

Superconducting Magnet system

Operator's Handbook

Supplied to: Dr Keifl
 TRIUMF
 Canada

Project number: 01247

Please quote this number whenever you contact the factory.

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1. Introduction

1.1. How to use this manual

Each of the main sections in the manual is separated from the others by a divider card. Use the divider card index which you see when you open the front cover or the table of contents to find the section that you want to read, quickly and easily.

Some diagrams are in a separate section. A few of these diagrams are folded so that you can pull them out and see them while reading the text which refers to them.

Additional manuals may be provided with your system to describe the details of some of the component parts. In particular, most electronic equipment is supplied complete with a manual, and you may need to refer to these separate documents to find out how to carry out some of the operations if you are not familiar with the equipment.

1.1.1. Safety

Warning It is your responsibility to ensure your own safety, and the safety of the people working around you.

Cryogenic fluids and high magnetic fields are potentially hazardous, and you must take precautions to ensure your own safety. The *Oxford Instruments* booklet *Safety Matters* has been included in this manual. It contains essential information and detailed recommendations about the precautions that you should take. Any additional information that applies specifically to your system is provided separately in the 'Safety' section of this manual.

1.1.2. Warnings

Please read this manual before assembling or commissioning the system. If you do not follow the correct procedures you might be injured or you might damage the system beyond repair. Oxford Instruments cannot accept responsibility for damage to the system caused by failure to observe the correct procedures laid down in this manual.

1.2. *Other manuals supplied with the system*

The following documents (marked ✓) are also supplied with the system.

Practical Cryogenics	✓
ILM200 family of cryogen level meters	✓
ITC502 temperature controller	
ITC503 temperature controller	
IPS120-10 superconducting magnet power supply	
PS120-3 superconducting magnet power supply	
ISS10 superconducting shim power supply	
SMC4 stepper motor controller	
Auto needle valve and Auto GFS for ITC502 and ITC503	
Data sheet for a PT100 thermometer	✓
RhFe reference scale 1.5 to 800K, for 3 point calibrated sensors (1985 revised to ITS-90)	
ObjectBench system control software	
DC-SQUID	
PHI-O RF-SQUID	

The following pages describe the system and give some information about the principle of operation. If you are experienced you may not need to read the Introduction in detail but you must read the warnings contained in it.

1.3. *Superconducting magnets*

The world's first commercial superconducting magnet was produced by *Oxford Instruments*, and now, more than 25 years later the company still leads the world, with fields higher than 20 T available. This technology allows customers to produce extremely high magnetic fields in laboratory scale cryostats without the kW to MW power supplies needed for non-superconducting magnets. In most cases the cost of refrigeration for a superconducting system is much less than the cost of the power required to run an equivalent resistive electromagnet.

The magnet consists of a number of coaxial solenoid sections wound using multi-filamentary superconducting wire. It is constructed using the Magnabond system, an integration of proprietary techniques developed by *Oxford Instruments*. It gives a structure which is both physically and thermally stable under the large Lorentz forces generated during operation.

Additional coils may be fitted to the basic windings to modify the shape of the field. 'Compensation coils' are often used to improve the homogeneity at the centre of field by reducing the rate at which the field drops at the ends of the coils (due to finite winding length effects). They are usually wired in series with the main coils so that they are energised with the magnet. 'Shim coils' (or shims) are used to remove residual field gradients; they may be wired in series with the main coils to give a basic level of correction or independently to give finer adjustment. Shims may be either cold superconducting coils or room temperature 'normal' coils. 'Cancellation coils' are often fitted to one end (or sometimes both ends) of a magnet to give a low field region quite close to the centre of field; for example < 10 mT (or 100 gauss) may be achieved over a region only 30 cm away from the centre of field of a 15 T magnet.

1.3.1. Persistent mode operation 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 1 part in 10^4 relative per hour is easily achieved in a typical small magnet, but this can be improved to 1 in 10^7 relative per hour for specific applications (for example, high resolution NMR spectroscopy). Persistent mode operation is achieved using a superconducting switch which is often fitted to the magnet in parallel with the main windings. The diagram on page 9 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 ohms, it is so much higher than that 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 a few tens of seconds the current in the magnet leads is slowly 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 gradually rises, until it carries the full current of the magnet.

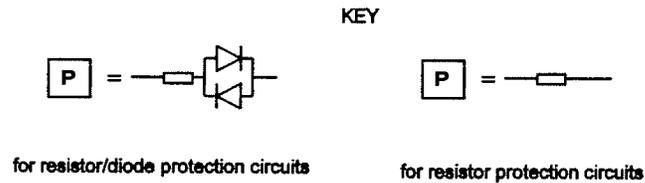
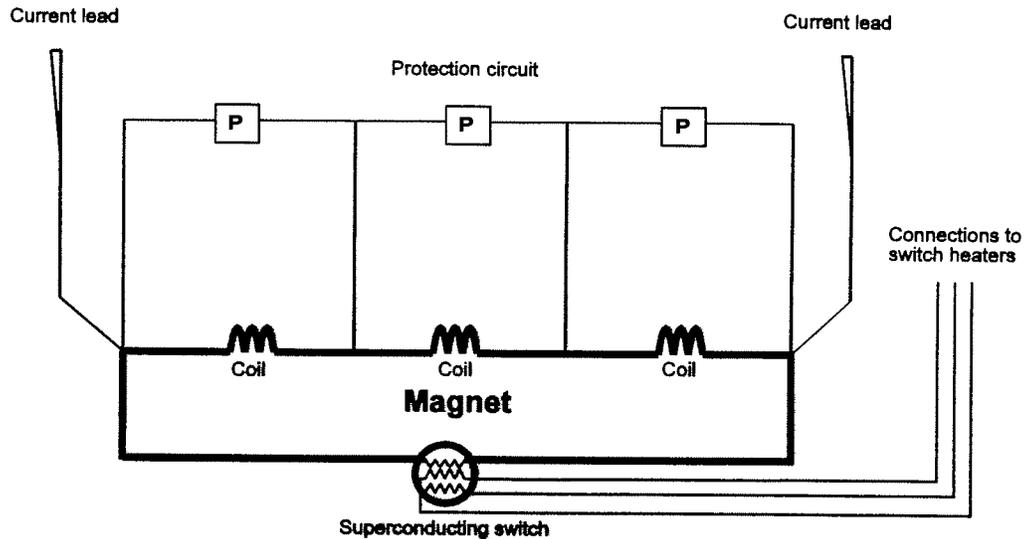


Figure 1 Simple resistor or resistor/diode protection circuit. (See the key at the bottom.)

1.3.2. 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 coil, and may spread into other parts of the magnet. All the stored energy in the magnet is dissipated rapidly, causing the liquid helium to boil off very quickly and often warming the magnet to a temperature significantly above 4.2 K. This is called a 'quench'.

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 a very large amount of stored energy. 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 refilling the cryostat with liquid helium. 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.

1.4. Low loss dewars for superconducting magnets

Oxford Instruments low loss liquid helium dewars are liquid nitrogen shielded. This means that there is a liquid nitrogen reservoir which cools a radiation shield surrounding the liquid helium reservoir. It is also linked to the neck of the helium reservoir to reduce the amount of heat conducted into the liquid helium.

The liquid nitrogen and liquid helium reservoirs are thermally isolated by using

- low thermal conductivity materials
- high vacuum chamber between the reservoirs and room temperature (OVC)
- multi-layer superinsulation

The dewar is vacuum insulated. The outer vacuum chamber (OVC) of the dewar will be fitted with a large diameter pressure relief valve at the base (or side) of the dewar. This ensures that it is not possible to build up a dangerously high pressure in the OVC.

Warning Do not tamper with this safety device or attempt to modify it.

1.4.1. Magnet support systems

Magnet support systems are used to provide mechanical support for a superconducting magnet and its current leads, protection circuit, and other accessories. It is built into the helium reservoir and there are no user serviceable parts.

The helium transfer tube (or siphon) entry port is used to transfer cryogens into or out of the liquid helium reservoir. During precooling the 'blow out tube' is inserted into this port. Below the port there is a 'siphon cone'. This is connected to the upper end of a tube which goes to the bottom of the helium reservoir. Plug the transfer tube or blow out tube into this cone if you want to transfer liquid to the bottom of the reservoir or blow out all of the liquid. This is particularly important when the liquid nitrogen is blown out at the end of the pre-cooling process and when liquid helium is transferred into the system to cool it to 4.2 K.

Warning All the service ports should be sealed when the system is cold, to prevent air from entering the system. If they are not being used fit the plugs that we have provided.

1.4.2. Magnet current leads

The magnet current leads are optimised to carry the maximum operating current of the magnet and introduce as little heat as possible to the liquid helium. In some systems the current leads are demountable. These can be removed to minimise the liquid helium boil-off while the magnet is in persistent mode, (but only at the expense of higher helium consumption while the current is sweeping).

1.4.3. Magnet protection circuit

The magnet protection circuit is used automatically in the event of a quench:

- to dissipate the energy stored in the magnet
- to make sure that high voltages are not produced.

Protection resistors (and diodes if appropriate) are provided for all magnet sections. The resistors are mounted in the helium reservoir. Diodes are used in the protection circuit to ensure that, all the current passes through the magnet under normal operating conditions so that the energisation current is proportional to the magnetic field. They also reduce the heat load from ohmic heating in the protection resistors and hence reduce system boil off while the magnet is sweeping. If the magnet quenches, the barrier voltage of the diodes is exceeded and the protection comes into operation automatically.

1.5. Fischer electrical connectors

High quality Fischer electrical connectors are used on most systems. These connectors have a self-locking mechanism to prevent the connection being accidentally broken if the cable is pulled.

Caution

Do not attempt to remove the connector by unscrewing the knurled black nut, as the wiring may be damaged. It is also likely that the nut maintains compression of a vacuum seal between the hermetic connector and the cryostat and that air will be admitted to a vacuum space.

To remove the Fischer connector from its mating part on the cryostat it is important to pull the correct piece. You will notice that part of the outside of the connector seems to be loose on the body of the connector. This is the locking mechanism. Pull this part away from the mating connector to break the connection. However, if you try to pull the connector out using the cable or another part of the body the connector and its mating part will remain locked together.

3. Assembly and thermometry

3.1. Unpacking the system

The system should be unpacked carefully and inspected for any damage that may have been caused during shipment from Oxford. It should also be checked to ensure that none of the components are missing. If any problems are encountered you should contact *Oxford Instruments* (through our agent or subsidiary if appropriate).

The dewar and other parts may be fitted with internal packing to prevent movement of the inner parts during shipment. If so, it will have a label on the outside to warn you, and to explain what has to be done to remove it. Keep these instructions and the packing in case you need to transport the system again in future.

3.2. Commissioning requirements for cryogenic systems

If you are planning to install a laboratory scale cryogenic system you are likely to need most of the following equipment. Some of it may be supplied with the system; other items may only be needed occasionally. If your system contains a superconducting magnet, ^3He refrigerator or dilution refrigerator there are additional requirements, and these are listed separately.

