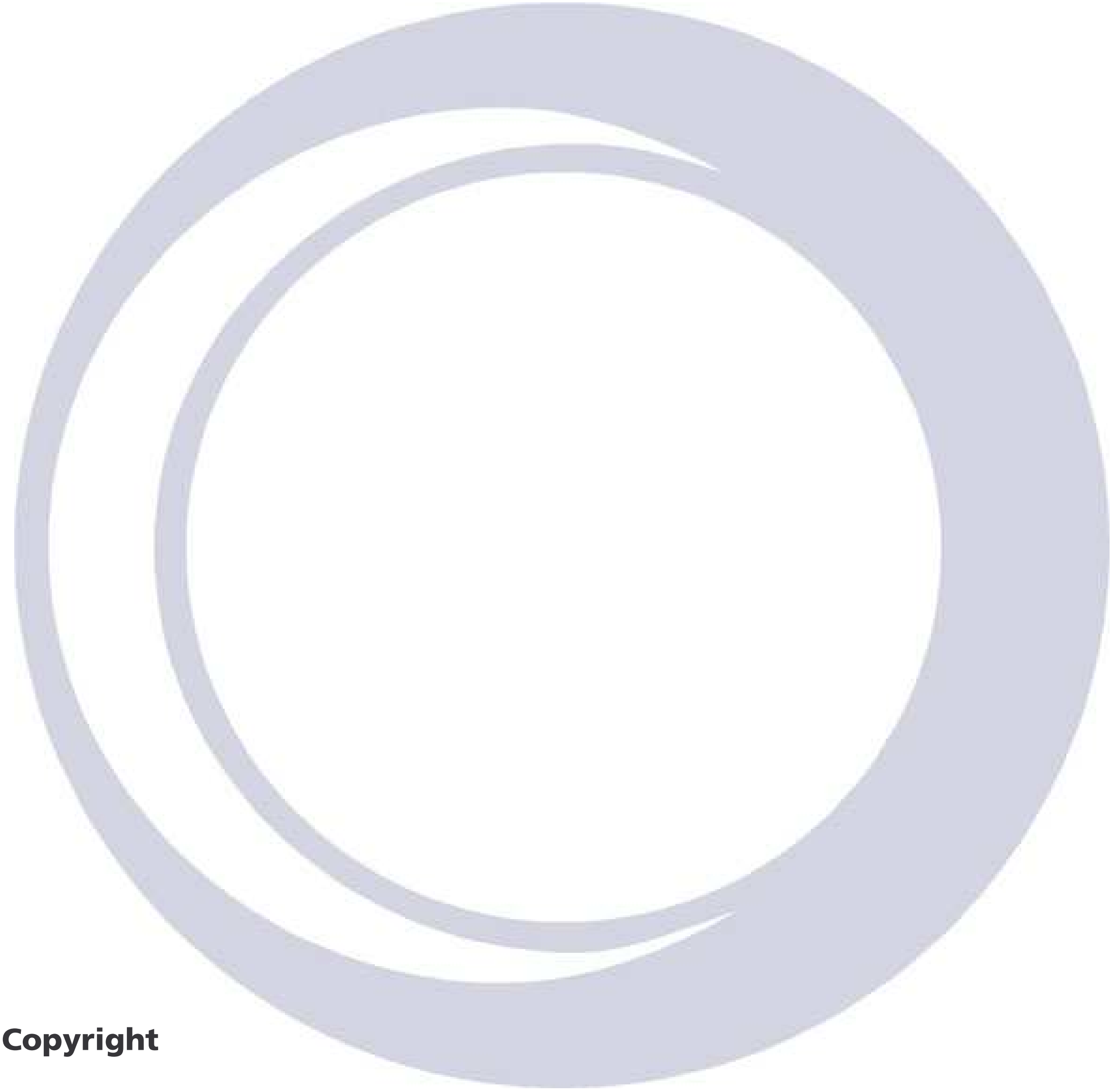
# MICROSTAT MO

*USER MANUAL*
Version 7.0



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### Technical and Customer Support

Contact details for Technical and Customer Support can be found on page 38.

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# *Welcome*

Thank you for choosing your equipment from Oxford Instruments, a company dedicated to providing world-class products and customer support. Our highly trained teams are available to help you with all your queries relating to your order, delivery or technical issues.

As an Oxford Instruments customer, you have access to a worldwide service and support package providing telephone and on-site technical and repair services. In the unlikely event that your product should require repair, our technicians will initiate service under the terms of your Oxford Instruments warranty.

At Oxford Instruments we know that your expectations are at the highest level. We aim to meet and exceed those expectations in the service that we provide, and in the quality you will see when you use Oxford Instruments equipment.

We are delighted you selected Oxford Instruments as your supplier and wish you success with your new equipment.

**Jim Hutchins, Managing Director, Oxford Instruments NanoScience**

# Introduction

## Scope

This manual contains user and technical information pertaining to the Microstat MO. It also contains reference information and includes details of your key contacts at Oxford Instruments who are available for help on repair matters and service. Please keep it close to your system.

## Abbreviations and Terms

| | |
|---|---|
| OVC | Outer Vacuum Chamber |
| SVC | Sample Vacuum Chamber |
| NV | Needle Valve |
| UMS | Unshielded Magnet Systems |
| LLT | Low Loss Transfer |
| HTS | High Temperature Superconductor |

# Safety

Full details of the safety precautions required when working with cryogenic liquids and superconducting magnets are given in the *Safety Matters* booklet that accompanies this manual. Additional copies of which may be purchased from Oxford Instruments Direct.

Safety procedures are vital to prevent

- Serious injury or death
- Serious damage to the equipment.

**This symbol indicates the presence of strong magnetic fields. Medical electronic implants (such as pacemakers) may be affected by a magnetic field and magnetic storage data may be corrupted (credit cards, storage disks etc). Ferromagnetic objects should be moved to a safe distance (outside the 5 gauss field contour).**

## Safety symbols used in this manual

Symbols are used in this manual to draw your attention to safety procedures that you must follow to protect yourself or the equipment. There are two types of hazard symbol used in this manual:

***Warning: The yellow warning triangle highlights dangers which may cause injury or, in extreme circumstances, death. Warnings and cautions must be followed to ensure your own safety.***

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

Additional safety symbols include:

**This symbol indicates that loose fitting, insulating gloves should be worn, suitable for protection against splashes of liquid helium and nitrogen.**

**This symbol indicates that protective goggles or (for cryogenic use) a face mask should be worn.**

**This is the symbol for protective earth.**

## Disclaimer

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

## 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 ($T_{HOT}$)' are given with the test results for the cryostat.

## Warnings

***Before you attempt to install or operate this system, please make sure that you are aware of all safety precaution listed in this document together with the warnings and cautions listed and the operating procedures set out in the associated documents.***

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

***All users of the Microstat MO must read the Oxford Instruments booklet Safety Matters that accompanies this manual.***

# Description

## Overview

The Microstat MO is a continuous flow liquid helium cryostat combined with a conduction cooled superconducting magnet. The design allows a sample to be cooled to a low temperature and studied in a magnetic field both optically and electrically simultaneously. 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.

A second window in the base of the cryostat enables the possibility of transmission measurements to be performed.

The cryostat may be operated in the horizontal or vertical orientation. When in the vertical orientation it is recommended that the side arm is kept horizontal (see Figure 1). Sample temperature stability and consumption can vary if the system is operated in other orientations.

The sample space and the magnet, plus radiation shields, are thermally insulated from the room temperature surroundings by two separate vacuum chambers, the Sample Vacuum Chamber (SVC) and the Outer Vacuum Chamber (OVC) respectively. This gives the possibility to change samples without the necessity to warm up the magnet. Both the OVC and SVC are pumped to a high vacuum before the cryostat is cooled down. Each chamber is protected against accidental build-up of high pressures by separate pressure relief valves.

**Figure 1 - System shown in horizontal position (horizontal magnetic field and access)**

Samples are mounted on separate sample holders with 12 electrical contact pins so that simultaneous electrical and optical measurements can be performed.

