User Manual - Original Instructions

MicrostatHires

EC Declaration of Conformity

This original Declaration of Conformity is suitable to Decision No 768/2008/EC of the European Parliament and of the Council of 9 July 2008 on a common framework for the marketing of products and contains the elements specified in the relevant modules set out in Annex II of that Decision for the applicable Directives.

This declaration relates exclusively to the equipment in the state in which it was placed on the market, and excludes components which are added and/or operations carried out subsequently by the final user.

Applied Council Directive(s):

The fulfillment of all the relevant provisions specified in the following Council Directive(s) have been demonstrated :

- 2014/30/EU Electromagnetic Compatibility (EMC)
- 2014/35/EU LOW VOLTAGE DIRECTIVE (LVD)
- 2011/65/EU RoHS Directive and Commission Delegated Directive (EU) 2015/863 (RoHS2)

We, The Manufacturer :

Oxford Instruments NanoScience, Tubney Woods, Abingdon, Oxon, OX13 5QX, United Kingdom, declare under our sole responsibility that the following equipment :

MicrostatHiRes

to which this declaration relates is in conformity with the relevant provisions of the following standard(s) or other normative document(s) when installed in conformance with the installation instructions contained in the product documentation :

- EMC
  - EN61326-1:2013 (Immunity)
  - EN61000-3-2:2006 + A2:2009
  - EN61000-3-3:2008
  - EN55011:2009 + A1:2010 (Emissions)
- LVD
  - IEC61010-1:2010 3rd Edition; This is harmonised with: ANSI/UL 61010-1:2012, CSA C22.2 No. 61010-1-12.
- RoHS2
  - EN 50581:2012 Technical documentation for the assessment of electrical and electronic products with respect to the restriction of hazardous substances.

The authorised compiler of the technical file is at Oxford Instruments NanoScience, Tubney Woods, Abingdon, Oxon, OX13 5QX, United Kingdom.

We, the undersigned, hereby declare that the product(s) specified above conforms to the listed directive(s) and standard(s).

M.N. Cuthbert.

Name : Dr Michael N Cuthbert
Position : Technical Director
Date of issue : 21st December 2017

IMPORTANT INFORMATION

Product description

The Microstat HiRes is a continuous flow liquid helium cryostat designed principally to allow a sample to be cooled to a low temperature and studied with an optical microscope. The sample mounted in vacuum and cooled by conduction, held at temperatures between 3.4K and 300K using the MercuryiTC temperature controller. Refer to the user manual for more details. The user manual can be downloaded from the website support.myoxinst.com.

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, use 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 NanoScience, as well as incorrect use or operation of the equipment, may relieve Oxford Instruments NanoScience 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.

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. Ensure that pressure relief devices are not obstructed.

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.

Health and safety information

The equipment operates at hazardous temperatures. Before working with the equipment, all personnel must read and become thoroughly familiar with the safety information given in the user manual. In particular, users must read, understand and strictly observe all : Warning notices; Caution notices; Safety labels and markings on the equipment. For ease of reference and rapid response in an emergency, it is advised that a copy of the user manual should be safely kept near the equipment when in operation.

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

LOW TEMPERATURE EQUIPMENT: Danger of death or serious injury. Contact with cold objects and cryogens can cause serious injury. Use personal protective equipment including hand and eye protection. Cryogens can displace the oxygen from air and cause death by asphyxiation. Ensure that adequate ventilation is provided. **ÉQUIPEMENT A BASSE TEMPÉRATURE**: Danger de mort ou de blessure grave. Le contact avec des objets froids et des cryogènes peut causer des blessures graves. Utilisez un équipement de protection individuelle, y compris la protection des mains et des yeux. Les cryogènes peuvent déplacer l'oxygène de l'air et provoquer la mort par asphyxie. Assurez-vous qu'une ventilation adéquate est fournie.

PRESSURE RELIEF: The equipment incorporates pressure relief

valves that exhaust directly to atmosphere. Do not modify or obstruct these devices.
RELIEF DE PRESSION: Cet équipement comprend des soupapes de secours qui s'écoulent directement dans l'atmosphère. Ne modifiez ni ne obstruez pas ces périphériques.

ELECTROSTATIC SENSITIVE EQUIPMENT: This equipment contains electrostatic sensitive devices (ESD). Use approved ESD procedures when installing or maintaining this product.
ÉQUIPEMENT SENSIBLE AUX DÉCHARGES ÉLECTROSTATIQUE : Cet équipement contient des dispositifs qui sont sensibles aux décharges électrostatiques (ESD). Utiliser des procédures ESD homologuées lors de l'installation ou de la maintenance de ce produit.

