OXFORD INSTRUMENTS

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User Manual - Original Instructions

MicrostatN

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 :

MicrostatN

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).

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

IMPORTANT INFORMATION

Product description

The Microstat N is a continuous flow nitrogen cryostat, designed principally to allow low temperature samples to be studied with an optical microscope. The sample is mounted in vacuum on a heat exchanger, with optical access available through up to two windows. The sample temperature is held at temperatures betweeen 77K and 500K 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.

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 N Manual

Oxford Instruments Nanoscience

Sep 2017

Contents

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MicrostatN - Principles of Operation

Continuous flow Nitrogen 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 MicrostatN 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.

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 MicrostatN is a continuous flow cryostat designed principally for to allow a sample to be cooled to a low temperature and studied with an optical microscope. The sample temperature is continuously variable between 77K and 500K. The special window arrangement allows the sample to be brought close to the objective lens of the microscope. The cryostat may be operated in any orientation. The sample is mounted in vacuum, and cooled by conduction.

Figure 4.1: Microstat N cryostat

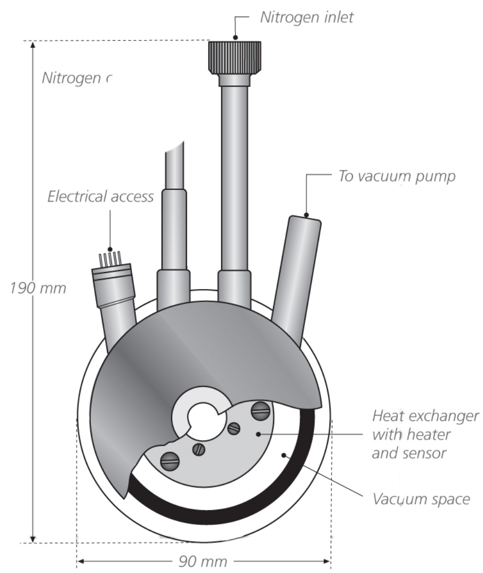

Figure 4.2: Microstat N schematic

The sample is mounted in vacuum on a heat exchanger, and optical access to the sample is available through the windows. The sample space is thermally isolated from the room temperature surroundings by the outer vacuum chamber (OVC). This space is pumped continuously with a two stage rotary pump whilst the cryostat is running.

Up to two windows can be fitted to the cryostat. Each window is permanently bonded into the OVC flange, which is designed to allow the sample to be mounted as close as possible to the inside surface of the window.

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

4.2 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 a insulated transfer tube. It flows through a heat exchanger at the bottom of the sample space, through the sample space and out of the cryostat to the pump. A thermometer and heater are mounted on the heat exchanger, and these can be used with a temperature controller to balance the cooling power of the cryogen and to control the temperature of the gas before it reaches the sample space.

4.3 The liquid Nitrogen transfer tube

A stainless steel, vacuum insulated transfer tube is provided with the system to transfer the liquid nitrogen from the storage vessel to the cryostat. This tube has been made as light and as flexible as possible in order to limit the strain transferred to the cryostat and the microscope stage.

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 MicrostatN cryostat
- Liquid Nitrogen storage dewar
- Liquid Nitrogen transfer tube with suitable storage dewar adapter
- Polythene tube (7mm inner dia.) for the gas exhaust
- High vacuum pumping system to evacuate the OVC continuously
- Oil free diaphragm pump (GF4)
- Temperature controller (Mercury iTC)
- Sensor/heater cable CQB0090
- Gas flow controller (VC-U) and pumping lines

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 schematic below shows the cryogen and exhaust connections required for the operation of the system:

5.3 Evacuating the outer vacuum chamber (OVC)

The OVC has to be pumped continuously with a two-stage rotary pump to make sure that it gives the required thermal isolation. 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.

