Operator's Handbook

Spectromag

Superconducting magnet system for optical spectroscopy

Revision 2

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1. Important Information

1.1. Safety

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

Danger: **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 with 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.**

1.2. Warnings

Before you attempt to install or operate this equipment for the first time, please make sure that you are aware of the precautions that you must take to ensure your safety. In particular, please read the 'Safety' section of this manual.

Caution: Please read this manual carefully before assembling or commissioning the system. It is possible to damage the system beyond repair if the correct procedures are not followed.

Caution: 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.

1.3. Important Note

This manual is part of the product that you have bought. Please keep it for the whole life of the product and make sure that you incorporate any amendments, which might be sent to you. If you sell or give away the product to someone else, please give them the manual too.

1.4. Important Health and Safety Notice

Important Health and Safety Notice

When returning components for service or repair it is essential that the item is shipped together with a signed declaration that the product has not been exposed to any hazardous contamination or that appropriate decontamination procedures have been carried out so that the product is safe to handle.

1.5. Conventions used in this manual

The following conventions have been followed in this manual:

Danger:	**Indicates that the hazard may cause death or severe injury if the instructions are not followed carefully.**
Warning:	**Indicates that the hazard may cause injury.**
Caution:	Indicates that the hazard may cause damage to equipment.
Note:	Something that needs to be brought to the customer’s attention.
Tip:	Indicates a helpful hint that may be of use to the customer.

1.6. Disposal and recycling instructions

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

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

2. Introduction

2.1. 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.

2.2. Stray Magnetic Field and Siting Issues

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.

2.2.1. 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 nonsymmetric 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.

2.2.2. 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.

2.2.3. 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. 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 5 kg iron mass centred on the 1000 G/m line will exert a force = 5 x 1000/500 = 10 kgf = approx. 100 N. However, note that this is only a guideline.

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

2.2.4. Field Limits for Devices (Unshielded Magnet Systems)

The following can affect the system and should be positioned outside 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

2.3. 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.

2.3.1. 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. Figure 1 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.

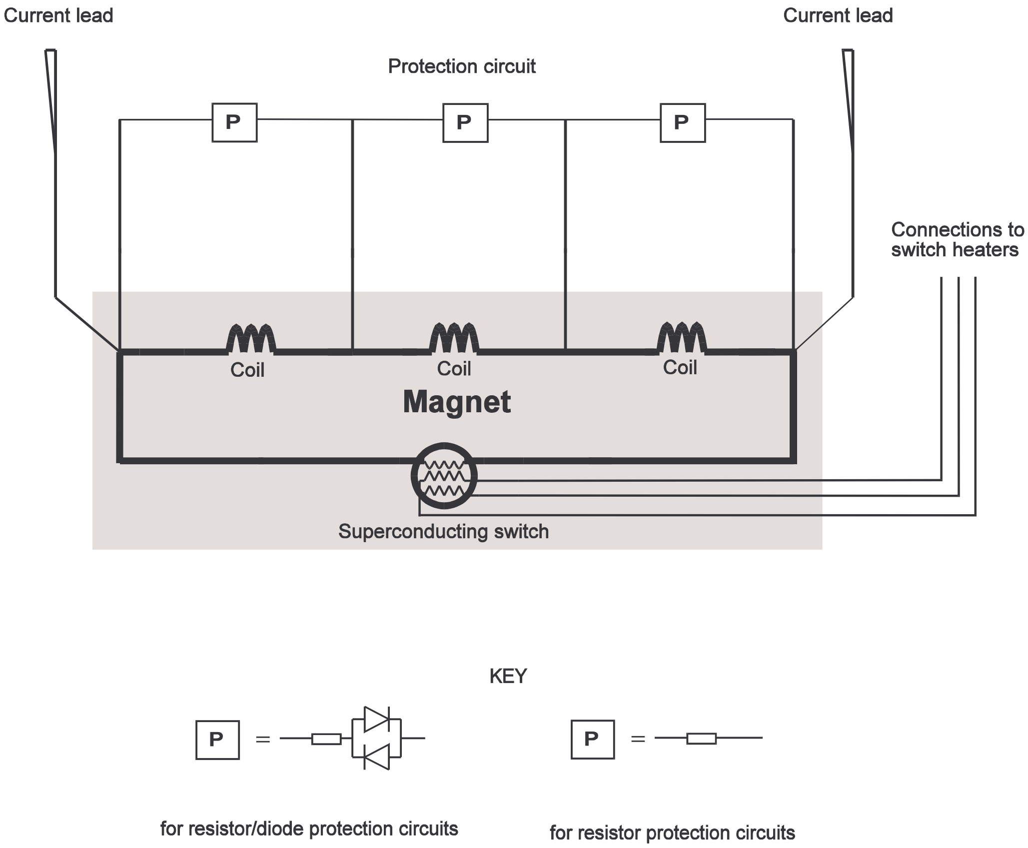


Figure 1 Simple resistor or resistor/diode protection circuit (see the key at the bottom).

2.3.2. 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.

2.4. Lambda point refrigerators

2.4.1. Enhanced performance of the magnet at 2.2 K

The magnetic field produced by a superconducting magnet is proportional to the current supplied. The maximum current that can be supplied to the magnet is typically limited by

- Electrical and magnetic limitations in the conductor (which are affected by the temperature of the magnet)
- Mechanical limitations of the magnet design

Oxford Instruments magnets are usually designed so that the field is limited by the performance of the conductor, not by the strength and stability of the magnet and this should make it difficult to damage the magnet accidentally.

Warning: **If you exceed the guaranteed field of your magnet you do so at your own risk. Observe any warnings given in the test results or elsewhere.**

Some magnets are designed to run at 4.2 K and their performance cannot be enhanced by cooling them to lower temperatures. However, the magnet supplied in your system can be energised to a higher field if you cool it to approximately 2.2 K. The test results give details of the current/field ratio and the maximum guaranteed field at 4.2 and 2.2 K.

2.4.2. Cooling the magnet to 2.2 K

The magnet is cooled by reducing the temperature of the liquid helium around it. This can be done by:

- Pumping the whole bath to reduce the vapour pressure of the liquid
- Cooling the liquid around the magnet with a lambda point refrigerator (LPF).

2.4.2.1. Pumping the whole liquid helium bath

If the whole bath is pumped to a pressure of 50 mbar with a rotary pump all of the liquid will be cooled to about 2.2 K. Although it is simple to set up a pumping system to do this for a short experiment there are many disadvantages. It is wasteful to cool the whole bath below 4.2K. It is difficult to maintain the temperature continuously and the process cannot be automated easily. Access to the main bath is restricted and you cannot refill the helium reservoir without running the magnet down to its 4.2 K field and venting the whole bath to 1 bar with helium gas.

2.4.2.2. Lambda point refrigerators

The lambda point refrigerator uses liquid helium from the main reservoir to cool the liquid around the magnet. Liquid helium has a very low thermal conductivity but strong convection currents can be set up in the liquid. If the liquid just above the magnet is cooled it convects down and displaces warmer liquid from the volume around the magnet. The liquid above the magnet is affected very little by the refrigerator, so the surface of the liquid remains at (or close to) atmospheric pressure and 4.2 K.