## Services

The services for the magnet and heat exchanger are shown in figure 2 below

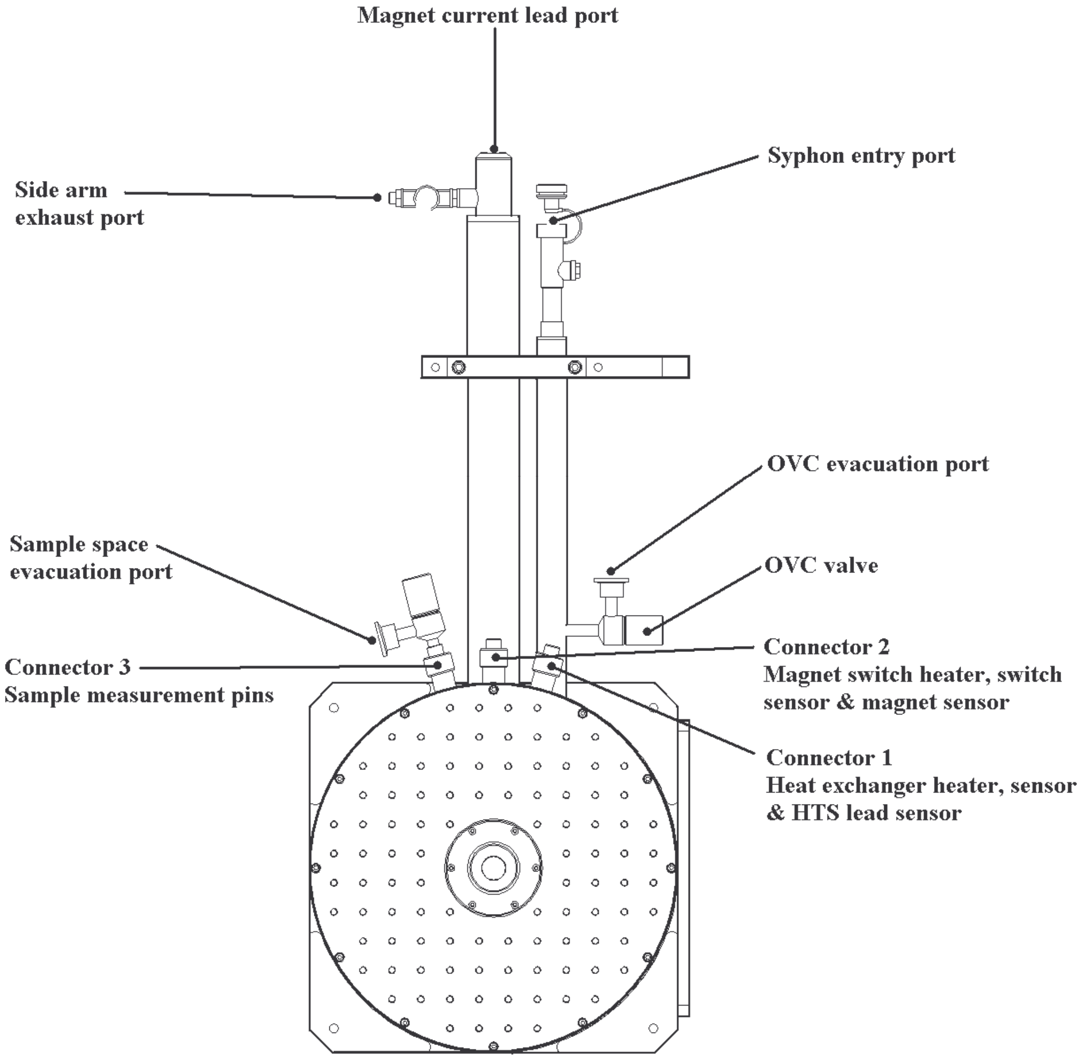

**Figure 2. Diagram of the Microstat MO system showing all service ports**

**All the service ports should be sealed when the system is cold, to prevent air from entering the system**

## Continuous flow cryostats

Continuous flow cryostats do not have an internal reservoir to store a supply of cryogens (although the Microstat MO system does have a small volume for condensed fluid). The liquid is supplied from a separate storage vessel through an insulated transfer tube. The transfer tube delivers the liquid helium to an annular volume around the magnet and to a heat exchanger forming part of the sample mount platform.

The cryostat is equipped with pressure relief mechanism and is therefore fully protected in case of vacuum failure or magnet quench.

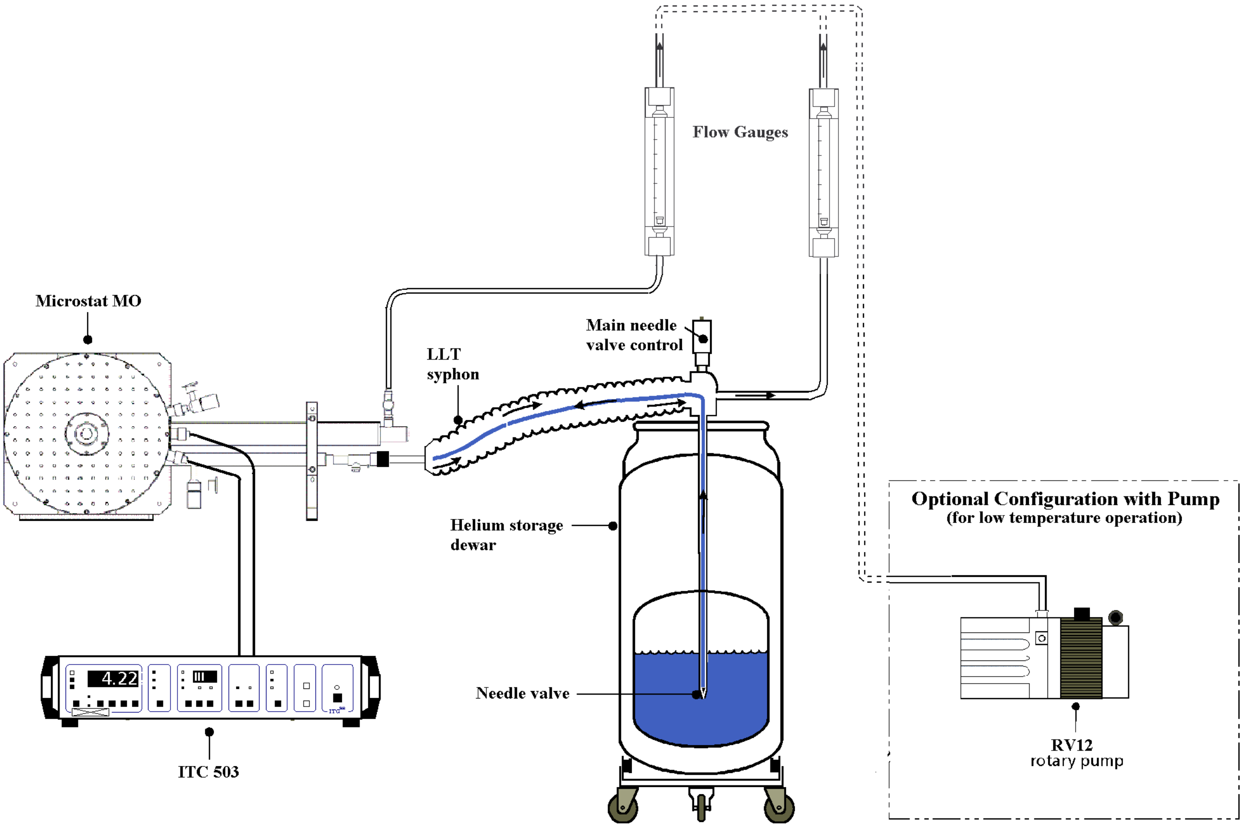

**Figure 3. Schematic of the Microstat MO system showing standard and optional configurations for continuous flow operation**

The Microstat MO has two circuits for the gas returning from the magnet volume. The first cools the HTS current leads and radiation shield before flowing out of the cryostat through the flow gauge valve. The second circuit carries gas to the sample heat exchanger and from there it is returned down the siphon jacket to improve the siphon transfer efficiency.

The exhaust gas flow rate in each circuit can be controlled using the flow gauge needle valve and siphon needle valve. A thermometer and heater are mounted on the sample heat exchanger, and these can be used with the ITC503 temperature controller to control the temperature of the sample.

It is possible to maintain a temperature below 6K continuously using the standard operating configuration shown below (with Helium supplied by pressurising the storage dewar at 5-7psi). Lower temperatures can be achieved by attaching a pump, to each of the helium exhausts. This is also shown in Figure 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 cryogen flow rate through the LLT 600 siphon is controlled manually using the needle valve.

