excess liquid helium in the heat exchanger. To prevent this, use the following procedure:

First put the heater control and the gas flow control of the MercuryiTC temperature controller into **Manual** mode, with zero heater voltage. When the cryostat has reached 4.2 K, close the needle valve on the transfer tube. The temperature will probably fall immediately, as helium in the heat exchanger is being boiled off. After a few minutes, the liquid would have boiled away and the temperature will start to rise. At this point, open the needle valve about a quarter turn. The temperature should stabilise around 20 K - point A in Figure 5-2. The needle valve should now be opened in very small increments, waiting for the temperature to stabilise after each change. As this is done, the temperature will gradually fall until the base temperature of the system is reached - point B in Figure 5-2. Be careful to not open the needle valve further than required, as this will increase the temperature of the system. Opening the valve fully will result in the system's temperature stabilising back at 4.2 K - point C in Figure 5-2. After reaching the system's base temperature, select the desired SET temperature on the MercuryiTC and switch the MercuryiTC heater control to **Auto**.

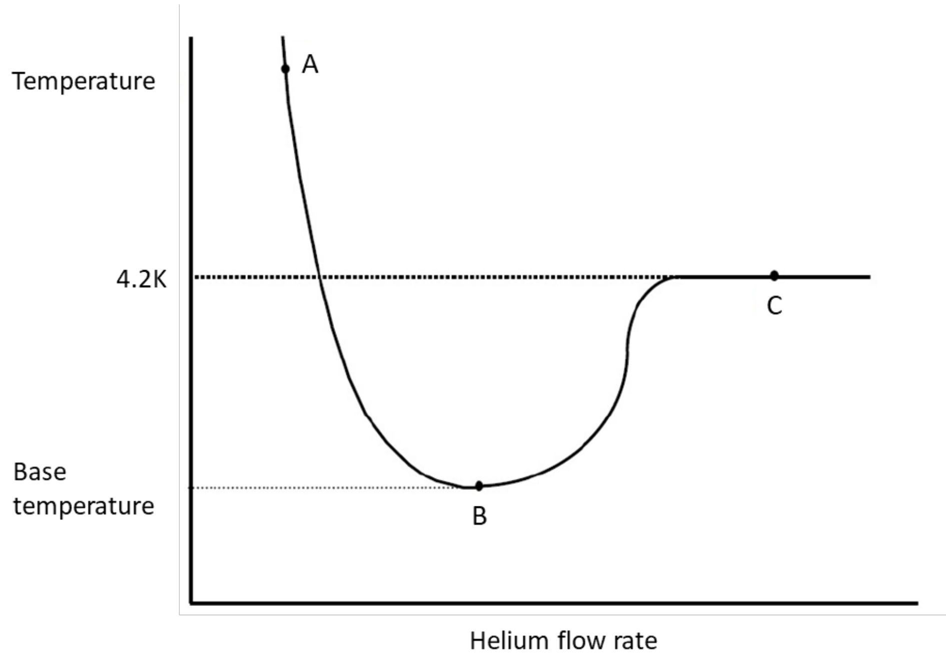

Figure 5-2: Temperature and helium flow rate relationship.

5.4.1 Transitioning from 4.2 K operation

HIGH PRESSURE IN HELIUM CIRCUIT
If you run the system near base temperature, it is possible for liquid to collect in the sample space heat exchanger. If too much heat is introduced to this liquid, it will boil quickly, developing a high pressure within the cryostat.

This situation can arise when setting the temperature controller to warm the system to a temperature above 4.2 K and allowing the heater voltage to increase automatically. The heater voltage will increase to the maximum available level introducing a large amount of heat and boiling off the liquid helium.

Oxford Instruments recommends using the following procedure to set a temperature above 4.2 K, after operating at or below 4.2 K:

1. Close the transfer siphon needle valve
2. Switch the MercuryiTC heater control to **Manual**
3. Set the heater to 5 V
4. Wait for the helium flow to stop
5. Switch the MercuryiTC heater control to **Auto**

The VC-U gas flow controller is fitted with an internal pressure relief valve, but it is best practice not to rely on this. If you are not using a VC-U, make sure that the gas line between the cryostat and the pump is fitted with a suitable pressure relief valve. If the gas is not allowed to escape quickly enough, the cryostat may be damaged.