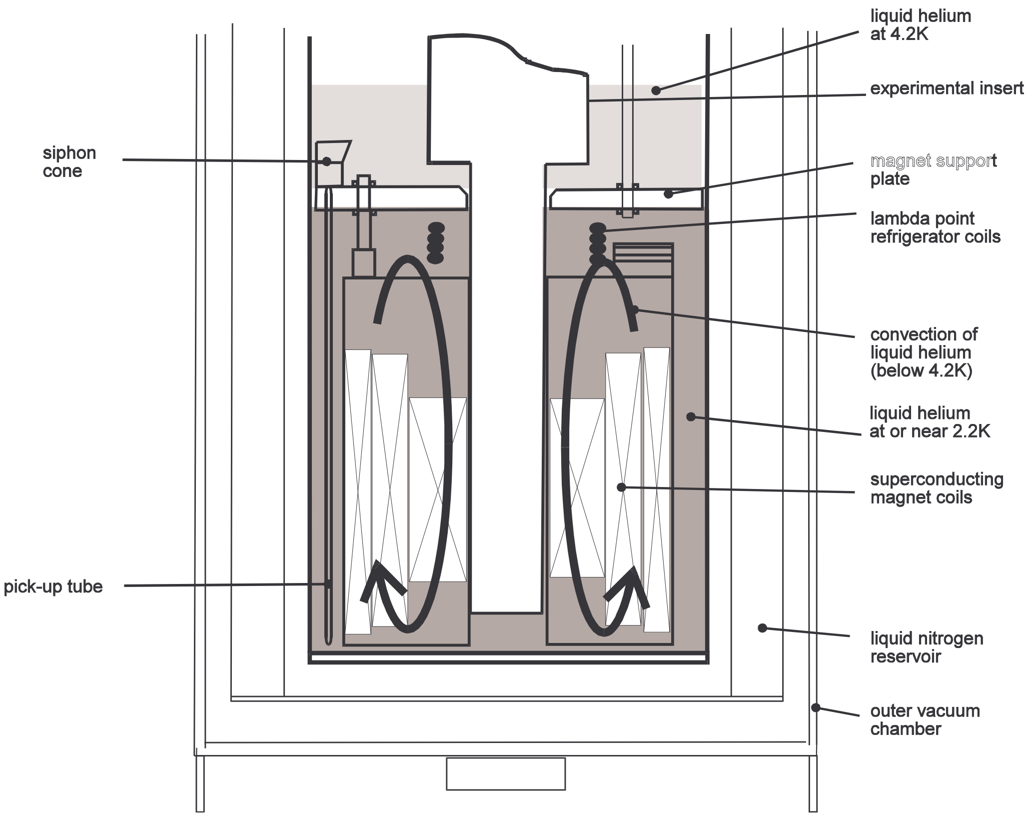


Figure 2 Schematic diagram of a lambda point refrigerator

Liquid is drawn through the lambda point refrigerator continuously and pumped to a low pressure by a rotary pump, reducing its temperature. This liquid is in good thermal contact with the main reservoir. A needle valve is used to control the flow rate through the lambda point refrigerator. When a high cooling power is required the refrigerator uses a high flow of liquid, and when the required temperature has been reached the flow rate can be reduced accordingly, minimising the running costs.

Resistance thermometers are fitted to the magnet and support system so that the performance of the lambda point refrigerator can be monitored. You can measure the temperature of these sensors with an ITC 501 temperature monitor or measure their resistances with a digital voltmeter (using a range with a low excitation current to minimise self heating in the sensor). Alternatively you can use a B-T environment Lambda Controller to give full automatic control of the refrigerator.

2.5. Dynamic variable temperature inserts

A variable temperature insert is used to allow you to adjust the temperature of a sample continuously over a wide range. It is vacuum insulated from its surroundings. On some systems the variable temperature insert (VTI) is mounted in the liquid helium reservoir. On others it is inside the outer vacuum chamber (OVC) of the cryostat; its insulating vacuum is then common with the OVC, and it may be indium sealed to a superconducting magnet. Both types of insert are operated in the same way but details of system assembly differ.

In a dynamic VTI the sample is supported on a top loading probe in the flowing helium gas. It can be removed while the insert is at any temperature. The sample temperature can typically be controlled over the range from 1.5 K to 300 K. This is done by balancing the cooling power of a flow of liquid helium with a heater, using an Oxford Instruments ITC temperature controller.

The liquid helium is drawn from the main reservoir through a needle valve. This valve is adjusted manually or by an auto needle valve stepper motor to control the flow. The heater and control thermometer can be mounted on the sample rod or on the heat exchanger. The needle valve is also fitted with a heater. If the valve or the small capillaries to the sample space become blocked with ice the heater can usually be used to clear the blockage without warming the whole system to room temperature.

Temperatures below 4.2 K are obtained by reducing the vapour pressure of liquid helium in the sample space. The needle valve can be set to fill the sample space with liquid helium continuously. If the flow rate is set correctly, this flow can just replace the evaporating liquid and maintain a constant liquid level. The base temperature in continuous fill mode is typically 1.5 K.

Lower temperatures can usually be achieved for a limited time by putting the insert into 'single shot' mode. The sample space is filled with liquid and the needle valve is closed completely. If a large enough rotary pump is used to reduce the vapour pressure of the liquid it is usually possible to cool the liquid and sample to approximately 1.2 K until all the liquid has evaporated. The volume of the sample space is sufficient to give a hold time of many minutes in single shot mode.

2.6. Setting temperature and voltage limits on an ITC temperature controller

If you have bought a cryostat and temperature controller together from Oxford Instruments the temperature controller should 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

When you first use the system it is wise to check that appropriate values have been set for your own application.

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' are given in the test results.

Read the ITC 'quick start' or the full ITC manual to find out how to set these values.

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

2.7. Spectromag dewars

Warning: **Windows may be ejected from cryostat without warning. Wear eye protection when system is in use.**

Spectromag dewars (or cryostats) are liquid nitrogen shielded. They are designed to give good optical access to a sample in a variable temperature environment and inside a split pair or solenoid superconducting magnet.

The magnet and protection circuit are welded into the liquid helium reservoir and they are therefore inaccessible.

The hold time when the system is running depends on the duty cycle. For example, if the magnet leads are carrying current the helium evaporation rate is increased.

The liquid nitrogen reservoir cools a radiation shield surrounding the 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 reservoir and liquid helium reservoirs are thermally isolated by using

- Low thermal conductivity materials
- High vacuum chamber between the reservoirs and room temperature
- 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 on the top 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.**

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 to air when the dewar is completely warm (which may take several days from the time when the cryogens are removed)
- Make sure that the helium reservoir has already been vented to atmospheric pressure
- Vent it very slowly to avoid any risk of collapsing the helium reservoir or moving the superinsulation.

2.8. 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. Safety

The following safety information is included. It is important that you read it.

Safety Matters	part number USC0001

4. Assembly and thermometry

4.1. Unpacking the system

The system should be unpacked carefully and inspected for any damage that may have been caused during shipment from Oxford Instruments. 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.

Warning: **Inspect any safety critical equipment (such as the relief valves and lifting eyes) prior to assembly. If any of this equipment shows sign of damage please contact Oxford Instruments NanoScience Customer Support before assembling the system.**

4.2. 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, ^{3}He refrigerator or dilution refrigerator there are additional requirements, and these are listed separately.

4.2.1. 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

4.2.2. 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).

4.2.3. 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.

4.2.4. 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.

4.2.5. Cryogens 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.

4.2.6. 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

4.2.7. 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

4.3. Additional requirements for variable temperature inserts

- GF4 diaphragm pump, valves and gauges to achieve temperatures down to about 3 K **OR**
- single stage rotary pump, valves and gauges if you want to run at temperatures below 3 K

4.4. 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.

4.4.1. 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).

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

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. If you have any doubts or concerns contact Oxford Instruments.