3.2.1. Safety equipment

- personnel protection equipment including gloves and goggles
- hazard warning signs to make sure that anyone approaching the system is aware of the potential hazards

3.2.2. Tools

- spanners or wrenches (open ended metric set). 5 to 19 mm
- Allen keys (metric set) 1.5 to 12 mm
- screw drivers, pliers, side cutters etc.
- hot air gun
- electrical soldering iron
- digital multimeter (with low current ohms range).

3.2.3. Lifting equipment

- suitable method of lifting the system from the delivery vehicle
- suitable hoist or crane for use in the laboratory
- lifting sling and shackles to suit the lifting points on the system

If you do not have access to lifting equipment above the position where you run the system you can use a trolley to transport the system to the hoist. It may be necessary to remove the system from the trolley when you are running it.

3.2.4. Vacuum equipment

- high vacuum pumping system to evacuate the insulating vacuum spaces, including a diffusion or turbomolecular pump and a liquid nitrogen cooled trap, flexible metal pumping lines for connection to the cryostat and a two stage backing pump. It should be capable of reaching a pressure of 10^{-6} mbar.
- a mass spectrometer leak detector system is required sometimes, especially when the system is commissioned, for routine leak testing operations.
- oil mist filters fitted to all rotary pump exhausts.
- a range of vacuum fittings (ISO KF fittings (also known as NW or DN) are used as standard)

Caution

It is important to remember that turbo-molecular pumps have a low compression ratio for helium gas. Therefore you should always use a two stage rotary pump as a backing pump.

3.2.5. Cryogenics and gas supplies

- liquid nitrogen in a self pressurising dewar
- liquid helium
- a supply of recovery grade helium gas with a regulator, at a pressure variable between 0 and approximately 1 bar gauge.

3.2.6. Consumables

- roll of mylar adhesive tape
- roll of aluminium adhesive tape
- tube of vacuum grease
- pair of cotton gloves for handling clean items
- 'Scotchbrite' or equivalent mild abrasive for polishing or removing old indium wire from joint faces.
- metal polish and degreasing agent or solvent for general cleaning.
- indium wire (1mm diameter)
- rubber soccer ball bladders (2 needed).
- assorted latex rubber and polythene tubing
- fishing line or dental floss

3.2.7. Other equipment

- helium transfer tube (or 'siphon')
- level meters for cryogen reservoirs (if required) or a suitable 'dipstick'
- suitable gas flow meters may be useful sometimes

3.3. Additional requirements for superconducting magnet systems

In addition to the items listed above, superconducting magnet systems typically have the following requirements.

- additional hazard warning signs, barriers or controlled entry systems appropriate for magnet systems
- a suitable power supply for the magnet and superconducting switch.
- if you have a helium recovery system in your laboratory it should be capable of handling (or safely releasing) the large amount of gas generated in the event of a quench.

3.3.1.1. If there are magnetic items in the floor (for example reinforcement in concrete floors)

- wooden or non-magnetic platform strong enough to support the system. It should typically be 25 cm high. (This may need to be higher for high field or high homogeneity systems.)

Note High homogeneity magnets are particularly susceptible to the presence of magnetic materials. The shape of the magnetic field can be altered significantly, and this may affect your experiment.

3.4. Assembling low loss dewars

Caution The cryostat must never be tilted away from the vertical. Internal parts could be permanently damaged, leaving the system inoperable.

The cryostat is delivered with the OVC already evacuated; therefore do not open the vacuum valve until you are ready to pump the OVC or check the vacuum. If possible the OVC should be kept under vacuum at all times. This helps to keep the superinsulation clean and minimise the boil off of the dewar.

Caution Do not evacuate the helium reservoir unless the OVC is already under vacuum. If you do, it will collapse and it may damage the magnet.

Caution Avoid venting the vacuum in the OVC if possible. If you have to vent the OVC take the following precautions:

- only vent the OVC when the dewar is completely warm
- make sure that the helium reservoir has already been vented to air
- vent it very slowly to avoid any risk of collapsing the helium reservoir or moving the superinsulation
- if there is a significant amount of helium in the atmosphere in your laboratory use dry nitrogen to vent the OVC.

If you do have to remove the base of the OVC make sure that you do not allow the superinsulation to become dirty. Avoid touching it with your bare hands, because grease and finger marks may affect the performance of the system. Clean all 'O' rings and check them for damage. Lightly grease them with vacuum grease before you re-assemble the dewar.

3.4.1. Inspecting the cryostat

The cryostat is delivered on a pallet. Examine the Tilt Watch and Shock Watch Indicators on the cryostat wrapping to determine whether the cryostat has been handled properly. If either is coloured, contact *Oxford Instruments*; internal damage may have occurred. All other components are in a separate packing case.

3.4.2. Removing the cryostat from the pallet

The cryostat is clamped to the wooden pallet by the base ring of the outer vacuum chamber (OVC).

- a) Unbolt the clamp plates.
- b) Unpack the legs of the cryostat.
- c) Connect lifting slings to the lifting points on the top of the cryostat OVC. Use a winch to lift the cryostat from the pallet, and support it so that it is safe for you to work underneath the cryostat. Bolt the legs to the cryostat and lower it to the ground.

3.4.3. Extracting the transit rods

Three transit rods are positioned on the base of the cryostat. These protect the cryostat's internal components during transport. They should now be released as follows.

Loosen the clamp ring around each transit rod. Rotate each transit rod handle anticlockwise to unscrew it from the base of the helium vessel. (Tip: if you feel too much resistance as you try to move the transit rods slightly loosen the bolts in the small flange that secures the transit rods to the OVC base.) When it is loose continue to turn it anticlockwise while you pull the transit rod about 100 mm out of the OVC (until they will come no further). Tighten the clamp ring on each transit rod. If extra clearance is required beneath the base of the cryostat these transit fittings may be replaced with the blanking plates supplied. However, the OVC vacuum must be vented to do this.

3.4.3.1. Systems with special packing

Some systems have removable internal packing. If so, there will be instructions on the outside of the cryostat. Keep these instructions and the packing in case you need to transport the system again in future.

3.4.4. Assembling the other fittings onto the cryostat

3.4.4.1. Assembling the helium manifold

The manifold and other fittings are shown on the general assembly drawing

- a) Check that the 'O' ring grooves in both stack assemblies are clean. Check that the 'O' rings are clean and lightly greased. Then fit the top-hat assemblies to the stacks, with the exhaust ports pointing in the direction shown on the drawing.
- b) Clean and lightly grease the 'O' rings and carriers that will seal the manifold to the top-hat assemblies, and seal the relief valve and non-return valve to the manifold. Ensure that all flanges on the manifold are clean.
- c) Fit the blue tube which connects the two top hat assemblies.
- d) Fit the pressure relief valve, non-return valve and helium bursting disc assembly to the manifold using their NW25 fittings.
- e) Tighten the top hat sealing nuts on each top-hat assembly by hand to form a gas-tight seal. Do not use a spanner (wrench).

3.4.5. Fitting the non-return valve to one of the nitrogen ports

At least one of the liquid nitrogen vessel ports must be fitted with a non-return valve while the dewar is cold. This ensures that it is not possible for all three ports to become blocked by moisture condensed from the air. Even if two of the ports are accidentally blocked the nitrogen gas is free to escape through the non-return valve. One of these valves is normally fitted in the factory, using a tamper-proof screw.

3.4.6. Evacuating the OVC

Check the vacuum in the OVC before you fill the cryostat with cryogenes.

Caution Always use a liquid nitrogen cold trap between a diffusion pump and the cryostat. Keep it full of liquid nitrogen during operation of the pump. The OVC could be seriously contaminated by back streaming diffusion fluid if this is not done. If a turbo-molecular pump is used instead of a diffusion pump, a liquid nitrogen cold trap is not essential, but it does help you to remove condensable vapours.

3.4.6.1. Checking the vacuum

If the cryostat is already under vacuum and you want to check the pressure in the outer vacuum chamber you can do so as follows. Pump the line up to the OVC valve with a diffusion pump (or turbo-molecular pump). Close off the diffusion pump and open the OVC valve. Read the pressure on a pressure gauge connected to the pumping line. If the OVC needs to be pumped you can now decide whether or not the pressure is too high for the diffusion pump. If so pump it with the rotary pump until the pressure is low enough.

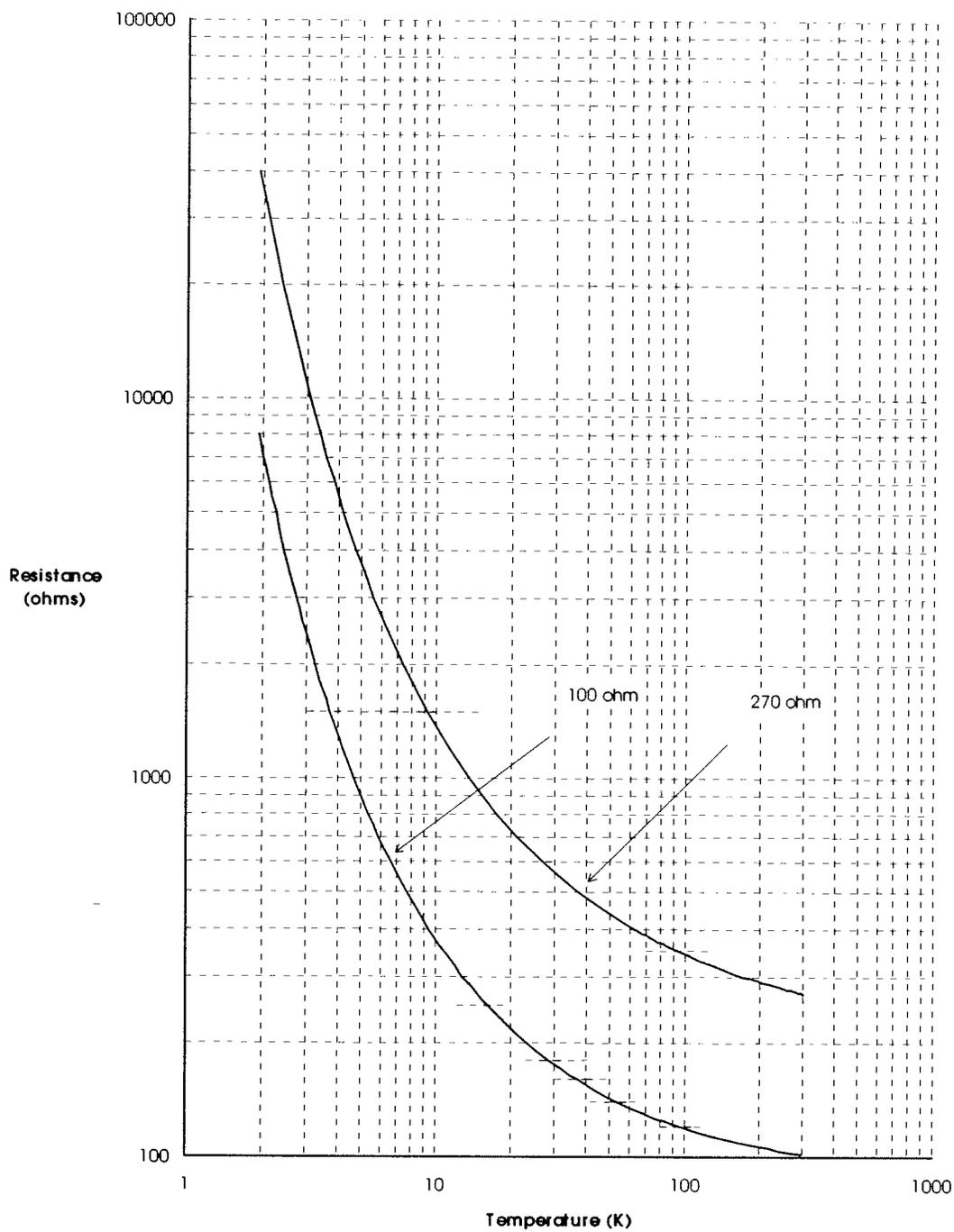
Before you use the dewar you should pump the OVC to high vacuum using a diffusion or turbomolecular pump system (fitted with a cold trap to collect condensable vapours – see Caution above). Even if the OVC has been left under vacuum since the last run the surfaces inside the OVC are likely to outgas, and the vacuum will not be sufficiently high. There may be a sorb in the OVC to help to maintain the vacuum while the system is cold, and pumping the OVC whenever the system is warm helps to keep it clean. If possible you should pump the OVC overnight (or longer), until the pressure at the pump drops to $< 5 \times 10^{-5}$ mbar.

