Oxford Instruments NanoScience Microstat He, He-R & Optistat CF-V Operators' Manual

System Manual / Issue 2.0 / December 2020





Page left intentionally blank

Contents

1	In	itroduct	tion	5
	1.1	Сор	yright	5
	1.2	Stat	ement of intended use	6
	1.3	Rest	trictions on use	6
	1.4	Mai	ntenance and adjustment	6
	1.5	War	ranty	6
	1.6	Ackı	nowledgements	6
	1.7	Tecl	nnical support	7
2	Н	ealth ar	nd safety	8
	2.1	Disc	laimer	8
	2.2	Disp	osal and recycling instructions	8
	2.	.2.1	WEEE	8
	2.	.2.2	RoHS compliance	9
	2.3	Mai	ntenance	9
	2.4	Gen	eral hazards	9
	2.	.4.1	Warning notices	9
	2.	.4.2	Caution notices	9
	2.5	Spe	cific hazards	9
	2.	.5.1	Hazardous voltages	9
	2.	.5.2	Low temperatures	10
	2.	.5.3	Pressure relief	10
	2.	.5.4	Weight and lifting	11
	2.	.5.5	Asphyxiation	11
	2.	.5.6	Fire	11
	2.	.5.7	Trip hazards	12
	2.	.5.8	Slip hazards	12
	2.	.5.9	Temperature and voltage limits	12
	2.6	Safe	ety equipment	12
	2.7	Risk	assessments	13
3	Sy	ystem d	escription	14
	3.1	The	cryostat	14
	3.	1.1	Microstat He	14
	3.	.1.2	Microstat He-R	15
	3.	.1.3	Optistat CF-V	16

	3.2	Sample holders	17
	3.3	The cryogen transfer siphon	18
	3.4	MercuryiTC temperature controller	18
	3.5	System wiring	18
	3.6	Sensor calibrations	20
	3.7	Gas flow pump and flow controller	20
	3.8	System dewar	20
	3.9	System components and layout	20
	3.10	Weights and dimensions	21
4	Syst	tem installation	22
	4.1	Unpacking the system	22
	4.2	Preparing the system for operation	22
	4.3	Evacuating the outer vacuum chamber	22
	4.4	Preparing the transfer siphon	23
	4.5	Exhaust gas connections	23
	4.6	Connecting the temperature controller	24
5	Syst	tem operation	25
	5.1	Preparations	25
	5.2	Cooling the system	25
	5.3	Cooling below 4.2 K	26
	5.3.	.1 Transitioning from 4.2 K operation	27
	5.4	Operation above 4.2K	27
	5.5	Controlling at a set temperature	29
	5.6	Operation above room temperature	30
	5.7	Warming up the system	30
	5.8	Removing the OVC and radiation shield	31
	5.8.2	.1 Microstat He and He-R	31
	5.8.2	.2 Optistat CF-V	32
	5.9	Changing windows	32
	5.9.	.1 Microstat He	33
	5.9.2	.2 Microstat He-R	33
	5.9.3	.3 Optistat CF-V	33
	5.10	Changing samples	35
	5.11	Adjusting the sample position	35
	5.12	Operating with liquid nitrogen	36

6	Se	ervice and maintenance	37
	6.1	O-rings	37
	6.2	Troubleshooting	37
7	M	1icrostat He specifications	39
	7.1	Performance	39
	7.2	Electrical power	39
	7.3	Physical	39
	7.4	Technical exclusions and assumptions	39
8	M	1icrostat HeR specifications	40
	8.1	Performance	40
	8.2	Electrical power	40
	8.3	Physical	40
	8.4	Technical exclusions and assumptions	40
9	0	ptistat CF-V specifications	41
	9.1	Performance	41
	9.2	Electrical power	41
	9.3	Physical	41
	9.4	Technical exclusions and assumptions	41
1	0	Appendices	42
	10.1	Checking the wiring	42
	10.2	Cleaning and general care	42

1 Introduction

This manual is designed to introduce you to the Microstat He, Microstat He-R and Optistat CF-V cryostats which are part of a range of continuous flow cryostats manufactured by Oxford Instruments. This manual contains important information for the safe operation of your system. You must read this manual carefully before operating the system for the first time.