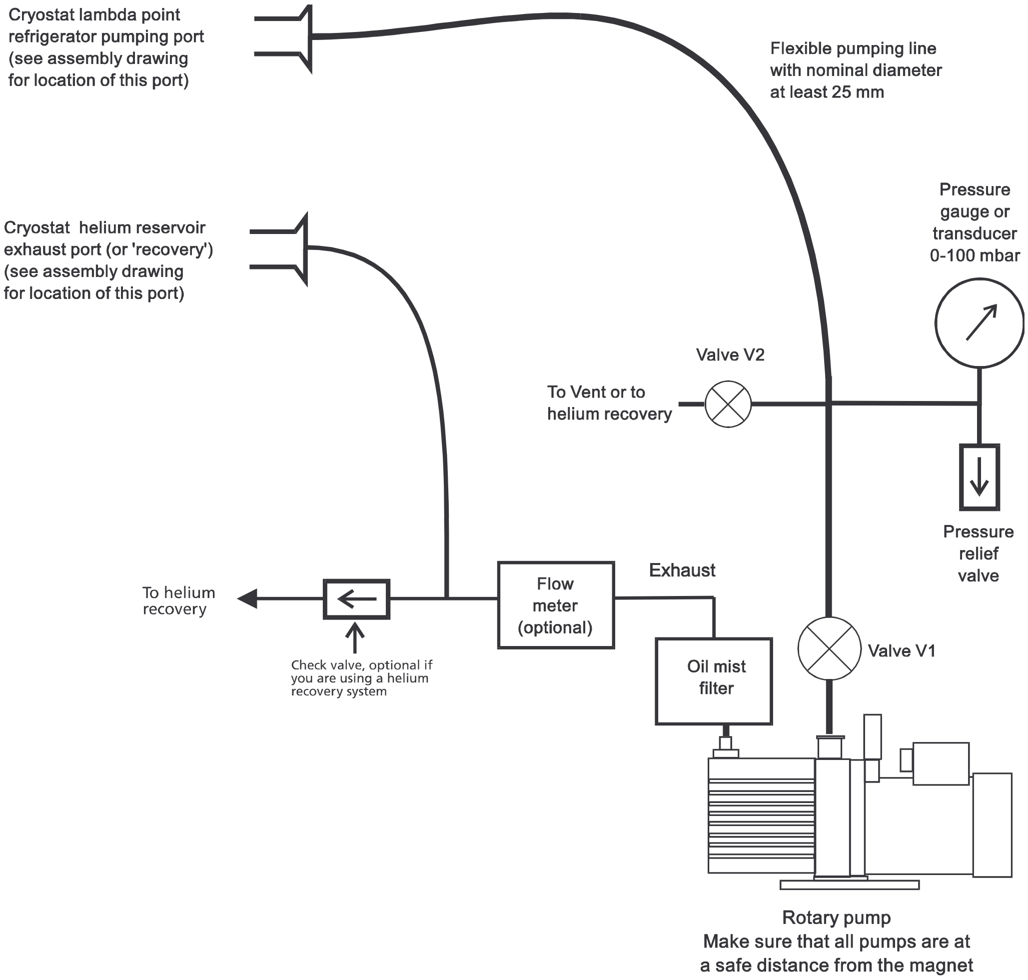
4.5. Additional requirements for lambda point refrigerators

A suitable single stage rotary pump or Leybold Sogevac pump with a displacement of 40m^3/hour or greater, (and preferably 60-80 m^3/hour for larger systems) with a connecting line from the pump to the cryostat, a valve to shut off the pump and a gauge for the pressure range from 0 to 100 mbar. An oil mist filter and a suitable exhaust line are also required, especially if you are connecting it to a helium recovery system.

4.6. Assembling lambda point refrigerator pumping system

The lambda point refrigerator is built into the magnet support system and cannot be removed from it.

The pumping system for a lambda point refrigerator is shown on the diagram below.



LPFPUMP.CDR/WMF

Figure 3 Pumping system for a lambda point refrigerator

Notes: Valve V1 is typically an NW25 diaphragm valve. See test results for recommended pump displacement

Caution: On B-T environment systems the pressure gauge shown on the diagram is a transducer. It is important that the transducer is positioned near the pump inlet, not near the cryostat, so that there is no risk of it getting too cold.

4.7. Assembling Spectromag systems

Caution Do not lay the system on its side at any time. If it is necessary to remove the tails for any reason, the system must be suspended from a suitable hoist.

Remove the outer tail from the system and remove the packing bungs which space the liquid nitrogen cooled shield from the outer vacuum chamber (OVC). All packing pieces are painted red. Remove the liquid nitrogen cooled shield tail and remove the packing pieces which you can see above the magnet.

Replace the tails of the liquid nitrogen cooled shield. Fit the windows (or re-entrant bore tubes) to the shield. Replace the OVC tail ensuring that the 'O' ring is clean and lightly greased with Fomblin grease. Fit the windows into the window mounts. Fit one 'O' ring in the groove behind the window as the vacuum seal and use the larger 'O' ring to hold the window in the frame. Fit another 'O' ring in the groove in the frame and push it carefully into the hole on the OVC.

4.8. Assembling variable temperature inserts (Spectromag systems)

4.8.1. Removing the variable temperature insert (VTI)

The VTI does not normally need to be removed. However, if it is necessary to remove it for any reason you should follow this procedure.

Caution The wiring on the insert is delicate and it is important that you take great care not to damage it.

Make sure that the entire system is at room temperature. Slowly vent the outer vacuum chamber (OVC) to atmospheric pressure with nitrogen gas or dry air.

Remove both tails from the bottom of the dewar.

You should them be able to see the indium seal in the capillary which feeds liquid helium from the main reservoir to the VTI. Break the indium seal by removing the bolts and prizing the flanges apart with a knife. Take care not to cut yourself or damage the indium seal flange faces. (This seal can be made again by following the procedure detailed in the *Background Information* section of this manual.)

Undo and remove the bolts on the top flange of the VTI.

Carefully withdraw the insert taking great care not to damage the wiring.

4.8.2. Replacing the VTI

Reassemble the insert into the dewar by following the same instructions in reverse.

4.9. Assembling the VTI pumping system

Assemble the pumping system as show in the diagram below.