5.5 Operation above 4.2 K

The MercuryiTC automatically controls the power supplied to the system's internal heater to maintain the set temperature. The MercuryiTC is a three-term controller, therefore the temperature control is optimised by setting the best values for:

- Proportional band (P)
- Integral action time (I)
- Derivative action time (D)

On the MercuryiTC home screen, as previously shown in Figure 4-4, the 'Control' button at the bottom of the screen can be selected to give the screen shown in Figure 5-3. From here, the system temperature can be set.

Figure 5-3: MercuryiTC 'Control' screen.

Tapping 'PID table' in the bottom left will display a screen like the one shown in Figure 5-4. This screen shows the current PID table loaded into the MercuryiTC. In this case, 'Optistat CF Mercury.pid' should already be loaded into the MercuryiTC.

Figure 5-4: MercuryiTC 'PID table' screen.

If an alternative PID table is desired, tapping 'Load' on the 'PID table' screen allows alternative PID tables to be viewed, as shown in Figure 5-5. These may be from the factory or created by the user. Tap a filename to select it and then tap 'Load' to load the selected PID table.

Figure 5-5: MercuryiTC 'select file' screen for loading alternative PID tables.

The PID values given in the test results for the system are suitable to give good stability. If you wish to improve the stability further, you may be able to do this by adjusting the three terms slightly. In Manual mode, individual PID values can be changed during operation. Control theory and the procedure for optimising the PID values are described in the MercuryiTC manual.

Tapping 'Close' on the 'PID table' screen will return the MercuryiTC to the 'Control' screen. Then tapping 'Home' will return the MercuryiTC back to the home screen.

5.5.1 Controlling at a 'set temperature'

To set a temperature, select the channel on the temperature controller corresponding to the sensor which will be used to control the system. Enter the desired temperature on the MercuryiTC and switch the MercuryiTC heater control to **Auto**.

It is not necessary to cool the cryostat to base temperature before you set the required temperature. If the temperature controller is set to the required temperature at the beginning of the cooldown, the cryostat should cool to the set temperature and the temperature controller should hold it at this point.

The helium flow should then be optimised so that the heater voltage of the temperature controller is not too high. If an automatic transfer siphon is being used the flow and heater voltage will be automatically optimised. However, if a standard transfer siphon is being used the flow will need to be manually optimised. This is done by reducing the flow with the transfer siphon needle valve until a reasonable heater voltage is attained. Some typical heater voltages for different temperature ranges are shown in Table 5-1 and should be used as a guide.

Temperature range (K)	Heater voltage (V)
4.2 – 20	3 – 5
20 – 300	8 – 12
> 300	> 8

Table 5-1: Typical heater voltages for different temperature ranges.

5.5.2 Operation above 300 K

To operate at temperatures exceeding 300 K, the OVC should be continuously pumped on using the OVC evacuation port. Pumping on the OVC continuously will help maintain the high vacuum required to operate the system.

SYSTEM WINDOWS
Before setting an operating temperature above 300 K, ensure that the high temperature sapphire windows have been fitted to the sample space. The procedure to change sample space windows can be found in Section 5.9.

After setting up continuous pumping and ensuring that the system's materials are of an appropriate material for the temperature desired, you may set a temperature exceeding 300 K, as described in Section 5.5.

MAXIMUM SET TEMPERATURE
Do not set a temperature higher than 500 K.

5.6 Changing samples

The sample holder can be removed and replaced while the cryostat is at any temperature above 4.2 K. The gas flow pump can be on or off when samples are changed. However, remember that the sample may be subjected to thermal shocks if it is warmed or cooled too quickly. Additionally, it will quickly become covered with ice if it is very cold when removed from the sample space. It is best to warm the sample to room temperature before you change it, this is easily done by setting the temperature at 300 K on the MercuryiTC temperature controller.

SAMPLE PROBE REMOVAL
If removal of the sample probe is attempted when cold, and cannot be removed easily, it is possible that the probe has become frozen into the system because of an air leak. In this scenario, the cryostat needs be warmed up with great care as the path to the pressure relief valve may be blocked. Immediately shut off the helium supply, set the heater voltage to 0 V and evacuate the surrounding area until the system returns room temperature. For more details refer to *Safety Matters* 1.2.4, Section 3.16.