In addition to this manual for the system cryostat, further manuals and documentation will have been supplied with the system. These additional 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
MercuryiTC manual	Electronic copy on USB
LLT Siphon manual	Electronic copy on USB
VCU manual	Electronic copy on USB
Practical cryogenics	Electronic copy on USB
Safety matters	Electronic copy on USB
Cryostat QuickStart guide	Electronic copy on USB
LLT QuickStart guide	Electronic copy on USB
Cryostat test results	Electronic copy on USB
MercuryiTC safety sheet	Hard copy

Table 1-1: Documentation supplied with the system.

Please keep all the manuals supplied with your system and make sure that you regularly 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

Copyright © Oxford Instruments Nanotechnology Tools Ltd, Tubney Woods, Abingdon, OX13 5QX, UK. All Rights Reserved.

Copyright in this document is owned by Oxford Instruments Nanotechnology Tools Ltd. Any person is hereby authorised to view, copy, print and distribute this document subject to the following conditions: the document may be used for informational purposes only; the document may be used for non-commercial purposes only; any copy of this document or portion thereof must include this copyright notice.

This document is provided "as is" without any warranty of any kind, either express or implied, statutory or otherwise; without limiting the foregoing, the warranties of satisfactory quality, fitness for a particular purpose or non-infringement are expressly excluded and under no circumstances will Oxford Instruments be liable for direct or indirect loss or damage of any kind, including loss of profit, revenue, goodwill or anticipated savings. All such warranties are hereby excluded to the fullest extent permitted by law.

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 lifecycle 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.

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 whilst 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: Microstat He / Microstat He-R / Optistat CF-V

Serial number: The Sales Order (SO) number and/or other identifiers of your system. **Contact information:** How we can contact you, email/telephone details, postal address. **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

Americas

OINS, 300 Baker Avenue, Suite 150, Concord, MA 01742, USA

Tel: +1 800 447 4717 (sales and support)
Fax: +1 978 369-8287 (sales and support)
Email: nanoscience@oxinst.com (sales)

Email: ServiceNSAmericas@oxinst.com (service and support)

Web: www.oxford-instruments.com

Asia

OINS, Floor 1, Building 60, No.461, Hongcao Road, Shanghai, 200233, China

Tel: +86 (0) 400 621 5191 (sales)
Tel: +86 (0) 400 622 5191 (support)
Email: nanoscience@oxinst.com (sales)

Email: ServiceNSAsia@oxinst.com (service and support)

Web: www.oxford-instruments.cn

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

All the materials and components used in the manufacture of the system 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 DN 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

Any 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 Vacuum	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 and transfer siphon have in-built pressure relief plates, as shown in Figure 2-1. This allows the system's vacuum chamber 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-V.

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 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 asphyxiation. 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 run-off. Additionally, warning signs should be placed around the system.

2.5.9 Temperature and voltage limits

The system cryostat 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 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 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.

3 System description

This manual is intended for use with the Microstat He, Microstat He-R and Optistat CF-V cryostats. These systems are described together in a single manual as the standard operating procedures for each cryostat are common as they use the same cold unit housed in a different OVC.

Each cryostat is a continuous flow cryostat designed principally to allow a sample to be cooled to a low temperature and studied with an optical microscope. Continuous flow cryostats do not have an internal reservoir to store a supply of cryogen, instead, the cryogens are supplied to the cryostat from a separate storage vessel using an insulated transfer siphon. The transfer siphon delivers the cryogen to the cryostat's heat exchanger which is thermally linked to the sample holder. The returning gas from the cryostat's heat exchanger then cools the radiation shield before flowing out of the cryostat. The cryostat's heat exchanger is fitted with a sensor and heater which are used to control the sample temperature.

The cryostats may be operated in any orientation, however, if inverted, the cryostat performance is likely to be reduced. Once fixed within the cryostat, a sample is mounted in vacuum and cooled by conduction where its temperature can be continuously controlled between the cryostat base temperature and 500 K.

3.1 The cryostat

Either liquid helium or liquid nitrogen can be used as a cryogen within the system cryostat. 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 by using the standard gas flow pump (GF4) and controller (VC-U). Lower temperatures can be achieved by using a larger pump, such as an EPS40 rotary pump.