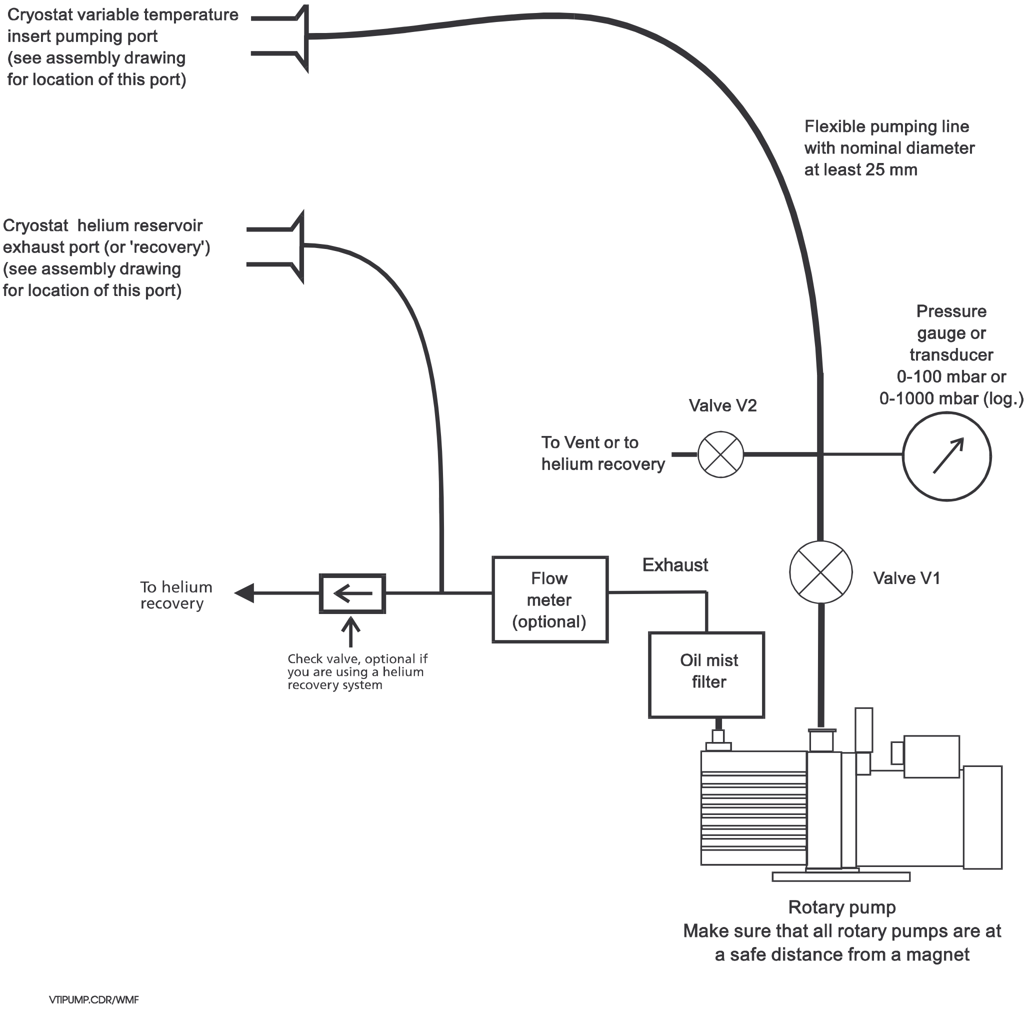
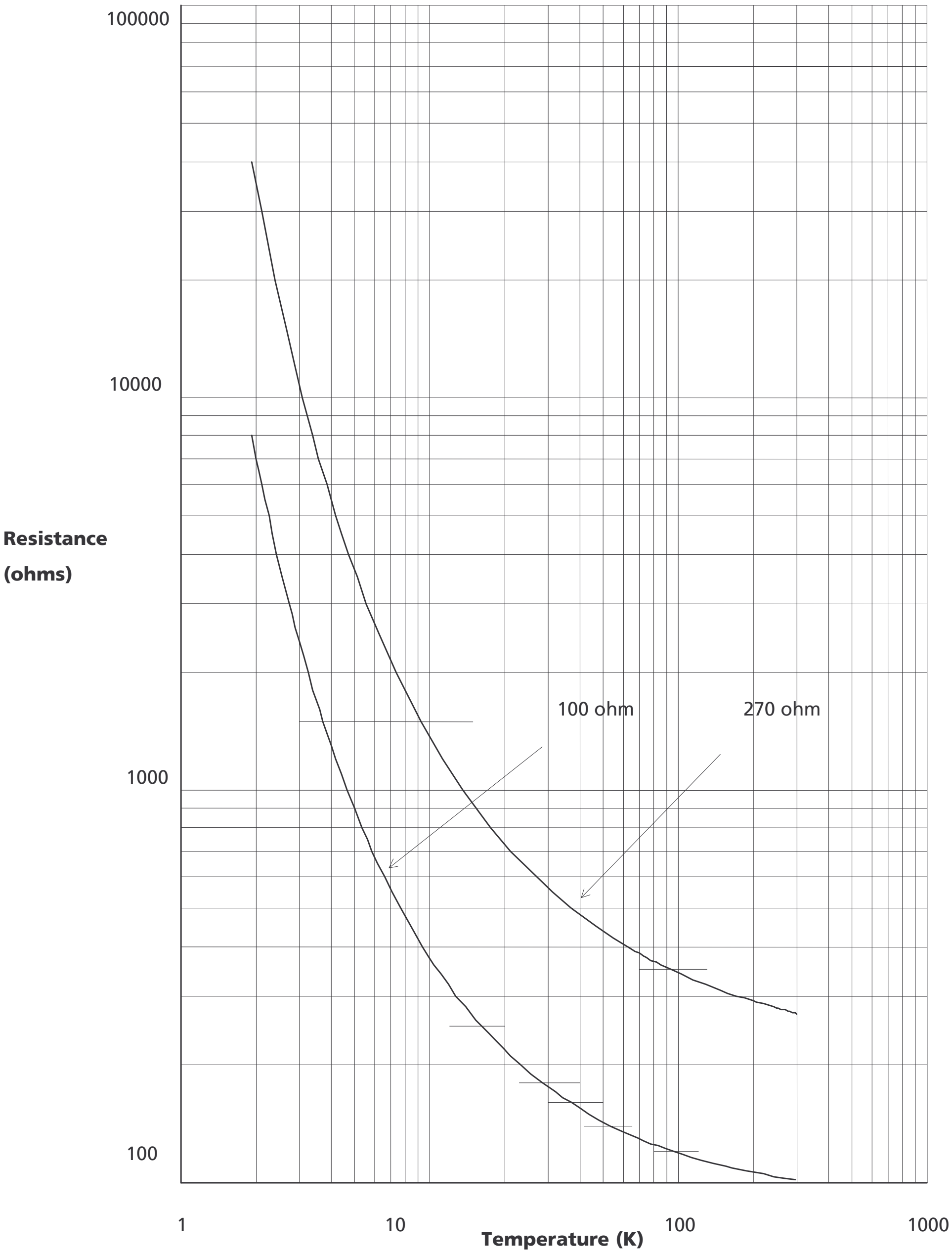


Figure 4 Variable temperature insert pumping system

Notes: Valve V1 is typically an NW25 diaphragm valve. See the test results for the recommended displacement of the pump.

4.10. Allen Bradley resistors as thermometers

Typical calibration curves of 100 and 270 ohm Allen Bradley resistors



5. Pre-cooling the system

5.1. Preparing a magnet for pre-cooling

5.1.1. 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 MΩ.

Check the following:

- Magnet to switch heater
- Magnet to dewar (ground)
- Magnet to all other diagnostic wiring

5.1.2. Normal pre-cool for NbTi magnets

Make sure that the liquid nitrogen is delivered to the bottom of the helium reservoir during precooling, so that the joints on the top of the magnet are not splashed with liquid nitrogen.

5.1.3. Slow pre-cool for Nb_3Sn magnets only

Caution: 10/11 Tesla magnets contain coils made from the brittle intermetallic compound Nb_3Sn. This material is fragile and it should not be subjected to thermal shock so you should cool it slowly as described below. It is typically used for magnets specified to reach a field above 9 T at 4.2 K or 11 T at 2.2 K.

The pre-cooling rate can be reduced by limiting the flow of liquid nitrogen into the helium reservoir. The simplest way to do this is to restrict the size of the exhaust line and the pressure in the liquid nitrogen storage dewar.

Fit a restriction to the helium reservoir exhaust port. This should have an inner diameter of approximately 6 mm, and you should fit a 300 mm length of rubber tube to this. Set the pressure in the liquid nitrogen storage dewar at approximately 250 mbar before you start to pre-cool the system.

5.2. Preparing the lambda point refrigerator for precooling

After you have pumped and flushed the main bath with helium gas you should pump and flush the lambda point refrigerator.

Connect the pumping system as shown in the diagram in the assembly section of this manual and evacuate the air from the lambda point refrigerator. Then close the valve in the pumping line. Monitor the pressure in the lambda point refrigerator for about 1 minute; if it starts to rise either the needle valve is not sealing properly or there is a leak somewhere in the system.

Open the lambda point refrigerator needle valve to fill it with helium gas (from the helium reservoir). Watch the pressure gauge. The pressure should quickly rise to approximately atmospheric pressure. Close the needle valve and repeat the process.