3.4.6.2. Evacuating the dewar from atmospheric pressure

If the OVC has been vented with air or nitrogen gas use a rotary pump to evacuate the vacuum line, then slowly open the vacuum valve. (Tip: if the spring on the pressure relief valve on the base of the OVC has been stretched you may need to hold into in place until the vacuum holds it for you.) When the pressure is low enough start to pump the OVC with a diffusion pump or turbomolecular pump. It will typically take at least 24 hours to reduce the pressure in the OVC to $< 5 \times 10^{-5}$ mbar, as measured by a Penning gauge. Initial leak tests can then be carried out. If all is well, we recommend that you continue pumping for a further 48 hours and then conduct the leak tests for a second time.

3.5. Allen Bradley resistors as thermometers

Typical calibration curves of
100 and 270 ohm Allen Bradley resistors



4. Pre-cooling the system

4.1. Preparing a magnet for pre-cooling

4.1.1. Electrical checks

Check the wiring carefully using a multi-meter. Make sure that the resistance between all pins on the electrical connectors is as expected. The magnet windings should be electrically insulated from all the other wiring to greater than 1 M Ω .

Check the following:

- magnet to switch heater (if fitted)
- magnet to dewar (ground)
- magnet to all other diagnostic wiring

If supplied, the wiring diagrams for the magnet and its control wiring are given in the wiring section of this manual. If you require further details please contact *Oxford Instruments*.

4.2. Preparing liquid nitrogen shielded dewars for pre-cooling

4.2.1. Evacuating the OVC

Evacuate the outer vacuum chamber (OVC) of the dewar for at least 24 hours before you precool the system. It may be under vacuum already. If you want the optimum boil off performance it is important to pump the OVC with a high vacuum pump, not just a rotary pump. A 50 mm (or larger) diffusion pump fitted with a cold trap to collect condensable vapours is best because it pumps all gases well, (including helium). A turbomolecular pump with a cold trap (and backed by a two stage rotary pump) can be used but if there is any helium in the vacuum space it will take a long time to pump it away because these pumps have a low compression ratio for light molecules. Always use pumping lines which are at least 25 mm diameter and as short as possible. Do not use lines which have previously been used to carry helium gas.

It is possible to cool the system down without pumping the OVC with a diffusion pump but the system boil off is likely to be increased. We recommend that you always pump the OVC to a high vacuum before you cool down the system.

4.2.2. Pumping and flushing the helium reservoir

This operation is typically part of the leak testing procedure for other parts of the system, so refer to the other sections describing how to prepare the system before carrying it out. Sometimes it is necessary to pump and flush the helium reservoir to purge air from other parts of the system (for example to prevent the risk of blockages caused by frozen air or moisture).

Warning Do not pump on the main helium reservoir unless the OVC is already under vacuum. It may collapse if you do!

Disconnect the helium recovery system from the cryostat. Connect a rotary pump to the exhaust of the helium reservoir and pump it to a rough vacuum (typically 1 mbar) to remove the air and moisture. Fill the helium reservoir with helium gas. If you want to check that there are no leaks from the helium reservoir to the OVC before you pre-cool the system you can do it as you vent it with helium gas.

4.2.3. Precautions to be taken before pre-cooling

- fill the liquid helium vessel with liquid nitrogen before pre-cooling the liquid nitrogen jacket
- make sure that at least one port on the liquid nitrogen jacket is fitted with a non-return valve

4.3. Pre-cooling the system

Make sure that you have carried out the preparations described for each part of the system before you start to pre-cool it. These are described in the other pages of this section of the manual.

Warning *Practical Cryogenics* gives some background information about transferring liquid nitrogen. Refer to it if you are unsure of the correct procedures. It is also important to be aware of the correct safety procedures, as described in the booklet *Safety Matters* which is included in the Safety section of this manual.

Disconnect the main helium bath from the helium recovery line (if you have one in your laboratory). Some systems have to be pre-cooled slowly to make sure that they are not damaged by thermal shock. If so, precautions are given in the description of the preparations that you should carry out before pre-cooling the system. Insert the liquid nitrogen "blow out tube" through the siphon port. If there is a siphon cone in the system it is best to push the blow out tube into it but it is not essential.

Connect a suitable tube from the top of the blow out tube to a liquid nitrogen storage dewar and transfer liquid nitrogen into the main bath. Fill the helium reservoir with liquid nitrogen and leave the cryostat to pre-cool. It may take several hours to pre-cool the system, depending on the type of system. This can conveniently be performed overnight.

Always wait until the liquid nitrogen has stopped boiling violently.

Caution: The OVC can be pumped during the pre-cooling procedure as long as there is a cold trap between the pump and the cryostat to prevent oil backstreaming. However, we advise that it should be isolated from the pump before the helium transfer is started.

5. *Cooling the system to 4.2 K*

Please read the whole of this section and make sure that you understand it before you proceed. This is possibly the most difficult part of the operation of the system because to do it most efficiently you have to carry out several operations at the same time. For example, you can carry out leak tests on several components together while the helium reservoir is pumped and flushed with helium gas.

5.1. *Preparing liquid nitrogen shielded systems for operations at 77 K*

Insert the liquid nitrogen blow out tube into the siphon port. If there is a siphon cone on your system push the blow out tube into it, and if there is a thread on the blow out tube screw it into the siphon cone. Connect the top of this tube to one of the nitrogen jacket ports on your dewar.

You can use this liquid to fill the liquid nitrogen jacket of the dewar until it is full. If it overflows, stop blowing out the liquid nitrogen, connect the blow out tube to a separate storage dewar and blow the rest of the liquid into it.

5.2. *Blowing out the liquid nitrogen*

Blow the liquid nitrogen out of the main bath using a slight overpressure of helium gas supplied through the exhaust port. 200 mbar should be sufficient. When all the liquid nitrogen has been removed withdraw the blow out tube and insert the bung in the siphon port.

You can see that liquid nitrogen is no longer being blown out of the helium reservoir by observing the following signs:

- the pressure drops in the main bath
- the flexible part of the blow out tube is no longer vibrating noticeably
- the metal part of the blow out tube nearest to the cryostat is no longer wet on the outside
- the plume of gas from the receiving vessel may change in character

If you are not planning to pump and flush the helium reservoir as described below it is wise to wait until the resistance of the Allen Bradley resistors (if fitted) drops by one or two ohms from the 77 K value measured when the reservoir was full of liquid nitrogen. This ensures that the reservoir has warmed slightly above 77 K and confirms that all the liquid nitrogen has been removed. Re-connect the main bath recovery line.

If you do not have a pressure gauge covering the range from 0 – 1000 mbar continue to blow warm helium gas through the helium reservoir for another 5 minutes to make sure that all the liquid nitrogen has been removed properly.

5.3. *Checking liquid nitrogen shielded dewars at 77 K*

5.3.1. *Leak testing the OVC*

If you want to check that there are no leaks from the liquid helium reservoir to the OVC you can do this by observing the helium signal in the OVC while the helium reservoir is filled with helium gas. Most cold leaks can be detected at 77 K, so there is little risk of a leak developing as the system is cooled to 4.2 K. However, if you have used the system without problems for a few weeks you may feel confident enough to run it without further testing. Close the OVC valve after you have completed the leak tests.

5.3.2. *Filling the liquid nitrogen jacket*

Fill the liquid nitrogen jacket with liquid nitrogen until it overflows.

5.3.3. *Preparing for the liquid helium transfer*

Fit a non-return valve to at least one of the liquid nitrogen jacket ports, and either non-return valves or Bunsen valves to the other ports. This will ensure that air is not condensed into the tubes during the liquid helium transfer, causing dangerous blockages.

5.4. *Pumping and flushing the helium reservoir*

It is wise to pump and flush the helium reservoir of your system (through the exhaust port) to carry out leak tests or to make sure that the liquid nitrogen has been removed completely. Complex systems with small capillary tubes could be blocked or superconducting magnets may be affected by frozen nitrogen.

If you are planning to test the system for leaks you can do many of the tests while you pump and flush the helium reservoir. Read the leak testing section for all the other parts of the system before you carry out this procedure.

Monitor the Allen Bradley resistors in the helium reservoir (if fitted) while you pump the reservoir. If you see their resistance rising as the pressure drops this is an indication that the liquid nitrogen has not been thoroughly removed, and you must try again to blow it all out.

Pump out the helium bath using the auxiliary pump to ensure that no liquid is left. The pressure should fall steadily to about 1 mbar. If this does not happen (for example, the pressure hesitates at 100 mbar) it indicates that the liquid has not all been removed. Vent the main bath to atmospheric pressure with helium gas, make sure that the blow out tube reaches the bottom of the helium reservoir, and try again to blow out any remaining liquid.

If you want to make sure that there is no liquid nitrogen in the reservoir after this process is complete, wait until the resistance of the Allen Bradley resistors drops by one or two ohms from the 77 K value measured when the reservoir was full of liquid nitrogen. This ensures that the reservoir has warmed slightly above 77 K and confirms that all the liquid nitrogen has been removed.

5.5. *Cooling systems to 4.2 K*

Liquid helium has a very low latent heat of evaporation but the gas has high enthalpy. This means that it is very easy to evaporate the liquid but it is difficult to warm up the gas so produced. Liquid helium therefore has to be transferred very carefully. If you do not transfer it properly you may lose all the liquid from your storage dewar without collecting any in your system. Follow these instructions to get an efficient liquid helium transfer.