To close the needle valve, turn the handle clockwise. To open it fully, turn it by 6 turns anticlockwise.

### Superconducting magnets

Superconducting magnets allow the production of extremely high magnetic fields in laboratory scale cryostats without the very high power consumption required by equivalent resistive electromagnets.

The MO magnet consists of a solenoid section wound using multi-filamentary superconducting wire. It is constructed such that it is both physically and thermally stable under the large Lorentz forces generated during operation.

The current carrying capacity of the superconducting wire varies with both applied field and temperature. In order to achieve the maximum 5T field the MO magnet must be cooled to 6.0K or less.

#### *Persistent mode using the superconducting switch*

One of the main advantages of the superconducting magnet is its ability to operate in 'persistent mode'. In this type of operation, the superconducting circuit is closed to form a continuous loop, and the power supply can then be switched off, leaving the magnet 'at field'. The field decays only very slowly, at a rate depending on the inductance, the design and number of superconducting joints and the choice of conductor. A decay rate of less than 1 part in $10^4$ relative per hour is easily achieved in the MO magnet. Persistent mode operation is achieved using a superconducting switch that is fitted in parallel with the main windings. Figure 4 shows a typical simple circuit with a switch fitted.

When the magnet is to be energised, the switch is warmed by the switch heater to hold it open  (that is 'normal' or non-superconducting). In this state, although the resistance of the switch is typically only a few 10s of ohms, it is so much higher than the impedance of the magnet that almost all of the current flows through the magnet. Soon after the magnet reaches the desired field the induced voltage across the switch drops to zero and all of the current then flows through the magnet. The switch is closed by turning off the heater, to allow it to return to the superconducting state. After approximately two minutes the current in the magnet leads can be reduced by 'running down' the power supply (this process is sometimes called 'running down the leads'). As the current in the leads drops, the current flowing through the switch rises, until it carries the full current of the magnet.

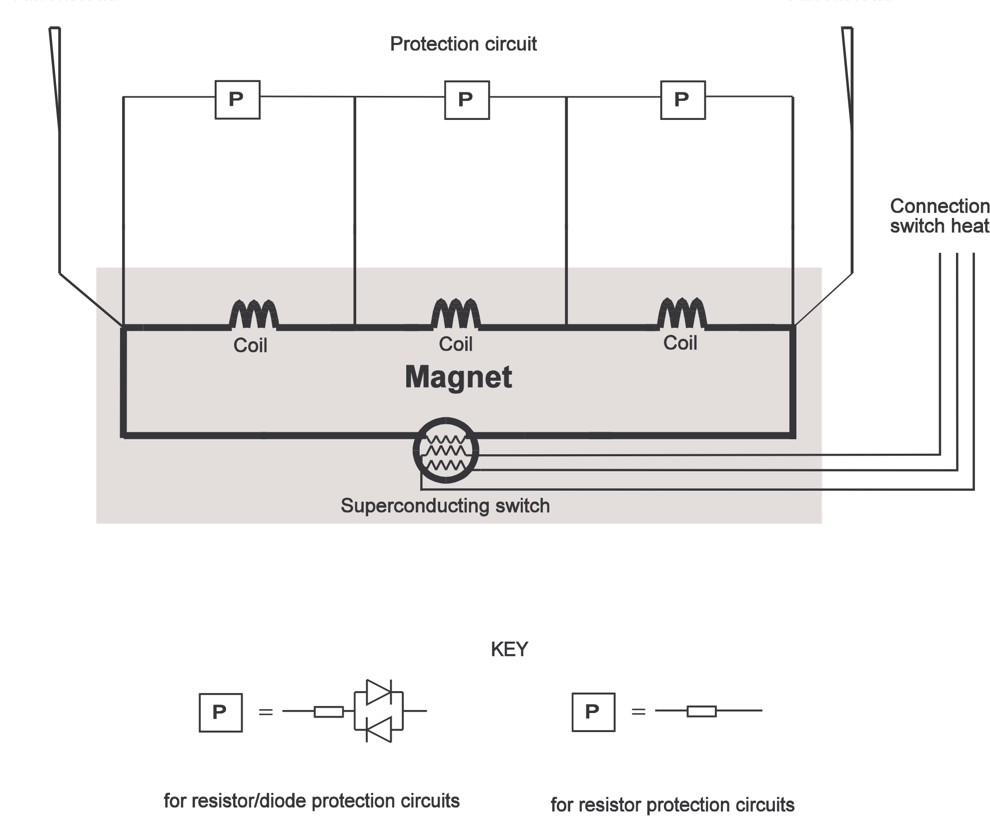

**Figure 4. Simple resistor or resistor/diode protection circuit.**

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### *Quenches*

The magnet will only function properly providing that all of the conductors remain in the superconducting state. If any part of the windings goes 'normal' or resistive, the current passing through it will cause ohmic heating ($I^2R$); in turn this heating increases the size of the normal zone. Once the process has started, it is possible to stop it only if the disturbance is very small, or the magnet is 'stabilised'. Otherwise, the normal zone propagates rapidly through the whole of the magnet. All the stored energy in the magnet is dissipated rapidly, causing the magnet to warm and any liquid helium in the system to boil off rapidly. This is called a 'quench'. If a quench occurs in this system, the pressure in the $^4$He circuit will rise.

The stability of the magnet is strongly influenced by the design of both the conductor and the windings. Only a very small amount of energy is required to start a quench, and this releases the energy stored in the magnet. Even microscopic movements of the wires in the coils may be sufficient to quench the magnet.

A quench often helps the windings to settle, and normal operation can continue after re-cooling the magnet. Indeed in a brand new magnet several quenches may be experienced before the magnet reaches its design field, and the quenches occur at progressively higher fields. This procedure is known as 'training', and it is quite normal. The training is carried out in the factory. It is unusual for the magnet to quench after it has left the factory, and you may run a superconducting magnet system for years without seeing a quench. However, if a new magnet quenches on its first run after transport (as occasionally happens) this should not be a great cause for concern, because it is possible that vibration has disturbed the magnet slightly. One or two training quenches should be sufficient to restore the magnet to its full specification.

### *Magnet protection circuit*

The magnet protection circuit is used in the event of a quench to make sure that high voltages are not produced outside the magnet. It helps to prevent the stored energy of the magnet from being dissipated in the quench region by diverting the current around it.

Protection resistors and diodes are provided for all magnet sections. They are attached across the magnet sections and bolted to the magnet cooling plates.

As diodes are used in the protection circuit then, under normal operating conditions, all the current passes through the magnet and ensures that the energisation current is proportional to the magnetic field. They also reduce heat load from ohmic heating in the protection resistors and hence reduce system heat load while the magnet is sweeping. If the magnet quenches, the barrier voltage is exceeded and the protection comes into operation automatically.

### *Magnet current leads*

This system is fitted with High Temperature Superconductor (HTS) magnet current leads. These are optimised to carry the maximum operating current of the magnet and introduce as little heat as possible to the $^4$He circuit.

### Stray Magnetic Field

The effects of stray magnetic fields on system performance and the environment can often require complex finite element modelling. The following information is provided as a guideline only.