3.1.1 Microstat He

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

- The outer vacuum chamber (OVC) and demountable radiation shield that isolates the sample from the room temperature surroundings.
- Two window flanges with bonded windows, shaped to allow a typical optical microscope objective lens turret to be rotated without withdrawing the microscope objective.
- The cryostat heat exchanger, with heater and sensor, onto which the system's sample holder is mounted.
- OVC pressure relief valve with DN16NW pump and flush port.
- Two 10-pin connectors; one for diagnostic wiring and one for experimental wiring.

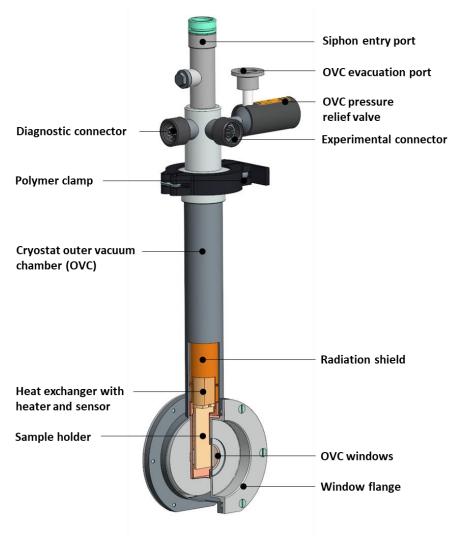


Figure 3-1: Microstat He Schematic.

3.1.2 Microstat He-R

A schematic of the Microstat He-R is shown in Figure 3-2. The main features of the cryostat are:

- The outer vacuum chamber (OVC) and demountable radiation shield that isolates the sample from the room temperature surroundings.
- Four permanently bonded windows into the rectangular OVC, shaped to provide a short working distance through all four windows.
- The cryostat heat exchanger, with heater and sensor, onto which the system's sample holder is mounted.
- OVC pressure relief valve with DN16NW pump and flush port.
- Two 10-pin connectors; one for diagnostic wiring and one for experimental wiring.

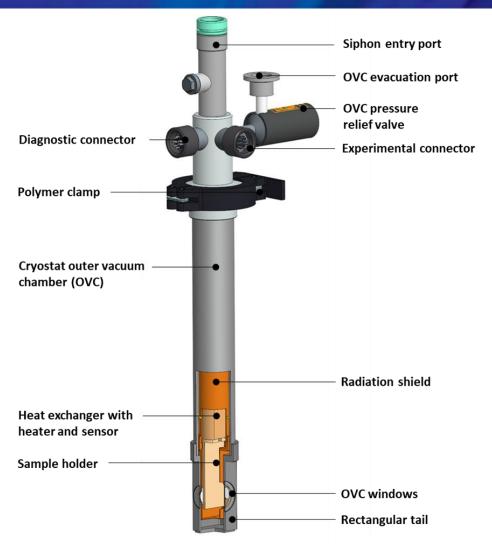


Figure 3-2: Microstat He-R Schematic.

3.1.3 Optistat CF-V

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

- The outer vacuum chamber (OVC) and demountable radiation shield with windows that isolates the sample from the room temperature surroundings.
- Four radial window ports and one axial port with optical access of f/0.9.
- The cryostat heat exchanger, with heater and sensor, onto which the system's sample holder is mounted.
- OVC pressure relief valve with DN16NW pump and flush port.
- Two 10-pin connectors; one for diagnostic wiring and one for experimental wiring.

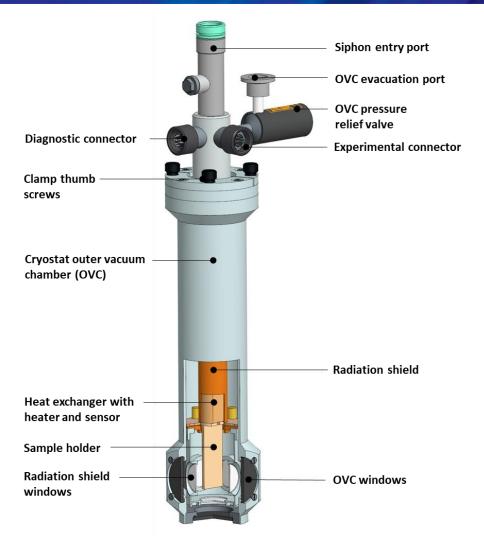


Figure 3-3: Optistat CF-V Schematic.