COLD OBJECT
If the system is not warmed before the removal of the sample probe, its temperature could be as low as 4 K. Contact with cold objects and cryogens can cause serious injury to the skin as it may adhere to cold surfaces. Ensure that appropriate PPE is worn during probe removal.

Once the sample space is opened, air and moisture can contaminate it and cause the windows to fog. To prevent this, connect a bladder of dry helium gas to the sample space valve, and maintain a continuous flow of gas into the sample space to purge it whilst it is open to the atmosphere. To remove the sample probe, unfasten the DN25NW clamp and lift the sample probe out of the sample space.

Close the sample space as soon as possible, using a DN25NW clamp and the blank supplied, until the sample probe can be re-inserted.

SAMPLE PROBE LOADING
Before loading the sample probe, inspect the DN25NW flanges on both the cryostat and sample probe for any damage or debris that could cause an air leak. Also, inspect the O-ring to ensure it is not damaged or contaminated with any debris that could cause an air leak. If required, clean the O-ring with isopropanol and apply a light coating of vacuum grease before re-fitting the O-ring.

Before reloading the probe, make sure that it is clean and dry. Connect a bladder of dry helium gas to the sample space valve so that a continuous flow of gas can be used to purge the sample space once it is opened to the atmosphere. Next, remove the DN25NW clamp and blank, opening the sample space to the atmosphere. Place the sample probe into the sample space and secure it in place with the DN25NW clamp. Leave the sample space valve open with the bladder connected, as this helps to monitor the pressure within the sample space.

CHECKING FOR LEAKS
The seal at the systems sample space entry port must be checked before cooldown. The test must be done using a leak detector and the leak rate must be below 10^{-8} mbarLS^{-1}. If the leak rate is not below this level the seal must be remade.

5.7 Warming up the system

SYSTEM WARM UP
Never put any part of your body directly above the sample probe whilst the system is warming up.

To warm up the system, first switch off the gas flow pump. This will allow the pressure in the helium flow circuit to rise to the level of the storage dewar. After the pressure has equalised, close the siphon needle valve and carefully open the dewar exhaust, this will stop the supply of helium into the system. The transfer siphon can then be removed from the cryostat.

COLD OBJECT
Upon removal of the siphon, its temperature will initially be 4 K, additionally a small amount of cold helium gas may be exhausted upon removal. Ensure appropriate PPE is worn during this process.

Once the transfer siphon has been removed from the cryostat, immediately fit the special slotted bung into the cryostat siphon arm, as shown in Figure 5-6.

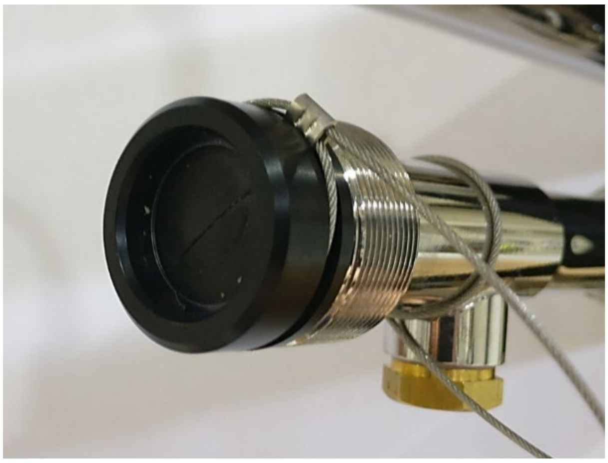

Figure 5-6: Special slotted bung fitted in the cryostat siphon arm.

SIPHON ARM SLOTTED BUNG It is essential to fit the slotted bung provided with the system as this ensures that the helium circuit within the cryostat is not contaminated, whilst also providing an escape path for any remaining helium within the circuit.

The system can then be left to warm up naturally. Whilst the system is warming, leave the bladder connected to the exchange gas valve and leave the valve open. This ensures that the exchange gas can expand safely as it warms up.

EXPANDING GASES When the cryostat is warming up, make sure that the exchange gas valve is opened to allow the exchange gas to expand into the bladder. The sample space is protected against dangerous pressure build up by a pressure relief valve, but it is not best practice to rely solely on it.