When you are cooling down a system to 4.2 K it is very important to transfer the liquid helium to the lowest point in the helium reservoir. If the system is warmer than 4.2 K the liquid boils almost immediately as it leaves the vacuum insulated transfer tube (or siphon). Very little cooling is obtained from this evaporation. However, this gas then has to pass over the equipment in the helium reservoir to reach the exhaust line, and this provides very useful cooling power. If you transfer the liquid helium into the system slowly you can make sure that the gas emerging from the exhaust line is not too cold. This ensures that you do not waste any cooling power. If you do transfer the liquid too quickly you may see liquid air running from the recovery line, indicating that the cooling power is being wasted.

5.5.1. *Preparations for the helium transfer*

Check that the leg lengths of the transfer tube are suitable. The storage dewar leg should be able to reach below the liquid level (and preferably reach the bottom of the dewar). The system leg should be able to reach the lowest point in the helium reservoir (or the siphon cone if one is fitted). Position the liquid helium storage vessel so that the transfer tube can be easily inserted to both the storage dewar and the system, and blow some helium gas through the transfer tube to remove the air.

Remove the non-return valve from the exhaust port of the helium reservoir.

If you have a helium recovery system, connect the exhaust line of the cryostat's helium reservoir and the storage dewar to it. It is important to make sure that the impedance of the recovery line is low enough to allow an efficient helium transfer. The recovery line should be at least 25 mm diameter. Contact your recovery system administrator for advice if you need it.

If you do not have a recovery system, make sure that the exhaust is free to vent but that there is no risk that the system will be filled with air condensed from the atmosphere. You can do this by connecting a flexible line a few meters long to the exhaust port. Let the other end lie on the floor. The helium in this line is lighter than air and tends to prevent air from rising to the exhaust port. However, when the helium transfer is complete, or if the system is to be left open to air for more than a few minutes, you should put a one way valve on the cryostat exhaust port.

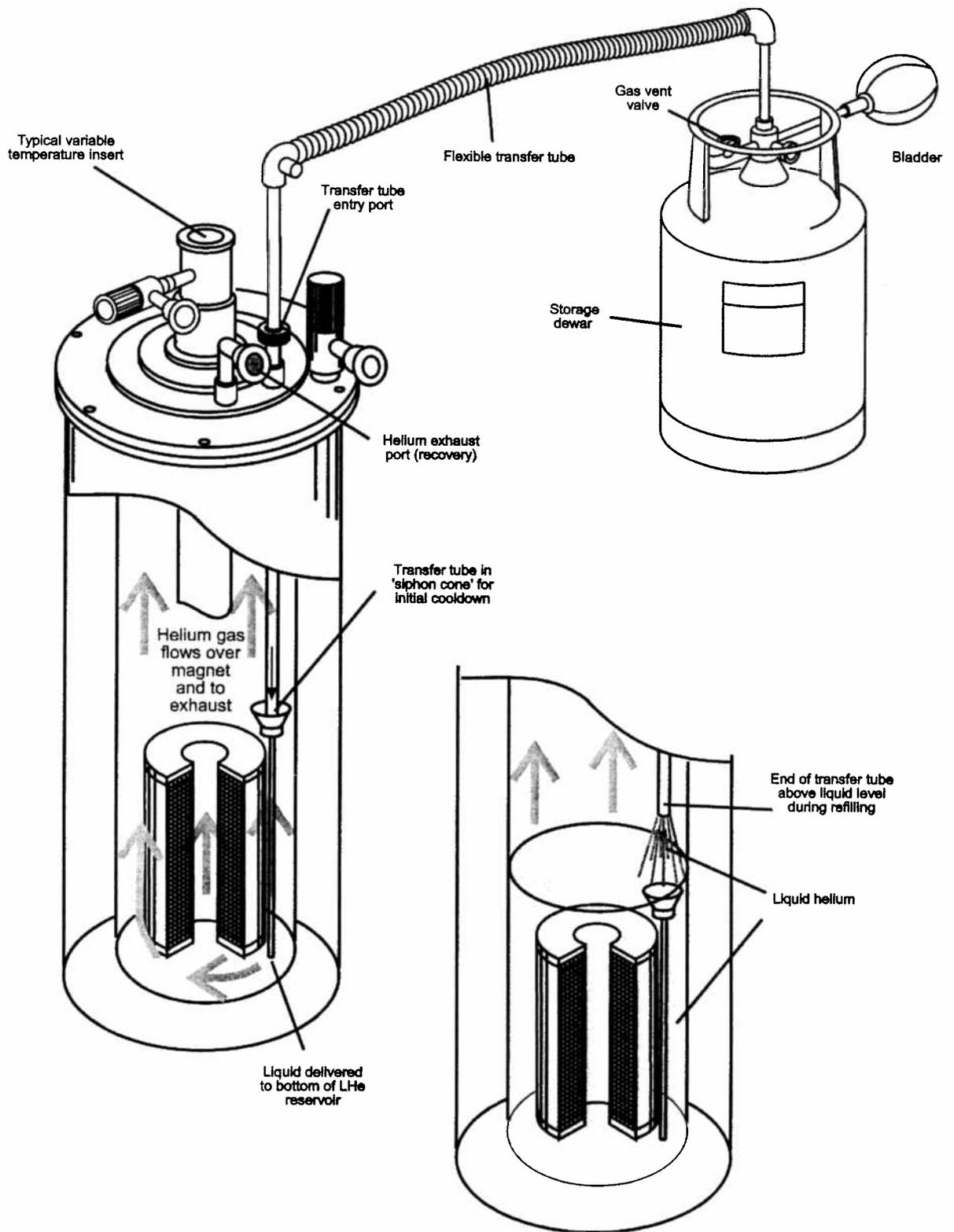


Figure 2 Transferring liquid helium into a typical laboratory cryostat.
Your system may not look like the one shown in the diagram.

5.5.2. *Transferring liquid helium*

Remove the plugs from the system's transfer tube entry port and the top of the storage vessel. Insert the transfer tube legs into the system and into the storage dewar slowly, allowing the dewar leg to cool gradually. Make sure that the end of the transfer tube in the cryostat reaches the bottom of the helium reservoir (or the siphon cone if fitted).

Close the exhaust line on the storage dewar and pressurise it slightly to start the liquid transfer. (This is generally done by gently squeezing a rubber bladder). The transfer rate should be such that the vent pipe is frozen for not more than 2 m (6 ft.) of its length. The initial transfer rate should be equivalent to between 4 and 10 litres of liquid per hour.

Close the OVC valve on the cryostat. There is no need to continue to pump it.

When liquid starts to collect in the helium reservoir the exhaust gas flow rate will be seen to drop noticeably (as the ice on the recovery line starts to melt). The pressure on the storage dewar can then be increased to transfer the liquid more quickly.

When the liquid helium reservoir has been filled, stop the transfer by releasing the pressure in the storage vessel. Remove the transfer tube and replace the bungs.

The booklet *Practical Cryogenics* contains a list of solutions to the problems commonly encountered in liquid helium transfers. Refer to this booklet if you are having problems.

Caution Remember to replace the non-return valve on the helium reservoir exhaust.

6. *Running the system*

6.1. *Running the magnet at 4.2 K*

6.1.1. *Introduction*

Many different types of power supply are available to run the magnet. The magnetic field is proportional to the current supplied to the magnet. The voltage available from the power supply determines the maximum sweep rate.

The current range of *Oxford Instruments* superconducting magnet power supplies (including the IPS120-10 and PS120-3) work in 'current control mode' with a voltage trip. This means that if the power supply's output voltage reaches the hardware voltage limit the power supply will trip and go into the HOLD state. The current will be kept constant in this state.

You can set a sweep rate limit in software to suit the maximum allowable sweep rate for the magnet or to prevent the output voltage reaching the hardware limit during normal operation. If the power supply reaches this limit it will reduce the sweep rate accordingly and a 'limiting' warning light will come on to indicate that the magnet is not sweeping at the set rate.

Older power supplies or those from other manufacturers work in different modes and a brief description of operation in 'voltage mode' is given later.

A magnet power supply is typically used to carry out the following operations:

- to supply power to the superconducting switch heater to open the switch (if fitted)
- to energise the magnet to the required field (or current) and hold it there
- to sweep the magnet to a set field at a defined rate
- to put the magnet into persistent mode if a constant field is required (so that the power supply can be switched off) and only if the magnet has a superconducting switch.
- to change the field direction by reversing the polarity of the current (not applicable to PS120-3)
- to run a programmed series of sweeps and holds

Power supplies can be run manually or under computer control. *Oxford Instruments* can supply software packages which are used to program the system. ObjectBench allows you to carry out a sequence of operations, so that experimental results can be taken automatically. Alternatively, Teslatron software provides a simplified user interface for the system through National Instruments LabView®, and you can write sequences of operations for the whole system and experimental apparatus.

Some magnets can be run to higher fields if they are cooled to 2.2 K. They are usually cooled to this temperature by a lambda point refrigerator (or by pumping the whole helium reservoir with a rotary pump). If your magnet is not specified to run to higher currents at 2.2 K do not attempt to energise it above the 4.2 K field. If you do this you could cause permanent mechanical damage to the magnet and invalidate the warranty.

6.1.2. Checking the system before operation

Before you run the magnet you should make the following electrical checks.

- magnet resistance
- magnet to cryostat isolation
- switch heater resistance
- switch heater to cryostat isolation
- magnet to switch heater isolation

Compare resistance values with the quoted values in the wiring section of the manual. Isolation values should typically be $> 1 \text{ M}\Omega$ unless otherwise stated.

6.1.3. Preparing to run the magnet

If you have bought a magnet and power supply together these instructions briefly explain how to run them. However, if you are planning to run a new magnet on a different type of power supply you may have to use another mode of operation (as described later). These instructions cover the basic principles of running the magnet. If you need to find other information refer to the manual supplied with the magnet power supply.

Most magnets are fitted with a superconducting switch. If a switch is fitted to the magnet the liquid helium consumption during a field sweep is increased slightly, because of the dissipation in the switch heater and resistive heating in the switch. However, if you plan to leave the magnet at a constant field for a long time the helium consumption is greatly reduced by putting the magnet into persistent mode.

If there is no switch on your magnet just ignore the steps that refer to the switch in the following text. This is an advantage if you only want to sweep the magnet continuously and do not want to leave it at a fixed field for long periods.

Ground the cryostat effectively so that it cannot accidentally reach a dangerously high voltage in the event of a failure in the insulation if the magnet quenches. Before you connect the power supply to the electricity supply, connect the magnet current leads and the superconducting switch heater lead to the terminals on the back of the power supply.

Warning Before you run the magnet make sure that you have taken the necessary steps to ensure your own safety and the safety of other people working around you. Refer to the booklet *Safety Matters*, which is included in the Safety section of this manual.

Connect the leads to the cryostat magnet terminals and fit the rubber boot over the connector. Connect the switch heater lead to the appropriate connector. Switch on the magnet power supply.

Warning Do not disconnect the power supply from the magnet while it is at field unless your system is fitted with special demountable current leads. You may be putting your life at risk (because of the chance of an electric shock) and you may damage the coil.

6.1.4. Energising the magnet from zero field

6.1.4.1. Preparations

The power supply will initialise by displaying the software version, then zero. The output is 'clamped' when the power supply is first switched on. Press the HOLD button to unclamp it and put the power supply into HOLD mode.