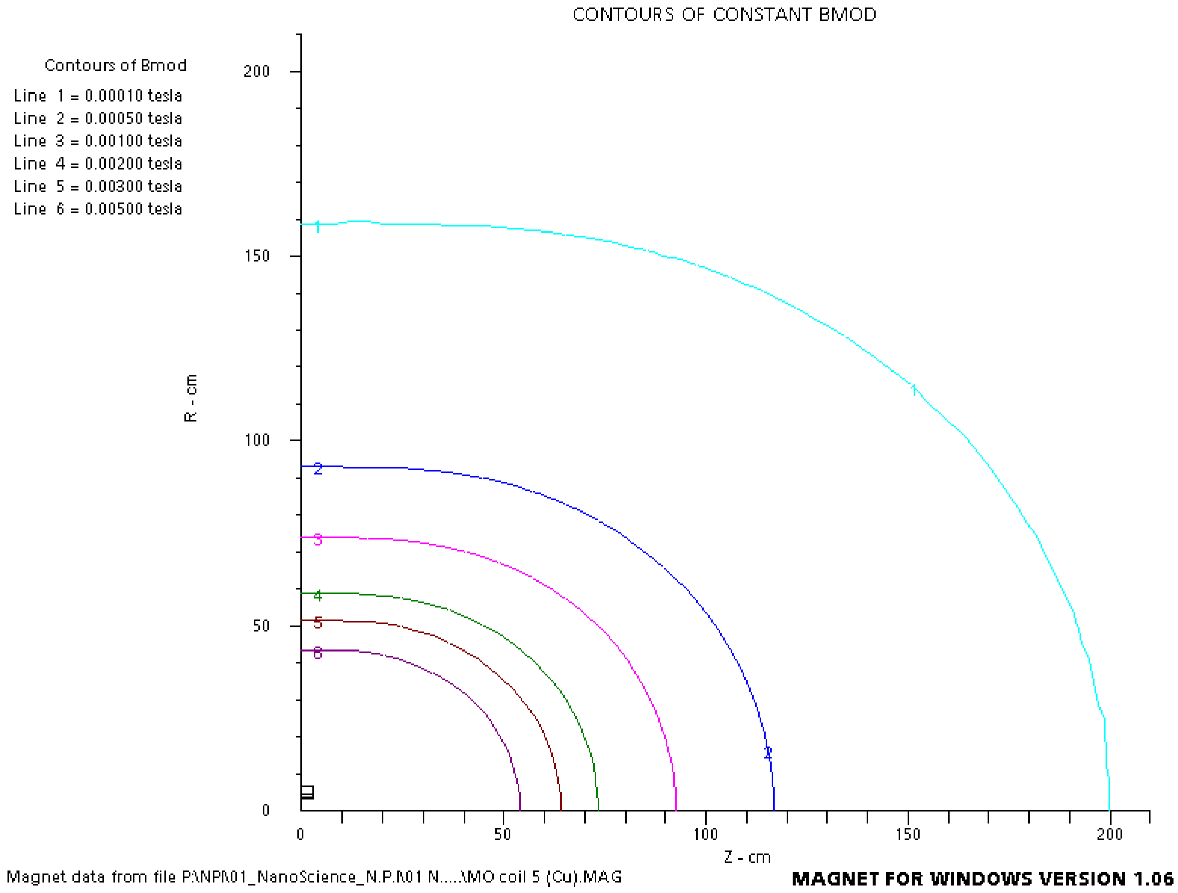

**Figure 5 Stray magnetic field map for the Microstat MO system showing contours at 5, 10, 20, 30 & 50 Gauss.**

### *Static Steel*

The presence of steel in close proximity to the magnet can cause two possible problems:

1. Excess force on the cryostat components leading to damage to the magnet support system or poor cryogenic performance
2. Perturbations of the magnetic field leading to poor homogeneity.

In order to avoid these problems it is necessary to ensure that there is no structural steel within the 30 gauss field contour. This distance can be determined from the system stray field plot.

Items such as steel beams or pillars and concrete reinforcing can cause problems, in particular any non-symmetric steel in the structure.

It is possible that steel in the building will become magnetised and cause areas of increased field at some distance from the system, in particular steel beams may cause increased field areas in adjacent rooms that may affect VDU’s.

### *Moving Steel*

The effect of moving steel can be the same as detailed above with the added problem of field disturbances that may be visible in experimental results. Objects such as vehicles or elevators should be well outside the 1 gauss contour. Large steel equipment, such as gas bottles or pallet trucks, should be kept well outside the 10 gauss contour, movement of this equipment should be controlled as even at this distance effects may be observed. These distances can be determined from the system stray field plot. The relative position and steel content of doors and windows should also be determined.

### *Approximations to force calculation*

The force between the magnet and a ferromagnetic object can be approximated if the magnetic field gradient is known. The critical limit is = 500 G/m; any iron located on this line is exerting a force on the magnet equal to its mass.

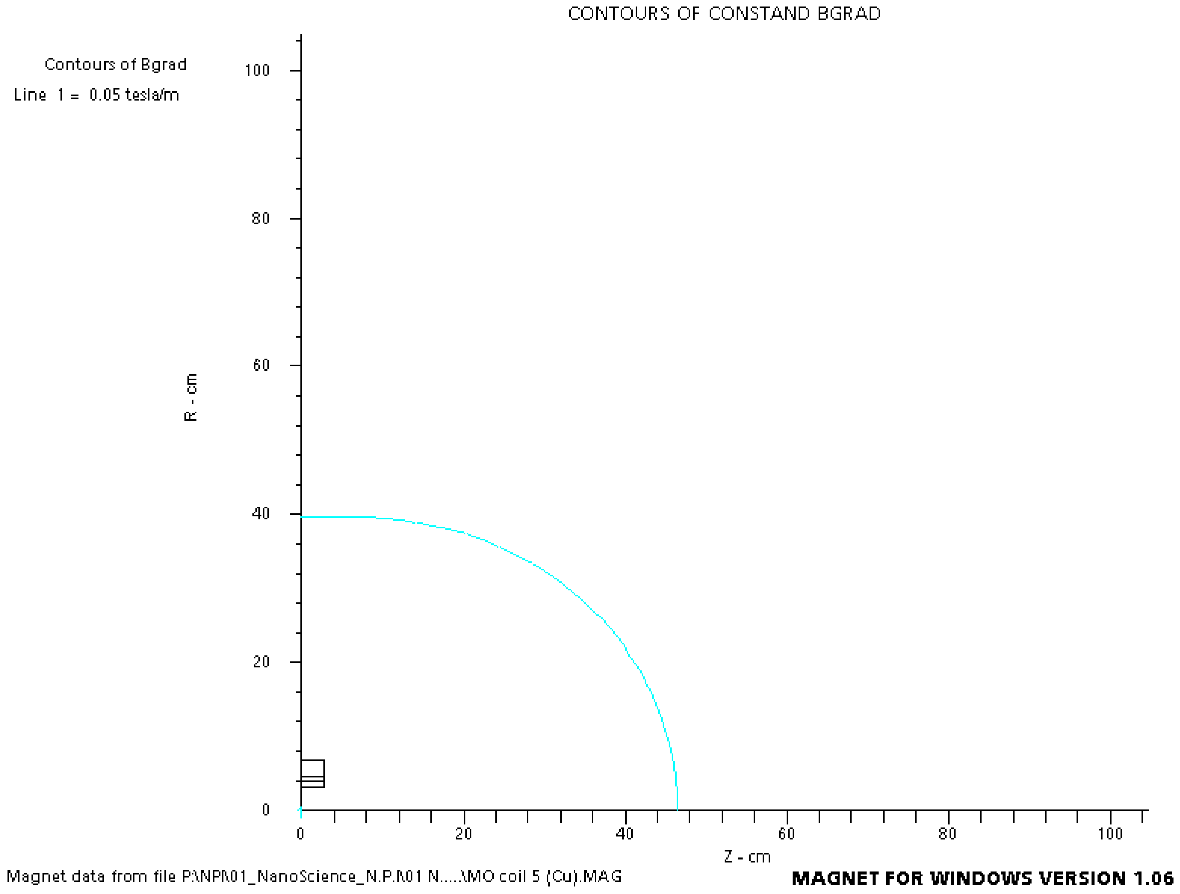

**Figure 6 Stray field gradient map for the Microstat MO showing the 500G/m contour**

Any iron within the region bounded by this line is exerting a greater force which can be approximated as force = (mass of iron object) x (field gradient at that point) / 500; e.g. A 5kg iron mass centred on the 1000 G/m line will exert a force = 5 x 1000/500 = 10 kgf = approx. 100N. However, note that this is only a guideline.

**If there is any significant amount of ferromagnetic material within 2 metres of magnet centre field, contact Oxford Instruments for advice.**

### *Field Limits for Devices*

The following can affect the system and should be positioned outside of the following limits:

**1 gauss**

- Motor vehicles
- Elevators.

**10 gauss**

- Large steel equipment.

**30 gauss**

- Typical structural steel beams.

The following will be affected by the magnetic field and should be outside of the following limits:

**1 gauss**

- Image intensifiers
- Electron microscopes
- Accurate measuring scales
- X-ray machines
- Graphics terminals.

**5 gauss**

- Pacemakers
- Public access without warning signs
- Cathode ray tubes.

**10 gauss**

- Computers
- Watches and clocks
- Credit cards.