To speed up the warming process, a temperature of 300 K can be set on the MercuryiTC, using the procedure described in Section 5.5. To warm the system even quicker, allow a small volume of dry nitrogen gas from a bladder into the OVC to break the vacuum, once the temperature sensor reads higher than 100 K.

BREAKING VACUUM When breaking the OVC vacuum, do not use a bladder that has previously been used with helium. Never allow helium gas into the OVC as it is difficult to pump out again.

5.8 Removing and replacing the OVC and radiation shield

The OVC and radiation shield only need to be removed from the system if you wish to:

- Change the windows
- Repair the wiring
- Repair mechanical damage

Before beginning, ensure that the cryostat has been warmed to room temperature by following the procedure described in Section 5.7. Next, vent the OVC and sample space with a dry nitrogen source through their respective valves and ensure that they are at atmospheric pressure.

To remove the OVC, remove the four screws which hold the OVC to the cryostat top plate. This will allow the OVC to be carefully removed from the rest of the cryostat.

OVC & RADIATION SHIELD REMOVAL
When removing the OVC and radiation shield, ensure the components remain concentric and parallel to each other until they are fully separated. Accidental tilting may cause mechanical damage to the cryostat.

Upon removal of the OVC, the small feed capillary tube which supplies liquid from the siphon arm to the heat exchanger can be seen. This capillary passes through the hole in the radiation shield thermal link, it is important that the capillary does not touch the radiation shield or any of the other tubes. The slightly larger tube takes the exhaust gas from the radiation shield's thermal link back to the siphon arm.

To remove the radiation shield, remove the four radial screws which attach it to the radiation shield's thermal link. This will allow the radiation shield to be carefully removed from the heat exchanger and sample space assembly.

To reassemble the radiation shield, slide it back over the sample space assembly and ensure it is in the desired orientation before replacing the four screws to secure it to the radiation shield thermal link. Before re-assembling the OVC, check the O-ring is clean, undamaged and lightly greased. Additionally, ensure that the O-ring is firmly pushed into place so it will form a vacuum-tight seal. To reassemble the OVC, ensure it is in the desired orientation and carefully lower the cryostat into the OVC. Secure the OVC in place by the replacing the four screws to secure it to the rest of the cryostat.

OVC & RADIATION SHIELD REPLACEMENT
When replacing the OVC and radiation shield, ensure that the feed capillary tube does not touch the OVC, radiation shield or any other tubes. Such a touch will reduce the performance of the system.

5.9 Changing system windows

All the windows; OVC, radiation shield and sample space, of the Optistat CF can be removed so that they can be cleaned or replaced. As each type of window is secured to the system in a different manner, the removal of each follows a different procedure.

5.9.1 OVC windows

To change an OVC window, the Optistat CF must first be warmed to room temperature and vented as previously described in Section 5.7. The OVC windows are held in place by four retaining screws as shown in Figure 5-7.

To replace an OVC window, carefully remove the four retaining nylon screws around the edge of the window, taking care to ensure the window does not fall out by itself when removing the screws. The window can then be removed. Select the new window or blank and clean it using a suitable lens cleaner and lens tissue. Inspect the OVC window mount and O-ring to ensure they are not damaged or contaminated with any debris that could cause an air leak. If required, clean the O-ring with isopropanol and apply a light coating of vacuum grease before re-fitting the O-ring. After replacing the O-ring in its groove, position the cleaned window into the OVC window mount and replace the four retaining nylon screws to secure it in place.

OVER-TIGHTENING Do not over-tighten the OVC window screws. Over-tightening could lead to window fracture or poor vacuum sealing.