3.2 Sample holders

There are two sample holder options supplied with each system. The first is a plain reflectance sample holder, as shown in Figure 3-4 (Left). Several small samples may be mounted on this sample holder at once. The second is a transmission sample holder which has a 12.5 mm diameter aperture and includes a sample retaining clamp, as shown in Figure 3-4 (Right).

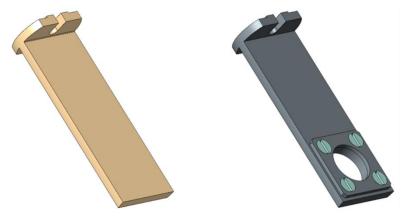


Figure 3-4: Reflectance sample holder (Left) and transmission sample holder (Right).

On all the systems, a sample can be mounted onto a sample holder within the cryostat's vacuum space. Optical access to the sample is then available through the windows of each cryostat.

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 and 700 are the standard transfer siphon options and are manually controlled. The LLT 650 and 750 are automated versions of the 600 and 700, respectively, which allow the gas flow rate to be automatically optimised using the MercuryiTC. 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 in Table 3-1.

Slot	Card Type	Function	Connection
Main Board	Sensor & Heater	Temperature Control	Diagnostic 10 Pin
1-8	Not used	n/a	n/a

Table 3-1: MercuryiTC configuration for the Microstat He, He-R & Optistat CF-V.

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

3.5 System wiring

The cryostats are all fitted with two 10-pin connectors on the cold unit by the siphon entry port. The first of these 10-pin connectors, used for the temperature control, is opposite the cryostat pressure relief valve. The second 10-pin connector, used for experimental wiring, is 90° from the cryostat OVC pressure relief valve. One is used for system control and the other is a spare. The pin configuration for both 10-pin connectors is shown in Figure 3-5. The seal is held in place by a black nut, this nut should not be removed, unless access to the wiring is required.

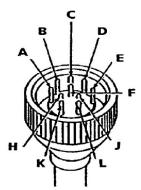


Figure 3-5: Pin configuration for the 10-pin connector.

The wiring configuration for the control and diagnostic connector is set out in Table 3-2, as per the pin configuration shown in Figure 3-5.

Pin	Function	Polarity	Туре
А	Hoat Eychangor Hoator	H+	Firerod heater
В	Heat Exchanger Heater	H-	(40 Ω nominal)
С		V+	Rhodium-Iron
D	Heat Exchanger Sensor	V-	resistance
E		I+	thermometer
F		I-	(27 Ω nominal)
Н			
J K	Not Used	N/A	N/A
	Not osed		IN/A
L			

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

The experimental 10-pin connector is wired to a Harwin pin ring that sits above the heat exchanger in the vacuum space. A schematic diagram of the Harwin pin ring configuration is shown in Figure 3-6 and the wiring configuration for the experimental wiring connector to the Harwin pin ring is set out in Table 3-3.

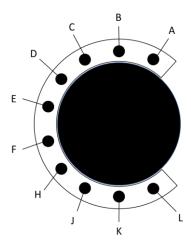


Figure 3-6: Harwin pin ring configuration as viewed from the bottom of the system.

Connector Pin	Harwin Ring Pin	Wire
Α	Α	34 SWG Cu
В	В	34 SWG Cu
С	С	40 SWG Cu
D	D	40 SWG Cu
E	E	40 SWG Cu
F	F	40 SWG Cu
Н	Н	40 SWG Cu
J	J	40 SWG Cu
K	К	40 SWG Cu
L	L	40 SWG Cu

Table 3-3: Pin configuration for the experimental 10-pin connector.

3.6 Sensor calibrations

The calibrations for the calibrated sensor on the system, a rhodium-iron resistance thermometer, will have been loaded into the MercuryiTC in 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 cm³min⁻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 System components and layout

A schematic showing the connections between the components of the system is shown in Figure 3-7.

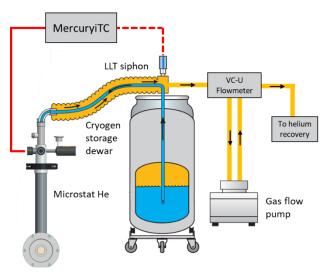


Figure 3-7: System components and example layout, shown with the Microstat He.