**20 gauss**

- Magnetic storage media.

**50 gauss**

- Magnet power supply.

# Unpacking

Examine the ‘Shockwatch’ sensors and Tip ‘n’ Tell on the outside of the packaging. If they show evidence of rough handling (i.e. have turned red) please contact Oxford Instruments immediately (refer to Technical Support on page 38).

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, as listed below. Visually inspect the components for signs of transit damage. If you find signs of damage please contact Oxford Instruments immediately (refer to Technical Support on page 38).

Supplied components are:

- Microstat MO cryostat
- Cryogen transfer tube LLT and suitable adapter for storage dewar
- Polythene tube (7 mm inner diameter) for the gas exhaust
- Magnet Power supply (IPS120-10) plus cables
- Temperature controller (ITC503) plus cables
- Helium gas flow gauges
- Sample Holder Guide
- Sample Holder Torque Wrench.

Optional additional items are:

- Rotary pump (such as the RV12) for enhanced low temperature operation.

Additional components required to run the system are:

- Liquid helium storage dewar
- High vacuum pumping system and lines to evacuate the OVC and SVC.

Documentation supplied:

- Superconducting magnet data
- ObjectBench CD ROM
- LLT Series Operator’s Handbook
- ITC503 Intelligent Temperature Controller Operator’s Handbook
- IPS120-10 Superconducting Magnet Power Supplies Operator’s Handbook
- ITC502 and ITC503 Temperature Controllers Quick Start

# Preparing for operation

**No special tools are required other than a slotted screwdriver and the sample holder tool supplied. However, all tools should be removed from within the 5 Gauss region (see Figure 5) before energising the magnet.**

## Summary of preparation

There are several steps required to prepare the Microstat MO for operation. These are:

- Securing the Microstat MO system to a bench
- Set up exhaust gas connections
- Set up electrical connections
- Remove the sample holder
- Preparing the sample holder
- Installing the sample holder
- Evacuating the OVC and SVC
- Evacuating the transfer tube (siphon).

## Securing the Microstat MO system to a bench

The Microstat MO system comes equipped with a fixing plate, pre-drilled for secured directly to a non-magnetic optical bench. If a magnetic bench is to be used the Microstat MO system must be raised above it by a minimum of 20cm using a suitable stand. An optional stand for this purpose is available for the Microstat MO system.

**Ensure the pressure relief valve is not obstructed (see Figure 7)**

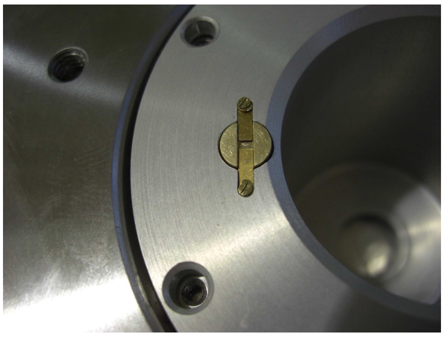

**Figure 7 - SVC pressure relief**

## Set up exhaust gas connections

The Microstat MO is designed for use with an LLT siphon. This plugs in directly to the side arm. The cryogen cools the magnet and exhausts via 2 routes. The first is down the magnet current lead sidearm and out via the flow gauge needle valve. The second is via the sample heat exchanger and back down the outer jacket of the siphon.

For the standard (push) mode of operation, a piece of polythene tube is used to connect the exhaust port on the current lead sidearm to a flow gauge and then to a suitable vent or recovery system. Similarly, another length of tube connects the siphon exhaust to the vent or recovery system via the second flow gauge. For typical operation with the sample heat exchanger at <50K the smaller (0-28l/min) flow gauge should be fitted to the side arm exhaust and the larger (0-60l/min) flow gauge to the siphon exhaust. For some operating conditions with the sample heat exchanger at >50K the flow through the side arm may exceed the scale of the smaller gauge. In this instance the gauges should be reversed.

For enhanced low temperature operation with the LLT transfer tube (optional configuration 2 in Figure 3), it is necessary to feed both exhausts from the flow gauges to a rotary pump such as the RV12 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 either be connected to a helium recovery system or vented to the atmosphere.

**In case of a magnet quench the polythene tubes can blow off the fittings. The tubes must be secured using the cable ties provided to ensure they are restrained.**

## Set up electrical connections

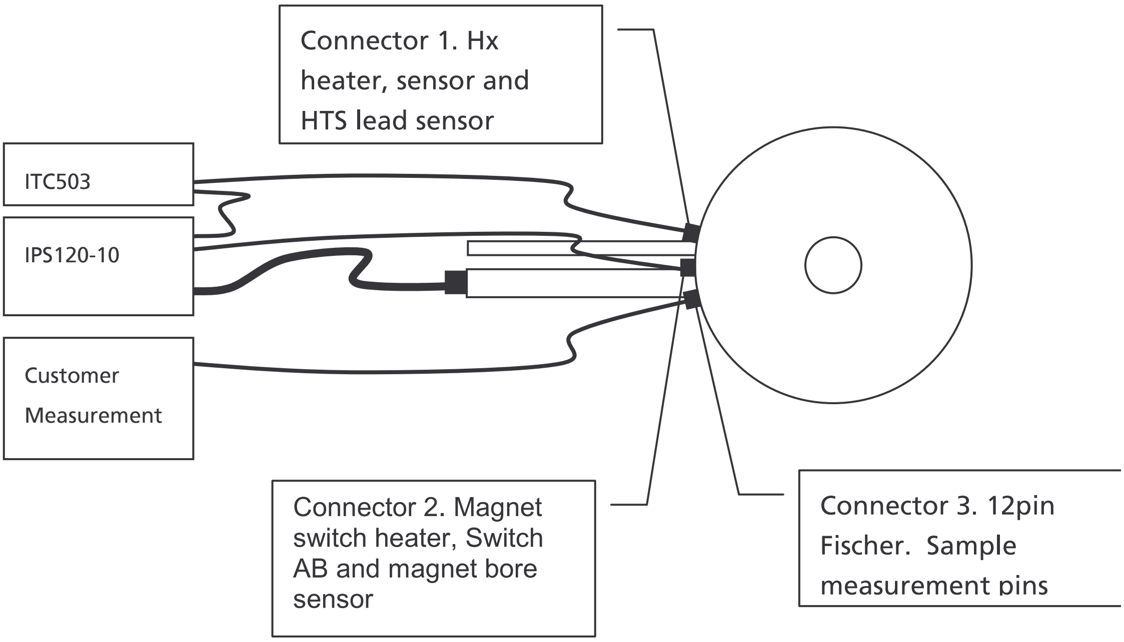

**Figure 8 Electrical connections to the Microstat MO system**

There are 3 multi-way connectors on the OVC in addition to a 2-way connector for the magnet current leads. The magnet current leads should be connected before the system is cooled. The three multi-way connectors are labelled with no.s 1-3. Leads are supplied for connectors 1 & 2. Connector 3 is supplied with a mating plug only.

The lead for connector 1 contains connections for the heat exchanger heater, heat exchanger temperature sensor and the HTS current lead temperature sensor. The heat exchanger sensor should plug into channel 1 of the ITC503 and the HTS lead sensor to channel 3 (both 9-way “D” type connectors). The connections for the heat exchanger heater are integral with the connector on channel 1.

The lead for connector 2 contains connections for the magnet switch heater (which should be connected to the switch heater terminals of the magnet PSU), the switch temperature sensor (not used during normal operation) and the magnet bore temperature sensor. The magnet bore temperature sensor should plug into channel 2 of the ITC503 (again a 9-way “D” type connector).

There is no lead for connector 3. This is for electrical connections to the sample and, if required, should be constructed by the customer to suit their requirements using the connector plug provided.

**Check that the mains voltage selector on the temperature controller is correct for your local power supply, After checking, connect it to the mains**

Switch on the temperature controller, and press the SENSOR button until Sensor 1 LED is lit. The main display should then show the code for the sensor fitted to the heat exchanger of the cryostat. The correct code is shown in the test results sheet. The calibration in the temperature controller has been set up for the thermometer in the cryostat. Pressing the sensor button will show the code for the sensor on that channel before displaying the temperature. If any of the codes displayed are incorrect, please refer to the temperature controller manual.

| ITC socket (back panel) | Cryostat or transfer tube plug | Function |
|---|---|---|
| Sensor 1 | Heat Exchanger cernox | Used to control and display the temperature of the heat exchanger. |
| Sensor 2 | Magnet bore un-calibrated cernox | Displays the temperature of the Magnet cooling plate to confirm the magnet is ready to run. |
| Sensor 3 | HTS lead un-calibrated cernox | Displays the temperature of the hot end of the HTS current leads to confirm the magnet is ready to run. |

Once connected, each sensor should read approximately room temperature (~295K).