Leave the needle valve and the valve in the pumping line closed with helium gas (at atmospheric pressure) in the refrigerator.

5.3. Preparing liquid nitrogen shielded dewars for pre-cooling

5.3.1. 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.

5.3.2. 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.

5.3.3. 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

5.4. 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 has been included with this system.**

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.

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.

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.

Warning: **Ensure that all the components are correctly bolted together before cooling down the system.**

6. 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.

6.1. 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.

6.2. 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.

Warning: **If the system is frozen into the cryostat with liquid nitrogen, it must be allowed to warm up naturally.**

6.3. Checking the VTI at 77 K

It is not essential that you check the insert for leaks at this stage, and after you have been using the system without problems for a few weeks you may feel confident enough to run it without further testing.

However, if the system is leaking it is better to find out before you use any liquid helium. Most cold leaks on cryogenic systems can be detected at 77 K, so if you want to ensure that there is no risk of wasting liquid helium we recommend that you carry out the following leak tests.

6.3.1. Leak testing the insert at 77 K

If the system is to be checked for leaks re-connect the leak detector to the IVC[1].

Repeat the leak tests that you carried out at room temperature. Some of the tests can be done while you are blowing out the liquid nitrogen and pumping and flushing the helium reservoir, and the sample space leak tests are best carried out after the main reservoir has been flushed with helium gas.

6.3.2. Cooling the insert to 4.2 K

Open the needle valve and draw cold gas through the VTI as you transfer liquid helium into the system to cool it to 4.2K using the pump to promote the flow.

6.4. Checking liquid nitrogen shielded dewars at 77 K

6.4.1. 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.

6.4.2. Filling the liquid nitrogen jacket

Fill the liquid nitrogen jacket with liquid nitrogen.

6.4.3. Preparing for the liquid helium transfer

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

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[1] If your VTI is mounted in the outer vacuum chamber (OVC) of the cryostat it does not have a separate IVC. Check for leaks into the OVC as described for the IVC above.

6.5. 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.

6.6. Preparing the lambda point refrigerator for cooling to 4.2 K

After you have pumped and flushed the main helium reservoir to make sure that all the liquid nitrogen has been removed, check that the lambda point refrigerator is not blocked and that the needle valve is free as follows.

Pump the helium gas out of the refrigerator and close the valve in the pumping line. Open the needle valve on the lambda point refrigerator (checking that it opens smoothly) and watch the pressure gauge. Check that the pressure rises quickly to approximately atmospheric pressure.

Leave the needle valve open as you cool the system to 4.2 K. This ensures that there are no closed volumes full of gas, in case the system is accidentally warmed to above 77 K.

6.7. 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.

6.7.1. 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.

Warning: **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 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.

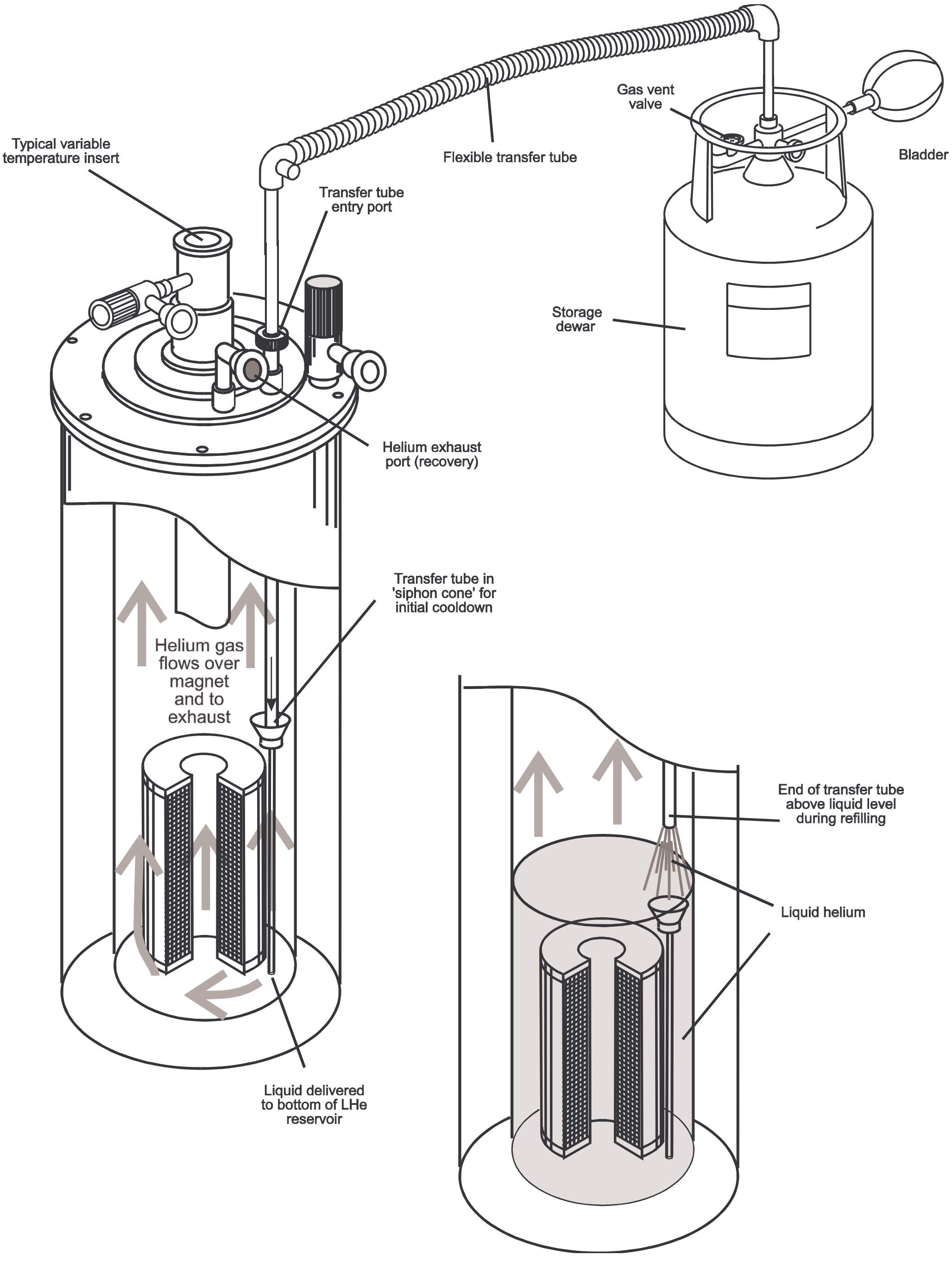

Figure 5 Transferring liquid helium into a typical laboratory cryostat. Your system may not look like the one shown in the diagram.

6.7.2. 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.

7. Running the system

7.1. Running the magnet at 4.2 K

7.1.1. 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) 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
- 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, B-T environment 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.

7.1.2. 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 MΩ unless otherwise stated.

7.1.3. 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.**

7.1.4. Energising the magnet from zero field

7.1.4.1. 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.

7.1.4.2. Energisation

Start to energise the magnet by pressing the GOTO SET button 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.

7.1.5. 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).

7.1.6. 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 IPS120-10 carries 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.)

7.1.7. 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 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.

7.1.8. 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 PUSS by pressing the power button.

7.1.9. Running the magnet in constant voltage mode

7.1.9.1. 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.

7.1.9.2. Preparation

Prepare the system as described in the Preparation section above.

7.1.9.3. 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 tenderisation 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.

7.1.9.4. 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 did 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.

7.1.9.5. 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.