3.10 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
Microstat He cryostat	455	160	75	1.8
Microstat He-R cryostat	435	150	75	1.5
Optistat CF-V cryostat	445	150	85	2
Mercury iTC	310	485	90	8
LLT600 (Siphon)	1950	1570	55	5
LLT700 (Siphon)	1560	1570	55	5
GF4 Pump	245	130	180	9
VCU Flowmeter	360	260	150	4

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

4 System installation

The setup of an Microstat He, He-R or Optistat CF-V 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.

To run this system, the following components are required:

- The cryostat: Microstat He, Microstat He-R or Optistat CF-V.
- 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 diameter).
- Sensor & heater cable CQB0090.
- Automatic needle valve cable CWA0112 (option).
- Liquid helium or liquid nitrogen storage dewar (customer provided).
- High vacuum pumping system to evacuate the OVC (customer provided).
- Rotary pump for enhanced low temperature operation (optional, customer provided).

4.2 Preparing the system for operation

Choose a suitable position to operate the cryostat safely, and if necessary, arrange for it to be supported so that it cannot accidentally fall. An example operating configuration for a system has been shown previously in Figure 3-7.



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, just that the new materials are being cleaned by the vacuum. The OVC should be pumped thoroughly before each cooldown, especially when the cryostat is new.

Each cryostat OVC is fitted with an evacuation port and safety relief valve, as previously shown in Figure 3-1, Figure 3-2 & Figure 3-3. 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 (lower than 10^{-4} mbar). 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. A high vacuum pumping system can be connected directly to this evacuation port. The transfer siphon must also be evacuated to a high vacuum before the system can be run. Please refer to the LLT Siphon manual.

4.5 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 4-1. 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.



Figure 4-1: Rear view of the VC-U gas flow controller showing the connectors.

For low temperature operation, < 3.2 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.

4.6 Connecting the 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 4-2.



Figure 4-2: 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/LLT750). After the cable connections have been made, turn on the MercuryiTC. The temperature should read approximately 295 K (room temperature).

Cable	From (Mercury)	То	Function
CQB0090	MB1 (Sensor Heater)	OVC body (Diagnostic 10-pin)	Heat Exchanger, Sensor & Heater
CWA0112	Auxiliary Socket	LLT650 / LLT750 (7 Pin)	Automatic Needle Valve

Table 4-1: System wired connections.

5 System operation

This section describes the operation of a cryostat 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.

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 and transfer siphon must be pumped thoroughly to a high vacuum before each cooldown.

The following procedures assume that liquid helium is being used with the system. The system can also be used with liquid nitrogen, but some of the techniques are different. Please see Section 5.12 for more details about the operation of the system 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 Cooling the system

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.

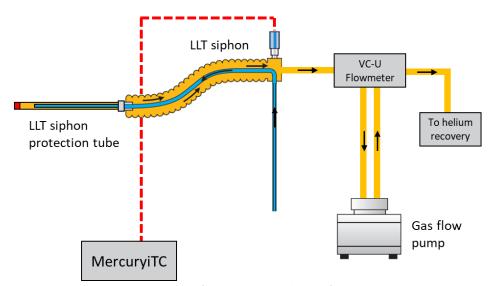


Figure 5-1: Transfer siphon pre-cooling configuration.

To start, fully close, then fully open the needle valve on the transfer siphon. If a manual transfer siphon (LLT600/LLT700) 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/LLT750) 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 entry arm of the siphon into the siphon entry port of the cryostat and engage the nut on the siphon with the thread on the cryostat arm. Take care not to overtighten the nut. Set up the desired operating configuration with either the GF4 or a rotary pump.

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.

5.3 Cooling 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'.

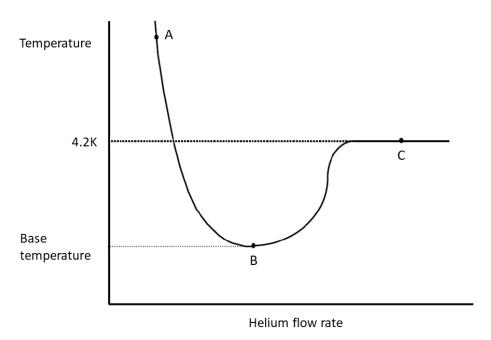


Figure 5-2: Temperature and helium flow rate relationship.

5.3.1 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.

5.4 Operation above 4.2K

The temperature of the system's heat exchanger can be controlled at any temperature between 4.2 K and 500 K using the MercuryiTC provided with the system. The MercuryiTC automatically controls the power supplied to the system's internal heater to maintain the set temperature.