The magnet current leads connect to the magnet terminals on the back of the PSU, red to +ve and black to –ve. The 2-pin connector plugs in to the current lead in the current lead side arm.

***Finally the green/yellow earth lead connects to the earth terminal on the PSU. Use the M6 bolt and brass washers provided to bolt the earth lead to the mounting point on the current lead side arm.***

## Remove the sample holder

**Ensure the sample heat exchanger is at ~295K or above and that the magnet is at zero field**

1. Twist the SVC vacuum valve anti-clockwise to vent the SVC (if the system is already cold dry Nitrogen gas must be used to minimise the chances of blockages through contamination of the SVC)

**Ensure dry Nitrogen is used to minimise the chances of blockages through contamination of the SVC**

2. Using a standard slotted screwdriver unscrew the 6 slotted screws holding the top window support tube in place
3. Remove the top window support tube by pulling it up gently
4. Insert the sample holder tool
5. Gently twist the torque wrench until it engages with the slots in the sample holder
6. Once engaged unscrew anti-clockwise until the sample holder comes free
7. Carefully remove the sample holder
8. Disengage the sample holder from the sample holder torque wrench by twisting it.

**If the system has already been cooled, and a new sample is not ready to insert immediately, the window support tube should be re-fitted and the SVC re-evacuated in order to prevent contaminants entering the system.**

## Preparing the sample holder

The sample should be mounted such that good thermal contact is made between the sample and sample holder. This is typically achieved using a very thin layer of grease (such as Apiezon N grease) between the sapphire window (or copper surface) of the holder and the sample.

For measurements that require electrical connections to the sample, the holder is fitted with 12 electrical pins. Upon insertion of the sample holder these pins will automatically pick-up on spring-loaded pins in the heat exchanger. These are wired through to the 12-way Fischer connecter on the OVC via a 12-way loom of 42-swg constantan twisted pairs. The standard sample holder and its electrical pins are shown below in Figure 9.

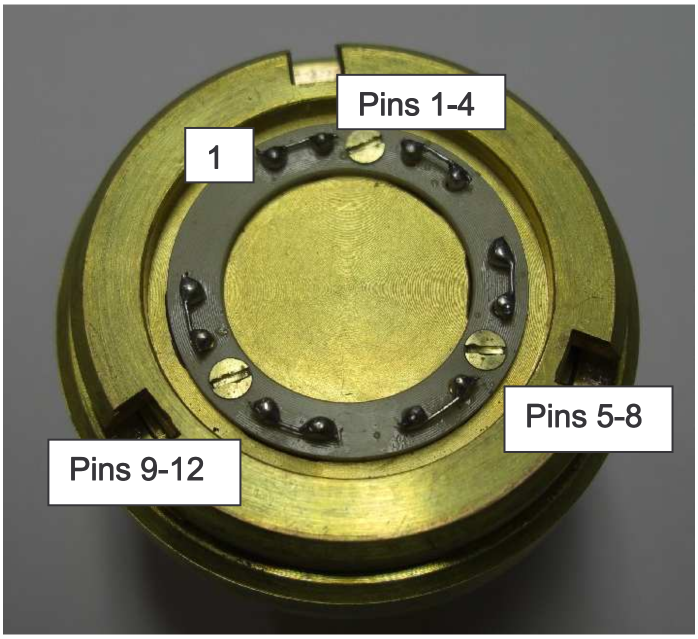

Figure 9 Sample Holder showing position of electrical pins

To make connections from the sample to the sample holder pins use low-temperature solder or silver dag (e.g. Acheson 1415M conducting silver paint) to connect to the pins provided on the sample holder.

Ensure that the sample will not contact the top window. Maximum height available for the sample is typically 5-6mm as shown in Figure below.

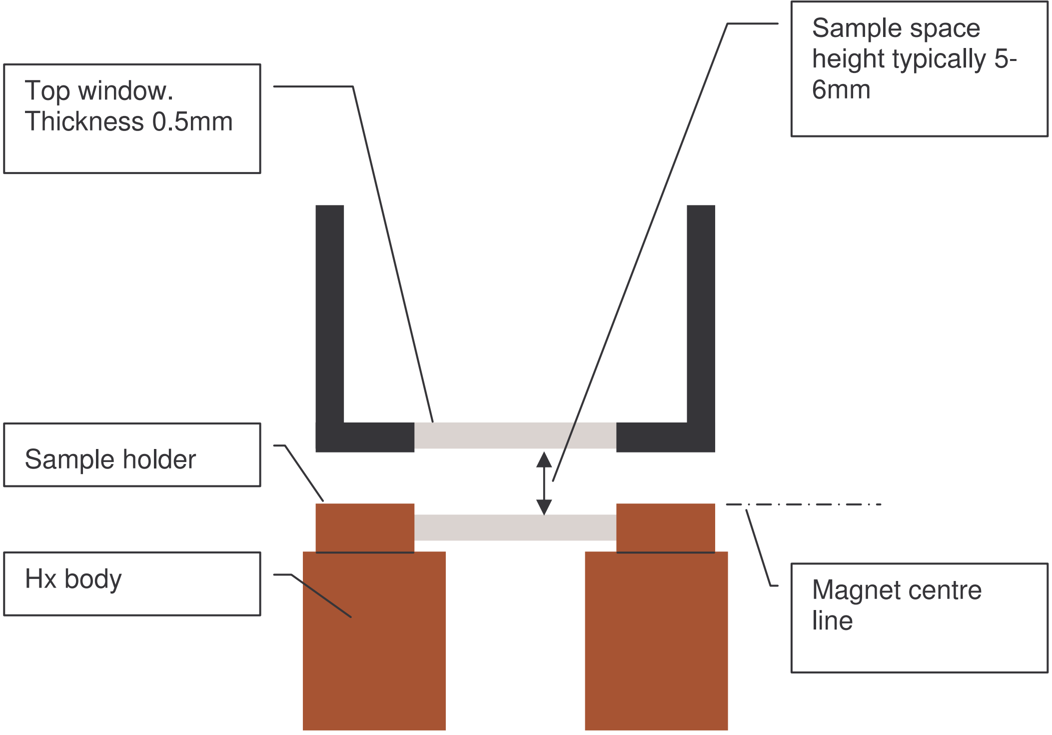

Figure10. Schematic to show the position of the sample relative to the top window

## Installing the sample holder

**Ensure the sample heat exchanger is at ~295K or above and that the magnet is at zero field**

Upon insertion of the sample holder its pins will automatically pick-up on the spring-loaded pins in the heat exchanger.

1. Engage the sample holder with the sample holder torque wrench
2. Align pin in heat exchanger with slot in sample holder
3. Gently twist the sample holder torque wrench so that the sample holder engages the thread on the heat exchanger
4. Tighten until the limit set on the torque wrench is reached
5. Twist the sample holder tool anticlockwise until it disengages with the sample holder
6. Withdraw the sample holder torque wrench
7. Using the six slotted screws, re-fit the top window support tube and secure.

## Evacuating the OVC and SVC

The OVC and the SVC must be pumped to high vacuum to make sure that they give 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 vacuum is cleaning the new materials.

The OVC should be pumped thoroughly before each cooldown, especially when the cryostat is new. Typically, you should pump until the pressure at the pump is $10^{-4}$ or $10^{-5}$ mbar. If the system is contaminated with water, the gas ballast facility on the rotary pump should be used.

To evacuate the OVC connect the pumping system to the OVC evacuation port (see figure 2). The OVC valve is turned clockwise to close and anticlockwise to open. Similarly, to evacuate the SVC connect the pumping system to the SVC evacuation port (see figure 2)

We recommend that you use a diffusion pump or turbo-molecular pump, backed by a suitable rotary pump. A diffusion pump should be fitted with a cold trap.