Solid waste: Dispose of this item according to local and national regulations.
Conformity: IEC61010-1: 2010 3rd Edition: Safety requirements for electrical equipment for measurement, control and laboratory use. EN61326-1:2013: EMC Immunity standard, EN55011:2009+A1:2010 Emissions standard Electrical equipment for measurement, control and laboratory use: EMC requirements. Please refer to the user manual for more details.

Contact us at : Oxford Instruments NanoScience. See www.oxford-instruments.com for details.

Microstat HiRes Manual

Oxford Instruments Nanoscience

Sep 2017

Contents

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MicrostatHiRes - Principles of Operation

Continuous flow Helium cryostat.

1.1 Revision history

Always use the latest issue of the manual. Check for updates online at https://support.myoxinst.com.

1.2 Contents

1.3 Copyright

- **Next** - Introduction

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Introduction

This manual contains user and technical information for the MicrostatHiRes system.

2.1 Documents supplied with the system

The following documents are supplied as electronic or paper copies:

- Operator's manual (this document)
- Factory test results
- Mercury iTC manual
- Practical Cryogenics
- Safety Matters
- Mercury iTC temperature controller
- VC-U Gas controller manual

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.

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

If you have bought a complete system from Oxford Instruments, separate manuals will have been supplied describing the other components. Please ensure you have reviewed the information supplied in all of the manuals before you attempt to operate your system.

2.2 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, use 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.

2.3 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.

2.4 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.

2.5 Support

If you have any questions, please contact us with the following details :

- **System type** :
- **Serial number** : the Sales Order (SO) number and/or other identifiers of your system.
- **Installation/Shipment Address** :
- **Contact information** : how we can contact you. email/telephone.
- **Details of your query** : The nature of your problem, part numbers of spares required, etc.

Please contact Oxford Instruments first before attempting to service, repair or return components.

2.6 Contact information

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) Tel: +1 800 447 4717 (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) 400 678 0609 (sales, service and 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

2.7 Acronyms

A number of acronyms may be used throughout this document. Please refer to the document Practical Cryogenics for a glossary of terms.

- **Next** - Safety Information

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Safety Information

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 the OINS' document Safety Matters. 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.

3.1 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.

3.2 Disposal and recycling instructions

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

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

3.3 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.

3.3.1 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: The warning triangle highlights dangers which may cause injury or, in extreme circumstances, death.

3.3.2 Caution notices

Caution notices draw attention to events or procedures that could cause damage to the equipment. Failure to obey a caution notice may result in damage to the equipment. A typical caution notice is shown below:


Caution: The general caution symbol highlights actions that you must take to prevent damage to the equipment. The action is explained in the text.

3.4 Specific hazards

Your system manual will indicate which of the following specific hazards relate to your system. Refer to Safety Matters for more information in each case.

3.5 Electrical hazards


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.

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.

3.6 Protective earth

The cryostat, electronics, pumps, and any other parts of the system fitted with earthing points must be connected to protective earth at all times when the system is in operation.

3.7 Low temperature

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 must be made available to all personnel expected to handle cryogenic liquids, including hand and eye protection.

3.8 Pressure relief

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.

3.9 Weight and lifting

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 at the installation site for the duration of the system installation.

3.10 Magnetic fields

Powerful magnetic fields can interfere with the operation of cardiac pacemakers. Do not approach the equipment if you wear a cardiac pacemaker.

Systems with the magnet option contain powerful electromagnets. These electromagnets can produce a powerful magnetic field, which can interfere with the operation of cardiac pacemakers in the vicinity of the system, causing death or serious injury.

Risk to personnel with cardiac pacemakers.

Where necessary, the appropriate warning signs should be in place around the installation site. Personnel who have a cardiac pacemaker must not approach the system at any time, even when it is powered off. The magnet can exert a force on nearby ferromagnetic objects, which presents a hazard to personnel if these objects are free to move.

3.11 Asphyxiation

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

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

3.12 Fire

Atmospheric oxygen can condense on cryogenicaly-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.

3.13 Safety equipment

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

- Personal protective equipment, including thermally insulated gloves, face protection and protective footwear.
- 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 you system includes a superconducting magnet.