The Mercury iTC 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 4-2, 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, 'Microstat He Mercury.pid' has already been loaded into the MercuryiTC.

PID Table		М	icrostat	He Mercu	ry.pid
Temperature(K)	To(K)	Р	I (min)	D (min)	
1.0000	15.0000	50.000	1.000	0.000	
15.0000	30.0000	40.000	1.000	0.000	
30.0000	60.0000	30.000	1.000	0.000	
60.0000	115.000	20.000	1.000	0.000	
115.000	225.000	15.000	1.000	0.000	
- Load		Save	2	Clos	se +

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 Controlling at a set temperature

Select the channel on the temperature controller corresponding to the sensor which will be used to control the system. Select 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 output of the temperature controller is not too high. If you are using an automatic LLT, the flow and heater voltage will be automatically optimised. However, if a manual 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. As a guide, some typical heater voltages for different temperature ranges are shown in Table 5-1.

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.

5.6 Operation above room temperature

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.



OPTISTAT CF-V WINDOWS

To operate the Optistat CF-V system above 300 K you must fit high temperature windows to the system's radiation shield. Instructions on how to change the Optistat CF-V radiation shield windows can be found in Section 5.9.3.

The siphon must be removed before operating the system above room temperature. Pump the OVC continuously to maintain the required high vacuum. The system can now be controlled at a 'set temperature' exceeding 300 K, as described in Section 5.5.



MAXIMUM SET TEMPERATURE

Do not set a temperature higher than 500 K.

5.7 Warming up the system

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 vented 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.

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 the OVC and radiation shield

The process for the removal of the OVC and radiation shields differs for the Microstat and Optistat systems. However, 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 with a dry nitrogen source through the evacuation valve and ensure that the OVC it is at atmospheric pressure.

5.8.1 Microstat He and He-R

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

- Change the sample.
- Adjust the sample position.
- Modify or repair the wiring.
- · Repair mechanical damage.

If the OVC is fixed to a microscope stage, it is not necessary to remove the OVC from the microscope stage to complete this process. To remove the cryostat from the OVC, release the DN40NW clamp from around the neck of the cryostat and carefully withdraw the cryostat from the OVC.



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.

The removal of the OVC gives access to the system's radiation shield. To remove the radiation shield, loosen the four screws that secure it to the cryostat and carefully pull the radiation shield off the cryostat. Note that the holes in the radiation shield are slotted so that the retaining screws do not need to be removed.

The removal of the radiation shield provides access to the system sample holder, heat exchanger and wiring. At this point, it is possible to modify or repair the system's wiring, change the sample and adjust the sample position, as described in Section 5.11.

Before reassembling, check the OVC O-ring is clean, undamaged, and lightly greased. Ensure that it is pushed firmly into place, to form a vacuum-tight seal.

5.8.2 Optistat CF-V

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

- Change the sample.
- Adjust the sample position.
- Modify or repair the wiring.
- Repair mechanical damage.
- Change the radiation shield windows.

To remove the OVC from the cryostat, remove the four thumb screws at the top neck of the cryostat, this allows the cryostat to be carefully withdrawn from the OVC.



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.

The removal of the OVC provides access to the system's radiation shield. The whole Optistat CF-V radiation shield or just the window assembly of the radiation shield can be removed, depending on the level of access required. The removal of the whole radiation shield will provide access to the system's sample holder, heat exchanger and wiring. The removal of the window assembly of the radiation shield will give access to the system's sample holder and radiation shield windows.

To remove the entire radiation shield, loosen the four screws that secure it to the cryostat and carefully pull the radiation shield off the cryostat. Note that the holes in the radiation shield are slotted so that the retaining screws do not need to be removed. At this point, it is possible to change the sample, adjust the sample position (as described in Section 5.11), modify or repair the system's wiring and repair any mechanical damage.

To remove just the window assembly of the radiation shield, undo the four brass thumb screws that attach the radiation shield window assembly to the upper section of the radiation shield. After removing the final thumb screw, the radiation shield window assembly will drop away from the upper section of the radiation shield. At this point, it is possible to change the sample and adjust the sample position (as described in Section 5.11) or change the radiation shield windows (as described in Section 5.9.3.2).