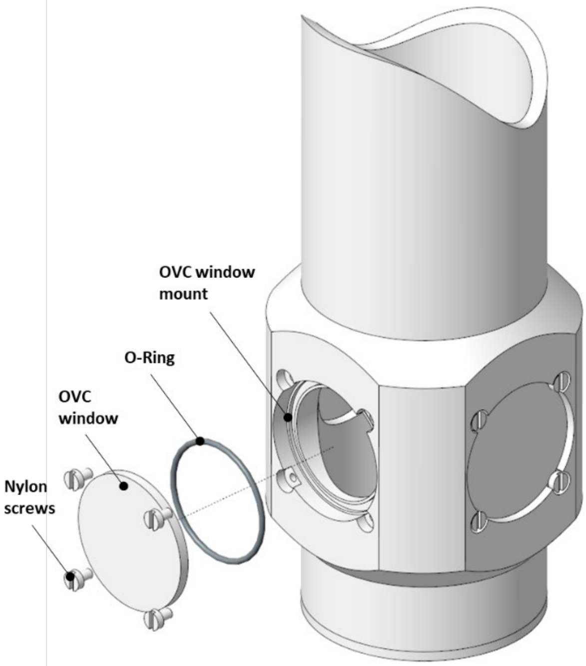

Figure 5-7: Changing Optistat CF OVC windows.

5.9.2 Radiation shield windows

To change or remove the radiation shield windows, the cryostat OVC must first be removed. The radiation shield windows can be changed with the radiation shield still attached to the system, however, it is often easier to change windows after removing the radiation shield from the cryostat. To remove the cryostat OVC and radiation shield, follow the procedure described in Section 5.8.

The radiation shield windows are held in place by wire clips, as shown in Figure 5-8. To remove a window, carefully prise the spring clip out of the groove in the window mount and turn the radiation shield over to allow the window drop out from a small height.

Select the new window and clean it using a suitable lens cleaner and lens tissue. Then apply a very thin layer of Apiezon-N grease edge of the window before replacing it in the radiation shield mount, as shown in the detailed view of Figure 5-8. This grease will improve the thermal contact of the window with the radiation shield. The spring clip should then be carefully pushed back into the window groove to secure the window in place. The radiation shield windows should fit tightly in their mounts and the force applied by the spring clips will be sufficient to ensure that the windows are properly cooled.

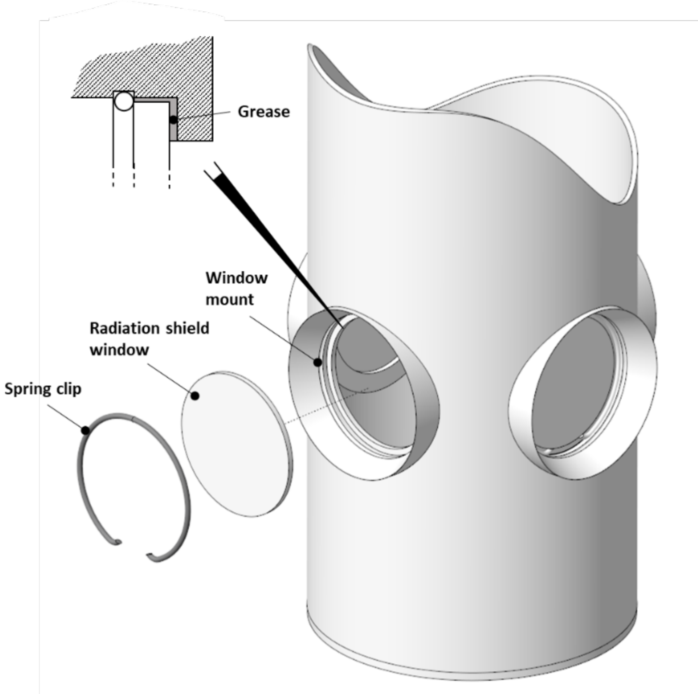

Figure 5-8: Changing Optistat CF radiation shield windows.

5.9.3 Sample space windows

To change the sample space windows the system OVC and radiation shield must be vented and removed from the cryostat. To remove the cryostat OVC and radiation shield, follow the procedure described in Section 5.8.

Each sample space window is held in place by six countersunk slot head screws and sealed with a copper gasket, as shown in Figure 5-9. These copper gaskets are suitable for use in temperatures up to 500 K. To remove a window, carefully undo its retaining screws and prise the window frame away from its mount. The copper gasket should be carefully removed and disposed of, as they are not reusable.

Clean the new window with an appropriate lens cleaner and lens tissue. Additionally, clean a new copper gasket thoroughly.

ANNEALED COPPER GASKETS
The spare gaskets supplied by Oxford Instruments are thoroughly annealed. If using gaskets supplied by a third party, ensure that they have been thoroughly annealed.

Place the gasket in the recess on the sample space window mount and position the new window frame over it. Use new high tensile steel screws to secure the window to the sample space. To ensure the window is secured evenly, tighten each screw and then the one opposite, so that the window frame is not distorted.