3.14 Maintenance

Observe the necessary maintenance schedule for the system. Consult Oxford Instruments if you are unsure about the required procedures.

- **Next** - System Description

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System Description

4.1 The cryostat

The MicrostatHiRes is a continuous flow liquid helium cryostat designed principally to allow a sample to be cooled to a low temperature and studied with an optical microscope. The pillared option allows the sample to be placed inside the bore of a magnet. The window arrangement allows the sample to be brought close to the objective lens of the microscope with the sample mounted in vacuum and cooled by conduction. The sample temperature is continuously variable between 3.4 and 300K (up to 500K with high-temperature window options).

Microstat HiRes schematic

The sample space and radiation shields are thermally insulated from the room temperature surroundings by the outer vacuum chamber (OVC). This space is pumped to a high vacuum before the cryostat is cooled down but it is protected against accidental build-up of high pressures by a pressure relief valve. The cryostat may be operated in any orientation.

Rotate the top window flange to bring it as close as required to the top surface of the sample. When the cryostat is evacuated this flange can still be rotated but some force will be required.

Up to two windows can be fitted to the MicrostatHiRes. Each window is permanently bonded into the OVC flange.

This manual describes the operation of the system in conjunction with an Oxford Instruments Mercury iTC temperature controller.

4.2 Dynamic continuous flow cryostats

Continuous flow cryostats do not have an internal reservoir to store a supply of cryogens. The liquid is supplied from a separate storage vessel through an insulated transfer tube. The transfer tube delivers the liquid helium to a heat exchanger close to the sample space. The gas returning from the heat exchanger then cools the radiation shield and flows out of the cryostat. A thermometer and heater are mounted on the heat exchanger, and these can be used with a temperature controller to balance the cooling power of the cryogen and to control the temperature of the sample.

Either liquid Helium or liquid Nitrogen can be used in these cryostats. Temperatures down to about 77K can be reached using liquid nitrogen, but 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 continuously using the standard gas flow pump (GF4) and controller (VC-U). Lower temperatures can be achieved using a larger pump such as the EPS40 rotary pump. Figure 1 illustrates the overall system configuration when the cryostat is run using an LLT transfer tube. The system can also be run using a TTL transfer tube, in which case the pump is attached to a port provided for this purpose on the cryostat. Figure 1 shows a transfer tube with an auto needle valve, but a manual needle valve can also be used.

4.3 The cryogen transfer tube

The LLT transfer tube 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 used for manual

Figure 4.1: Microstat HiRes cryostat

control, and the LLT 650 and 750 are automated versions, which allow the gas flow rate to be optimised automatically using the Mercury iTC Temperature Controller.

To operate the needle valve manually, set the gas flow control on the Mercury iTC to Manual. To close the needle valve, set the gas flow to 0%. To open the needle valve, set the gas flow to 100%.

4.4 The 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 42 litres per minute. The air leak rate is guaranteed to be less than 10cm^3/min. 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.