Make sure that there is sufficient liquid helium in the system to ensure that the magnet is still covered with liquid at the end of the field sweep.

Select the display mode that you require (in amps or tesla) by pressing the button labelled CURRENT/FIELD. The relationship between the current and field for your magnet and the appropriate sweep rates can be set in software. If you bought the power supply with the magnet this should have been set up in the factory and does not need to be adjusted. However, some magnets have to be run at slower sweep rates the first time they are energised after cooldown or after transport. See the test results for details.

You can set the power supply to run the magnet either to a 'set field' or to a 'set current' depending on the display mode that you chose earlier. Press and hold the SET POINT button and use the RAISE and LOWER buttons on the ADJUST panel to select the set point. You can set the 'sweep rate' in a similar way by pressing and holding the SET RATE button and using RAISE and LOWER. On IPS120-10 power supplies you can also choose the polarity of the current supplied to the magnet, and thus the direction of the field.

Caution The current for maximum field and the recommended maximum sweep rates for your magnet are given in the magnet data sheet supplied in the test results section of this manual.

Tip You can change the SET RATE while the magnet is being energised (if necessary). Energise the magnet in one of the following ways:

- a) run the power supply from a computer and program the changes of sweep rate
- b) choose the SET POINT to be the value that you want to reach at the end of the sweep and change the sweep rate as the magnet is energised
- c) or set sweep rate limits for the power supply (for different current ranges) so that the sweep rate changes automatically as the magnet sweeps.

If the magnet has a superconducting switch, press the HEATER ON button (on the SWITCH HEATER panel). The power supply makes several checks to ensure that it is safe to run the magnet and if it finds no problems the switch heater light is illuminated immediately.

If you have not bought a complete system it may be necessary to adjust the switch heater current to suit the magnet. Set it to the value given in the test results. Note that the heater current may have to be increased slightly if you run the magnet at a lower temperature.

If the power supply finds a problem the light will not come on. Check that everything is properly connected and that there is no reason not to energise the switch. Typical problems include:

- the power supply thinks that the magnet is already at field
- the switch heater is not connected properly

If you decide to override the power supply's checks, press and hold the HEATER ON button for about five seconds until the indicator light comes on. Wait for 15 seconds to make sure that the switch is open.

6.1.4.2. Energisation

Start to energise the magnet by pressing the GOTO SET button (or the SET POINT button on PS120-3 power supplies) on the SWEEP CONTROL panel. The current or field will start to change on the digital display. The output voltage will vary with the sweep rate, the inductance of the magnet, and the resistive voltage drop in the current leads.

When the power supply reaches the set point wait until the output voltage reaches a steady value. If your protection circuit is of the resistor/diode type (and the superconducting switch has a relatively high normal resistance) this will happen quickly. Resistor protection circuits have a longer time constant and it may be necessary to wait for a minute or longer.

6.1.5. Establishing persistent mode

Turn off the switch heater by pressing the HEATER ON button again. Wait for about 30 seconds for the switch to become superconducting. Press the ZERO button on the SWEEP CONTROL panel. The current in the magnet leads will decrease to zero leaving the magnet in persistent mode. The leads can be swept faster than the magnet, and the power supply software automatically runs the leads up or down at a higher rate (typically 240 amps/minute).

6.1.6. Taking the magnet out of persistent mode

The magnet can be taken out of persistent mode by using the following procedure. This assumes that you are using the same power supply and that none of the settings have been changed since you put the magnet into persistent mode. The power supply will remember the settings even if it has been turned off.

Press the SET POINT button on the SWEEP CONTROL panel. The current leads will be swept quickly to the Set Point value. Turn the switch heater current 'on' by pressing the HEATER ON button. The PS120-3 and IPS120-10 carry out various checks before the switch heater is turned on. Normally you will not notice this happening, and the switch heater light will light up when you release the button. Wait for about 15 seconds.

If the switch heater does not come on immediately, the power supply thinks that the current it is supplying does not match the current in the magnet. Check to make sure that you have done everything properly. If you decide that the current in the leads matches the current in the magnet, (and is in the same direction), you can override the power supply's checks. Press and hold the HEATER ON button for about five seconds until the indicator comes on. (However, note that if you wrongly override the power supply in this way it is possible that you will quench the magnet.)

6.1.7. Running the magnet to a new set point (or running it down)

If you want to change the field, press and hold the SET POINT button and RAISE and LOWER to change the set point to the new desired value. IPS120-10 also allows you to choose a set point of the opposite polarity. Choose the sweep rate and press the GOTO SET button (or SET POINT button on PS120-3) on the SWEEP CONTROL panel and the magnet sweeps to the new set point.

When the magnet has reached the new set point and the voltage has stabilised you can turn off the switch heater to put it into persistent mode again.

6.1.8. *Running the magnet down*

If you want to run the magnet to zero field press the ZERO button on the SWEEP CONTROL panel. The magnet will start to run down at the rate defined . The sweep rate can be changed without stopping the sweep (if required).

When the current has run to zero, wait for about 1 minute, or until you are sure that the voltage across the magnet terminals has also dropped to zero. Turn off the persistent mode switch by pressing the switch heater button

Turn off the PSU by pressing the power button.

6.1.9. *Running the magnet in constant voltage mode*

6.1.9.1. *Introduction*

If you are not using one of the current range of *Oxford Instruments* power supplies you may have to run the magnet in constant voltage mode. It is difficult to control the sweep rate accurately or to automate operation of the system.

The magnet is swept to a set current (or to zero) at a constant voltage, measured at the power supply terminals (or sometimes at the magnet itself using a 'four wire' type of measurement technique where the voltage is sensed through an additional pair of leads). There are two components to the voltage that the power supply must provide:

- the resistive voltage drop in the current leads (which varies with the current)
- the induced voltage due to the inductance of the magnet (which varies with sweep rate)

If you use a four wire measurement it is possible to eliminate the resistive voltage drop from the measurement so that you can set a constant sweep rate. You can then neglect the steps taken to measure the resistive component of the voltage in the instructions that follow.

6.1.9.2. *Preparation*

Prepare the system as described in the Preparation section above.

6.1.9.3. *Energising the magnet in constant voltage mode*

With the switch heater off, sweep the power supply to the required current manually. Measure the voltage at the power supply output terminals. This is the resistive voltage drop in the magnet leads. If you want the magnet to reach the set current it is necessary to set an energisation voltage higher than this value.

Sweep the power supply back to zero amps.

Turn the superconducting switch heater on and wait for 15 seconds for the switch to open.

Turn the positive voltage setting to a value higher than the resistive voltage drop measured above. The next section describes how to choose the right voltage. The higher the voltage you set the faster the magnet will sweep to field. Do not exceed the maximum sweep rate recommended for your magnet.

Allow the power supply to sweep the magnet to field. Turn the switch heater off and wait for 30 seconds. Run down the leads manually to leave the magnet in persistent mode.

6.1.9.4. Choosing the voltage for the required sweep rate

You can calculate the induced voltage corresponding to the required sweep rate from the following equation.

$$\text{Induced voltage} = -L \frac{dI}{dt}$$

where dI/dt is the sweep rate in amps per second and L is the inductance of your magnet.

Neglect the negative sign from this calculation. If you want to sweep the magnet up add this value to the resistive voltage measured earlier. If you want to sweep the magnet down, subtract it from the resistive voltage.

6.1.9.5. To take the magnet out of persistent mode in constant voltage mode

Run up the leads to the set current manually. Turn the switch heater ON and wait for about 15 seconds.

Set an appropriate negative voltage for the rate at which you want to run the magnet down. Sweep the power supply back to zero amps. When the voltage has dropped to zero you can turn off the switch heater.

6.2. *Leaving the system unattended*

6.2.1. *Running the system unattended*

If you plan to leave the system to run unattended you must take the following precautions. Remember that it is your responsibility to make sure that no one is put into danger by the system. Read and learn the contents of the Safety section of this manual and take appropriate actions.

- erect suitable warning signs to prevent tampering by other people
- try to make sure that only competent people have access to the system
- make sure that there are sufficient cryogenics in the system
- arrange for the cryogenics to be re-filled if necessary
- connect the exhaust of the helium reservoir to a recovery system or fit an appropriate one way valve to prevent air or moisture from entering
- make sure that the system can vent safely, even if it is accidentally warmed up or pumps stop running unexpectedly
- leave a telephone number so that you can be contacted in an emergency
- make sure that there is sufficient ventilation in the laboratory to avoid a potential asphyxiation hazard when you return

If there are any closed volumes that are pumped during normal operation make sure that they are free to vent either into the cryostat reservoirs or through the pumping line. If there are valves in the pumping line and on the inlet to these volumes make sure that you do not leave them both closed.

6.2.2. *Leaving the system static*

If you are not using the system for a few days (for example over the weekend) it is often possible to close it down and leave it in a static condition. This could save liquid helium or reduce some of the potential hazards associated with the system. To leave a typical system in static mode:

- de-energise the superconducting magnet
- close down the lambda point refrigerator (if one is fitted) and vent it safely
- close down any variable temperature insert, Heliox insert or Kelvinox insert

6.3. *Re-filling the liquid helium*

When the liquid helium level drops close to the minimum working level you should carefully re-fill it. When you refill the liquid helium you should take care to pre-cool the transfer tube thoroughly before you put it into the system. Otherwise the warm gas passing through the tube will evaporate liquid in the helium reservoir. The booklet *Practical Cryogenics* contains a list of practical solutions to the problems commonly encountered in liquid helium transfers.

Important note This describes the easiest method of transferring liquid into a cold system for beginners. However, some laboratories have strict rules about recovering all helium gas. If you have a helium recovery system ask the administrator to show you the preferred method of transferring helium.

Caution If your system contains a superconducting magnet:

- make sure that the liquid helium level does not drop below the minimum level shown on the drawing while it is energised.
- run down the magnet, if in doubt
- beware of the stray magnetic field while you are working close to the cryostat.

Some transfer tubes are supplied with special fittings for refilling the liquid helium. These fittings are screwed onto the end of the transfer tube and divert the gas and liquid from the transfer tube up and away from the liquid surface. The gas passes out of the cryostat and the heavier liquid falls into the reservoir.

6.3.1. Pre-cooling the transfer tube (or siphon)

Prepare the storage dewar and transfer tube as described in the section about "Cooling systems to 4.2K". Insert one leg of the transfer tube into the helium storage vessel, but leave the other leg outside the cryostat. Unscrew the cryostat 'siphon entry' fittings (the O-ring and the knurled nut) and slide it onto the leg of the transfer tube which will go into the cryostat. Put the bung loosely in the transfer tube entry port on the system to prevent gross contamination with air. Pressurise the transport dewar slightly, in the normal way. After about 20 seconds you should hear oscillations in the tube, gradually increasing in frequency and intensity. When these stop you should see white vapour and when liquid starts to emerge you may see a white cone (like a gas flame).

6.3.2. Transferring the liquid helium

If you have a rigid transfer tube quickly release the pressure in the transport dewar, lift the transfer tube and insert the open end into the cryostat. If you have a transfer tube with a flexible section it is easy to do this without releasing the pressure or moving the leg in the storage dewar.

