Practical Cryogenics

An Introduction to Laboratory Cryogenics

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This document is intended to be used only as background information, and not as an instruction manual. No liability will be accepted for any damages incurred because of the information contained in, or implied by, the contents.

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1 Foreword

This booklet has been written to help you to learn more about the basic principles of cryogenics, so that you can design your experiments to make the best possible use of your system. A little training often makes the difference between success and failure for a low temperature experiment.

The booklet is a collection of practical notes to introduce beginners to the fundamentals of good practice. It contains a 'glossary of terms' to explain some of the jargon commonly used in cryogenics. The descriptions are intended to translate these terms into plain English so that beginners can understand them.

The other sections give general advice on a range of relevant topics. A strong emphasis is placed on practical information rather than theoretical details. Previous editions of this booklet (then called *Elementary Practical Cryogenics)* contained some of this information. Several small errors have been corrected and more information has been added.

The subject of 'safety' has deliberately been omitted. All cryogens are potentially hazardous. Before you try to use a cryogenic or high magnetic field system you should receive training from a competent person who knows your laboratory and the laws in your country. You may then like to use the booklet *Safety Matters* (available from Oxford Instruments) to remind you about this training when you are using a system.

N H Balshaw

2 Vacuum equipment

Vacuum systems are used most commonly in laboratory scale cryostats and superconducting magnet systems for the following purposes:

- To pump out the high vacuum insulation spaces in the cryostat and transfer tube
- To pump out an exchange gas
- To set up a pressure gradient along a pumping line so that the flow of cryogen through the cryostat can be controlled
- To reduce the vapour pressure over liquid helium surfaces where temperatures below 4.2 K are required
- To pump out the nitrogen gas from a pre-cooled helium vessel, after the liquid has been blown out

All gases, except helium, hydrogen and neon, will condense on surfaces cooled to below about 60 K. Therefore, once liquid helium at 4.2 K is introduced into a vacuum vessel, all the residual gases that are normally present will condense (or cryopump), reducing the pressure in the vacuum space by one or two orders of magnitude. Therefore, the function of a vacuum system is to reduce the pressure in the vacuum space to a point where the thermal insulation is sufficiently good to allow liquid helium to be held in the vessel. In a typical laboratory scale system the pressure then drops to 10^{-5} mbar or less.

In cryostats that contain only liquid nitrogen, the coldest surface is at 77 K, which is above the temperature for effective cryopumping by a metal surface. If the cryostat is not pumped continuously by an external pumping system, a sorption pump is mounted on the liquid nitrogen reservoir to maintain the integrity of the vacuum. Occasionally it has to be cleaned by warming it to a temperature around 100°C and pumping the vacuum space. It pumps air to a very low pressure when cooled with liquid nitrogen.

A booklet is available from Leybold to describe how to do most common vacuum calculations. (See section 12).

2.1 Vacuum pumps

2.1.1 Single stage rotary pumps

Rotary pumps are used as roughing pumps (to reduce the pressure to a rough vacuum) or as backing pumps (with a diffusion pump or turbomolecular pump). If the rotary pump's sole function is to back a small oil diffusion pump or a turbomolecular pump then a single stage rotary pump with a base pressure of about 10^{-2} mbar and a displacement of about 5 m^3/hour is adequate. However, some laboratories prefer to use a two stage rotary pump with a diffusion pump because of the risk of stalling the diffusion pump if the backing pressure exceeds a critical value (about 10^{-1} mbar). If the diffusion pump stalls oil back-streams into the vacuum system and can permanently affect the performance of the cryostat.

If the rotary pump is also to be used as a roughing pump or to reduce the vapour pressure over a liquid surface it may be necessary to choose a higher displacement pump to suit the requirement. Most vacuum equipment manufacturers supply the information needed to calculate the pump size in their sales brochures.

In most cases, it is best to use a pump fitted with a 'gas ballast' facility. This helps the pump to remove condensable vapours from the vacuum space of a cryostat. It is common for water to accumulate in the vacuum spaces of cryostats if the cold surfaces are ever exposed to air or if the cryostat is left unused for some time. Most surfaces release absorbed water vapour when the pressure is reduced.

Practical base pressure:	10^{-2} to 10^{-3} mbar
Max. working pressure:	1 bar (for a limited period), few hundred mbar continuously
Ideal for:	Rough pumping, backing high vacuum pumps, lambda point refrigerators, variable temperature inserts, 1 K pots

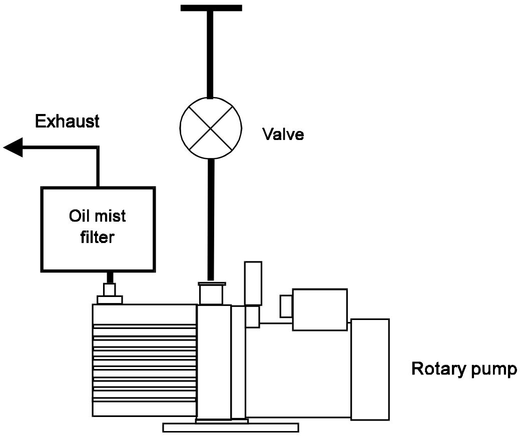

Figure 1 Rotary pump system

2.1.2 Two stage rotary pumps

In some cases, it is possible to replace a rotary / diffusion pump combination with a two stage rotary pump. A base pressure of 10^{-4} mbar can be achieved in ideal conditions and using a cold trap, but in practice the pressure in the cryostat will probably only be 10^{-1} to 10^{-2} mbar.

This type of pumping system is very simple but it cannot reach a low enough base pressure to give good thermal isolation . A large amount of liquid helium would be required to cryopump the residual gas and the static boil off of the system would be slightly increased. In addition, if the cryostat is used above 60 K and there is no sorption pump in the vacuum space, condensation or frosting may be seen on the outside of the cryostat.

2.1.3 Diffusion pumps

A 50 to 75 mm diameter oil diffusion pump is sufficient for pumping laboratory scale cryostats. An air-cooled pump with an air pumping speed of about 50 litres/second and an ideal ultimate pressure of about 10^{-7} mbar is commonly used.

It is advisable to use a cold trap with a diffusion pump (although some people do not consider it to be essential). You should never pump the vacuum space of a cold cryostat without a cold trap between the pump and cryostat. This trap helps to remove water vapour from the vacuum space and prevents back streaming of oil vapour from the pump. Two types of trap are commonly used, liquid nitrogen filled traps and thermo-electric (Peltier effect) cooled baffles. The latter require less attention and are better for very long-term unattended operation.

Practical base pressure:	10^{-7} mbar
Max. working pressure:	10^{-2} to 10^{-1} mbar
Ideal for:	Pumping insulating high vacuum spaces in cryostats (e.g. OVCs)

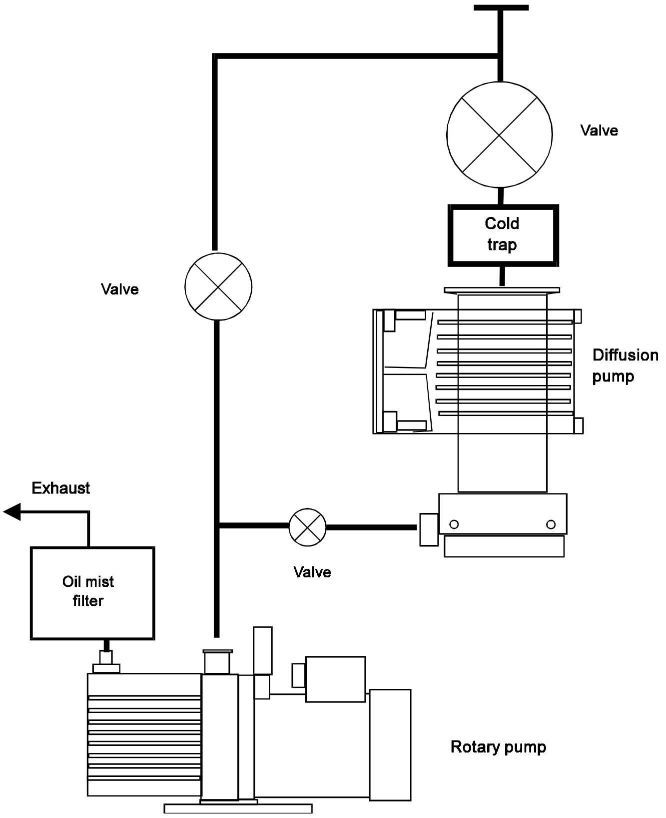

Figure 2 Diffusion pump system

2.1.4 Turbomolecular pumps

These high vacuum mechanical pumps can be used instead of diffusion pumps. They are especially useful if a very clean high vacuum is needed because the compression ratio is strongly dependent on the mass of the molecules. The large hydrocarbon molecules are pumped so well that there is virtually no backstreaming of oil. Many of the modern pumps incorporate a molecular drag stage within the pump and can tolerate a backing pressure of 10 mbar or higher. An oil free diaphragm pump can then be used as the backing pump for some applications.

Practical base pressure:	10^{-8} mbar
Max. working pressure:	A few mbar (for conventional turbomolecular pumps). About 30 mbar (for some pumps with a molecular drag stage).
Ideal for:	Pumping clean high vacuum spaces (with or without cold trap).

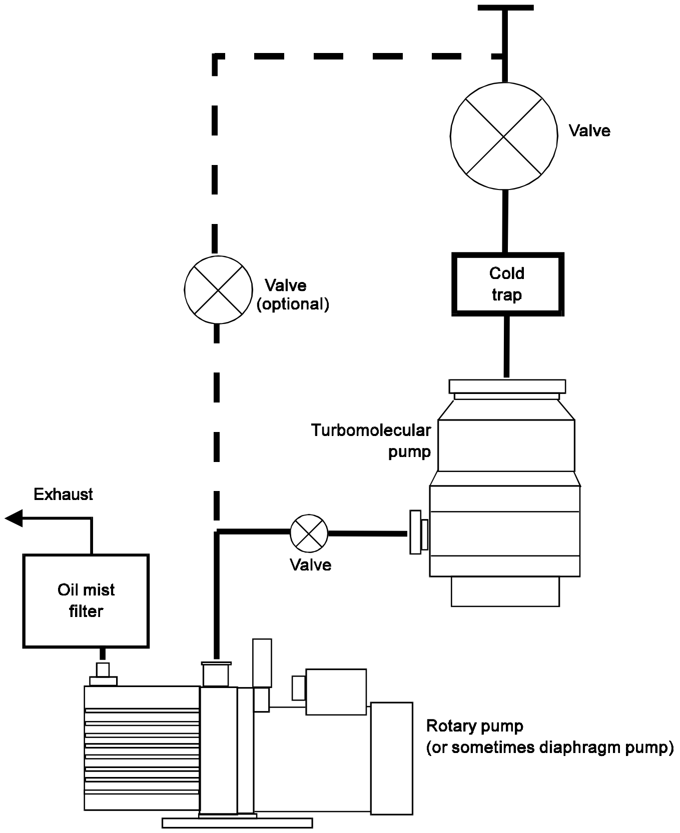

Figure 3 Turbomolecular pump system

The pumping speed for helium is about 20% higher than that for nitrogen, but the compression ratio is much lower. Therefore if the pump is to be used to pump helium from a vacuum space it is best to use a two stage rotary pump, so that the backing pressure is as low as possible. A diffusion pump is still better at pumping helium!