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 Nitrogen transfer tube

Although sufficient thermal insulation can be achieved by pumping the transfer tube 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

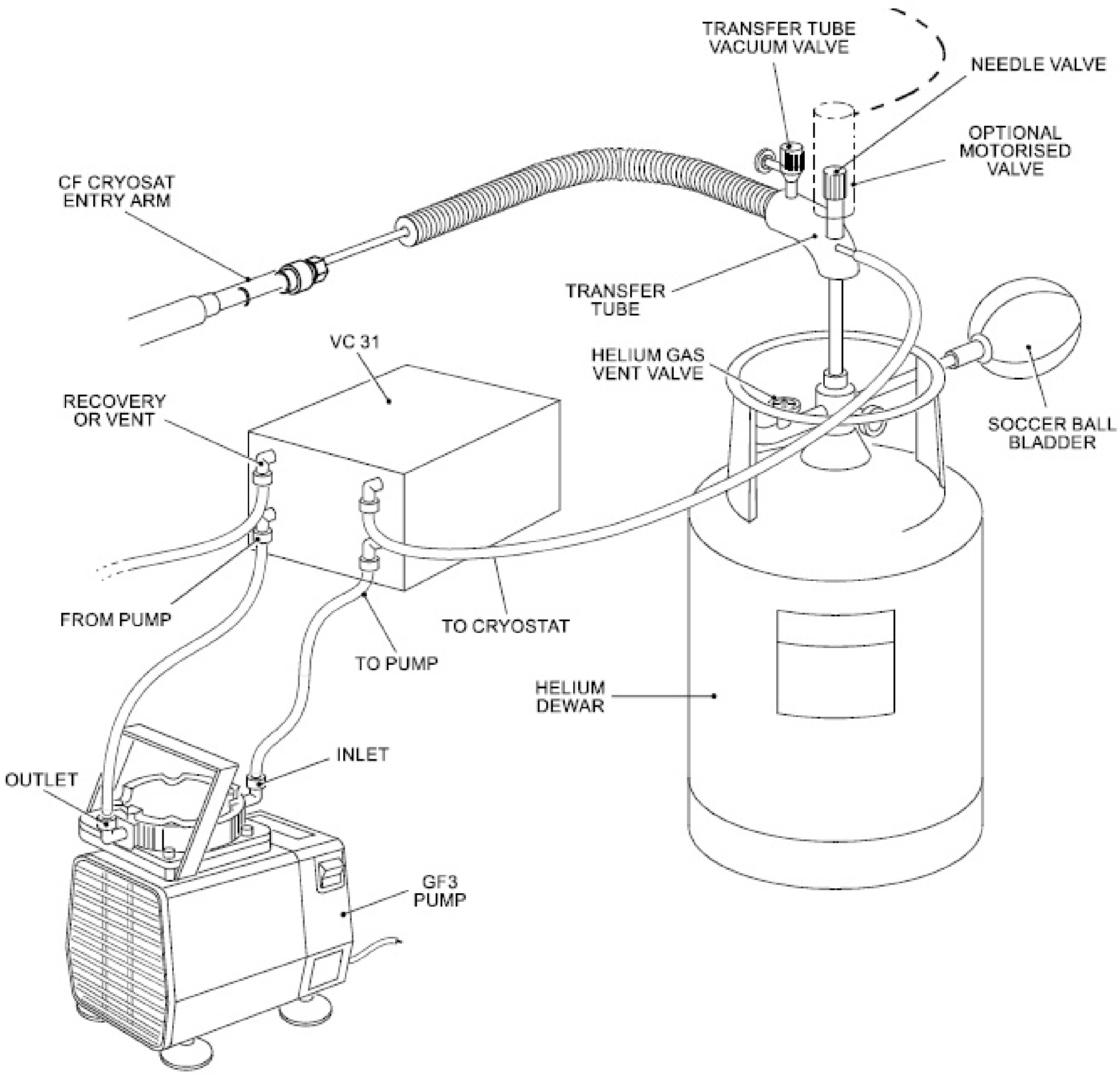

Figure 5.1: Typical system set-up

valve and the pumping system can be connected to it directly.

5.5 Exhaust gas connections

A piece of polythene tube is used to connect the Nitrogen outlet on the cryostat to the VC-U gas flow controller.

5.6 Loading the sample

The sample is mounted on the heat exchanger within the OVC. The top flange of the OVC has to be removed to change the sample. The procedure for changing samples is given later on.

Do not attempt to remove the screws from the bottom of the cryostat.

5.7 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:

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).

5.7.1 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.

Figure 5.2: Mercury home screen

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 Preparations

Make sure there is good ventilation in the room, to avoid any risk of asphyxiation. This can arise from spillage of liquid nitrogen or knocking over the Nitrogen storage dewar.

Ensure that the cryostat OVC is being pumped and that the transfer tube has recently been pumped to a high vacuum.