Push the transfer tube into the system to approximately the maximum helium level. Do not push it to the bottom of the helium reservoir or into the siphon cone (if there is one on your system).

Caution Do not push the transfer tube below the maximum helium level if you have a superconducting magnet in the system. You may quench the magnet.

Quickly increase the pressure in the storage dewar again. It is most efficient to transfer the liquid quickly to reduce the losses in the transfer tube. However, 200 mbar is usually sufficient pressure to do this.

7. *Warming up the system*

7.1. *Warming up the system - liquid nitrogen shielded dewars*

7.1.1. *Preparations*

Before you start to warm up the system you must make sure that it is safe. The Safety section of this manual gives some guidelines.

Make sure that there are no trapped volumes of liquid, gas or condensed solids inside the system. You may not know that they are there if they have accidentally been condensed into the system while it has been cold. Therefore you must make sure that all closed volumes are free to vent or that they are pumped continuously as the system warms up.

Close down any other parts of the system. In particular if your system contains any of the following items prepare them properly.

- superconducting magnets must be de-energised
- lambda point refrigerators must be closed down and pumped out (and pumped continuously during warm-up) or vented to the main helium reservoir
- variable temperature inserts, Heliox inserts or Kelvinox inserts must be closed down and vented (or pumped continuously during warm-up)

7.1.2. *Allowing the system to warm naturally*

When you have prepared the system you can leave it to warm up naturally. When the cryogens have all evaporated the system will warm slowly to room temperature. If you do not need to use it again soon this is the easiest way to warm the system up.

7.1.3. *Warming the system quickly*

If you want to warm up the system more quickly you have to blow out the cryogens and break the insulating vacuum in the outer vacuum chamber.

The liquid helium can be blown out of the system either into a storage vessel for use elsewhere or into a helium gas recovery system. Liquid nitrogen can be blown into a storage vessel or disposed of safely. The system will then begin to warm up.

If possible you should avoid warming the system more quickly than this. However, if it is essential that it is warmed up more quickly the best way is to wait for the helium reservoir and its contents to warm to above 65 K and then vent the OVC slowly with clean dry nitrogen gas. Use a volume of gas smaller than the volume of the OVC. Make sure that the OVC is free to vent safely through a non-return valve in case it contains more gas than you think.

8. Background information

8.1. Making indium seals

Oxford Instruments uses two main types of indium seal, as illustrated in the diagram below. They both use 1mm diameter wire, retained

- either in a groove by a flat surface
- or in a corner between two flanges

In both cases, the indium wire is overlapped by bending one end of the wire sharply outwards and laying the other end across the corner of the bend. The wire is so soft that the joint will be compressed into a cold weld.

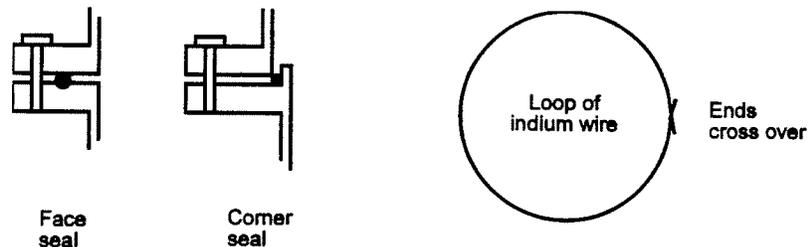


Figure 3 Indium seals

8.1.1. Preparations

Before you make the seal ensure that the groove and the mating surfaces are clean. Thoroughly remove any old indium wire from the seal faces. If necessary a solvent can be used for cleaning. Some people like to grease the metal surfaces with silicone vacuum grease to make it easier to remove the wire later, but this is not necessary.

8.1.2. Making the seal

Lay a new piece of indium wire in the groove or round the male spigot on one of the flanges and overlap it as shown on the diagram. There are usually alignment marks on the flanges to indicate the correct orientation. Carefully bring the two flanges together and hold them loosely in place with two bolts while you put the other bolts into the flanges and tighten them by finger only. Slowly and evenly tighten all of the bolts with a small spanner (wrench) or Allen key. Do not tighten them too much. There is no need to use an extension on the tool to give extra leverage. On large seals (typically > 50mm diameter) it is then best to leave them for about an hour. The indium flows slightly during this period so it is often possible to tighten the bolts slightly more.

8.1.3. *Separating indium seal flanges*

It is often difficult to separate indium seal flanges because the indium metal seems to glue them together. Most large indium seals made by *Oxford Instruments* have two or more threaded holes in one of the flanges for 'jacking screws'.

Remove the bolts that hold the indium seal together (leaving two of the bolts loosely in place so that the flanges do not fall apart when they separate). Use another two of these bolts to jack the flanges apart by screwing them evenly into the jacking screw holes from the same side of the flange. This will push the flanges apart.

If there are no jacking screw holes (as often happens on small diameter indium seals), the flanges can be separated by inserting a sharp blade between the flanges. Make sure that the blade does not slip and cut you as the flanges separate.

8.2. *Emergency run down procedure for magnets*

If it is not possible to run the magnet down conventionally using the magnet power supply it is possible to do it safely by dumping the energy into a pair of high power diodes. This might be necessary:

- if you cannot remember the polarity of the current in the magnet when it is in persistent mode
- if you cannot remember the current in the magnet when it is in persistent mode
- if no power supply is available

Warning Never touch the current lead terminals while the magnet is at field. The protection circuit is built to prevent the development of high voltages in the event of a magnet quench, but it is not good practice to rely on it.

Choose a pair of high power diodes capable of carrying the full operating current of the magnet and fix them to an adequate heat sink. Remember that the magnet stores a very large amount of energy so the heat sink must be well cooled. Connect a pair of diodes across the terminals as shown in the figures below. Activate the switch heater using either the power supply or a separate 6 volt battery. The switch heater current required is given in the test results section of this manual. The magnet will run down at a rate determined by the forward voltage drop of one of the diodes. The de-energisation will be slow, (for example, typically about 100 minutes using silicon diodes). Do not disconnect the diodes before the magnet is completely de-energised.

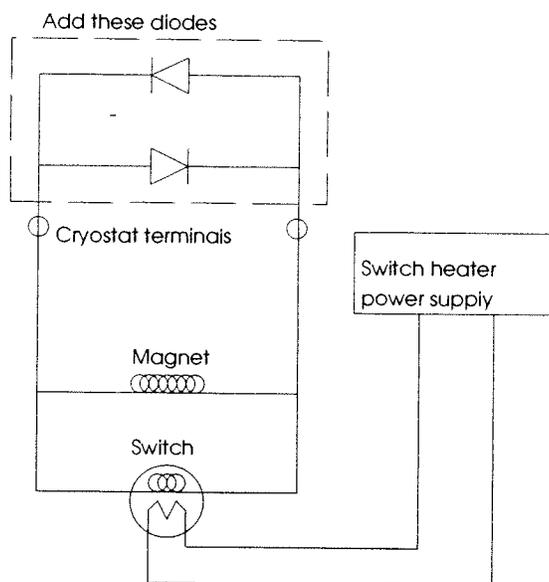


Figure 4 Emergency de-energisation circuit diagram

8.3. Cryostat fault finding

Symptom	Possible cause	Solutions
Poor vacuum in OVC	Leak on pumping system	Close the cryostat OVC valve and check pumping system base pressure.
	Leak on dewar or insert	Obtain a mass spectrometer leak detector and identify the source of the leak. The booklet <i>Practical Cryogenics</i> gives advice on this subject.
	Excessive moisture in OVC	Pump and flush the OVC with dry nitrogen several times, then pump to high vacuum again.
Condensation or frost on the OVC when the system is cooled down	Poor vacuum in the OVC	Pump the OVC again. Check with a mass spectrometer leak detector for leaks including leaks from the helium reservoir.
Transfer tube gets frosty	Poor transfer tube vacuum	Pump its vacuum space to high vacuum again.

Symptom	Possible cause	Solutions
Transfer tube shows ice "spots"	Internal capillary touches outer tube	If the transfer tube is still under warranty and it has not been not damaged contact us for a replacement. Otherwise consider replacing the transfer tube if the liquid helium consumption is unacceptably high.
Difficulties transferring liquid helium into the system.		See the chapter on this subject in the booklet <i>Practical Cryogenics</i> .

8.4. *Helium recovery systems*

Helium gas recovery systems are often used to collect the exhaust gas from cryostats. They are useful for the following reasons:

- to allow the gas to be liquefied and recycled
- to collect gas for other uses (for example vacuum leak detection)
- to prevent air from entering and contaminating the cryostat
- to conserve the Earth's helium supply.

A typical recovery system consists of a low pressure gas collector, a compressor and high pressure gas cylinders to store the gas. Many different cryostats are usually connected to a central low pressure gas collector. The recovery system typically has non-return valves at strategic points to make sure that the cryostats do not interact, and the system operates slightly above atmospheric pressure to reduce the risk of contaminating the gas if there is an air leak. The compressor should be specifically chosen for use with helium because a large amount of heat is generated when it is compressed.

Many factors affect the financial implications of building and using a helium recovery system. In particular it is important to consider:

- the cost of liquid helium in your laboratory
- the cost of installing and running a recovery system and liquefier

If you do use a recovery system you should take precautions to make sure that you recover as much gas as possible and avoid contaminating the gas with air or other substances.

8.5. *Operating the Room Temperature Bore heater*

$$R = 34.4 \Omega \quad (\text{Feb 02})$$

The room temperature bore is fitted with a Thermocoax heater and two PT100 temperature sensors. A suitable voltage/current source is required to operate the heater and a suitable DVM is required to measure the temperature sensors. The temperature sensors are wired to enable 4-way measurement although 2-way measurements can be made with reasonable accuracy. The heater is not fitted with a thermal fuse so it is important not to overdrive the heater. The heater is guaranteed to 150 degrees celsius and must not exceed 180 degrees celsius as permanent damage may be caused. The bore tube bake out should be done after pre-cool (see section 4.3) with **both the N² and He⁴ cans nitrogen filled**. Bakeout should never be done without nitrogen as this protects the magnet from radiated heatloads. Also the bore should always be under vacuum to prevent oxidisation. During test conditions at Oxford the bore tube temperature was held constant at 150 degrees celsius constant temperature with 55.5V/50Hz Ac/1.6A

8.6. *Useful reference books*

The following books may be found useful as background reading.

Experimental Techniques in Low Temperature Physics,
by G.K.White, Oxford University Press, ISBN 0-19-851381-X

Experimental Principles and Methods below 1 K,
by O.V.Lounasmaa, Academic Press, ISBN 0-12-455950-6

Low Temperature Laboratory Techniques,
by A.C.Rose-Innes,
London: English Universities Press, ISBN 0-34004778-X
(Probably out of print, but worth looking in the library).

Properties of Materials at Low Temperature, A Compendium.
General Editor Victor J. Johnson, National Bureau of Standards.
Pergamon Press, 1961.

Vacuum Technology its Foundations Formulae and Tables
Leybold Heraeus GMBH.