Turbomolecular pumps should only be vented (from the high vacuum side) while they are still spinning slowly. This helps to prevent contamination backstreaming from the high pressure side of the pump. Most pumps can be fitted with an automatic venting device which is activated by the pump controller. The gas can be drawn through a drier cartridge to prevent contamination with water.

2.1.5 Roots pumps

Roots pumps (or roots blowers) are mechanical booster pumps, used (in conjunction with a backing pump) to reach the medium to high vacuum range with very high gas throughputs. Two (or more) pumps can be used in series with some advantage. For example, if you need a pumping speed of 1000 m^3/h it may be best to use a 1000 m^3/h roots pump backed by a 250 m^3/h roots pump, which in turn is backed by a 65 m^3/h rotary pump. Vacuum companies often recommend a 1000 m^3/h roots pump backed by a 250 m^3/h rotary pump, but this option is usually more expensive.

Practical base pressure:	10^{-4} mbar
Max. working pressure:	Few hundred mbar
Ideal for:	High volume flow rates in the pressure range 10^{-4} to 50 mbar (for example, in dilution refrigerator systems)

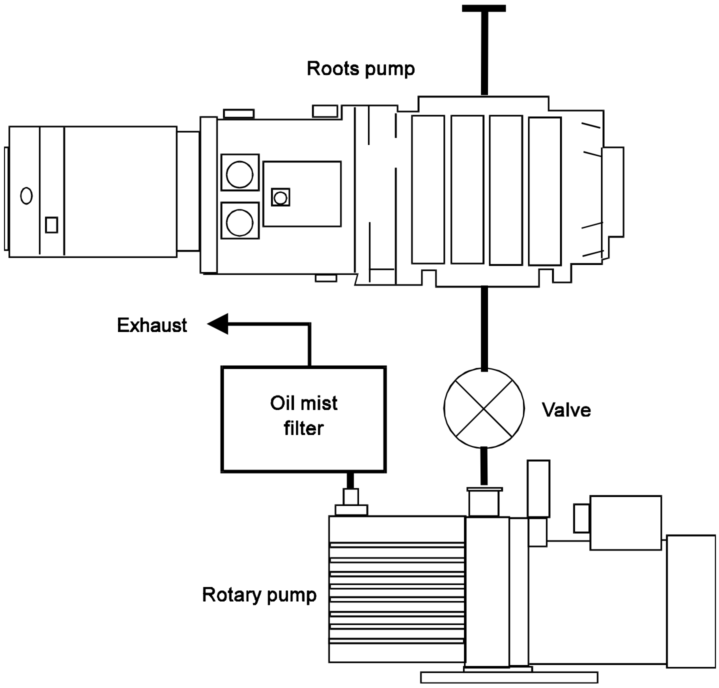

Figure 4 Roots pump system

2.1.6 Sorption pumps (or sorbs)

Sorption pumps are often used in vacuum spaces because they are cheap and reliable, and they require little maintenance. The adsorbent material (usually activated charcoal or a molecular sieve) has a very large surface area, and the gas molecules are trapped onto the surfaces when the sorb is cold.

Liquid nitrogen cryostats usually have a sorb fitted to the outside of the nitrogen vessel to maintain a good insulating vacuum. If a sorb is not used the vacuum slowly deteriorates as the warm surfaces outgas. A 77 K sorb will not trap helium gas, but if it is cooled to 4.2 K helium gas may be pumped to below 10^{-5} mbar. Liquid helium vessels are cold enough to freeze any gas except helium onto the metal surfaces, so a sorb is not usually fitted because it may hinder leak detecting operations.

These pumps are single shot devices. They eventually become saturated and have to be warmed (and sometimes evacuated with a suitable high vacuum pumping system) to regenerate the absorbent material. The amount of gas that can be pumped before the sorb is saturated depends on the type of gas and the temperature of the pump, but in a high vacuum environment, they may be expected to last for a period of months or years before they need to be regenerated.

2.1.7 Cryopumps

A cryopump usually consists of a large number of metal plates cooled to a temperature close to 4.2 K, (either by liquid helium or by a closed cycle cooler). Like sorption pumps, these are single shot pumps and they have to be regenerated when a layer of ice has collected on the metal surfaces. The pump relies on the fact that the vapour pressure of most materials at a temperature below 10 K is negligible. This type of pump is essentially clean and it is suitable for use in ultra high vacuum systems.

2.2 Vacuum accessories

2.2.1 Oil mist filter

An oil mist filter is used to remove the fine mist of oil from the exhaust gas of a rotary pump. It is desirable to remove this mist for the following reasons.

- The vapour represents a health hazard if inhaled
- It may contaminate any flow meters or other fittings behind the pump, changing their calibration
- It is desirable to avoid contaminating the helium recovery system with pump oil.

Several different types of filter are available. The following are the most common:

a) Coalescing filters, which only need to be replaced if they are dirty. The oil normally runs back into the pump continuously.

b) Centrifugal filters or 'catch pots', which usually have a transparent bowl to collect the oil, and have to be emptied occasionally.

2.2.2 Vacuum gauges

High vacuum is normally measured using a combination of Pirani and Penning type gauges. Typically, the Pirani gauge operates in the range 10 mbar to 10^{-3} mbar, and the Penning in the range 10^{-2} mbar to 10^{-7} mbar. The calibration of these gauges (and some others) depends on the type of gas in the system.

Vacuum in the range from 1 to 1000 mbar is normally measured with reasonable accuracy using a simple capsule or dial gauge. Other types of gauge are available, for example, Piezoelectric gauges and Baratron gauges, which allow accurate remote measurement.

2.2.3 Pumping lines

The pumping lines may have as large an effect on the efficiency of the vacuum system as the pumps themselves. Check the following points:

a) The lines must be leak tight. Plastic or rubber materials are sometimes used as pumping lines but they may be permeable to helium gas.

b) The throughput of the lines must be at least as high as that of the pumps. Otherwise their impedance limits the flow of gas and may affect the base pressure at the cryostat end of the line. It will certainly affect the amount of time required to pump down to the required pressure.

c) The lines should be clean inside. If there is any moisture in the lines it will limit the pressure that can be reached. If the lines are heavily contaminated with helium gas it will be difficult to perform the normal leak tests.

2.2.4 Mass spectrometer leak detectors

Although these machines are expensive, it is very useful to have access to one. The leak detector need not be dedicated to one system; it can be shared by the lab or the department. In general, the more complicated the system is, the more useful the leak detector will be. For example, a complex dilution refrigerator system may have 500 to 2000 joints that must be leak tight. Many of these are subject to thermal cycling, and a leak from almost any of them could cause a system failure. It is clearly important that any leaks can be traced and cured as quickly and easily as possible. A sensitivity of 10^{-8} mbar l/s (or standard cm^3/s) is sufficient for most purposes, but 10^{-10}mbar l/s is preferred when looking for very fine leaks or superleaks.

If you have little or no experience of using these leak detectors , refer to section 3.

2.2.5 Foreline traps

A foreline trap is sometimes used on the inlet of a rotary pump to reduce the amount of oil backstreaming up the pumping line. The active material in the foreline trap must be changed regularly so that it remains effective.

2.2.6 Choosing an appropriate 'O' ring material

Silicone rubber is often used for 'O' rings in electrical equipment, but it is not generally suitable for cryogenic equipment because the material is porous to helium gas. However, it is probably suitable for dynamic seals at temperatures down to -60°C, or static seals down to -100°C. It can also be used up to 250°C.

Butyl rubber was an old favourite material for vacuum applications, and it was often used because of its low gas permeability. Suitable for temperatures down to -60°C.

Nitrile rubber (or Buna N) is probably the best material for most common vacuum applications. It is cheap, easily available in a range of sizes, and appropriate for temperatures slightly below room temperature. It is also resistant to silicon grease (for example, vacuum grease). Its working temperature range is from -40°C to +120°C.

Fluoroelastomer (for example, 'Viton') is also suitable for vacuum. It is better than nitrile rubber for high temperature applications, but it is more expensive and tends to be deformed permanently after being compressed for a length of time. Its working temperature range is from -20°C to +200°C. Beware: if it is subjected to temperatures much higher than 200°C, the black sticky residue contains hydrofluoric acid!

Teflon (or PTFE) can also be used to make vacuum seals. However, the joint has to be designed to prevent the Teflon 'creeping' when it is compressed.

3 Detecting vacuum leaks

3.1 Introduction

These notes describe how to locate leaks in complex vacuum systems using a helium sensitive mass spectrometer leak detector. They do not describe how to use the leak detector in detail, because so many different models are available. Consult the instruction booklet for this information (and good luck!). Better still, ask someone to show you how to use the leak detector.

Warning: **Before you attempt to carry out a leak test, it is important to check that it is safe to evacuate a vessel, and that there is no risk of it collapsing because of the external pressure of the atmosphere. This is especially important for vessels which have thin walled tubes (for thermal reasons) and for large vessels. If you collapse a vacuum vessel you might be badly injured by the shock wave or by flying fragments.**

Helium sensitive leak detectors are used because:

- Helium atoms are small and mobile, so they can pass through small holes easily
- Helium gas is inert and safe to use
- There is very little helium in the air allowing the leak to be located precisely

Vacuum leaks are most commonly associated with:

Welds	Leaks caused by imperfect welds, cracked welds, or corrosion around the weld.
Soldered joints	Leaks caused by imperfect joints, or corrosion.
'O' ring seals	Dry, damaged or broken 'O' rings, or scratches or hairs lying across the seal are the most common sources of problems. (As a rough guide, a hair lying across an 'O' ring may cause a leak in the range 10^{-6} to 10^{-3} mbar ls^{-1}, depending on many factors.)
Indium seals	Insufficient compression of the indium wire, dirt on the metal faces or scratches across the seal may cause leaks. Problems after thermal cycling might point to poor flange design.
Glued joints	Leaks may be caused by bad joint design, bad surface preparation, inappropriate choice of glue, or rapid thermal cycling.
Porosity of metals	Gas sometimes leaks along the grain of a metal (especially in some grades of brass). Small flanges are commonly made of plate rather than bar for this reason.
Diffusion	Many plastics and composites are porous to helium gas at room temperature but not when cooled down. Materials must be chosen appropriately for the working environment and temperature range.
Thermal cycling	Leaks may be undetectable at room temperature but only open when the component is cooled to liquid nitrogen temperature. Sometimes the leak will still be detectable when it is warmed up again. If not, repeated thermal cycling may help to open the leak path and make detection easier.

Superfluid leaks

Components that are leak tight at room temperature and even in liquid helium may leak when subjected to superfluid helium (which has zero viscosity). This is the most difficult type of leak to find!