Before reassembling the OVC, ensure that the thumb screws that attach the window assembly of the radiation shield to the upper section are tightly fastened. Additionally, check that the cold unit OVC O-ring that seals with the OVC is clean, undamaged, and lightly greased.

5.9 Changing windows

The Microstat He, Microstat He-R and Optistat CF-V are supplied with Spectrosil B quartz windows as standard. The processes in this section describe how to change the windows of each system in turn.

Before changing the windows, it is essential that the cryostat has been warmed to room temperature, as described in Section 5.7. After the system has been warmed up, the OVC pumping port needs to be opened to allow air into the system. If the internal parts of the cryostat are still be cold, use dry nitrogen gas instead and ensure the system is warm throughout before attempting to change windows.

After fitting any new OVC windows, they should be carefully tested for leaks, Oxford instruments recommends using a mass spectrometer leak detector to do this.

5.9.1 Microstat He

The windows for the Microstat He are bonded directly into window mounts that can be detached from the system's OVC. To change the window, the whole window mount must be replaced. To remove the current window mount first remove the six countersunk M4 screws from around the circumference of the window mount. Next, remove the window mount from the OVC. Remove the O-ring, check it for any damage, clean it and then lightly grease it with vacuum grease before replacing it back into the groove on the OVC. Take the replacement window mount and clean its window with a suitable lens cleaner and lens tissue. Place the window mount onto the OVC with the flat edge in the correct orientation before securing it by replacing the six countersunk M4 screws.

5.9.2 Microstat He-R

It is not possible to replace the windows of the Microstat He-R as they are bonded directly into the system's OVC tail. If different OVC windows are required, please contact Oxford Instruments.

5.9.3 Optistat CF-V

Unlike the Microstat systems, the Optistat CF-V has windows on both the OVC and radiation shield. All the windows of the Optistat CF-V can be removed so that they can be cleaned or replaced. As the OVC and radiation shield windows are secured to the system in different manners, the removal of each follows a different procedure.

5.9.3.1 OVC windows

To change an OVC window, the Optistat CF-V must first be warmed to room temperature and vented with dry nitrogen gas, as previously described in Section 5.7. Each Optistat CF-V OVC window is held in place by four nylon retaining screws, as shown in Figure 5-7.

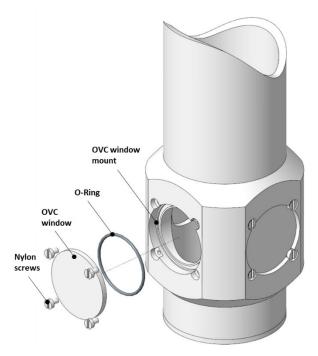


Figure 5-7: Changing the Optistat CF-V OVC windows.

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.

5.9.3.2 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 around the 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.

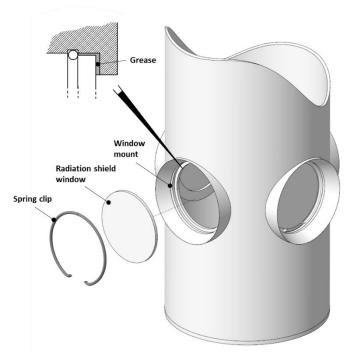


Figure 5-8: Changing Optistat CF-V radiation shield windows.

5.10 Changing samples

To change the sample in all the system cryostats, the cryostat must first be warmed to room temperature, as described in Section 5.7. The sample in the system is mounted on the heat exchanger within both the OVC and radiation shield.

To change samples, both the OVC and radiation shield need to be removed. The process for removing the OVC and radiation shield differ between the Microstat and Optistat systems and are described separately in Sections 5.8.1 & 5.8.2, respectively. When the sample has been replaced, re-assemble the cryostat and evacuate the OVC. Proceed to cool the system down as described in Section 5.2.

5.11 Adjusting the sample position

The sample holder can be adjusted to position the sample as close as possible to the inside of the OVC window. This adjustment must be made before the radiation shield and OVC are fitted.

To adjust the position of the sample holder the screw that secures the sample holder to the heat exchanger should be loosened. The sample holder is located into a groove in the heat exchanger. The groove allows for the sample holder to slide up and down and prevents rotation during adjustment. Additionally, the heat exchanger has two tapped holes, this allows for the sample holder to be turned 180° to face the opposite window if desired.