Superconducting Magnets
Martin N. Wilson,
Clarendon Press, Oxford, 1983, ISBN 0-19-854805-2.

Eléments de Cryogénie,

R.R. Conte (in French).

Masson & Co, Paris, 1970. (Probably out of print, but very useful).

Experimental Techniques in Condensed Matter Physics at Low Temperatures.

Robert C Richardson and Eric N Smith,

Addison Wesley Publishing Company Inc, 1988, ISBN 0-201-15002-6

Matter and Methods at Low Temperatures

Frank Pobell,

Springer Verlag, 1992, ISBN 0 540 53751 1 and 0 387 53751-1

Practical Cryogenics

An Introduction to Laboratory Cryogenics.

N.H.Balshaw, Oxford Instruments Ltd, 1996.

Introduction to Thermometry below 1 K

(A review of the available techniques)

Oxford Instruments Ltd., Ultra Low Temperature Group, 1990.

9. Drawings

The following drawings are included. Some of these drawings have been folded so that you can see them while you are reading text elsewhere in the manual.

General assembly drawing of the system, PRA4583

Also included:

Equipment return form (for use if you have to return any equipment to the factory).

Equipment return form (ERF)



An ERF must be filled in for any goods returned to the factory.

Please call our Customer Support Group to obtain a 'Return number', complete the declaration and return it before you dispatch the equipment. Enclose a copy with the equipment, (along with a standard proforma detailing the number of items, gross weight, and value for customs if you are outside the UK). If we have not received one of these forms work may be delayed.

Return Number R	Project number or Order number
Description of the equipment being returned to Research Instruments: Serial number:	
Please attach a detailed list of all items returned to the factory, (including electrical leads, vacuum fittings etc.)	
Why are you returning the equipment?	
Is the equipment under warranty? Yes/No	
Does the equipment contain or has it been contaminated by hazardous materials? Check box if YES Radioactive <input type="checkbox"/> Chemically active <input type="checkbox"/> Biologically Active <input type="checkbox"/>	
If so, what are the hazardous materials?	
What appropriate decontamination has been carried out?	
Please attach a certificate which verifies that decontamination has been completed.	
Any other relevant information	

Declaration

I declare that I have supplied complete and accurate information on this form and that the returned components are safe to handle.

Name Position.....

Organisation Fax number

Signature..... Date.....

We will use this information to protect the health of our employees. If any of the information is false, this may lead to a claim for subsequent damage or personal injury, and you may be held liable. If you are in any doubt about the information we require, please tell us.

10. Specifications, wiring and test results

10.1. Magnet services - control wiring

10.1.1. Earthing the cryostat

Before you run the magnet you must make sure that the cryostat is firmly earthed using a low resistance cable. During normal operation the magnet and its protection circuit are electrically isolated from the cryostat so there is little danger of the cryostat reaching a high voltage. However, if the magnet quenches and the electrical insulation fails at any point the cryostat could reach a dangerously high voltage, causing a hazard.

10.1.2. Magnet current leads

Single current terminals or coaxial pairs are provided for the magnet current leads. Attach the room temperature current leads to these and fit the rubber boot over the leads to make sure that you cannot accidentally touch the exposed terminals. The current leads are optimised to give the best possible electrical and thermal performance, and they are cooled by helium gas from the main reservoir of liquid helium.

Current terminal pairs are wired as follows:

Centre or red terminal = +ve = start of magnet

Outer or black terminal = -ve = end of magnet

Warning Do not modify the wiring in any way.

Do not disconnect the power supply from the magnet while it is at field. On most systems the power supply must be connected to guarantee that the voltages at the magnet terminals are kept at a safe level.

10.1.3. Protection circuit

Warning A superconducting magnet must always be fitted with a protection circuit, otherwise the magnet may be permanently damaged.

If the magnet is supplied as part of a complete system this protection circuit will have been installed on the magnet support system and it is only necessary to connect it when you assemble the system. Magnets supplied independently have to be protected as specified in the protection circuit diagram. Magnets which are welded into cryostats have had their protection circuits fitted in the factory and no access is possible.

10.2. 4 and 10 way Fischer connector pin labels

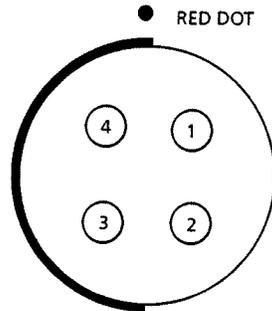


Figure 5 Pin numbers on a 4 way hermetically sealed Fischer connector viewed onto pins from the outside of the cryostat. Fischer part number 103 Z053 (DBEE).
(Mating connector Fischer part number SE103 Z053)

Tip The connector on the cryostat is a plug and the pins are accessible.

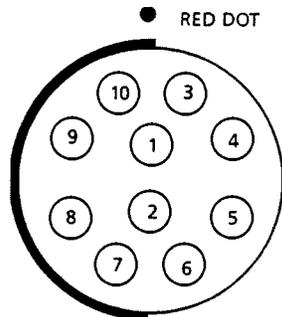


Figure 6 Pin numbers on a 10 way hermetically sealed Fischer connector viewed onto pins from the outside of the cryostat. Fischer part number 1031 Z010 (DBEE).
(Mating connector Fischer part number SE1031 Z010)

Tip The connector on the cryostat is a plug and the pins are accessible.

10.3. Fischer connectors for magnet systems

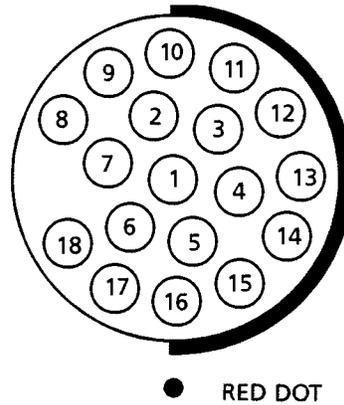


Figure 7 Pin numbers on an 18 way hermetically sealed Fischer connector viewed onto pins from the outside of the cryostat. Fischer part number.105 Z038 (DBEE).
(Mating connector Fischer part number SE105 Z038)

Tip The connector on the cryostat is a plug and the pins are accessible.

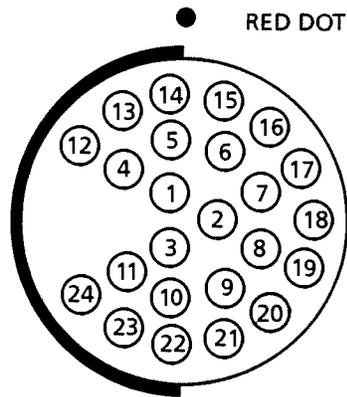


Figure 8 Pin numbers on a 24 way hermetically sealed Fischer connector viewed onto pins from the outside of the cryostat. Fischer part number 105 Z093 (DBEE).
(Mating connector Fischer part number SE105 Z093)

Tip The connector on the cryostat is a plug and the pins are accessible.

10.4. Flying leads for Fischer connectors

Leads attached to Fischer connectors by *Oxford Instruments* are colour coded according to the following convention.

1	Red	11	Turquoise	21	Blue/Black
2	Blue	12	Grey	22	Orange/Blue
3	Green	13	Red/Blue	23	Yellow/Green
4	Yellow	14	Green/Red	24	White/Green
5	White	15	Yellow/Red		
6	Black	16	White/Red		
7	Brown	17	Red/Black		
8	Violet	18	Red/Brown		
9	Orange	19	Yellow/Blue		
10	Pink	20	White/Blue		
				Body	Screen

Note that leads with less than 24 wires are colour coded in the same way. If you have a 10 way lead you should simply ignore the information given for pins 11 to 24.

10.5. Test results

The system test results are given after this page.

Superconducting Magnet

Description

Magnet type S9/132/15

Persistent mode switch fitted.....Yes

Type of protectionResistor Diode

Specifications and measured values for the magnet are combined in the following tables.

Main coils

Guaranteed maximum central magnetic field at 4.2 K	9 tesla
Current for full 4.2 K field	89.534 amps
Field / Current ratio	9.948 tesla/amp
Homogeneity (Radial over 5mm Radius)	20 PPM
Homogeneity (Axial over 10mm)	3 PPM
Magnet clear bore diameter	101.68 mm
Nominal inductance	69.639 henries
Switch heater current for open state	80 mA
Current decay in persistent mode (Better than)	1 part in 10^{-7} per hour

Energisation rates

Slow rates for use after thermal cycles

Energisation Current (A)		Energisation Rate	(tesla/minute)	Temperature
From	To	(amps/minute)		
0	65	6	0.60312	≤ 4.2 K
65	80	3	0.30156	≤ 4.2 K
80	89.534	1	0.10052	≤ 4.2 K

Fast rates

Energisation Current (A)		Energisation Rate	(tesla/minute)	Temperature
From	To	(amps/minute)		
0	89.534	6.96375	0.7	≤ 4.2 K

The field may be swept up or down and in either direction at these rates.

Persistent mode

Maximum rate of change of current in the magnet leads with the magnet in persistent mode (switch heater off) 240 amps/minute

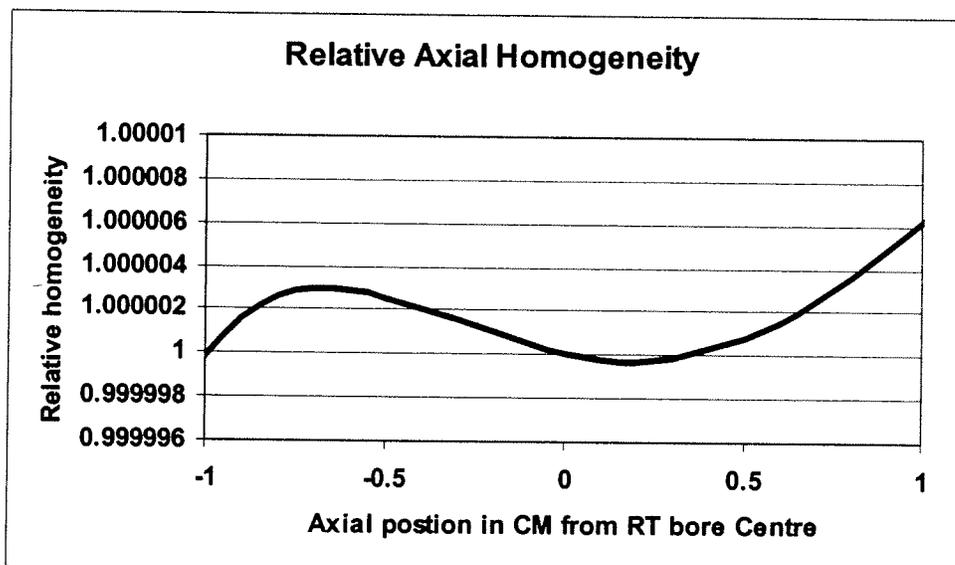
Oxford Instruments power supplies have a default rate of 240 amps/minute