3.1.1 Getting started

Vacuum leak detection is an art, but a scientific approach helps. When you start to learn how to use the leak detector you will almost certainly find yourself looking for leaks that do not exist, and you could waste hours if you are not careful. These notes should help you to avoid most of the common problems.

Most leak detectors have an audible signal and a visual display. Both of these are useful. The visual display is used to quantify a leak and detect a slow change in the signal, so it is especially useful to help you locate small leaks. The audible signal is much easier to use for general leak testing, because you do not have to look at the leak detector. You can then concentrate on looking at the equipment that you are testing and if you hear the signal rise you can go back over the same area again more slowly, and try to pin point the position of the leak.

From time to time the sensitivity of the leak detector should be checked and reset using a 'standard leak', since the sensitivity peak may drift.

3.2 Leak testing a simple vessel

3.2.1 Preparations

Consider first how to test a simple vessel for leaks: for example, a flexible pumping line. The principles learnt here can then be extended to more complex systems.

Evacuate the line to a rough vacuum using a suitable rotary pump, and then pump it to a sufficiently high vacuum for the mass spectrometer to be used (typically 10^{-5} mbar). Many leak detectors will evacuate the vessel and switch on the mass spectrometer for you automatically.

Select a suitable sensitivity range so that a small leak can be detected. For most cryogenic systems the 10^{-8} mbar l/s range (or 10^{-8} standard cm^3/s) is best. If you use a more sensitive range than this the background helium signal in the vacuum space may exceed full scale on the leak detector. If you use a less sensitive range you may not notice the leak.

If the background signal is too high to allow you to use a sensitive range, pump the vessel until the signal has been reduced sufficiently. You can sometimes reduce the signal more quickly by 'pumping and flushing'. Pump the air out of the system until it reaches a pressure of a few mbar, allow dry nitrogen (or air if this is not available) into the vacuum space again (slowly to avoid damaging the vacuum vessel), and repeat the process as often as necessary.

3.2.2 Leak testing the pumping line

Spray the pumping line with helium gas, paying special attention to any joints. If possible, place it in a plastic bag, and fill the bag with helium, so that there is no chance of missing a small leak in an unexpected position. However this is likely to be impractical for large vessels.

If the signal on the leak detector rises at any time during the test, a leak should be suspected, and you should methodically check to find out whether the leak is real, or an artefact caused by the outgassing of some trapped gas within the vacuum system.

3.2.3 Work from the top

Remember that helium gas is lighter than air so it rises. Therefore you should start by spraying gas on the highest point, and slowly work downwards. If the signal on the leak detector rises at any time, go back over the area that you have just covered, and check again. If you do not start at the top you can get misleading results when you are checking an area below the position of the real leak.

When you have found the approximate location of the leak, fit a fine nozzle to the end of the helium gas line, and reduce the flow of gas. Check the suspect areas in detail. You can locate leaks very precisely. Usually (but not always) you can see a small hole, flaw in the material or dullness of the surface at the leak position.

If you want to test a long weld on a large vessel you can fix a tunnel of plastic sheet to the vessel with adhesive tape and fill the tunnel with helium gas.

3.3 Locating 'massive' leaks

Occasionally you may find a leak that is so big that you cannot reduce the pressure sufficiently to use the mass spectrometer. How can you find the position of the leak?

3.3.1 The safe method

Connect a large displacement medium vacuum pumping system in parallel with the mass spectrometer, as shown in Figure 5.

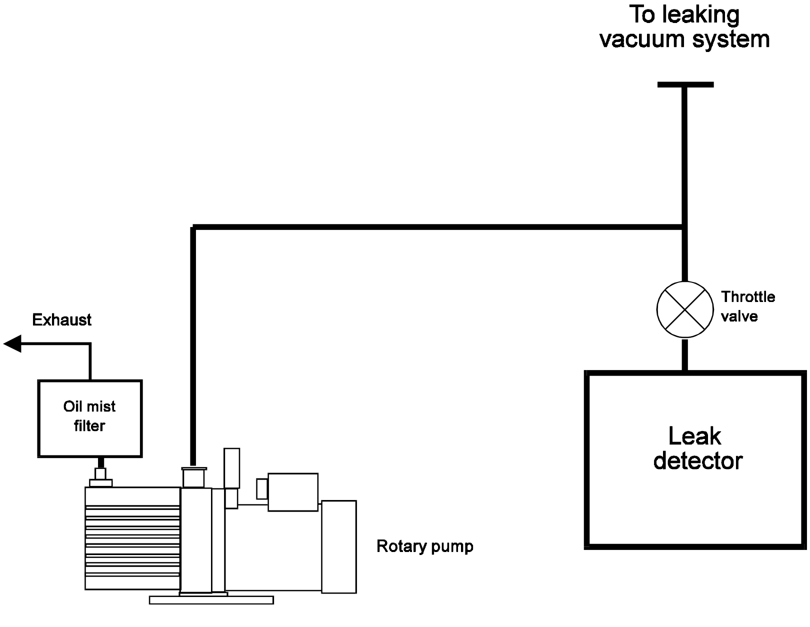

Figure 5 Locating a massive leak in a vacuum system

Pump the vacuum system to a rough vacuum with the rotary pump. Then slightly open the throttle valve on the pumping port of the mass spectrometer. Make sure that the pressure at the inlet of the leak detector is not too high for it to work properly.

It is sometimes possible to check for a leak by slightly pressurising the vessel with helium gas as described in section 3.8.

3.3.2 The other way (at your own risk)

Some people use water, acetone or methanol to locate leaks on small systems. Open the gas ballast valve on the pump to make sure that contamination does not collect in the pump oil.

Hazard: Acetone or methanol are flammable so you must not use them in large quantities. Take care not to create a fire hazard - contact your safety officer first.

Pump the vessel to a rough vacuum and measure the pressure. Brush liquid onto the outside of the vessel. The pressure rises quickly when the area of the leak is found.

These liquids may also be used to block a 'massive' leak temporarily, so that the rest of the vessel may be tested. Apply the liquid with a brush. Initially the pressure rises, but soon the liquid freezes and blocks the leak. You can remove the ice by gently warming the area with a hot air blower.

3.4 Leak testing sub-assemblies

If you are building a complex system you can test the sub-assemblies before you join them together. In this way you can locate leaks before the system is assembled. Components that are not fitted with standard vacuum fittings can be sealed to suitable plates using a product such as Apiezon ™ 'Q compound'. This is a malleable material that can be used to make a temporary seal, but it is only suitable for use at room temperature.

Occasionally you may find a component that has a detectable leak in one direction but not in the other. Therefore it is best to test components by evacuating the side that will be under vacuum in the finished assembly. In any case you must check that it is safe to evacuate the vessel, and that there is no danger of it collapsing.

3.5 Testing more complex systems

Most real cryogenic systems are quite complex. It may be useful to consider a liquid nitrogen shielded liquid helium dewar, which would usually be tested at room temperature and at liquid nitrogen temperature. If a vessel is leak tight at 77 K it is unusual for it to develop a leak as it is filled with liquid helium at its normal boiling point. This is probably because most materials undergo very little thermal contraction below 77 K, so thermal stresses induced by cooling to 4.2 K are smaller than those caused by the initial pre-cooling process.

The dewar must be leak tight in the following ways:

- Outer vacuum chamber (OVC) to air
- Liquid helium reservoir to OVC
- Liquid nitrogen reservoir to OVC

Evacuate the OVC, and set up the leak detector to monitor it. Check the outside of the dewar as described in section 3.2. Then flush the liquid nitrogen and liquid helium vessels with helium gas. The best way to check them thoroughly is to pump the air out of each vessel using a small rotary pump, and then to fill each vessel in turn with helium gas.

Sometimes the signal on the leak detector rises and falls again as the pressure in one of the reservoirs changes. This might not indicate a leak; small movements of the vessels can release gas from the surfaces. You can check this by repeating the test using air instead of helium gas.

If there is any doubt about the presence of a minute leak, use a chart recorder to monitor the signal from the leak detector. You should then see a step on the chart when the helium gas is allowed into (or pumped out of) the suspect space. This makes it easier to distinguish between a real leak signal and noise on the signal.

When you have finished the room temperature leak tests, you can pre-cool the cryostat to 77 K. Then blow the liquid nitrogen out of each space in turn using helium gas, while you monitor the OVC with the leak detector.

3.5.1 Pumping and flushing with helium gas

When you have removed all of the liquid, pump the vessel to a pressure of a few mbar, and then fill it with helium gas. This ensures that small leaks are not blocked by remaining droplets of liquid nitrogen.

3.5.2 Temperature effects

If you see a signal rise it may have been caused by a temperature change. Helium gas (trapped on the surfaces) may be released as warm gas is allowed into the vessel.

3.5.3 Masking cold leaks

If you discover a cold leak, you might be able to determine the approximate position of the leak by refilling the vessel with liquid nitrogen and then blowing the liquid out again with helium gas. When you see the signal rise again stop blowing out the liquid, and measure the level of the liquid. The leak is probably at this height in the vessel.

3.5.4 More complicated systems

You can check even more complicated systems in a similar way; for example, dewars with variable temperature inserts or dilution refrigerator inserts in the liquid helium reservoir. Think in advance about the best order to carry out the leak tests. In this way you can often reduce the number of pumping operations. You may be able to test several vessels at once. It is only necessary to check them individually if you discover a leak.

3.6 Leaks at 4.2 K and below

Any leak found at a temperature below 4.2 K is difficult to locate, because helium gas tends to be absorbed onto the cold surfaces. It may take a long time for the leak detector to respond to a small leak, so patience is essential. Do not be tempted to hurry the tests. It may take many hours for a positive result to be obtained. It is sometimes best to warm up the system to 77 K and pump the helium gas away thoroughly before cooling to 4.2 K again, because this removes the gas absorbed on the cold surfaces. Check each possible source of the leak in turn, with the others under vacuum to eliminate any possibility of confusion. At best, you will only be able to determine which space is responsible for the leak.

If the leak is very small, it is possible for the leak detector to pump away the helium gas at the same rate as the leak, and so the signal may not be seen to rise at all. In this case, close off the vacuum space for a few hours, and compare the signals before and after the test period. Make sure that the conditions for both readings are identical, so that thermal effects on the outgassing rate can be neglected.

3.7 Superfluid leaks (or superleaks)

Superleaks will only be seen at temperatures below the lambda point (2.2 K). Superfluid liquid ^{4}He (also known as helium II) has zero viscosity and can pass through very small holes quickly. Fortunately these leaks are quite rare, because it can take days or weeks to cure them.

Location of the precise position of the leak is extremely difficult. It may be possible to open up the leak sufficiently to detect it at 77 K (or even room temperature) by rapid thermal cycling, but this technique is not always successful. Failing this, the only other course of action is to replace joints or components, starting from the easiest operations.