7.2. Running the magnet at 2.2 K using a lambda point refrigerator

Warning: **Before you attempt to run the magnet to a higher field at a temperature below 4.2 K, check the test results to make sure that it was designed for this type of operation. If these results do not specify a current for a 2.2 K field and you expect to be able to run it to higher fields please contact Oxford Instruments for clarification before you proceed.**

7.2.1. Preparing the lambda point refrigerator

Connect the pumping system as shown in the assembly section of this manual. Make sure that the exhaust of the pump is connected to the helium reservoir exhaust as shown, or arrange for a suitable low pressure gas supply to be connected to it. This makes sure that the pressure in the reservoir does not drop below atmospheric pressure while the lambda point refrigerator is running. This is especially important while you are cooling the magnet from 4.2 K to 2.2 K, because the flow rate through the lambda point refrigerator is high and the boil off from the main bath is unlikely to be high enough to fill the volume left when the liquid is pumped away. The oil mist filter is used to clean the gas.

Use an ITC501 temperature monitor, a B-T environment Lambda Controller or a digital voltmeter with a low excitation current to monitor the resistance thermometers fitted in the helium reservoir.

7.2.2. Preparing the magnet

The operation of the lambda point refrigerator will be most efficient if the magnet is run to its 4.2 K field and put into persistent mode as described in "Running the magnet at 4.2 K". If the magnet is in this state the heat load on the helium reservoir is minimised.

If you prefer, or if the magnet does not have a superconducting switch (and cannot be put into persistent mode), you can leave the magnet at zero field while you cool it to 2.2 K. However, when you start to sweep the magnet heat will be introduced to the helium reservoir, and you may have to adjust the lambda point refrigerator's flow rate as described below.

Alternatively, if the magnet does not have a superconducting switch, you can hold it at the 4.2 K field using the magnet power supply. The current in the leads may produce noticeable ohmic heating at the soldered joints, and will take longer (and use more liquid helium) to cool the magnet to 2.2 K.

On some systems the maximum sweep rate of the magnet is limited by the cooling power of the lambda point refrigerator, not by the performance of the magnet itself. To some extent it is possible to increase the cooling power by increasing the flow rate through the refrigerator and/or using a larger displacement pump.

7.2.3. Running the lambda point refrigerator automatically

The B-T environment Lambda controller helps you to monitor and control the lambda point refrigerator economically and automatically if you run the VI called "Run Interlocks". You can then set the system up to take a range of measurements unattended. The B-T environment controller will check the helium level and ensure that the magnet is cold enough to operate at the required field. If necessary it will sweep the magnet down to a safe field automatically.

7.2.4. Running the lambda point refrigerator manually

Fill the liquid helium reservoir so that there is sufficient liquid in the reservoir for the cooldown.

Make sure that the pumping system is connected to the cryostat as shown on the diagram in the assembly section of this manual. Start the pump and fully open the valve in the pumping line. Measure the thermometers R1 to R3 (or sometimes R4), which are fitted to the magnet and support system. This gives a 4.2 K calibration for future reference.

Slowly open the lambda point refrigerator needle valve until the pressure on the gauge in the pumping line is between 35 and 45 mbar. After a few minutes you should start to see some cooling on R2, and then on R3. R1 should not cool noticeably unless the refrigerator has been running for an extended period with a high helium level.

As the magnet cools down, the pressure in the refrigerator will rise slowly. Gradually close the needle valve to maintain the pressure in the range 35 to 45 mbar.

The magnet will typically cool to approximately 2.2 K over the time shown in the test results. However, this time can be affected by several factors. In particular the cooldown will take longer if:

- Current is flowing through the leads to the magnet, (especially if the field is sweeping)
- The switch heater is on
- The pressure in the lambda point refrigerator is lower
- You are using a smaller pump

When the magnet reaches a temperature of 2.2 K the flow through the lambda point refrigerator can be reduced further to reduce the consumption of liquid helium. Experiment with the needle valve to determine the minimum flow that will keep the magnet at 2.2 K. When you feel confident that you can keep the magnet at 2.2 K consistently proceed to run it to its 2.2 K field. You will need to increase the flow slightly while there is current in the leads and while the switch heater is on. When the magnet is put into persistent mode you should be able to reduce the flow again. Make sure that the magnet does not warm above about 2.3 K while it is at its maximum field. If in doubt run it down to its 4.2 K field rather than risk quenching the magnet because it has warmed up too much.

Make the following checks regularly while you are running the lambda point refrigerator:
Check the liquid helium level and re-fill if necessary
Check the temperature of the magnet and run it down immediately if it warms too much
* Reduce the flow rate to slightly above the minimum flow needed to maintain the temperature at 2.2 K

7.2.5. Running the magnet to its 2.2 K field

When the magnet is at a stable temperature and you are confident that you understand the operation of the lambda point refrigerator you can run it above its 4.2 K field.

Set the required field on the magnet power supply as described in "Running the magnet at 4.2 K". A lower sweep rate is normally recommended as the magnet reaches higher fields. The recommended sweep rates are given with the test results.

As you sweep the magnetic field make sure that the temperature of the magnet does not rise significantly. If it starts to rise, open the needle valve slightly. If you cannot keep the magnet at 2.2 K and the pressure in the pumping line has reached 50 mbar or more try the following to reduce the heat load on the system:
Check that the switch heater current is at the recommended value
Check that you are not sweeping the field faster than recommended

* Check that you are using a pump of the recommended displacement and that the diameter of the pumping line is large enough .

Warning: **If you cannot keep the magnet cold enough run it down to its 4.2 K field until you find the cause of the problem.**

7.2.6. Closing down the lambda point refrigerator

When you have finished running the magnet above its 4.2 K field, (and if you do not intend to run it above this field again for a long time), you can close down the lambda point refrigerator by closing the needle valve. Either leave the pump running or vent the refrigerator to the main bath so that there is no risk from gases trapped in a closed volume in case the system warms up accidentally. The magnet is likely to stay below 4.2 K for a long time (perhaps one or two days for a large system). This does not affect normal operation up to the 4.2 K field.

7.3. Running a dynamic VTI

These instructions should help you to operate the variable temperature insert. However you may also need to refer to the manual for the temperature controller and the auto needle valve if you encounter unusual problems.

A range of pumps is available to promote the flow of liquid helium through a variable temperature insert:

- Small diaphragm pumps (for example Oxford Instruments GF4) for low flow rates and temperatures down to approximately 3 K (or 10 K in low loss dewars)
- Rotary pumps for higher cooling powers and lower base temperatures

It is also possible in some conditions to allow gravity to promote the flow of liquid through the insert. However, it is difficult to control the flow and obtain good temperature control in this way.

7.3.1. Operating the VTI above 4.2K

Above 4.2 K the temperature of the insert and sample is controlled by adjusting the liquid helium flow rate and applying heat. As the liquid helium enters the sample space it flows through a heat exchanger. The liquid is boiled and the gas is warmed to the required temperature by this heat exchanger, which is controlled by the temperature controller.

Connect the insert exhaust to a suitable pumping system as shown in the diagram in the assembly section of this manual. Connect the temperature controller and switch it on. Evacuate the sample space with the needle valve closed.

7.3.1.1. Manual operation

Refer to the test results to find the flow rate and PAD settings for a temperature close to the value that you want to set. Set the PAD values on the temperature controller by holding down PROP, INTO or DERIVE button while using RAISE and LOWER until the required value is displayed. Then open the needle valve slightly to set the required flow. Set the required temperature by holding SET and using RAISE and LOWER.

7.3.1.2. Automatic control

If an auto needle valve stepper motor is fitted to the needle valve on the insert the whole control process can be carried out by an ITC temperature controller. This sets a suitable flow rate and PAD values, and adjusts the heater power to suit the set temperature. If you bought the VTI and temperature controller together, the ITC will have been set up in factory and the settings will be stored in it.