6.2 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 Nitrogen 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.

As soon as the dewar leg has been loaded into the liquid Nitrogen, turn the GF4 pump off and disconnect the polythene tube from the transfer siphon. Push the other end of the siphon into the entry arm of the cryostat and tighten the nut on the cryostat arm. Connect the “from cryostat” quick-connect on the VC-U to the Nitrogen outlet port on the cryostat, and turn the GF4 pump back on.

The cryostat heat exchanger and sample should now cool steadily. The cryostat cool down time to 77K is typically 10 minutes.

When base temperature is reached, slowly close the needle valve on the VC-U to reduce the consumption of liquid Nitrogen. If you close the needle valve too much the cryostat will begin to warm after a short delay, and you should open it slightly further. During this short delay the cryostat may cool slightly, because liquid sometimes collects in the cryostat and this cools as the pressure drops. However, this ‘single shot’ effect only lasts until this liquid has all evaporated.

6.3 Temperature control above base temperature

You can control the temperature of the heat exchanger (and sample) between base temperature and 300K (or 500K with high temperature windows fitted). The Nitrogen 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 6.1: 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.

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. 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.

6.4 Controlling at a set temperature

On the Mercury iTC home screen (see above), tap **Control**. 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

Figure 6.2: Mercury PID table

Figure 6.3: List of PID tables

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 Nitrogen 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 less than 12V.

6.5 Warming up the system

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

If you do not need to warm the system quickly it 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.

Do not disconnect the pumping line from the OVC while the cryostat is still cold. If this occurs, the top plate of the OVC may be dislodged from the cryostat.

6.6 Changing samples

Warm the cryostat to room temperature, as described above, then open the OVC pumping port to allow air into the system. If the internal parts of the cryostat might still be cold, then use dry Nitrogan gas instead and ensure the system is warm throughout before opening the OVC.

Ensure the cryostat cable and any other electrical connections are disconnected.

To access the sample holder it is necessary to remove the top flange of the cryostat. This is held in place by a circumferential ’O’ring and should be lifted off the cryostat.

Do not attempt to remove the screws from the bottom of the cryostat.

The sample holder, cold finger and wiring are now accessible. To remove the sample holder the retaining screws need to be removed.

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

Take care not to break the window during this process.

When the sample has been replaced, re-assemble the cryostat and evacuate the OVC. Proceed to cool the system down as described previously.

6.6.1 Sample holders

Various sample holders are available for use with the cryostat, including:

- A solid flat holder covering the entire clear aperture. Several small samples may be mounted on this sample holder, so that they can all be measured without having to warm up to change samples.

6.6.2 Adjusting the sample position

Different sample sizes can be accommodated by the use of different thickness copper sample holder adaptor plates.

- **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.

7.2 Removing the OVC

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

- Change the sample
- Modify or repair the wiring
- Repair mechanical damage

Follow the procedure set out in the *Changing samples* section of the Operation chapter.

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Microstat N Specifications

8.1 Performance

- Temperature range: 77 to 500 K
- Temperature stability: ± 0.5 K
- Liquid Nitrogen consumption rate: 0.5 l/hr (nominal)

8.2 Operating Cycle

System Cooldown	Specification
Room Temperature to base	< 10 min with pre-cooled transfer siphon
Sample change time	approx. 30 min (cryostat must be warmed up to access the sample space)

8.3 Sample Position Stability

Designed to give positional stability of the sample holder of approximately 1 μm; the stability is neither measured nor guaranteed and will be dependent on the final system configuration and the environment in which the equipment is used.

8.4 Physical

Parameter	Nominal Value
Cryostat Weight (kg)	0.4

8.5 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

Component	Power Consumption

8.6 Notes

- None

8.7 Further Information

- Test Specifications
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Appendices

9.1 Electrical connections on the cryostat

The standard cryostat is fitted with a 10-pin seal on the OVC body. 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 Platinum resistance thermometer (four-wire measurement) and Watlow "firerod" cartridge 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	20 - 30 Ω
C - D	130 Ω approx.
C - E	< 20 Ω
C - F	130 Ω approx.
D - E	130 Ω approx.
D - F	< 20 Ω
A - C	> 1 MΩ
A - ground	> 1 MΩ
C - ground	> 1 MΩ