This would normally be done in the following order (probably with a leak test after each step):

- Indium seals replaced
- Wood's metal joints re-run
- Soft soldered joints re-run
- Silver soldered joints re-run[1]
- Welds re-made
- Components or sub-assemblies replaced

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[1] You can only re-run silver soldered joints if there is no soft solder nearby. If soft solder is heated to the melting temperature of silver solder it can dissolve other materials into solution. It is then almost inevitable that the joint will leak, even if a large hole has not appeared.

3.8 Overpressure leak detection

Overpressure helium sensitive leak detectors ('sniffers') can be used to detect helium gas in the air. You can use these to detect very large leaks on vacuum systems but they should not be relied upon for the routine testing of cryogenic equipment. The vessel should be slightly pressurised with helium gas, and the sniffer is then used to detect where the gas is escaping. It is also important to check that it is safe to over-pressurise the vessel before trying to use this technique.

Sniffers are especially useful to detect the location of a leak in a helium recovery system, or on the (room temperature) fittings on the helium reservoir of a cryostat.

4 Cryostats and coolers

Various types of cryostat are available, and each type has advantages and disadvantages compared with the others. The following notes may help you to decide on the best type of system for your application.

In general, if the cryostat has to be very large (for example, to contain a superconducting magnet or conventional ^{3}He/^{4}He dilution refrigerator) it is best to use a 'bath' cryostat. You can fit a suitable continuous flow insert within the bath cryostat to achieve the sample temperature range required for your experiment.

However, if the cryostat has to fit into a small space or has to be thermally cycled rapidly and often, and the experimental equipment does not need a self contained reservoir of cryogen, it may be better to feed liquid from a remote storage dewar through a special transfer tube. This is called a 'continuous flow' cryostat.

The different types of cryostat that are widely available are described individually in the following sub-sections.

4.1 Bath cryostats

Bath cryostats contain large enough supplies of cryogens for a convenient period of operation. There is no need to refill the cryostat continuously from a storage dewar. The 'hold time' depends on a number of factors, (for example, size, experimental heat load and cryogen consumption rate). They are typically designed to give operating periods between 10 hours and 4 months.

Two types of bath cryostat are commonly used for laboratory scale liquid helium temperature systems. Both types are vacuum insulated to reduce the heat load due to conduction and convection. However, the helium reservoir is shielded from the room temperature radiation heat load in different ways. According to Stefan's Law, the amount of heat radiated from a warm body to a cold body varies with the difference between the fourth power of their temperatures. Therefore a 300 K surface radiates 230 times more heat to a 4.2 K surface than a 77 K surface would radiate onto the same 4.2 K surface.

Therefore liquid helium reservoirs are always shielded from room temperature radiation by a cooled shield. In most cryostats, the radiation load is further reduced by the use of 'multi-layer superinsulation'. This consists of many thin layers of low emissivity material, in the insulating vacuum space.

4.1.1 Liquid nitrogen shielded cryostats for liquid helium

In this type of cryostat, the liquid helium reservoir is surrounded either by a reservoir of liquid nitrogen, or by a shield cooled by this reservoir. The liquid nitrogen vessel is thermally linked to the neck of the liquid helium vessel to form a thermal barrier to heat conducted down from room temperature. Figure 6 shows a typical small liquid nitrogen shielded cryostat used to cool an infra-red detector to 1.5 K.

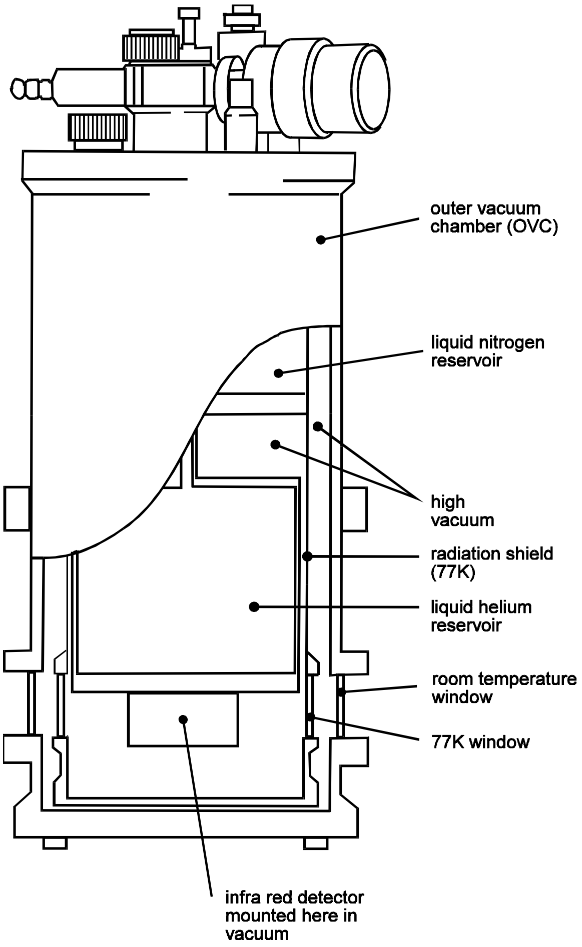


Figure 6 Low loss infra-red detector cryostat

The advantages and disadvantages of liquid nitrogen shielded cryostats are summarised in Table 1 on page 23.

4.1.2 Vapour shielded cryostats for liquid helium

As an alternative to liquid nitrogen cooled shields, it is possible to link several thermal shields to the neck of the liquid helium vessel. The cold gas that has evaporated from the reservoir is then used to cool these shields. This type of cryostat typically has between two and six shields (depending on the required performance) linked to different points on the neck. The space between the shields is filled with superinsulation.

The boil off rates of the two types of cryostat are similar, providing that there are no dimensional constraints on the system. Table 2 shows the advantages and disadvantages of vapour shielded cryostats.

Advantages of liquid nitrogen shielding	**Disadvantages of liquid nitrogen shielding**
The shield forms a firm point to fix the temperature of windows or thermal anchors, and the temperature of the shield is fairly constant. The system may be warmed up quickly by allowing gas into the vacuum space. The small amount of superinsulation does not become badly contaminated, and the gas can be pumped out to an acceptable level. Comparatively short systems can be made, because of the firm 77 K thermal link in the neck of the helium vessel.	Liquid nitrogen must be filled regularly. Boiling liquid nitrogen creates intermittent vibration since it tends to boil in bursts. The gas flow from the LN_2 exhaust port may be very low for an extended period as liquid in the cryostat stratifies. Liquid near the bottom of the reservoir can become warmer than the surface because of the hydrostatic pressure of the liquid above it. When this stratification is disturbed the evaporation rate increases dramatically. This is sometimes sufficient to blow liquid out of the cryostat.

Table 1 Liquid nitrogen shielded cryostats for liquid helium

Advantages of vapour shielded cryostats.	**Disadvantages of vapour shielded cryostats.**
Liquid nitrogen does not need to be re-filled. Vibration levels may be reduced, since there is no vibration from the intermittent boiling of liquid nitrogen.	Warming up the system may take longer than it would take if the vacuum space could be 'softened' with gas. Very short systems may have a higher boil off than a corresponding liquid nitrogen shielded system. The temperature of the shields varies with the liquid helium level.

Table 2 Vapour shielded cryostats for liquid helium

4.1.3 Bath cryostats for liquid nitrogen

In some ways it is more difficult to make a reliable bath cryostat for liquid nitrogen than for liquid helium. Unlike liquid helium, liquid nitrogen is not cold enough to freeze (or cryopump) all the contaminating gases in the surrounding vacuum space onto a cold metal surface. The quality of the vacuum is critical for operation of the cryostat, so a sorption pump (containing charcoal or molecular sieve) is normally fitted to the outside of the liquid nitrogen reservoir to maintain the vacuum. The vessel is usually superinsulated, but other insulation techniques are occasionally used; for example, filling the vacuum space with a low conductivity material such as a suitable mineral powder.

4.2 Lambda point refrigerators

Superconducting magnets are usually operated in liquid helium at 4.2 K. Their performance can often be enhanced by cooling the magnet to lower temperatures as described in section 6.3 on page 41. The simplest way to achieve temperatures below 4.2 K is to pump the whole liquid helium reservoir with a rotary pump, to reduce the vapour pressure above the liquid. If the bath is cooled to 2.2 K in this way, about 35% of the helium is evaporated to cool the remaining liquid. Temperatures below 2.2 K can be achieved, but if the bath is cooled below the lambda point, the liquid helium consumption increases significantly (both to reach the low temperature and to maintain it).

This simple approach has several disadvantages. A large amount of liquid is used to cool the magnet down, and since the reservoir is then below atmospheric pressure, access to the reservoir is difficult and all the fittings on the top plate have to be reliably leak tight. The liquid helium can only be re-filled by de-energising the magnet to its 4.2 K field and filling the reservoir to atmospheric pressure with helium gas, which interrupts the experiment.

Lambda point refrigerators (also known as 'lambda plates' or 'pumped plates') are used to cool superconducting magnets to about 2.2 K and maintain this temperature continuously. See Figure 7. They consist of a needle valve (to control the flow of liquid helium into the refrigerator) and a tube or chamber with a pumping line. They are normally built into the 'magnet support system'. The refrigerator is in good thermal contact with the liquid helium just above the magnet.

Liquid is continuously fed into the refrigerator and pumped to a low pressure so that it cools. The cooling power is determined by the liquid flow rate and the size of the pump, and it can be adjusted using the needle valve. High flow rates are typically used at high temperatures to cool the system quickly or to obtain high cooling power, but when base temperature is reached, the flow can be reduced to make operation as economical as possible.

The density of liquid helium changes rapidly with temperature, so strong convection currents are set up, around the magnet. The cold liquid from the refrigerator sinks to the bottom of the reservoir, cooling the magnet and keeping it at about 2.2 K. Meanwhile the warmer liquid above the refrigerator is affected very little. The thermal conductivity of the liquid is so low that the region immediately above the plate has a steep temperature gradient, and the liquid surface remains at 4.2 K and at atmospheric pressure. It is important to make sure that this thermal gradient is maintained, and not short circuited by high conductivity components.