- **Next** - Installation

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Installation

5.1 Unpacking the system

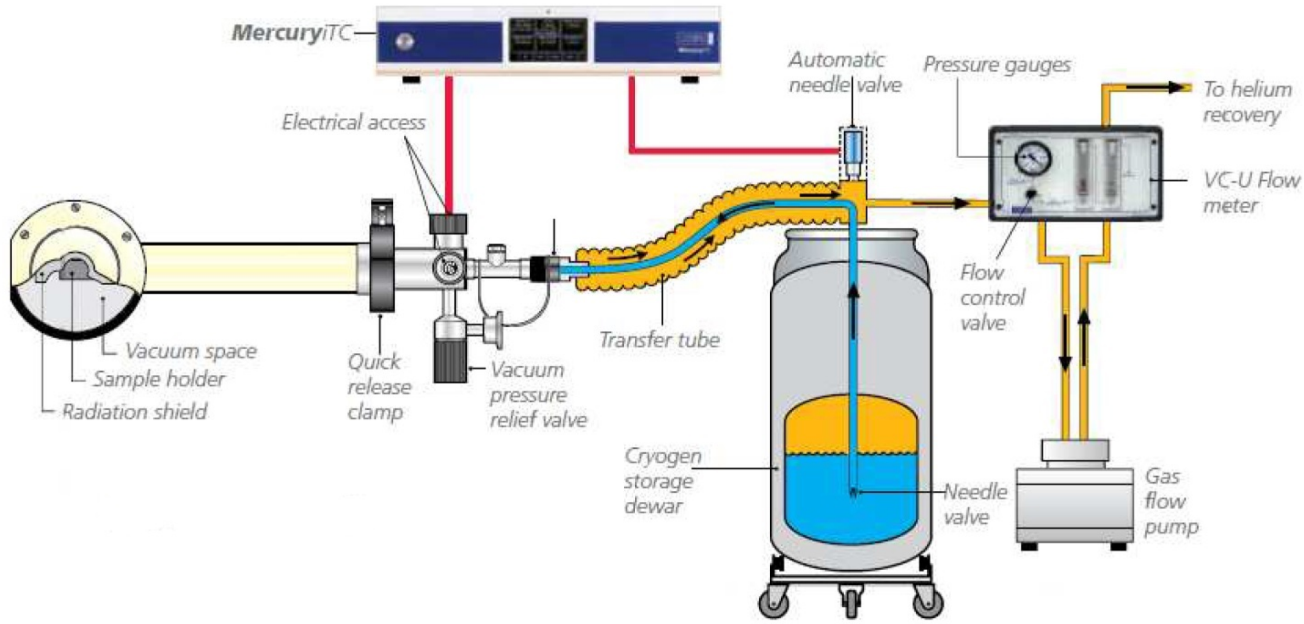
Carefully remove the cryostat and all the accessories from the packing case, and check the packing list to make sure that you have found all of the components. Examine the system to make sure that it has not been damaged since it left the factory. If you find any signs of damage please contact Oxford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

To run this system you need the following components:

- The MicrostatHiRes cryostat
- Liquid Helium or liquid Nitrogen storage dewar
- Cryogen transfer tube (LLT) with suitable storage dewar adapter
- Polythene tube (10mm outer dia.) for the gas exhaust
- High vacuum pumping system to evacuate the OVC occasionally
- Oil free diaphragm pump (GF4) or EPS40 rotary pump for low temperature operation
- Temperature controller (Mercury iTC)
- Sensor/heater cable CQB0090
- Gas flow controller (VC-U) and pumping lines
- Motor controlled needle valve cable CWA0112 (option)

5.2 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. The below image shows the operating configuration of the system.



5.3 Evacuating the outer vacuum chamber (OVC)

The OVC has to be pumped to high vacuum to make sure that it gives the required thermal insulation. When the system is new, all of the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This 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.

Connect the pumping system to the cryostat vacuum valve on the top plate of the cryostat. We recommend that you use a turbo-molecular pump, backed by a rotary pump, and fitted with a cold trap which helps the system to pump water vapour. If the system is badly contaminated with water vapour, the gas ballast facility on the rotary pump should be used.

5.4 Evacuating the transfer siphon

Although sufficient thermal insulation can be achieved by pumping the transfer siphon with a two-stage rotary pump, performance will improve significantly if it can be pumped overnight with a high-vacuum pumping system. The transfer tube vacuum space has a separate evacuation valve and the pumping system can be connected to it directly.

5.5 Exhaust gas connections

During standard operation, a piece of polythene tube is used to connect the exhaust port on the cryostat or transfer tube to the VC-U gas flow controller. The other connections should be made as shown in the figure above. The exhaust line from the VC-U can either be connected to a Helium recovery system or vented to the atmosphere.

For low temperature operation, remove the “christmas tree” adaptor (ribbed metal connector for plastic hose) clamped to the NW16 fitting on the transfer siphon exhaust port. Attach a pumping line directly between this fitting and a rotary pump (for example, the EPS40 pump).

Make sure that an oil-mist filter is attached to the exhaust of the pump. The outlet of the oil-mist filter can either be connected to a Helium recovery system or vented to the atmosphere.

5.6 Electrical connections to the temperature controller

The Mercury iTC has been configured by Oxford Instruments to suit the system ordered. When you first switch on the Mercury iTC you will see the instrument home screen, similar to that shown below:

Figure 5.1: Mercury home screen

The Mercury iTC temperature controller should be connected to the cryostat as follows:

- The sensor/heater cable CQB0090 is connected between the ten pin seal on the cryostat and the “Sensor/Heater” socket on the temperature controller.
- The indicated temperature should now read approximately 295K (room temperature).

5.6.1 Temperature and voltage limits

If you have bought a cryostat and temperature controller together from Oxford Instruments, the temperature controller will have been set up in the factory:

- To prevent you from accidentally exceeding the maximum safe operating temperature of the cryostat
- To limit the maximum heater voltage to a safe level.