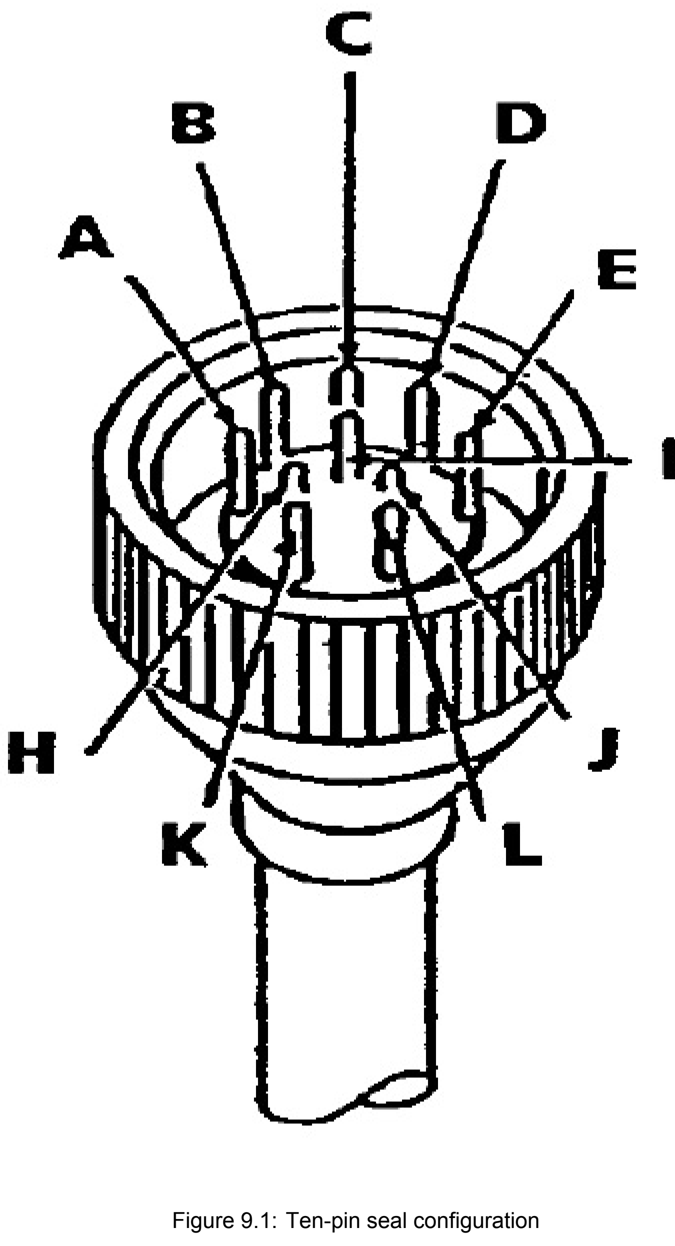

Figure 9.1: Ten-pin seal configuration

9.1.2 Additional Wiring - LX4 connector

The additional LX4 option connects four of the pins on the 10-way connector to pins on the heat exchanger. These pins may be accessed using the special split cable, CWA0111. The 4-way cable is connected to the 10-pin connector as follows:

Pin	Wire colour
H	Red
J	Blue
K	Green
L	Yellow

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 Platinum resistance thermometer

The Platinum Resistance Thermometer is a low cost industrial grade miniature temperature sensor, suitable for cryogenic use over the range 70 to 900K. It is the ideal choice where temperature measurement or control is required to liquid nitrogen temperatures.

The sensor conforms to BS 1904:1984 Class A band 4 with the temperature/resistance relationship shown below. Class A band 4 refers to the manufacturing resistance tolerance of the sensor.

- Ensure sensor is completely clean and dry before assembly and always dry before cooling.
- For cryogenic use, it is sufficient to solder the leads to platinum wire. For high temperature and higher accuracy, extension leads should be connected by welding.

The specifications for the sensor are given in the table below:

Specification	Value
Part No.	PRZ0015
Temperature Range	70-900K
Stability/year	130 mK
Reproducibility	60 mK
Nominal Resistance	100Ω ohms at 273K
	138.5Ω ohms at 373 K

File “RP51.dat” is provided with the system information on your USB stick. This file contains the generic calibration data for the PT100 sensor.

9.3 Troubleshooting

The following list summarises the most common faults on the system.

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.

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.

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.

Sensor not reading correctly

Check the wiring.

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