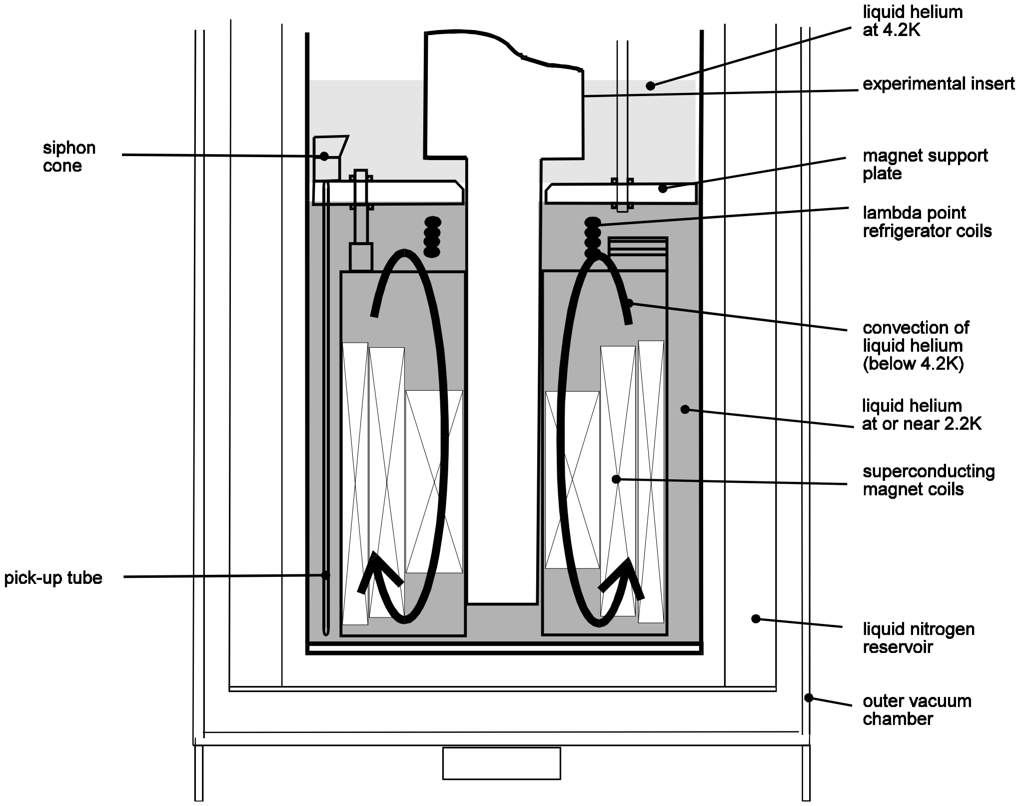


Figure 7 Lambda point refrigerator

Lambda point refrigerators have several advantages. In particular:

a) Since only a small proportion of the liquid in the reservoir is cooled by the lambda plate less liquid has to be used, and this reduces the cost of operation.

b) Operation can be automated (using a Teslatron Lambda controller).

c) The reservoir can be refilled without stopping operation of the system, as long as the transfer tube does not stir the liquid and upset the temperature gradient above the lambda plate.

The performance of these systems is dominated by the amount of liquid helium that has to be cooled. Although the mass of the magnet is much larger than that of the liquid, its heat capacity is very much lower. It is possible to calculate the amount of heat that has to be removed if the magnet and liquid are cooled from 4.2 K to 2.2 K. In a typical system, containing a 50 kg magnet, there may be about 3 litres (0.5 kg) of liquid below the lambda plate. Only 5 J has to be removed from the magnet, but about 3 kJ has to be removed from the liquid. Therefore it is important to minimise the amount of liquid around the magnet so that it will cool quickly and cheaply.

In most systems the magnet can only be cooled to 2.2 K in this way, because liquid helium has a phase change (the lambda point) at this temperature. Below the lambda point, the liquid becomes 'superfluid' and has a very high thermal conductivity, so the phase transition can only occur if the whole reservoir is cooled to the lambda point.

The heat from any warmer region in the reservoir would be rapidly conducted to the colder region, keeping its temperature above the critical level. However, in a few specialised applications, the refrigerator is built into the top of a separate chamber around the magnet. The refrigerator is fed from a 4.2 K liquid reservoir, but thermally isolated from it. The lambda plate then cools the whole of this chamber, and temperatures below the lambda point can be reached and maintained continuously, while the liquid is at atmospheric pressure. The optimum temperature is about 1.8 K, as the superfluid is then able to carry heat away from the magnet most effectively.

4.3 Continuous flow cryostats

A wide range of continuous flow cryostats is available. Some of these are supplied with cryogens from a storage vessel; others are mounted in a bath cryostat which supplies liquid. In most of these systems the cooling power available from a flow of cryogen (LN_2 or LHe) is balanced by power supplied electrically to a heater near the sample (usually by a temperature controller).

4.3.1 Variable temperature inserts (VTI)

Variable temperature inserts are used in bath cryostats to adjust the temperature of a sample without affecting the helium reservoir. 'Dynamic' and 'static' types of VTI are available, and the advantages and disadvantages of each type are described in section 4.4. The inner parts of the insert are vacuum insulated from the liquid helium. There may also be a radiation shield between the sample space and the liquid reservoir to reduce the radiated heat load on the reservoir when the sample is at a high temperature. This shield is usually cooled by the exhaust gas or the boil off from the main bath.

The temperature range of a VTI is typically from 1.5 to 300 K, but in certain circumstances this range may be extended. The sample temperature can be controlled continuously at any point in this range. Lower temperatures can often be achieved in single shot mode: the sample space is filled with liquid and the needle valve is closed to allow the pump to reduce the vapour pressure above the liquid to the lowest possible level.

4.3.2 Independent continuous flow cryostats (CF)

The operating principles of CF cryostats are generally the same as those of VTIs. Dynamic and static versions are available as described in section 4.4. However, they normally have their own independent thermal shielding, and they are supplied with coolant from an independent storage vessel through a 'low loss' or 'gas flow shielded' (GFS) transfer tube. These cryostats are sometimes used with a superconducting magnet if it has a room temperature bore. They are also used with resistive magnets.

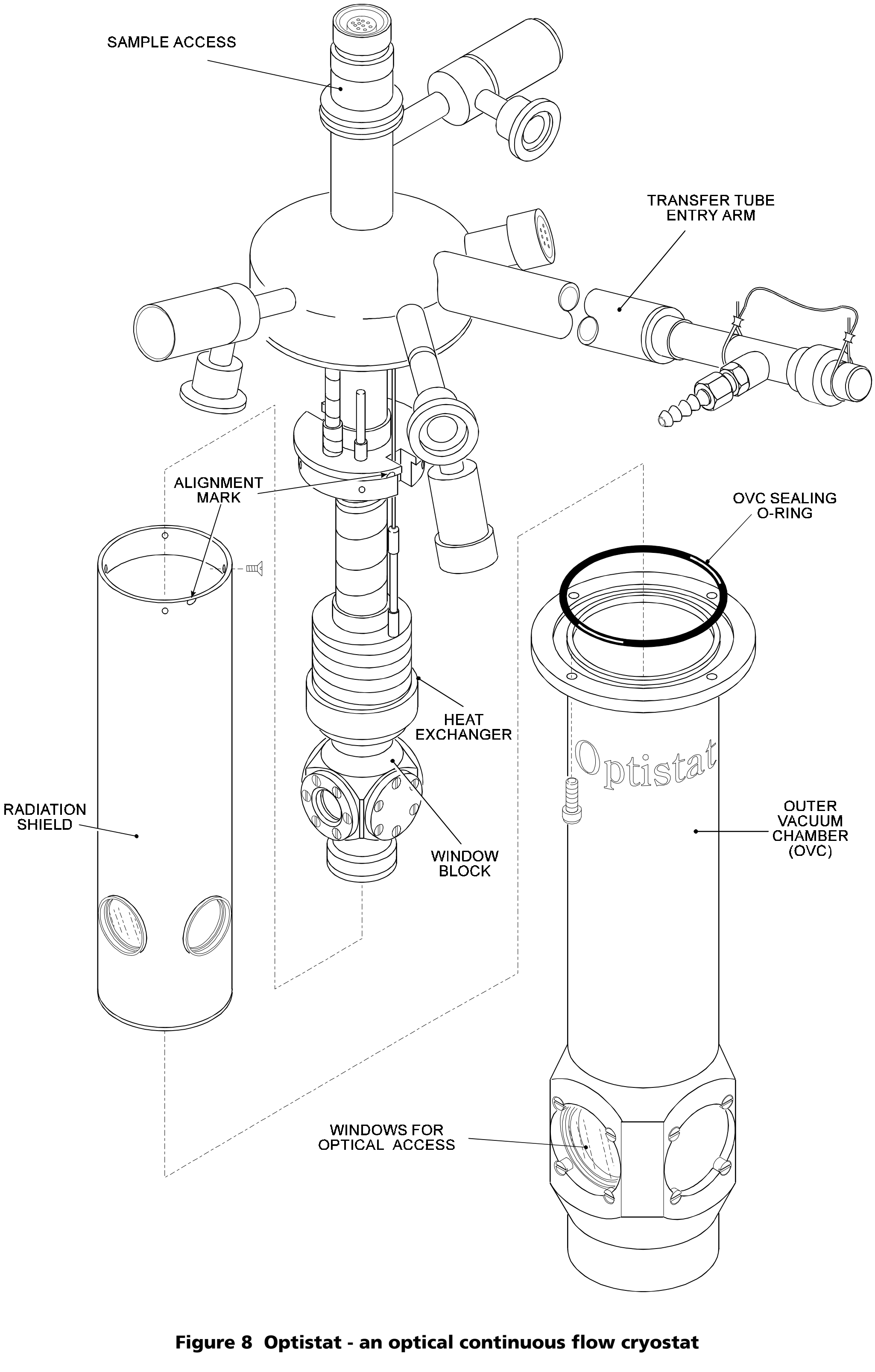


Figure 8 Optistat - an optical continuous flow cryostat

The transfer tubes are always vacuum insulated. In order to reduce the losses in the tube, GFS type transfer tubes use the enthalpy in the exhaust gas from the CF cryostat to cool a radiation shield in the tube.

The temperature range of CF cryostats is typically < 4 to 300 K in continuous mode, with lower temperatures available for limited periods in 'single shot mode'. However the range may be extended to give higher or lower temperatures if necessary. In general it is difficult to achieve temperatures as low as those available in VTIs because of the thermal losses in the transfer tubes, but some cryostats are designed to reach 1.6 K continuously. Figure 8 shows one of them schematically.

4.4 Static and dynamic continuous flow systems

Although all continuous flow cryostats work on the principle of balancing the cooling power of a flow of cryogen with electrical power from the temperature controller, there are several distinct types of cryostat: the most important are referred to as 'dynamic' and 'static'.

4.4.1 Dynamic systems

In a dynamic continuous flow cryostat, the sample is mounted in a flowing gas or in liquid, and its temperature is strongly influenced by the fluid. The temperature of the fluid is controlled by passing it through a heat exchanger (usually placed at the bottom of the sample space). The heat exchanger temperature is set by simultaneously controlling the cryogen flow rate and the heater on the heat exchanger. A temperature controller is usually used to do this automatically. Providing that the flow of cryogen through the heat exchanger is not too high the temperature of the flowing fluid can be controlled quite accurately. The fluid flows past the sample and out of the exhaust port of the insert to the pump.

This type of insert is easy to operate and it responds very quickly if the set temperature is changed to a new value. However, the temperature stability is not as high as that of a static insert. It is also possible to block the small capillary that feeds the cryogen to the heat exchanger with frozen water or air during the sample changing operation if care is not taken.