If you are planning to use an existing temperature controller, or a power supply or controller made by another manufacturer, you should take the same precautions. The recommended values for the “Heater Voltage Limit” and the “Temperature Limit” are given with the test results for the cryostat.

If you do not safeguard the system it is possible to cause serious damage.

- **Next** - System Operation

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System Operation

6.1 Loading the sample

With the cryostat at room temperature, open the vacuum valve and remove the top window flange by unscrewing it. Place the sample on the sample holder. A very thin layer of grease (such as Apiezon N grease) will improve thermal contact to the heat exchanger.

The sample holder can also be removed if required. To do this, first remove the top part of the copper radiation shield. Then remove the sample holder by undoing the four screws which hold it to the heat exchanger.

When replacing the sample holder or radiation shield, do up the screws tightly to ensure good thermal contact. Before re-installing the top window flange check the O-ring is clean, undamaged and lightly greased.

6.2 Preparations

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 the section at the end of the next chapter for more details.

Before you start to use liquid Helium make sure that you are aware of the precautions that are necessary to ensure your safety. Refer to *Safety Matters* for more information.

Ensure that the cryostat's OVC and the transfer tube have recently been pumped to high vacuum. Connect the system together and prepare it as described previously.

Check that the white PTFE seal near the end of the transfer tube is clean and undamaged. There should be no grease on it.

6.3 Cooling the system

Close the needle valve on the transfer siphon fully, then open it by six turns. If the transfer siphon has been supplied with the cryostat it should fit the system without any modification. If not, you may need to remove a PTFE washer from the transfer tube arm.

Fully open the needle valve on the VC-U gas flow controller and connect the "from cryostat" connection to the transfer siphon using the polythene tube.

Open the exhaust valve of the liquid Helium dewar to release any pressure, keeping your hands and face away. Remove the plug in the transfer siphon entry fitting. Slowly lower the dewar leg of the siphon into the dewar, engaging the nut on the syphon with the dewar fitting. Switch on the GF4 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, and there is a risk of being burnt by the cold gas.

Once the siphon leg has been loaded into the Helium dewar, monitor the pressure gauge on the VC-U until you see the flow of Helium, then turn off the pump and disconnect the polythene tube from the syphon. Push the other end of the siphon into the cryostat entry arm and engage the nut on the siphon with the thread on the cryostat arm. Take care not to over-tighten the nut. Now connect the polythene tube from the VC-U “from cryostat” connection to the christmas tree fitting on the rear of the transfer siphon. Turn the GF4 pump back on. The cryostat heat exchanger and sample should now cool steadily, typically cooling to 4.2K within 10 minutes.

6.4 Cooling below 4.2K

Temperatures below 4.2K 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 the figure below. 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:

Put the heater control and the gas flow control of the Mercury iTC temperature controller into **Manual** mode, with zero heater voltage.