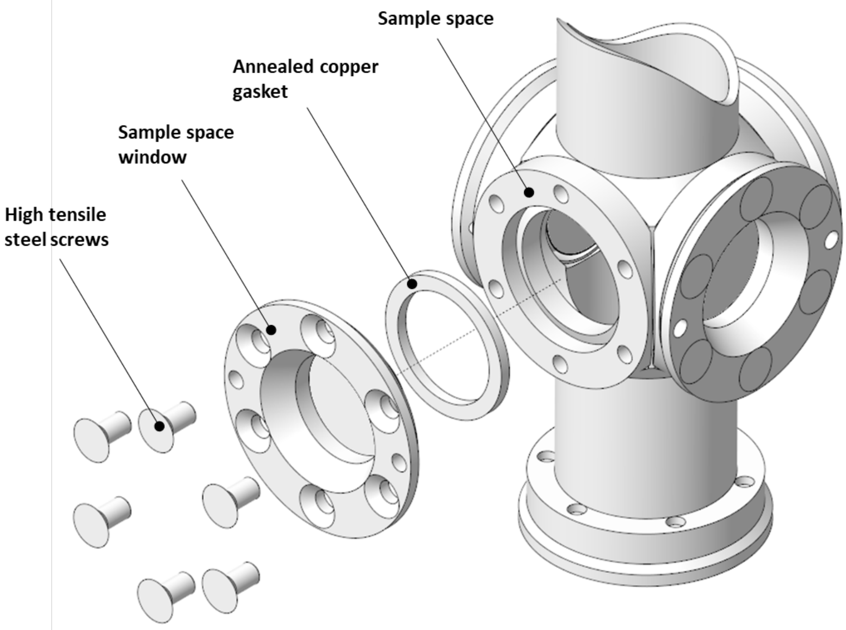

Figure 5-9: Changing Optistat CF sample space windows.

5.10 Operating with liquid nitrogen

The Optistat CF can be operated with liquid nitrogen instead of liquid helium. The basic operating procedure is the same, but there are a few differences:

- Ensure that the flow gauge calibrated for air is used on the VC-U.
- The viscosity of liquid nitrogen is greater than that of helium, so the flow rate through the cryostat is lower. This will increase the cool-down time.
- If you pump the liquid nitrogen to a pressure below 150 mbar, it may freeze and block the cryostat. A GF4 pump is unlikely to reduce the pressure sufficiently, but a rotary pump could.
- It is more difficult to control the temperature of the sample, and the stability specification is typically increased to ±0.2 K. It may be more difficult to control the temperature below 90 K because liquid collects in the heat exchanger and boils intermittently.
- Liquid nitrogen is not cold enough to cryopump air effectively, so it is more difficult to maintain a good vacuum in the OVC and transfer siphon. It may be necessary to pump the OVC and transfer siphon continuously.
- It is best to use the minimum flow possible to achieve good stability at low temperatures (especially below 90 K). If the temperature remains stable for a short time then suddenly becomes unstable, try reducing the flow. Adjust the flow rate slowly so that any liquid collected in the heat exchanger has time to boil away.
- The optimum flow rate for base temperature should be suitable for the whole temperature range. The flow rate can be increased to cool down more quickly but should be reduced again as base temperature is approached to prevent the cryostat filling with liquid.
- If you are using an auto-LLT transfer siphon, it is best to run it in **Manual** mode. When the system is in **Auto** mode, the flow rate may change too rapidly, and good stability may not be achieved.
- The PID settings on the temperature controller may be different from those given in the test results. Typically, the P and I values should be increased slightly.

6 Service and maintenance

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

6.1 Rubber 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 Troubleshooting

Should you encounter a problem with your system, it is first important to establish the source. This could be with the MercuryiTC, the transfer siphon or within the cryostat itself.

Diagnosis of MercuryiTC temperature controller faults should be made using the MercuryiTC manual T*roubleshooting* chapter. Diagnosis of the transfer siphon should be made using its respective manual. 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 analysis, along with details of any additions or modifications that you may have made to the system.