4.4.2 Static systems

Static systems are also fitted with heat exchangers, and the temperature of the heat exchanger is controlled in a similar way. However, the exhaust gas does not flow over the sample, but it passes out of the cryostat to the pump through a separate pumping line. The heat exchanger usually forms an annulus around the sample space, and thermal contact is made to the sample through exchange gas. The exchange gas pressure can be adjusted to suit the conditions. The sample temperature follows the temperature of the heat exchanger, but rapid temperature fluctuations tend to be filtered out, and the temperature stability of the sample can be improved considerably. In some cases, a heater is fitted to the sample block for fine control of the temperature or to warm the sample quickly.

Static inserts are as easy to operate as the dynamic type, and have the advantage that it is not possible to block the heat exchanger during the sample changing process. Indeed, quite large amounts of air may be frozen into the sample space without affecting the operating procedure. However, the increased sample temperature stability has to be traded off against the increased time taken to change the sample temperature to a new value. In particular, it is not possible to cool the sample as quickly, and static systems are generally used for small sample spaces.

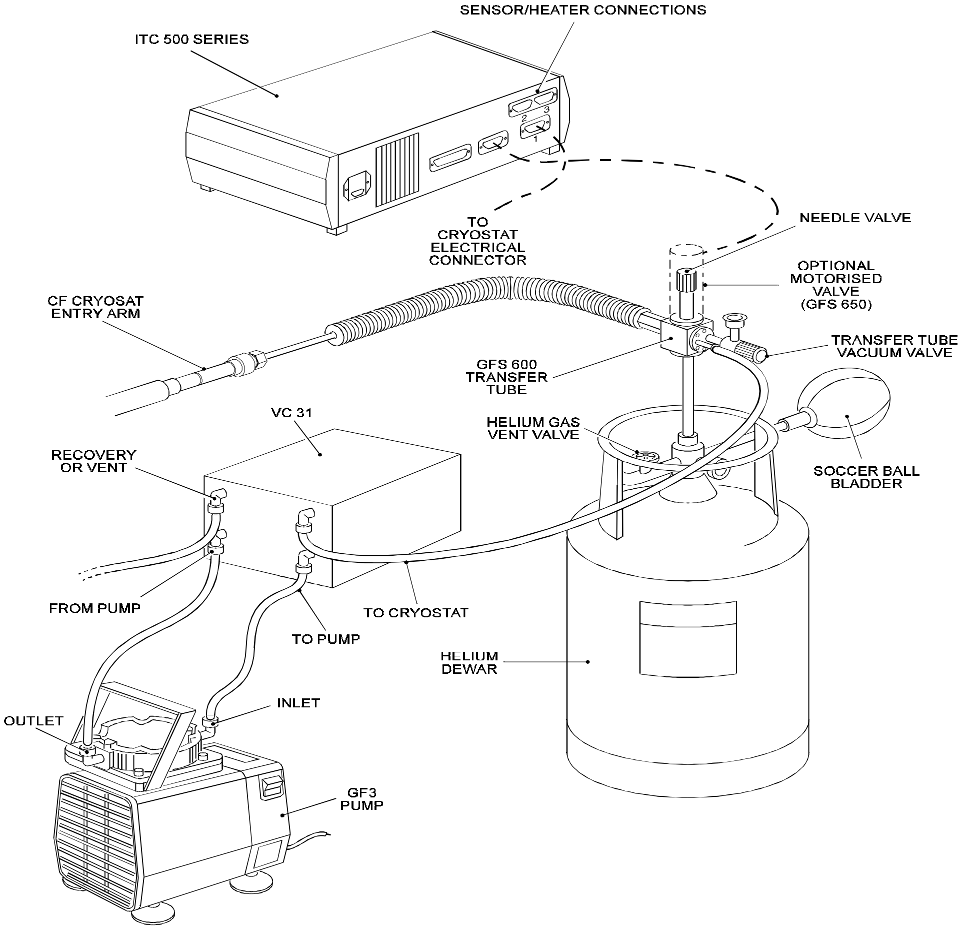


Figure 9 Flow system for a continuous flow cryostat

4.5 Storage/transport dewars

Storage (or transport) dewars are generally only suitable for supplying cryogens to the cryostat, (whether it is of the bath or continuous flow type). They are designed to be robust and to have a low evaporation rate. They usually have very narrow necks and a large amount of superinsulation. A few liquid helium storage dewars are fitted with liquid nitrogen jackets (especially older dewars).

However, some small variable temperature inserts are available to fit into storage dewars, providing that the diameter of the neck is sufficiently large, (50 mm). In particular, Oxford Instruments can supply a variable temperature inserts (the Compact VTI), a ^{3}He refrigerator (Heliox 2VL) or a ^{3}He/^{4}He dilution refrigerator insert (Kelvinox15) to fit into a storage dewar. These inserts give temperature ranges from 0.03 to 300 K.

4.6 Closed cycle coolers

Modern closed cycle coolers offer a highly reliable method of achieving low temperatures. They may either be used alone, to cool a sample and a radiation shield, or with a bath cryostat to cool one or two radiation shields and thus reduce the evaporation rate of the cryostat. This can considerably extend the hold time of a low loss cryostat, but it is not usually appropriate if the equipment inside the cryostat has a high consumption rate which has a dominant effect on the hold time. It is now possible to build cryogen free systems containing superconducting magnets.

However, this type of cooler has a high initial cost and the pay back time (in terms of reduced cryogen costs) may be very long. They also need to be serviced regularly (typically every 5,000 hours). There is also the possibility of introducing unwanted vibration into the experiment if it is not mounted very carefully.

4.7 'Stinger' systems

Some closed cycle cooler systems are used to re-condense helium gas into a bath cryostat continuously. They take the form of a cold finger that fits into the helium reservoir. They need quite high cooling powers both at the 4.2 K stage and at higher temperatures because they have to provide enough cooling to replace the enthalpy of the boil off gas, which usually helps to cool the neck of the reservoir. The helium reservoir is normally pressurised slightly so that the gas recondenses effectively, and so the liquid helium is held at a temperature close to 4.5 K.

4.8 Peltier effect coolers

Peltier effect coolers work by the thermoelectric effect; they are a thermodynamically reversible low impedance devices, operating at a high current from a d.c. power supply. A single stage cooler can typically achieve a temperature of -40°C, and lower temperatures can be achieved using several stages. A six stage device may achieve -100°C and give a cooling power of around 1 mW at -80°C. They do not introduce vibration into the cryostat. Although they have a small temperature range and limited cooling power, they offer a cheap solution for some requirements, (for example, Peltier effect cooled baffles, see section 2.1.3).

4.9 Making indium seals

Oxford Instruments uses two main types of indium seal, as illustrated in Figure 21 on page 80. 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.

4.9.1 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.

4.9.2 Making the seal

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

4.9.3 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.

5 Ultra low temperatures

Refrigerators working at temperatures below 1 K are used for a surprisingly diverse range of applications in research establishments. A range of specialised techniques is used to achieve these temperatures. Most of the systems described in the previous chapters use liquid helium and liquid nitrogen to reach and maintain low temperatures, but it is difficult to achieve temperatures significantly below 1 K using these cryogens alone. However, most ultra-low temperature systems are immersed in liquid helium (^{4}He) at 4.2 K, so that the heat load from the surroundings is minimised.

It is possible to reach temperatures slightly below 1 K by pumping liquid ^{4}He to a low pressure but very large pumps are required and it is not usually economically viable. ^{4}He may also be used to give very low cooling powers at temperatures down to 0.7 K in 'vortex refrigerators' which rely on the special properties of superfluid ^{4}He.

However, the valuable lighter isotope of helium, ^{3}He, is usually used in refrigerators working below 1 K. Evaporating ^{3}He is used in some systems, and temperatures slightly below 0.3 K can be achieved by reducing its vapour pressure. Temperatures below 0.3 K are usually reached by continuously diluting a flow of ^{3}He in liquid ^{4}He using a ^{3}He/^{4}He dilution refrigerator.

5.1 ^{3}He Refrigerators

^{3}He refrigerators are usually designed for routine operation in the temperature range from 0.3 to 1.2 K, and they use evaporating ^{3}He as the refrigerant. Their operating range can often be extended to 100 K or higher. Some of these systems can run continuously, returning the liquid ^{3}He to the system to replace the evaporated liquid. Others work in 'single shot' mode, by pumping on a small charge of liquid ^{3}He condensed into the system. In an efficient cryostat a 20 cm^3 charge of liquid ^{3}He may last for longer than 50 hours. Small laboratory refrigerators may give a cooling power of a few milli-watts at 0.5 K, but very large and high powered machines can give cooling powers of several watts at this temperature.

5.1.1 Sorption pumped ^{3}He systems

Sorption pumped ^{3}He systems are usually single shot refrigerators, capable of high performance operation for a limited time. Several types of system are available to suit the majority of laboratory requirements. Most of them can be used with high field superconducting magnets if required. The top loading systems allow the sample to be mounted on a probe which is loaded directly into liquid ^{3}He. They may also be designed to operate in rapidly sweeping magnetic fields, and a wide range of special services may be fitted to make connections to the sample. The maximum temperature limit is typically 100 K.

The Heliox 2VL insert is a low cost miniature ^{3}He system designed to allow inexperienced users to cool samples to 0.3 K. It is designed for operation in a liquid helium storage dewar, or with a superconducting magnet system. The sample is mounted in vacuum, and wiring can be connected easily. The whole insert is removed from the cryostat to change the sample, but since it is small, the time scale for sample changing is similar to that on the top loading systems. The Heliox system can be run up to about 200 K if it is used with a superconducting magnet, but higher temperatures (up to 300 K) can be reached if the insert is pulled up into the neck of the cryostat.

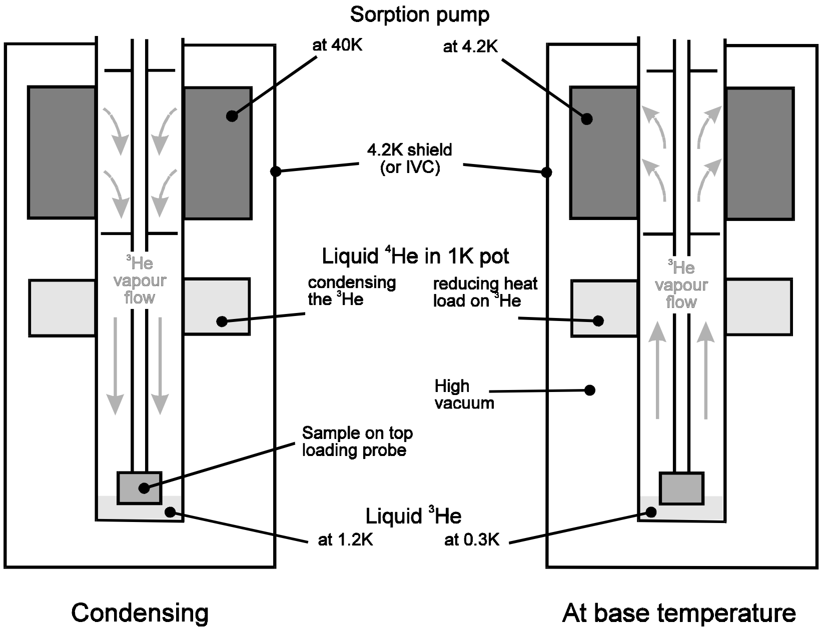


Figure 10 Principle of operation of a typical sorption pumped ^{3}He system (top loading type)

Figure 10 shows the working parts of a typical system. Although a top loading insert is shown, the principle of operation is similar for all Oxford Instruments' sorption pumped inserts. The insert has an inner vacuum chamber, (IVC), to provide thermal isolation from the main liquid helium bath.