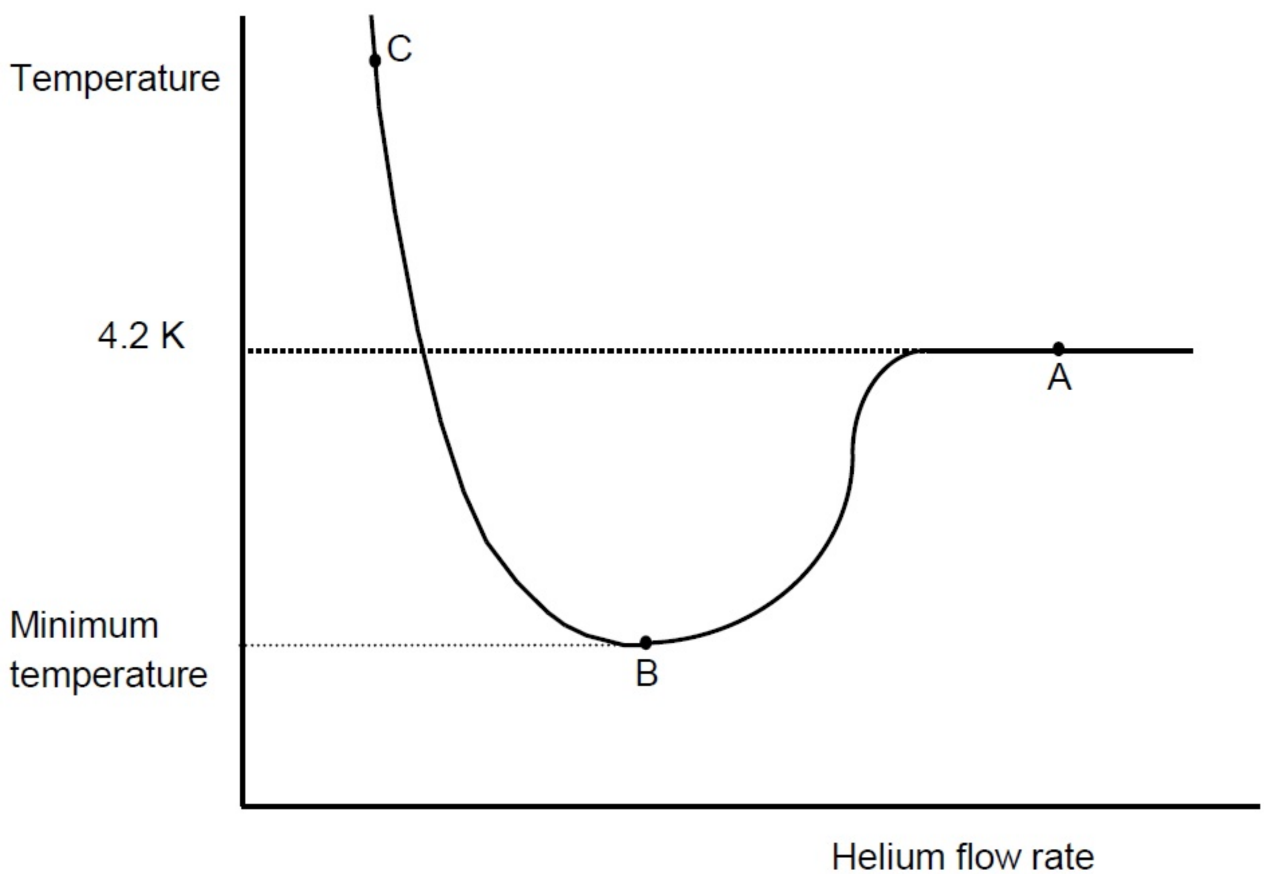

Figure 6.1: Optimising the flow rate

When the cryostat has reached 4.2K (point A), 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 will 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 below about 20K (point C). Now open the needle valve in very small increments, waiting for the temperature to stabilise after each change. As you do this, the temperature will fall, until you reach the base temperature of the system (point B).

Now select the desired SET temperature on the Mercury iTC, and switch the Mercury iTC heater control to **Auto**.

6.5 Temperature control above 4.2K

You can control the temperature of the heat exchanger between 4.2K and 500K. The Helium flow and heater power have to be adjusted in order to reach the desired set point.

The Mercury iTC can control the heater power automatically, adjusting the applied power to maintain the set temperature. The Mercury iTC is a three-term controller - the temperature control is optimised by setting the best values for:

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

On the Mercury iTC home screen (see above), tap **Control** to give the following screen:

Figure 6.2: Mercury control screen

Tap **PID Table** to display a screen similar to the following:

This screen shows that a standard PID table, in this case, “Microstat He Mercury.pid”, is already loaded into the iTC.

Tap **Load** to view alternative PID tables. These may be from the factory or created by the user.

Figure 6.3: Mercury PID table

Figure 6.4: List of PID tables

Tap a filename to select it and then tap **Load** to load the selected PID table.

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 mercury iTC manual.

Tap **Close** to return to the control screen, then tap **Home** to return to the home screen.

6.6 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 Mercury iTC and switch the Mercury iTC 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. In general, the flow should be reduced until the steady heater output is at a suitable level.

If you are using an Auto LLT system, the flow and heater voltage will be automatically optimised. As a guide, when manually optimising the flow, typical heater voltages are as follows:

Temperature range / K	Heater voltage / V
4.2 - 20	3 - 5
< 300K	8 - 12
> 300K	> 8

6.7 Operation above room temperature

Before you set a temperature above room temperature, check that your system is suitable for this temperature range.

The siphon must be removed. Pump the OVC continuously to maintain the required high vacuum. You can now control the system at a ‘set temperature’ as described above, exceeding 300K.

6.8 Warming up the system

Switch off the gas flow pump. After a few seconds, the pressure in the Helium flow circuit will rise to approximately the pressure of the storage dewar. The transfer tube can then be removed from the cryostat.