## Evacuating the transfer tube

The transfer tube vacuum space should be pumped out before use. It has a separate evacuation port and valve similar to that on the cryostat. The pumping system should be connected to it directly.

# Operation

**Any tools used in preparing the system should be removed prior to running the magnet.**

## Running the system

Ensure that the system has been properly prepared, as per Preparing for operation on page 19.

Set the 'set temperature' of the temperature controller to the desired temperature:

1. Press and hold the SET button
2. On the main display use RAISE/LOWER to adjust the set point
3. Check that each of the sensors is working (i.e. reading approximately room temperature) prior to starting the cooldown.

### Cooldown by pushing helium through the system

**Appropriate personal protective equipment should be used when handling cryogens, see Safety Matters.**

*The OVC and SVC should be pumped thoroughly before each cooldown, especially when the cryostat is new.*

In this configuration the $^4$He storage dewar is over-pressurised by 5-7psi (typical relief valve pressure). Flow is regulated by the needle valve (NV) on the siphon and the balance between the flow branches is controlled by the regulator valve, on the current lead exhaust.

1. Make sure the needle valve on the transfer tube is fully open initially. (See The cryogen transfer tube on page 12)
2. Check that the white PTFE seal near the end of the transfer tube is clean and undamaged. There should be no grease on it
3. Open the exhaust valve of the liquid helium dewar to release any pressure, keeping your hands and face away
4. Remove the plug in the transfer tube entry fitting
5. Slowly lower the dewar leg of the transfer tube into the liquid helium. Some liquid will be used to cool the leg, and the dewar exhaust must be open to allow this gas to escape. If you try to cool the leg too quickly a large amount of liquid will be wasted, and the cold gas could burn you
6. As soon as the dewar leg has been loaded into the liquid helium, push the other end into the entry arm of the cryostat until the knurled nut just touches the thread on the arm. Do not engage the thread yet. This allows liquid helium to bypass the cryostat, passing straight from the transfer tube into the entry arm and back into the exhaust, cooling the transfer tube quickly

7. After a few minutes, engage the nut on the transfer tube on the thread on the cryostat arm and tighten it. This brings the PTFE seal into contact with its seat in the cryostat, forcing the helium to pass through the cryostat. If the total gas flow does not reach >10 l/min after 20 minutes the transfer tube may be blocked, or the needle valve may not be opening correctly. Refer to the transfer tube manual for further details.

For a fast cooldown, set the NV to give a total flow in excess of 25-gas l/min. Adjust the flow gauge needle valves so that approximately 40% of this (i.e. ~10l/min) flows through the current lead branch with the remainder flowing back down the siphon jacket.

The cryostat should now be cooling steadily, and the transfer tube and cryostat arm may contract by different amounts. The knurled nut on the cryostat arm should be tightened again occasionally, to make sure that it maintains the seal in the cryostat, so that the liquid helium does not by-pass the cryostat. Monitor the heat exchanger temperature (ITC Ch1) and magnet temperature (ITC Ch2) to check the cooldown progress.

**A small amount of ice, depending on the flow rate, will form on the end of the side arm around the current lead exhaust.**

### Enhanced low temperature operation

Temperatures lower than base temperature in the standard “push” mode of operation (typically 5K) can be 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.

Total flow through the system can be reduced to 18-gas lts/min by adjusting the NV:

1. Use the flow gauge needle valve to reduce the flow in the current lead branch to 6 gas lts/min
2. Connect the RV12 rotary pump to the siphon return and start pumping
3. Make fine adjustments on the NV to reduce the heat exchanger temperature to establish base temperature
4. Adjust the balance regulator to keep 6 gas lts/min through the current leads to ensure the shield and magnet are sufficiently cooled.

If the ITC is being controlled via ObjectBench then with the ITC in MANUAL:

1. Set the control mode of the ITC to:
   Gas = Manual
   Heat = Auto
2. Now select the desired SET temperature on the ITC
3. Switch the ITC heater control to AUTO.
   Gas control should be MANUAL.

### Temperature control above base temperature

You can control the temperature of the heat exchanger between base temperature (typically around 5K) and the specified maximum temperature of your cryostat using a temperature controller. The flow of liquid helium and the heater power have to be adjusted to reach the required set point. The ITC503 temperature controller is used to control the heater power automatically, and adjusts the power to maintain the set temperature. These temperature controllers are three term controllers. The temperature control is optimised by setting the best values for:

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

The values given in the test results for the system are suitable to give good stability. If you want to improve the stability further you may be able to do this by adjusting the three terms slightly. The autotune facility on the ITC503 can be used to optimise these values, and the auto PID feature can be set up to allow the temperature controller to choose the best values for the three terms to suit the set temperature. The procedure for optimising the PID values and control theory are given in the ITC manual.

### Controlling at a 'set temperature'

Check that the cryostat has been connected to the temperature controller as described in

Set up electrical connections on page 21.

*Note: It is not necessary to cool the cryostat to 4.2 K before you set the required 'set temperature'.*

1. Select the channel on the temperature controller corresponding to the sensor that will be used to control the system
2. Ensure that the light on the heater control panel corresponds to the control sensor
3. Set the required 'set temperature' by pressing and holding the SET button on the temperature controller
4. On the main display use the RAISE/LOWER buttons to adjust the value
5. Set the PID values and the cryogen flow rate to those shown for the nearest temperature in the test results
6. Press the AUTO button once, and the temperature controller should adjust the heater output to warm the heat exchanger to the 'set 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 then hold it at this point.

You should then optimise the flow of liquid helium 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. As a guide, if you are optimising the flow manually the heater voltage should typically be as follows:

- 3 to 5 volts when the system is working in the region 4.2 K to 20 K
- 8 to 12 volts in the region 20 - 300 K.

**The 'set temperature' must not exceed 300K.**

**The ITC temperature limit should be set at 305K. For systems supplied with an ITC this is set at the factory. For systems supplied without an ITC consult section 7.2 of the ITC 503 operators handbook.**

## Running-up the Magnet

Before running-up the magnet first ensure:

**Ensure that the earth is bolted to the system and the power supply.**

**Check that the mains voltage selector on the power supply is correct for your local power supply.**

1. Current leads are connected to the system and the magnet power supply (IPS120-10)

2. Services lead is connected to the system and the IPS (switch heater terminals) and ITC (Ch2)
3. Magnet temperature is below 5.5K (ITC Ch2). If not, increase the total flow of Helium via the siphon NV
4. HTS hot end sensor reads below 40K (ITC Ch 3). If not, increase Helium flow through the current lead side arm via the side arm NV
5. $^4$He flow is stable. Note that this may take several minutes to stabilise following adjustments to either the flow gauges or siphon NV's.

To run-up the magnet:

1. If the IPS is clamped press HOLD on the IPS front panel to unclamp the IPS output
2. Use the CURRENT/FIELD button to show magnet current or central field
3. Set the field or current desired by holding in the SET POINT button and adjusting using the RAISE and LOWER buttons
4. Switch on the switch heater from the front panel of the IPS. Both the SELECT and CONFIRM LED's should light. Wait 30 seconds
5. Press GOTO SET on the IPS front panel to run up the magnet. This will ramp up at a preset ramp rate. If a different ramp rate is required this can be set in a similar way setting the target field using the SET RATE and RAISE and LOWER buttons. The IPS will not ramp faster than preset ramp rate limits. See IPS manual for more details.

*Note.* *All of the above operations are available as software commands either using ObjectBench or LabView applications. The command syntax is listed in the IPS manual.*

**The current supplied to the magnet must not exceed 0.5A above that required for 5T. For systems supplied with an IPS120 this is set at the factory. For systems without an IPS120 consult section 11.10 of the IPS120 operators handbook.**

**Running magnet with Hx temps >100K.**

**For heat exchanger temperatures of >100K the magnet must be run only after the heat exchanger has reached the set temperature. Failure to do this may result in magnet quench.**

## Running in persistent mode

To set the magnet persistent once it has been run-up:

1. Ensure flow is stable and magnet temperature is not drifting up and HTS lead temperature is <40K
2. Switch off the switch heater. Both LED's should go out. Wait a minimum of 120 seconds for the switch to cool and become superconducting

3. Press GOTO ZERO on the IPS front panel to run down the IPS and current leads. Current will remain in the magnet coils flowing through the superconducting switch. The magnet is now “persistent”.
4. If the magnet is to be left persistent for a period of several hours it would be advantageous to reduce helium flow through the current lead side arm slightly as the heat load on the current leads will reduce when they are no longer required to carry current. Ensure the magnet temperature remains stable.