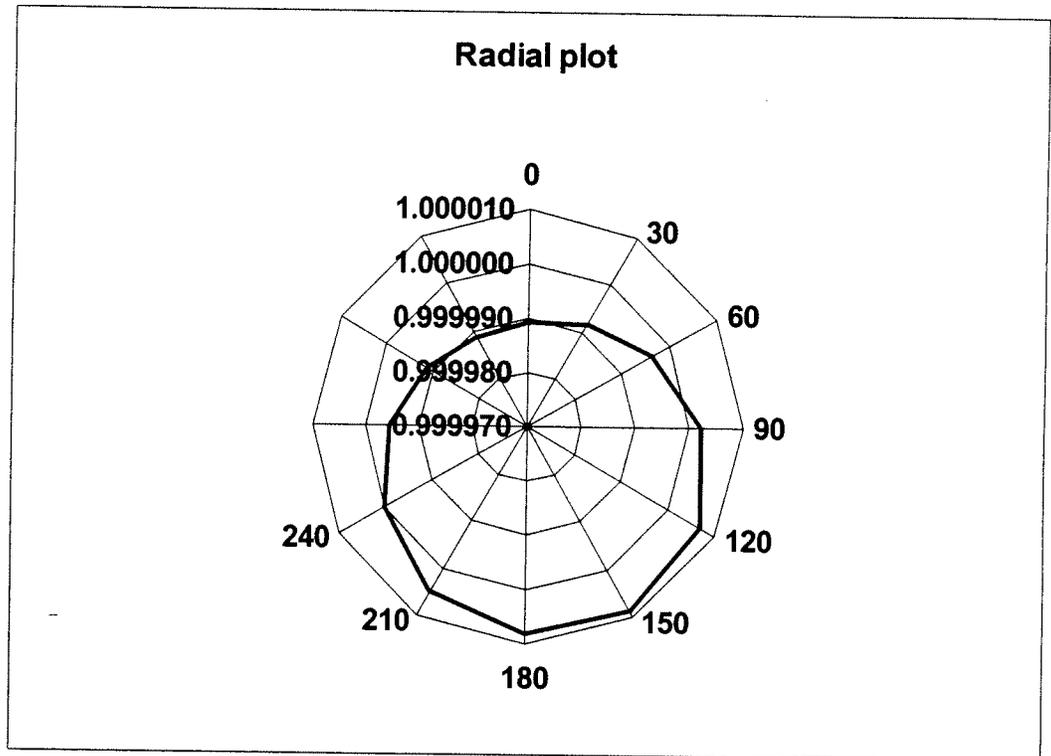
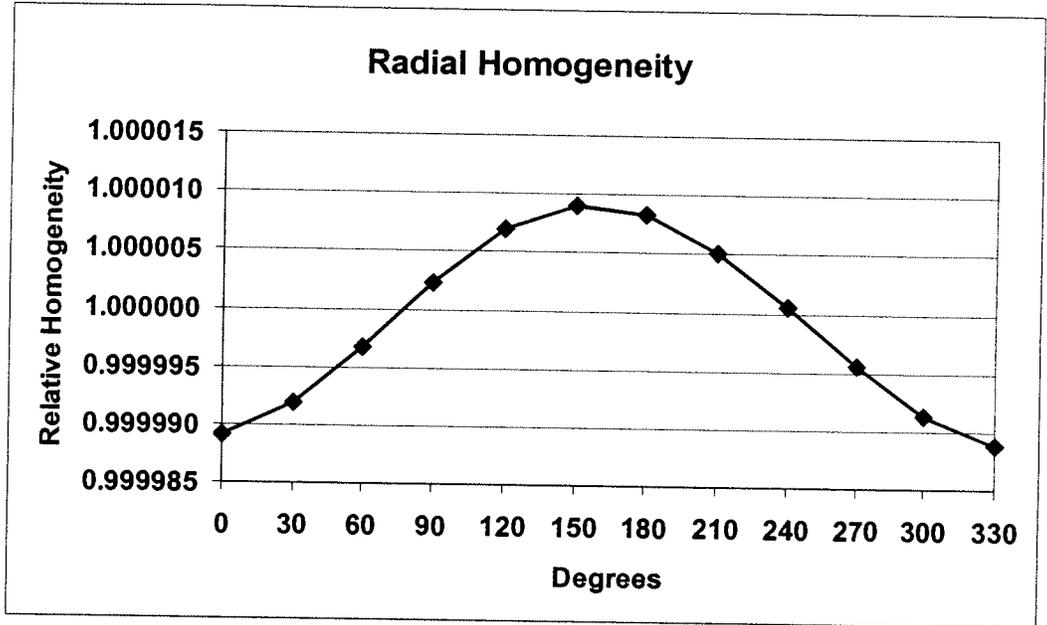
Measured magnet resistance values (ohms)

Temperature	Room temperature	77 K	4.2 K
Main magnet resistance Start-End (switch connected)	47.2	38.2	0.1
Switch heater resistance	117	115.7	108.3
Spare switch heater resistance	117	115.4	108.1
Switch heater(s) to cryostat isolation	∞	∞	∞
Magnet to cryostat isolation	∞	∞	∞
Main magnet to switch heaters isolation	∞	∞	∞
Allen Bradley on Magnet $\delta + G$	346	428	5.16K

Field plots

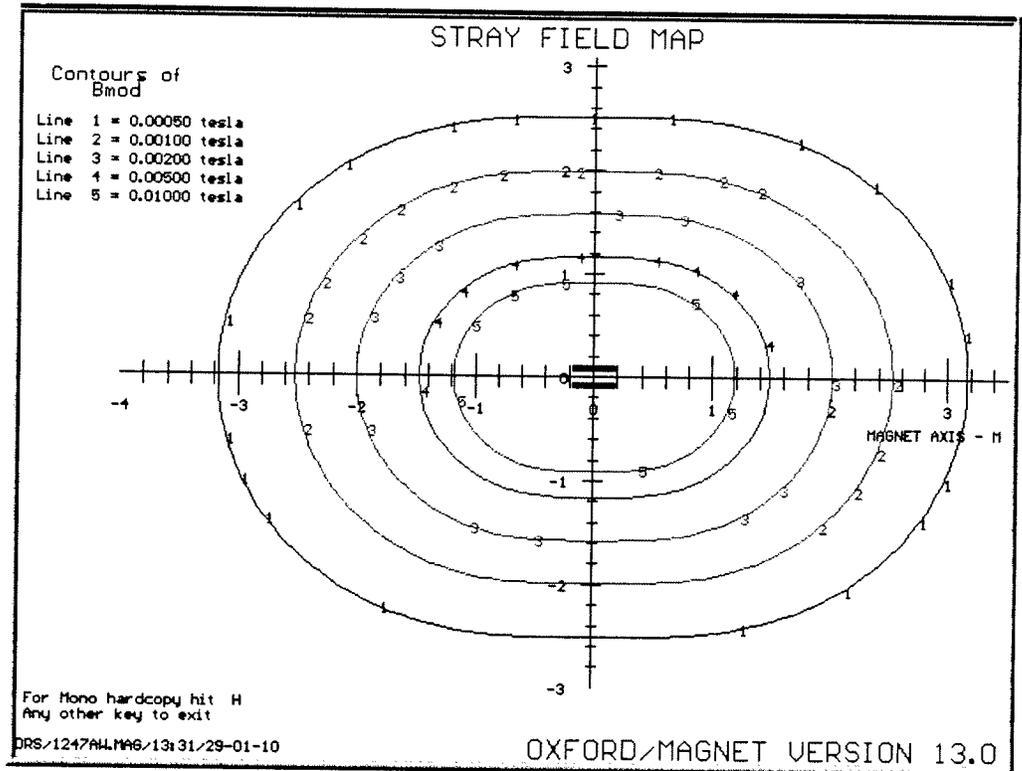
The magnet was plotted with an NMR probe in a test cryostat which contains a set of room temperature insert tails. The NMR probe uses a Deuterium source.





Note: The 0° reference is the 12oclock position on the cryostat.

Stray field plots



Dewar

Specifications (static conditions)

Dewar type

Liquid helium reservoir evaporation rate 90 cm³/h

Liquid nitrogen evaporation rate 450 cm³/h

Dewar test results

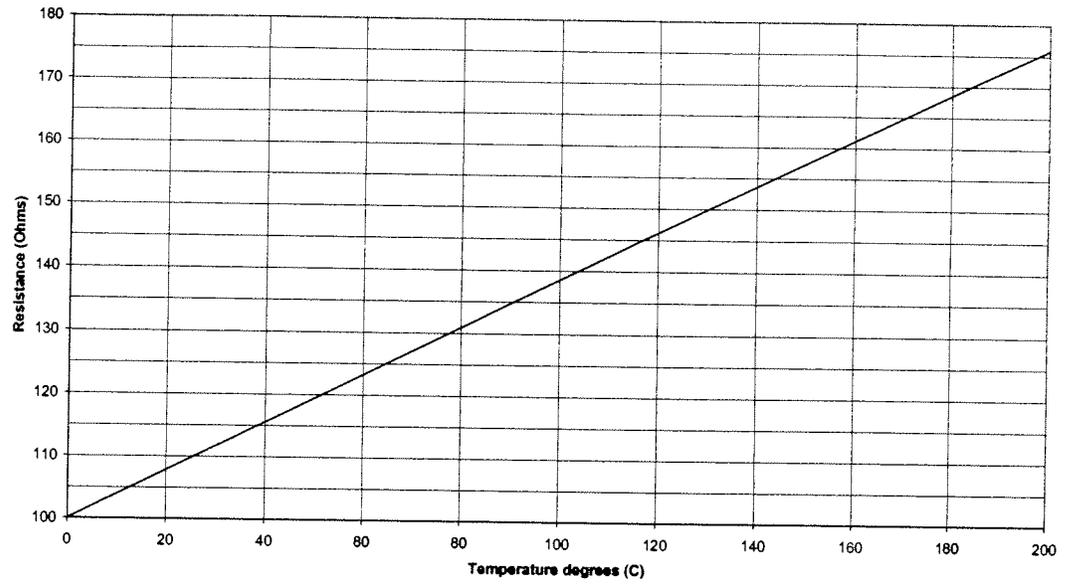
Useful liquid helium volume	68 litres
Liquid helium evaporation rate (static) at 82.4 %	56 cc/hour
Liquid helium evaporation rate (static) at 59.6%	65 cc/hour
Total consumption with full current in the leads	224 litres/hour
Intial cool down volume used to fill to 100%	120 litres

Liquid nitrogen volume	50 litres
Liquid nitrogen evaporation rate	837 cc/hour

Heated bore tube operation

Heater resistance at room Temperature (20°C)	36 Ohms
Voltage required to maintain 150°C approx with N2 in helium bath	52 Volts
PT 100 main @ room temperature (20°C) main bath filled with N2	113.9 Ohms
PT 100 spare @ room temperature (20°C) main bath filled with N2	113.7 Ohms
Ambient temperature in RT bore at 20°C	9.5 °C
Voltage required to maintain 15°C @ room temperature (20°C) with He in main bath	10 Volts

Temperature vs Resistance of a PT100



Weight and dimensions of the system

Approximate weight of the system

<300 kg

Minimum ceiling height requirement for helium transfer

m