After the sample holder has been moved to the desired position tighten the securing screw back up and reassemble the system. When reassembling the system ensure that neither the sample nor the sample holder touch the radiation shield, OVC, or window.

5.12 Operating with liquid nitrogen

The cryostat can also be operated with liquid nitrogen instead of 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 VCU.
- The viscosity of liquid nitrogen is greater than that of Helium, so the flow rate through the cryostat is lower. This increases the cooldown 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 90K 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 the base temperature is approached to prevent the cryostat from 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 Microstat He, He-R & Optistat CF-V will deliver repeatable and reliable performance if maintained properly. This section contains basic and essential maintenance information.

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 Troubleshooting

Should you encounter a problem with your system, it is important to establish the source first. 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's Troubleshooting chapter. Diagnosis of the transfer siphon and VC-U should be made using their respective manuals. 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 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 gas ballast valve open.
High base	Heater still on	Check the heater is switched off.
temperature	Poor vacuum	Check both the quality of the vacuum in the cryostat and transfer siphon.
	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.
	Wiring heat load	Check that too much heavy wiring has not been added to the sample holder, introducing a high heat load.

Issue	Possible cause	Recommendation
High base temperature	Transfer siphon seal	Check the transfer siphon nut is tight enough. Check the PTFE seal has not been damaged.
(continued)	Low gas flow	Check whether there is any flow of gas through
		the system using the gauge on the VC-U. Check
		there is sufficient liquid in the dewar.
	Mechanical damage	Warm the system to room temperature and
		check for touches between the OVC, radiation shield and sample holder.
Condensation or frost on OVC	Poor cryostat vacuum	Check poor cryostat vacuum actions.
Condensation on windows	Air humidity	Use a dry nitrogen source to shield the windows.
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 wiring fault	Check that the heater circuit is not open or shorted by checking the resistance between pins
		A and B on the diagnostic connector, checking
		for isolation between these pins and ground.
	Low heater voltage	Check that the heater voltage limit on the
	limit	temperature controller is high enough. Normal
		settings are limited to: 40 V, Resistance: 20 Ω .

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

7 Microstat He specifications

7.1 Performance

Performance	Specification
Temperature control range	3.2 K† - 500 K
Temperature control stability	± 0.1 K
Liquid helium consumption at 4.2 K	< 0.45 L/hr
Liquid helium cooldown consumption	1.3 L (nominal)
System cooldown time	10 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)	1.8
Dimensions W x D x H (mm)	455 x 160 x 75

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.

- † Base temperature may be extended down to 2.2 K by using a rotary pump, such as the EPS40.
- ‡ When using a pre-cooled transfer siphon.

8 Microstat HeR specifications

8.1 Performance

Performance	Specification
Temperature control range	3.2 K† - 500 K
Temperature control stability	± 0.1 K
Liquid helium consumption at 4.2 K	< 0.45 L/hr
Liquid helium cooldown consumption	1.3 L (nominal)
System cooldown time	10 minutes‡

8.2 Electrical power

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

8.3 Physical

Parameter	Nominal Value	
Weight (kg)	1.5	
Dimensions W x D x H (mm)	435 x 150 x 75	

8.4 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.

- † Base temperature may be extended down to 2.2 K by using a rotary pump, such as the EPS40.
- ‡ When using a pre-cooled transfer siphon.

9 Optistat CF-V specifications

9.1 Performance

Performance	Specification
Temperature control range	3.2 K† - 500 K
Temperature control stability	± 0.1 K
Liquid helium consumption at 4.2 K	< 0.45 L/hr
Liquid helium cooldown consumption	1.3 L (nominal)
System cooldown time	10 minutes‡

9.2 Electrical power

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

9.3 Physical

Parameter	Nominal Value	
Weight (kg)	2	
Dimensions W x D x H (mm)	445 x 150 x 85	

9.4 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.

- † Base temperature may be extended down to 2.2 K by using a rotary pump, such as the EPS40.
- ‡ When using a pre-cooled transfer siphon.

10 Appendices

10.1 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	35 – 45 Ω
C - D	30 – 40 Ω
C - E	3 – 6 Ω
C - F	30 – 40 Ω
D - F	3 – 6 Ω
A - C	> 1 MΩ
A - ground	> 1 MΩ
C - ground	> 1 MΩ

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

10.2 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 as this may lead to an O-ring failure and may result in vacuum loss which could damage the system.