Immediately fit the special pressure relief valve (supplied with the system) into the cryostat so that it is not contaminated with ice (condensed from the air) which could block the transfer tube next time the system is cooled down.

The system may be left to warm up naturally. To speed up the process, set a temperature of 300K.

To warm the system more quickly, allow a small volume of dry Nitrogen gas from a bladder into the OVC to break the vacuum. 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. Never allow Helium gas into the OVC as it is difficult to pump it out again. Do not use a bladder on the OVC that has previously been used with Helium.

6.9 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:

1. Ensure that the flow gauge calibrated for **air** is used on the VCU.
2. 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.
3. If you pump the liquid Nitrogen to a pressure below 150mbar it may freeze and block the cryostat. A GF4 pump is unlikely to reduce the pressure sufficiently, but a rotary pump could.
4. It is more difficult to control the temperature of the sample, and the stability specification is typically increased to ±0.2K. It may be more difficult to control the temperature below 90K because liquid collects in the heat exchanger and boils intermittently.
5. 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.
6. It is best to use the minimum flow possible to achieve good stability at low temperatures (especially below 90K). 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.
7. 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 from filling with liquid.
8. 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.
9. 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.

- **Next** - Service and Maintenance

7

Service and Maintenance

A number of simple procedures can be followed to resolve issues which may be encountered during routine operation. With proper care and maintenance, the system should provide years of reliable operation.

7.1 Rubber O-rings

Whenever you remove part of the cryostat, or should you suspect that there is a leak on the system, check the O-rings on the sample space and OVC. Ensure that the O-ring is clean, undamaged and lightly greased. Replace any damaged O-rings.

- **Next** - Appendices

8

Microstat HiRes Specifications

8.1 Performance

- Temperature range: 3.4 to 500 K measured at the heat exchanger
  - May be extended down to 2.7 K (see notes)
- Temperature stability: ± 0.1 K
- System may also be run with liquid Nitrogen temperature range 77 to 500 K
- Liquid Helium consumption at 4.2 K: < 0.7 l/hr
- Cool down consumption: 0.8 litres (nominal)

8.2 Operating Cycle

System Cooldown	Specification
Room Temperature to base	approx. 15 minutes with pre-cooled transfer siphon

8.3 Sample Position Stability

Typical sample position drift	Drift
at constant temperature of 4.2 K	0.15 μm per hour
on cooling from 300 to 4.2 K	13.0 μm

8.4 Vibration performance

The vibration of the type of cryostat being offered has been measured using a Leo 1450VP scanning electron microscope at Oxford Instruments NanoAnalysis UK. The results obtained showed that under operating conditions where all system components had been securely held the vibration recorded was better than 20 nm. As the vibration is strongly dependent on the customer's experimental environment and it is not a performance criteria that is measured as part of the standard factory test it would not be prudent for Oxford Instruments to guarantee a level of vibration. However although we cannot make a guarantee of performance we can state that the same design and build have been proven in the past to be capable of obtaining better than 20 nm.

8.5 Physical

Parameter	Nominal Value
Cryostat Weight (kg)	approx 1.5

8.6 Electrical Power

Single Phase

1PWR-U All single phase powered units supplied with the system can be used worldwide without the need for configuration. The maximum power consumption of each unit in this application is:

Component	Power Consumption
MercuryiTC controller	450 W

8.7 Notes

- Base temperature may be reduced by using a rotary pump, such as the EPS40. Please enquire for further details.

8.8 Further Information

- Test Specifications
- Microstat-Optistat Index

9

Appendices

9.1 Electrical connections on the cryostat

The standard cryostat is fitted with a 10-pin seal on the side of the cryostat. This is used for the connection to the cold finger. The seal is held in place by a black nut - do not remove it unless you need to gain access to the wiring. The heat exchanger is fitted with a Rhodium-Iron resistance thermometer (four-wire measurement) and heater. The wiring configuration of the 10-pin seal is given in the table below:

Pin	Function
A	Heat exchanger heater
B	Heat exchanger heater
C	Heat exchanger sensor (V+)
D	Heat exchanger sensor (V-)
E	Heat exchanger sensor (I+)
F	Heat exchanger sensor (I-)
H	Spare
J	Spare
K	Spare
L	Spare

9.1.1 Checking the wiring

A resistance meter can be used to check the wiring of the cryostat - the following readings should be expected across pins:

Pins	Nominal resistance
A - B	42 Ω approx.
C - D	72 Ω approx.
C - E	42 Ω approx.
C - F	72 Ω approx.
D - F	42 Ω approx.
A - C	> 1 MΩ
A - ground	> 1 MΩ
C - ground	> 1 MΩ

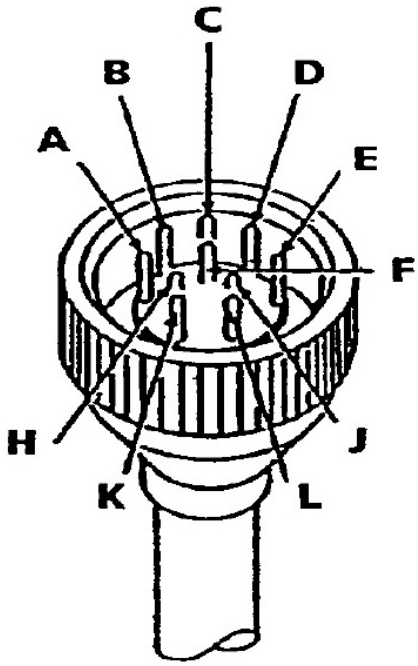

Figure 9.1: Ten-pin seal configuration

9.1.2 Rhodium-Iron resistance thermometer

The RhFe sensor has a 3-point calibration (taken at 4.2K, 77K and 300K) which is used to scale a generic calibration curve covering the temperature range from 1.2K to above 500K (note that the maximum operating temperature of the heat exchanger is 500K).

This scaled generic curve is loaded into the MercuryITC temperature controller to allow monitoring and control of the heat exchanger temperature. In order to confirm the base temperature of the cryostat, a fully calibrated Cernox resistance sensor is fitted for the factory test. The Cernox sensor is mounted on a Copper sample holder, in good thermal contact with the heat exchanger. The Cernox sensor has a calibration accuracy of ±8mK in the range 1.4K to 4.2K. The cryostat base temperature readings, as measured using the system's RhFe sensor and the fully calibrated Cernox, are given in the test results.

9.1.3 Additional Wiring - LX10 connector

An additional 10-pin connector, fitted to the top plate of the cryostat, is wired to terminals located just above the sample holder.

Any wiring made to the sample should be thermally anchored to the heat exchanger or sample holder in order to reduce the heat load on the sample.

9.2 Troubleshooting

The following list summarises the most common faults on the system. You may also need to refer to the troubleshooting guide in the transfer siphon manual.

Cryostat OVC cannot be pumped to high vacuum *OR* **Water condenses on the cryostat body when it is cold**

Check the cryostat OVC for leaks. In particular check the top plate seals.

If there is no leak there may be too much moisture in the OVC and it should be pumped with a rotary pump, with the gas ballast valve open.

Cryostat will not cool down

Check whether there is any flow of gas through the system, using the gauge on the VC-U if applicable.

Poor temperature stability

Check that the PID settings on the temperature controller and the cryogen flow rate are as suggested in the manual and test results.

Cryostat cannot be warmed up from base temperature *OR* **Heater not working**

Check that the *set temperature* is higher than the present sample temperature, or switch the heater on manually.

Check that the heater voltage limit on the temperature controller is high enough. Normal settings are Limit: 40V, Resistance: 20Ω. Check that the high temperature limit of the temperature controller has not been exceeded.

Check that the heater is not open circuit by checking from pins A to B. If so, the wiring will have to be repaired.

Cryostat will not reach base temperature

Check that the heater is switched off. Check that the flow rate is high enough, and that there is sufficient liquid in the storage dewar. If the flow is high the liquid flow may be by-passing the cryostat.

Check that the transfer siphon nut is tight enough, and if so check that the PTFE seal has not been damaged.

Check the connections to the thermometer and make sure that it is working properly and in good thermal contact with the cryogen flow.

Check that you have not added too much heavy wiring to the sample holder, introducing a high heat load.

Check the quality of the vacuum in the OVC and in the transfer siphon.

Check that the sample or sample holder are not touching the OVC.

Check the cryostat for mechanical damage. Warm it to room temperature and remove the OVC to check whether the radiation shield touches the sample space or OVC.

Check that the radiation shield has been fitted correctly and that the sample or sample holder are not touching the radiation shield or OVC.

Sensor not reading correctly

Check the wiring.

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