The sorption pump, (or sorb), will absorb gas when cooled below 40 K, and the amount of gas that can be absorbed depends on its temperature. It is cooled by drawing some liquid helium from the main bath through a heat exchanger. The flow of ^{4}He through the heat exchanger is promoted by a small diaphragm pump and the rate of flow is controlled by a valve in the pumping line. A heater is fitted to the sorb so that its temperature can be controlled.

The 1 K pot is used to condense the ^{3}He gas and then to reduce the amount of heat conducted to the sample space. It is fed from the main liquid helium bath through a needle valve, and it can be filled continuously.

During condensation, the sorb is warmed above 40 K. When it is at this temperature it will not absorb any ^{3}He (see Figure 10). The ^{3}He condenses on the 1 K pot assembly and runs down to cool the sample and ^{3}He pot to the temperature of the 1 K pot. When most of the gas has condensed into the insert, the 1 K pot needle valve is closed completely so that the pot cools to the lowest possible temperature for optimum condensation. At this stage the ^{3}He pot is full of liquid ^{3}He at approximately 1.2 K. The sorb is now cooled, and it begins to reduce the vapour pressure above the liquid ^{3}He, (see Figure 10), so the sample temperature drops. As the limiting pressure is approached, the temperature of the liquid ^{3}He can be reduced to below 0.3 K.

The temperature of the sample can be controlled by adjusting the temperature of the sorb. If the sorb temperature is set between 10 and 40 K it is possible to control the pressure of the ^{3}He vapour, and thus the temperature of the liquid ^{3}He. However, if the best stability is needed, a temperature controller can be set up to measure the sample temperature and control the power supplied to the sorb heater. No heat is supplied directly to the liquid ^{3}He; this would evaporate it too quickly. The temperature of the sorb is continuously adjusted by the temperature controller, and the temperature of the sample can typically be maintained within 1 mK of the set temperature for the full hold time of the system.

These systems have limitations both in their cooling power and base temperature, and if high cooling powers (> 5 mW) are required, or operation must be continuous, it may be more appropriate to choose a continuously circulating ^{3}He refrigerator. If however, the base temperature is not low enough, a dilution refrigerator should be chosen. In general it is found that a dilution refrigerator has a better performance below 0.4 to 0.5 K, and a continuous ^{3}He system is better above this temperature. In either case, these refrigerators typically have large room temperature pumping systems, and they are therefore rather more expensive.

5.1.2 Continuously circulating ^{3}He refrigerators

Continuously circulating ^{3}He refrigerators are capable of giving high cooling powers and of operating continuously for a long period. They use an external room temperature pumping system (including a rotary pump and a booster pump).

The ^{3}He gas is injected into the cryostat and it is cooled to approximately 4.2 K by the liquid helium bath before it enters the IVC. It is then cooled to 1.2 K and condensed by the 1 K pot.

The liquid ^{3}He then passes through a special heat exchanger where it is cooled by the outgoing ^{3}He gas. Below this heat exchanger an impedance is used to keep the pressure in the condenser high enough even if the pressure in the ^{3}He pot is very low. On some systems a needle valve is used here as a variable impedance to set the ^{3}He flow rate. Since the liquid has already been cooled to a temperature close to that of the ^{3}He pot in the heat exchanger, only a small fraction of it evaporates as it expands through the needle valve. This ensures that the maximum amount of latent heat is available from a given flow rate of ^{3}He. The liquid and gas then enters the ^{3}He pot, which has a large surface area to give good thermal contact to the sample.

The flow rate determines both the base temperature and the cooling power available from the system. In general, a low flow rate will be required for a good base temperature, and a high flow rate will allow a high cooling power to be achieved.

5.2 ^{3}He/^{4}He Dilution refrigerators

The principle of operation of the dilution refrigerator was originally proposed by H. London in 1951, but the first working systems were not built until more than ten years later. Since that time, the performance of these systems has steadily improved, and the physical processes involved have become much better understood.

When a mixture of the two stable isotopes of helium is cooled below a critical temperature it separates into two phases. The lighter 'concentrated phase' is rich in ^{3}He and the heavier 'dilute phase' is rich in ^{4}He. The concentration of ^{3}He in each phase depends upon the temperature. Since the enthalpy of the ^{3}He in the two phases is different, it is possible to obtain cooling by 'evaporating' the ^{3}He from the concentrated phase into the dilute phase.

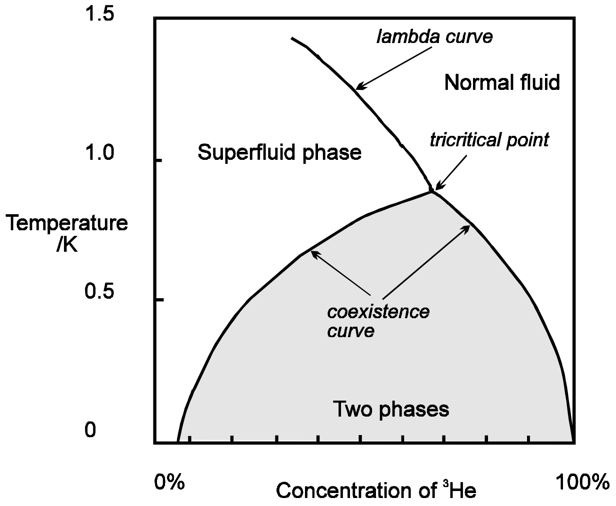


Figure 11 Phase diagram of ^{3}He/^{4}He mixtures

The properties of the liquids in the dilution refrigerator are described by quantum mechanics and the details will not be described here. However, it is helpful to regard the concentrated phase of the mixture as liquid ^{3}He, and the dilute phase as ^{3}He gas. The ^{4}He which makes up the majority of the dilute phase is inert, and the ^{3}He 'gas' moves through the liquid ^{4}He without interaction. This 'gas' is formed in the mixing chamber at the phase boundary. This process continues to work even at the lowest temperatures because the equilibrium concentration of ^{3}He in the dilute phase is still finite, even as the temperature approaches absolute zero. However, the base temperature is limited by other factors, and in particular by the residual heat leak and heat exchanger performance.

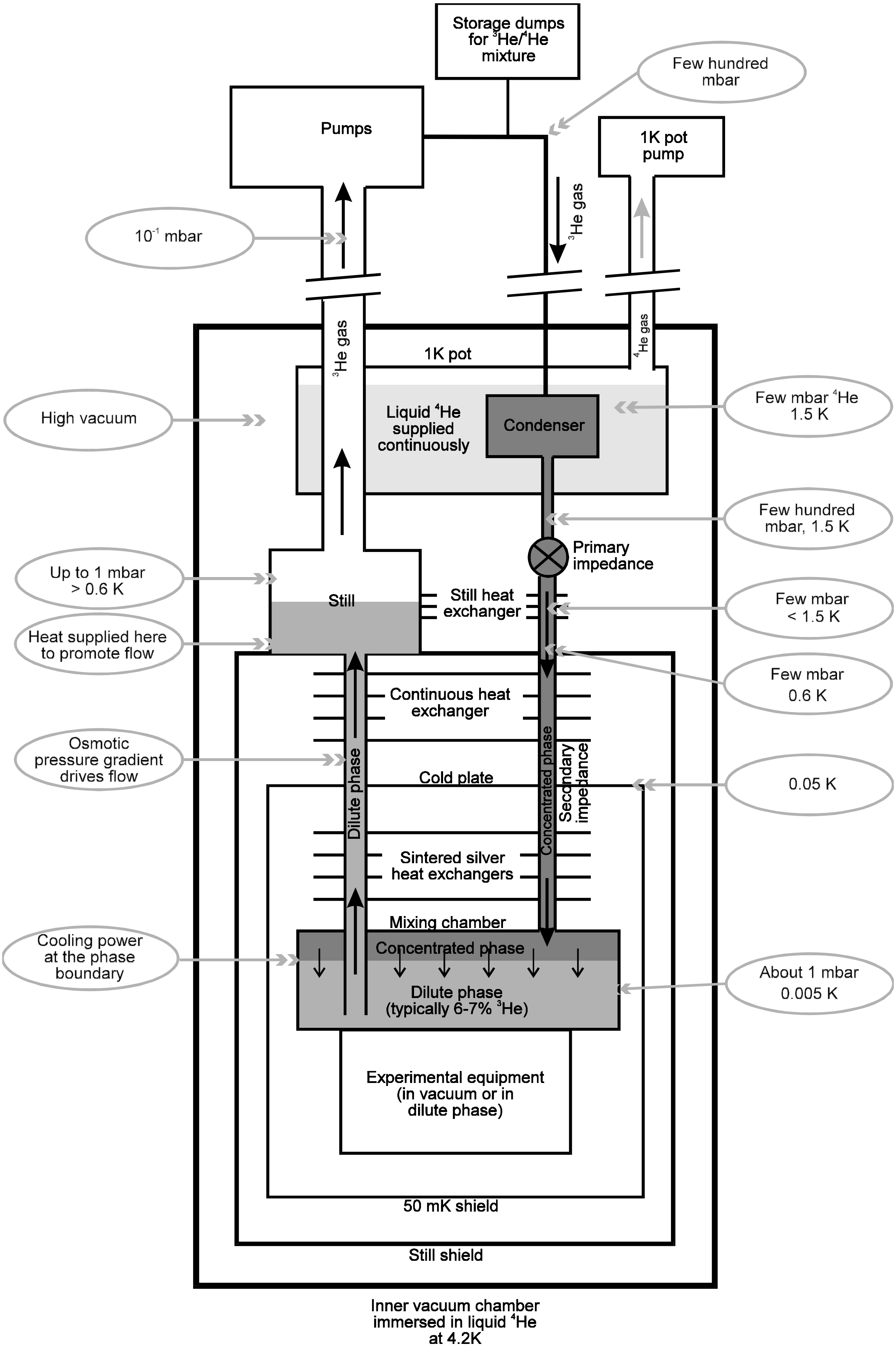


Figure 12 Schematic diagram of a dilution refrigerator

When the refrigerator is started the 1 K pot is used to condense the ^{3}He/^{4}He mixture into the dilution unit. It is not intended to cool the mixture enough to set up the phase boundary but only to cool it to 1.2 K. In order to get phase separation, the temperature must be reduced to below 0.86 K (the tri-critical point). The still is the first part of the fridge to cool below 1.2 K. It cools the incoming ^{3}He before it enters the heat exchangers and the mixing chamber, and phase separation typically occurs after a few minutes. Gradually, the rest of the dilution unit is cooled to the point where phase separation occurs.