To take the magnet out of persistent mode:

1. Ensure helium flow through the current lead side arm will be sufficient to maintain HTS lead temperature at <40K once the current is applied. This is typically ≥10l/min

**Ensure the IPS set point value matches the field the magnet is energised to.**

2. Press GOTO SET on the IPS front panel to run up the IPS and current leads
3. Switch on the switch heater. Both LED’s should light. Wait 30 seconds for the switch to warm up
4. The magnet current is now fully under the control of the power supply.

*Note.* *If the set point current of the IPS does not match the magnet current a fast change in magnet current may occur as the switch opens. If the change in current is too fast the magnet may quench.*

### Warming up the system

**Appropriate personal protective equipment should be used when handling cryogens, see Safety Matters.**

1. Switch off the gas flow pump if used
2. Close the valve on the flow gauge
   After a few seconds the pressure in the helium flow circuit will rise to approximately the pressure of the storage dewar
3. Remove the transfer tube from the cryostat
4. Immediately fit the special slotted bung (supplied with the system) into the cryostat so that it is not contaminated with ice (condensed from the air). This could block the transfer tube next time it is cooled down.

If you do not need to warm the system quickly it may be left to warm up naturally. If you want to warm it more quickly allow a small volume of dry nitrogen gas into the OVC to break the vacuum.

# Technical Data

## Electrical connections

The cryostat is fitted with two 10-way and one 12-way connector on the side of the cryostat. The plugs are held in place by black nuts. Do not remove these unless you need to gain access to the wiring, as this will allow air into the vacuum chamber.

Connector 1 carries the connections for the heat exchanger sensor, the heat exchanger heater (for control of sample temperature) and the temperature sensor for the “hot” end of the HTS current leads. Each of the temperature sensors is a cernox device.

| Connector 1 Pins | Function | |
|---|---|---|
| 1 | I+ | Hx heater: Watlow Firerod cartridge heater |
| 2 | I- | Hx heater: Watlow Firerod cartridge heater |
| 3 | V- | Heat exchanger sensor |
| 4 | V+ | Heat exchanger sensor |
| 5 | I+ | Heat exchanger sensor |
| 6 | I- | Heat exchanger sensor |
| 7 | V- | HTS hot end sensor |
| 8 | V+ | HTS hot end sensor |
| 9 | I+ | HTS hot end sensor |
| 10 | I- | HTS hot end sensor |

Connector 2 carries connections for the magnet switch heater, the Allen Bradley temperature sensor attached to the switch and the cernox temperature sensor fitted to the bore of the magnet. The bore sensor used as the reference sensor for the magnet temperature. The Allen Bradley switch sensor is not used during normal running of the system – it is present for diagnostic purposes only.

| Connector 2 Pins | Function | | Wire colour |
|---|---|---|---|
| 1 | V+ | Switch heater | Orange |
| 2 | V- | Switch heater | Pink |
| 3 | V- | Switch heater Allen Bradley | |
| 4 | V+ | Switch heater Allen Bradley | |
| 5 | I+ | Switch heater Allen Bradley | |
| 6 | I- | Switch heater Allen Bradley | |
| 7 | V- | Magnet bore sensor | Green |
| 8 | V+ | Magnet bore sensor | Blue |
| 9 | I+ | Magnet bore sensor | Violet |

| Connector 2 Pins | Function | | Wire colour |
|---|---|---|---|
| 10 | I- | Magnet bore sensor | White |

The spring contact pins on the top of the sample heat exchanger are wired through to connector 3. The pins connect to the pins on the sample holder and the wiring is pin to pin i.e. pin 1 on the sample holder connects to pin 1 on the fischer connector, pin 2 to pin 2 etc. The standard wiring is 42-swg constantan in twisted pairs. Pin 1 and pin 2 wires twisted together, pin 3 and pin 4 wires twisted together etc.

| 12-pin Fischer Pin# | Heat exchanger / sample holder Pin# |
|---|---|
| 1 - 12 | 1 – 12 respectively |

## Checking the wiring

A resistance meter can be used to check the wiring of the cryostat. You should expect to measure the following values. These readings may be affected if the cryostat is damp or if your fingers are in contact with one or more of the pins.

| Pins of connector 1 | Expected resistance (at room temperature). |
|---|---|
| 1 to 2 | 40Ω approx. |
| 3 to 4 | 220Ω approx. |
| 3 to 5 | 220Ω approx |
| 3 to 6 | 170Ω approx. |
| 4 to 6 | 170Ω approx. |
| 7 to 8 | 220Ω approx. |
| 7 to 9 | 220Ω approx |
| 7 to 10 | 170Ω approx. |
| 8 to 10 | 170Ω approx. |
| 1 to 3 | > 1 MΩ |
| 1 to 7 | > 1 MΩ |
| 1 to ground | > 1 MΩ |
| 3 to ground | > 1 MΩ |
| 7 to ground | > 1 MΩ |

| Pins of connector 2 | Expected resistance (at room temperature). |
|---|---|
| 1 to 2 | 105Ω approx. |
| 7 to 8 | 220Ω approx. |
| 7 to 9 | 220Ω approx |
| 7 to 10 | 170Ω approx. |
| 8 to 10 | 170Ω approx. |
| 3 to 5 | 280Ω approx |
| 1 to 3 | > 1 MΩ |
| 1 to 7 | > 1 MΩ |
| 3 to 7 | > 1 MΩ |
| 1 to ground | > 1 MΩ |
| 3 to ground | > 1 MΩ |
| 7 to ground | > 1 MΩ |

| Pins of fischer connector | Expected resistance. |
|---|---|
| All to ground | > 1 MΩ |

# Faultfinding

The following table shows the most common faults. Refer also to the fault-finding table in the transfer tube manual.

| Symptom | Diagnosis and suggestions |
|---|---|
| Cryostat OVC cannot be pumped to high vacuum<br>*or*<br>Water condenses on the cryostat body when it is cold | Check the OVC for leaks. In particular check the main O-rings<br>If there is no leak there may be too much moisture in the OVC and it should be pumped with a rotary pump whilst the gas ballast valve is open. |
| Cryostat SVC cannot be pumped to high vacuum | Check the SVC for leaks. In particular check the top O-ring for contamination.<br>If the sample or sample mounting grease is outgassing excessively leave the SVC on pump during cooldown |
| Cryostat will not cool down | Check whether there is any flow of gas through the system, using the gauge on the VC31. If not see the transfer tube manual. |
| Poor temperature stability | Check that the PID settings on the temperature controller and the cryogen flow rate are as suggested in the test results. |
| Cryostat cannot be warmed up.<br>*or*<br>Heater not working | Check that the 'set temperature' is higher than the present sample temperature, or switch the heater on manually.<br>Check that the high temperature limit of the temperature controller has not been exceeded, (as indicated by the message "Hot 1" on the display).<br>Check that the heater voltage limit on the temperature controller is high enough.<br>Check that the heater is not open circuit by checking from pin 1 to pin 2. If so the wiring will have to be repaired. |

| Symptom | Diagnosis and suggestions |
|---|---|
| Cryostat will not reach specified minimum temperature | Check that the heater is switched off.<br>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.<br>Check that the transfer tube nut is tight enough, and if so check that the PTFE seal has not been damaged.<br>Check the connections to the thermometer and make sure that it is working properly.<br>Check the quality of the vacuum in the OVC.<br>Check the vacuum in the transfer tube.<br>Check the cryostat for mechanical damage. Warm it to room temperature and remove the OVC to check whether the radiation shield touches the sample holder or OVC.<br>Check that the sample or sample holder are not touching the window mount tube. |
| Sensor not reading correctly | Check the wiring.<br>Refer to ITC manual for sensor calibration set up. |
| Magnet will not run up | Check magnet temperature is below 5.5K (ITC Ch2).<br>Check the current leads and side arm current leads are properly connected.<br>Check switch heater is on and the CONFIRM LED is lit.<br>Check the set field is greater than zero.<br>Check the switch heater is not open circuit.<br>Measure the magnet Start – End resistance from the current lead fischer connector. When the magnet is properly cold it should be < 0.4 ohms. |

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