ITC temperature controllers can be controlled from a computer, allowing a temperature sweep to be programmed and carried out automatically. This can be done in conjunction with a complex magnetic field sweep so that a set of experimental results can be taken while the system is unattended. Oxford Instruments Object Bench and B-T environment software packages are available to help you do this.

When you are controlling the temperature of the sample by setting a temperature on the Vita’s heat exchanger there may be a small temperature offset between the set and measured temperature. If you find that this happens try to use the thermometer on the sample holder to control the temperature, or adjust the set temperature on the heat exchanger slightly to obtain the required sample temperature.

7.3.2. Operating the VTI below 4.2 K

Temperatures below 4.2 K are achieved by reducing the vapour pressure of the liquid helium in the sample space. You can set the flow through the needle valve for continuous operation, or close the needle valve to put the insert into 'single shot' mode, (for the lowest possible temperature).

Cool the insert to 4.2 K as described above.

7.3.2.1. Continuous operation

The ITC temperature controller can control the temperature in this range automatically using and auto needle valve to set the flow to the optimum value. Operate the insert as described above.

7.3.2.2. Manual operation using a pressure controller

If you prefer, you can control the temperature by setting the pressure in the sample space. Use an Oxford Instruments Manos tat (or equivalent pressure controller) to do this. The base temperature of the insert will typically be between 1.5 and 2 K, depending on the total heat load and the type and size of pump.

7.3.2.3. Single shot operation

Open the needle valve fully to fill the sample space with liquid. Allow it to fill until the pressure in the pumping line rises to a high value and becomes unsteady.

Close the needle valve and continue to pump the sample space to reduce the vapour pressure of the liquid helium. Base temperature should be reached after a few minutes. The operating time is limited by the capacity of the sample space. When the liquid helium has all evaporated the sample will begin to warm. The best base temperature will be achieved by using a large pump and pumping lines.

7.3.3. Changing samples

Set the sample temperature to approximately 300 K (or the maximum temperature limit of the insert if this is less than 300 K). This will not warm the entire sample space but it does reduce the risk of blocking the heat exchanger at the bottom of the sample space and reduces the amount of moisture condensed on the sample as it is removed.

Stop pumping the sample space and fill it to a pressure of 1 atmosphere with clean helium gas, either by opening the needle valve or by connecting the sample space to the recovery line. Quickly remove the sample holder and immediately seal the sample space with the baffle set provided.

Follow the same procedure to load the sample into the insert. It may be necessary to rotate the sample holder slightly as it enters the sample space.

7.4. Leaving the system unattended

7.4.1. 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 cryogens in the system
- Arrange for the cryogens 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.

7.4.2. 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

7.5. 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.

7.5.1. 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).

7.5.2. 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.

Note: The reading on the helium level probe may be affected when using the siphon entry on the dewar to top up with helium. For best results use the siphon entry on the insert (if fitted).

8. Warming up the system

8.1. Warming up the lambda point refrigerator

Warning: **It is important to make sure that the lambda point refrigerator is free to vent safely while the system warms up. Remember that there may have been an air leak in the pumping line while the system was cold. If so, solid air may have condensed in the refrigerator and this must be allowed to expand freely. You cannot tell whether this contamination is present or not.**

Either - pump the refrigerator to a pressure less than 1 mbar, close the valve in the pumping line, open the needle valve and vent the exhaust to the main helium reservoir. This allows any liquid or gas remaining in the refrigerator to expand into the main reservoir safely.

Or - open the valve in the pumping line and continue to pump the lambda point refrigerator until the system is at room temperature. The pump can then be switched off. Vent the refrigerator with 1 atmosphere of helium gas by opening the needle valve.

8.2. Warming up a dynamic VTI

Warning: **It is important to make sure that the variable temperature insert is free to vent safely while the system is warmed up. Remember that there may have been an air leak in the pumping line while the system was cold. If so, solid air may have condensed in the insert and this must be allowed to expand freely. You cannot tell whether this contamination is present or not.**

Either - open the valve in the pumping line and continue to pump the VTI until the system is at room temperature. The pump can then be switched off. Vent the insert with 1 atmosphere of helium gas by opening the needle valve.

Or - pump the insert to a pressure less than 1 mbar, close the valve in the pumping line and open the needle valve, allowing any liquid or gas remaining in the insert to expand into the main reservoir safely.

8.3. Warming up the system - liquid nitrogen shielded dewars

8.3.1. 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)

8.3.2. 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.

8.3.3. 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.

9. Background information

9.1. 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.

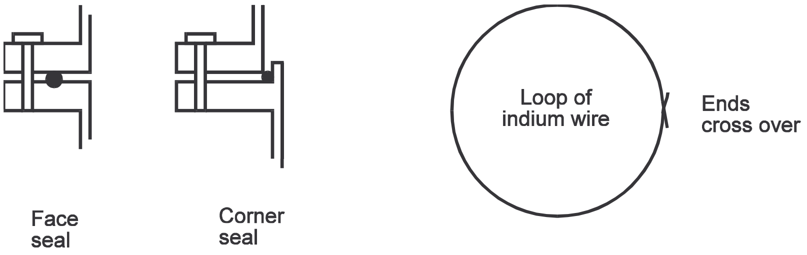

Figure 6 Indium seals

9.1.1. 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.

9.1.2. 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 > 50 mm 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.

9.1.3. 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.

9.2. 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.

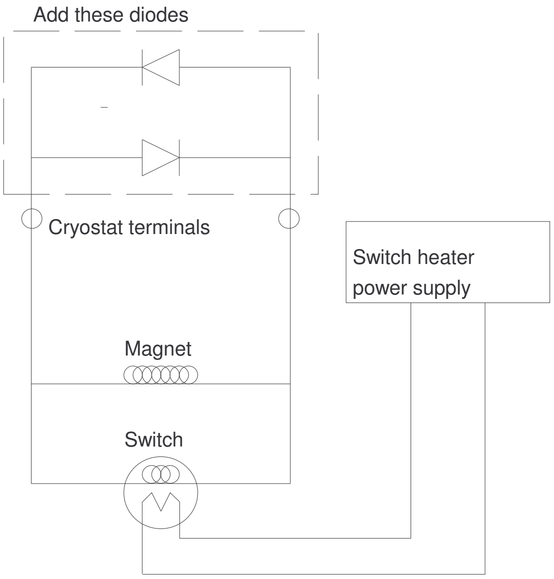

Figure 7 Emergency de-energisation circuit diagram

9.3. 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 *Practical Cryogenics* 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.
Transfer tube shows ice "spots"	Internal capillary touches outer tube	If the transfer tube is still under warranty and it has not been 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 *Practical Cryogenics.*

9.4. VTI fault finding

This is a summary of the common faults and a description of the most likely cause of a problem. If these guidelines do not help you to solve the problem a full set of test data should be taken and sent to Oxford Instruments for diagnosis, along with details of any modifications that you have made to the system.

9.4.1. Blockages in the variable temperature insert

If the pressure does not rise when needle valve is opened it is likely that the insert is blocked. Try to clear the blockage by warming the sample to 300 K (if this does not exceed the maximum temperature limit of your insert), or using the needle valve heater as described in the section called "Clearing blockages in VTIs".

If this does not clear the blockage warm up the whole system. Pump the VTI and thoroughly flush the needle valve with dry helium gas.

9.4.2. Needle valve will not move

If the needle valve is fitted with an auto needle valve stepper motor it is possible that the motor cannot supply sufficient torque to open the valve. Refer to the manual for the auto needle valve.