Issue	Possible cause	Recommendation
Poor temperature control	Incorrect temperature controller PID settings	Refer to the system's test results and the MercuryiTC manual.
Heater or sensor wiring fault	Wiring short or break	Check wiring resistances and compare with values in the factory test results.
Poor cryostat OVC vacuum	Vacuum leak	Examine O-rings for contamination or damage. Check windows and electrical connectors are correctly sealed and undamaged. Check pressure relief valves are sealing correctly. Use a leak detector to identify the leak.
	Water contamination	Warm up thoroughly to ensure all internal surfaces are free of condensed water. Additionally, pump the outer vacuum chamber with a rotary pump, with the gas ballast valve open.
	Sample space leak	Check the inner windows by pumping the sample space. If the pressure in the OVC then drops, check the inner windows individually and replace the gaskets as appropriate.
Poor sample space vacuum	Vacuum leak	Examine O-rings for contamination or damage. Check pressure relief valves are sealing correctly. Use a leak detector to identify the leak.
High base temperature	Heater still on	Check the heater is switched off.
	Poor vacuum	Check both the quality of the vacuum in the cryostat and transfer siphon.

Issue	Possible cause	Recommendation
High base temperature	Transfer siphon seal	Check the transfer siphon nut is tight enough. Check the transfer siphon is the correct length. Check the PTFE seal has not been damaged.
	Sensor fault	Check sensor resistance at base temperature and compare with the supplied sensor calibration data. Remember to account for the resistance of the wiring.
	Low gas flow	Check whether there is sufficient flow of gas through the system using the VC-U gas flowmeter. Check there is sufficient liquid in the dewar.
	Wiring heat load	Check that too much heavy wiring has not been added to the sample holder, introducing a high heat load.
	Mechanical damage	Check the radiation shield is not touching the sample space or OVC by warming to room temperature and removing the OVC.
Condensation on OVC	Poor cryostat vacuum	Check poor cryostat vacuum actions.
Sample cannot be warmed up	Low set temperature	Check the set temperature is higher than the present sample temperature or switch the heater on manually.
	Heater fault	Check that the heater circuit is not open or shorted by checking the resistance between pins 1 and 2 on connector 1 whilst checking for isolation between these pins and ground.
	Low heater voltage limit	Check that the heater voltage limit on the temperature controller is high enough. Normal settings are limited to: 40 V, Resistance: 20 Ω.
Moisture on the inner windows	Valve or O-ring leak	Check whether the sample space valve is shut. Examine O-rings for contamination or damage.
	Air from sample change	Warm the system and pump the sample space to remove contamination.
Sample rod cannot be removed	Condensed air in sample space	Air may have condensed in the sample space, freezing the sample holder in place. Connect a pump to the sample space valve and pump the sample space while you slowly warm the cryostat to room temperature. Check the sample space for leaks before you run the system again.

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

7 Optistat CF specifications

7.1 Performance

Performance	Specification
Temperature control range	3.4 K - 300 K *
Temperature control stability	± 0.1 K
Helium consumption during system cooldown	1.5 L
Helium consumption at 4.2 K	< 0.55 L / Hour
Sample change time	5 minutes
System cooldown time	25 minutes

7.2 Electrical power

Component	Power Consumption	Voltage	Frequency
MercuryiTC temperature controller	450 W	100 - 240 VAC	50 - 60 Hz

7.3 Physical

Parameter	Nominal Value
Weight (kg)	3.7
Dimensions W x D x H (mm)	230 x 480 x 480

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.

- The standard cryostat is fitted with 4 optical radial windows and 1 blank axial window.
- * Base temperature may be reduced by using a rotary pump.
- * Maximum operating temperature can be increased to 500 K by fitting high temperature windows.

8 Appendices

8.1 Checking the wiring

A resistance meter can be used to check the wiring on the cryostat. You should expect to measure the following values.

Pins	Approx. resistance (at 300K)
A - B	15 – 25 Ω
C - D	30 – 40 Ω
C - E	< 15 Ω
C - F	30 – 40 Ω
E - F	30 – 40 Ω
A - C	> 1 MΩ
A - ground	> 1 MΩ
C - ground	> 1 MΩ

Table 8-1: Expected resistance measurements for 10-pin cryostat connector.

8.2 Cleaning and general care

All stainless-steel surfaces may be cleaned with water or IPA (Isopropanol Alcohol), 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.