If the needle valve does not have an auto needle valve motor fitted or if it still cannot be freed, try warming the needle valve with the heater as described in the section called "Clearing blockages in VTIs".

9.4.3. Poor cooling

If the sample does not cool as quickly as you expect, check the following.

- Is the flow rate high enough, and if not, is the VTI blocked?
- Have you fitted very heavy wiring to the sample probe?
- If the flow rate is very high is it possible that the radiation shield is touching the sample space and conducting heat into it?
- On static inserts only, have you introduced sufficient exchange gas?

9.4.4. Poor temperature stability

If the temperature stability is not as good as you expect refer to the test results and try to control at one of the set points used during the test. Check that you are using the correct settings on the temperature controller and that the helium flow rate is approximately the same as on the test results.

9.4.5. Sample rod cannot be removed

If the sample rod cannot be inserted or removed from the insert it is likely that there is ice in the sample space. This may indicate an air leak or that air has been allowed into the sample space during sample changing.

Set the maximum permissible sample temperature and pump the sample space for 15 to 30 minutes. Then try again to remove the sample rod. If you still cannot move it you will have to warm the whole insert to room temperature. Make sure that the sample space is free to vent as you warm it up, and if possible pump it continuously to remove the contamination.

9.4.6. OVC becomes frosty and helium boil-off increases when sample exchange gas is admitted.

On systems where the VTI is insulated from the helium reservoir by the OVC (known as common vacuum systems) this may indicate a leak from the sample space to the OVC.

Dismantle the system and replace the window seals and any other indium seals. Repeat the leak test at room temperature and during pre-cooling.

9.5. Clearing blockages in VTIs

If you are careful when you run a variable temperature insert it is unlikely to become blocked. However, if a large amount of air is allowed into the sample space during sample changing (on dynamic VTIs only) it may freeze in the heat exchanger and block it. If the liquid helium contains a large amount of ice it may block the filter on the end of the pick up tube and the only way to clear this type of blockage is to warm up the insert.

9.5.1. Checking for blockages

Connect the pump and a pressure gauge to the pumping line as in the diagram in the assembly section of this manual. Close the needle valve and pump the VTI to the base pressure of the pump. Close valve V1 and read the pressure from the gauge. It should not rise, but if it does it suggests that there is an air leak in the system and this could be the cause of the blockage.

Open the needle valve and observe the pressure on the pressure gauge. If the pressure does not rise quickly the insert is blocked.

The needle valve is vacuum insulated. Most variable temperature inserts have a heater and sensor on their needle valves. It may be possible to free a blockage by using this heater. Pump the VTI exhaust continuously throughout this procedure and monitor the flow of gas from the exhaust of the pump. (However, if you do not have a suitable oil mist filter and flow meter you can leave the VTI under vacuum and watch for a pressure rise.)

The heater resistance is 68 ohms. Connect a suitable power supply to the heater. (You can use an ITC temperature controller under manual control if you like.) Apply approximately 10 to 15 volts across the heater. The sensor on the heater block is a 270 ohm Allen Bradley resistor. You can use this as a temperature indicator[2] to see how quickly the temperature is rising.

Caution: If you exceed 15 volts or maintain it for more than 30 minutes it is possible that the heater or cryostat may be damaged.

If you see the flow start to increase, (or the pressure rise) the blockage is clearing. Continue to heat the needle valve until the flow reaches a steady value and then switch it off.

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[2] The resistance reading from an Allen Bradley resistor above room temperature is meaningless, because its resistance passes through a minimum close to room temperature.

9.6. 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.

9.7. 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.

Eléménts 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

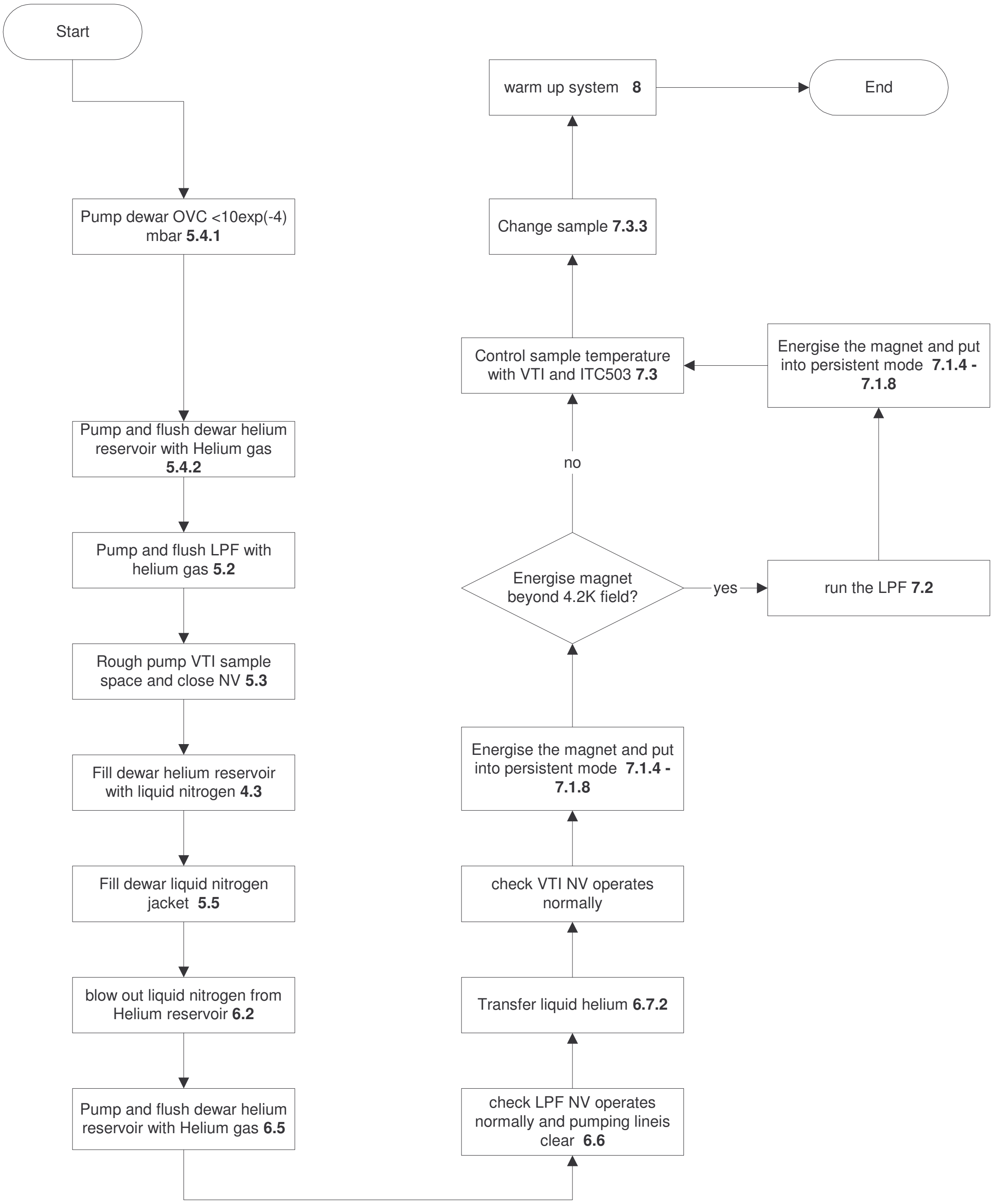
Matter and Methods at Low Temperatures
Frank Pobell,
Springer Verlag, 1992, ISBN 0 540 53751 1 and 0 387 53751-1

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.

9.8. Summary flow chart for experienced users

Numbers in bold type refer to the section number in this document.



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