It is important for the operation of the refrigerator that the ^{3}He concentration and the volume of mixture is chosen correctly, so that the phase boundary is inside the mixing chamber, and the liquid surface is in the still. The concentration of ^{3}He in the mixture is typically between 10 and 20%.

In a continuously operating system, the ^{3}He must be extracted from the dilute phase (to prevent it from saturating) and returned into the concentrated phase keeping the system in a dynamic equilibrium. Figure 12 shows a schematic diagram of a typical continuously operating dilution refrigerator. The ^{3}He is pumped away from the liquid surface in the still, which is typically maintained at a temperature of 0.6 to 0.7 K. At this temperature the vapour pressure of the ^{3}He is about 1000 times higher than that of ^{4}He, so ^{3}He evaporates preferentially. A small amount of heat is supplied to the still to promote the required flow.

The concentration of the ^{3}He in the dilute phase in the still therefore becomes lower than it is in the mixing chamber, and the osmotic pressure difference drives a flow of ^{3}He to the still. The ^{3}He leaving the mixing chamber is used to cool the returning flow of concentrated ^{3}He in a series of heat exchangers. In the region where the temperature is above about 50 mK, a conventional counterflow heat exchanger can be used effectively, but at lower temperatures than this, the thermal boundary resistance (Kapitza resistance) between the liquid and the solid walls increases with T^{-3}, and so the contact area has to be increased as far as possible. This is often done by using sintered silver heat exchangers, which are very efficient even at the lowest temperatures.

The room temperature vacuum pumping system is used to remove the ^{3}He from the still, and compress it to a pressure of a few hundred millibar. The gas is then passed through filters and cold traps to remove impurities and returned to the cryostat, where it is pre-cooled in the main helium bath and condensed on the 1 K pot. The primary impedance is used to maintain a high enough pressure in the 1 K pot region for the gas to condense.

The experimental apparatus is mounted on or inside the mixing chamber, ensuring that it is in good thermal contact with the dilute phase. All connections to the room temperature equipment must be thermally anchored at various points on the refrigerator to reduce the heat load on the mixing chamber and give the lowest possible base temperature. If the experiment is to be carried out at higher temperatures, the mixing chamber can be warmed by applying heat to it directly, and a temperature controller can be used to give good stability.

5.3 Sorption pumped dilution refrigerators

It is possible to build continuous dilution refrigerators which do not have external pumps for the ^{3}He/^{4}He mixture. Instead, two sorption pumps are used to pump the still to a low pressure. A cold valve is fitted between each sorb and the still. While one of the sorbs is pumping, the other is regenerating. The temperatures of the sorbs are adjusted by electrical heaters to control the pumping cycle.

A special 'collector' is fitted below the 1 K pot to hold the liquid condensed by the pot. The pressure in this collector is controlled by maintaining a constant temperature, so that the flow of ^{3}He to the dilution unit is kept constant even though the flow from the pumps to the condenser is not constant.

The advantages of these systems are that the vibration levels can be significantly reduced, and the refrigerator system is compact. Since the ^{3}He/^{4}He mixture remains in the cryostat it is less likely that air can leak into it and block the system. They are controlled by a computer, so they can be automated easily.

5.4 Nuclear demagnetisation systems

Temperatures below approximately 4 mK cannot be achieved easily or cheaply. Dilution refrigerators capable of reaching temperatures below 5 mK are available but they are large and expensive. Although temperatures as low as 2 mK have been achieved in this type of system, most experimentalists use other techniques.

Most experiments carried out below 4 mK rely on adiabatic demagnetisation of a nuclear paramagnet. This is a single shot process, but very long hold times can be achieved. However, the total amount of heat that can be absorbed from the sample by the demagnetisation stage is limited. Demagnetisation stages are typically pre-cooled to approximately 10 mK in a magnetic field of 8 to 10 T by a powerful dilution refrigerator. They are then isolated from the mixing chamber by a superconducting heat switch, and the magnetic field is slowly reduced. Temperatures slightly below 1 mK can be achieved using $PrNi_5$ (an enhanced nuclear paramagnet), but copper can be demagnetised to around 10 μK.

6 Superconducting magnet technology

6.1 Introduction

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 non-superconducting system.

Many types of magnet are available, but solenoids and split pairs (sometimes referred to as split solenoids) are the types most commonly encountered in the laboratory. These two types of magnet are shown schematically in Figure 13. Solenoids are generally simpler, and it is cheaper to produce a magnet with a given field using a solenoid than it is using a split pair. It is also generally possible to achieve better homogeneity of the magnetic field using a solenoid. The very high forces between the coils make it difficult to produce fields higher than 15 T using a split pair magnet. However split pairs give access to the sample perpendicular to the magnetic field. They are commonly used for optical experiments which require this access.

Fields up to 9 T are usually produced using NbTi superconductor at 4.2 K (or 11 T at 2.2 K); higher fields (up to 20 T) require the use of the expensive and brittle intermetallic compound Nb_3Sn. However the Nb_3Sn is only used for the inner sections of such a magnet (where the field is highest) and the outer sections use the cheaper NbTi for economic reasons. Many kilometres of wire are used in the winding of even a modest magnet.

Fields up to about 40 T can be achieved with hybrid magnets. In this type of system a large bore superconducting magnet provides the background field (up to 16 T) for a high power, water cooled inner winding.

It is also likely that 'high T_c' superconducting inner coils will soon be commercially available to enhance the field produced by a Nb_3Sn magnet. Although these materials cannot yet tolerate very high current densities they have exceptionally high critical fields when cooled to 4.2 K.

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). '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.

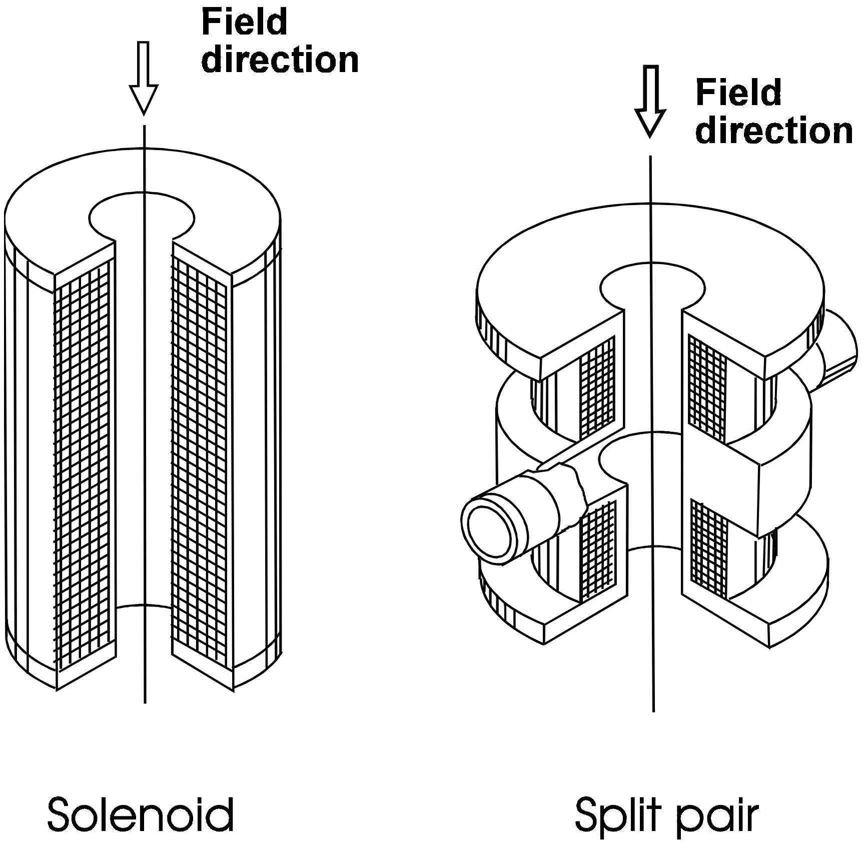


Figure 13 Schematic diagram of a simple solenoid and split pair

6.2 Construction of the magnet

Superconducting magnets are typically constructed from a number of coaxial coils. They are wound from different grades of superconductor so that the cost is reduced as far as possible. The coils are impregnated to give a high mechanical stability and thus to prevent relative movement of the components as the field is changed. The Oxford Instruments 'Magnabond' system has been developed to achieve this.

The coils of split pair magnets have to be supported especially carefully to resist the large forces between and within the coils. These forces are typically tens of tonnes. Compensation coils, shim coils and cancellation coils also have to be fixed very firmly to the main coils.

Electrical connections between the coils have to be made using superconducting joints so that the residual resistance of the magnet is reduced to a minimal level, (typically lower than 10^{-8} ohms).

The wire used in the construction of most laboratory magnets is multi-filamentary, because this improves the stability by preventing 'flux jumping' which dissipates energy in the superconductor. However, some low decay rate magnets are wound using single core wire because it is possible to make lower resistance joints in conductors of this type, (typically 10^{-14} ohms or lower).

6.3 Basic physics of the magnet

Although the basic physics taught to a 16 year old is sufficient to explain many of the phenomena observed in a magnet, the production of a reliable magnet is extremely challenging. The magnet is effectively a pure inductor with zero resistance, and the circuit theory taught in schools explains that the magnet stores energy, that there is a time constant associated with a circuit containing an inductor and a resistor in parallel, and that it is difficult to change the current flowing in the inductor because of the induced back e.m.f. (or voltage).

$$Stored\ energy = \frac{1}{2}LI^2 \qquad\qquad Induced\ 'back\ e.m.f'. = -L\frac{dI}{dt}$$

$$Time\ constant = \frac{L}{R}$$

where L is the inductance of the magnet, I is the current in the magnet, and R is the resistance in parallel with the windings. As an example, a magnet with an inductance of 100 H is not unknown in a laboratory cryostat, and if it was operating at a current of 100 A, the stored energy would be ½ MJ!

The induced (or 'inductive') voltage observed when the magnet is energised or de-energised is explained further in the section on the typical operation procedure (see section 6.9 on page 46). In many cases this induced voltage limits the rate at which the magnetic field can be changed (or 'swept'), because of the limitations of the power supply. However, there are several effects within the windings of the magnet that cause heating (for example, eddy currents, hysteresis and diamagnetism), and ultimately these limit the sweep rate.

Large stresses are induced in the windings of the magnet because of the Lorentz forces between the field and current. These forces lead to large hoop stresses (trying to explode the magnet) and axial compression in the windings.

For simple operation the magnet is cooled to 4.2 K using liquid helium at its normal boiling point. However, the properties of the superconducting materials in the windings of the magnet are improved when their temperature is reduced further. In many cases it is possible to obtain an enhanced performance by cooling the magnet to 2.2 K and energising it to a higher current. Enhancements of the order of 20 to 25% can typically be achieved. However, it is important to check that the magnet is designed to withstand the increased stresses before attempting to run it in this way, otherwise it may be badly damaged. The temperature is commonly reduced by a 'lambda point refrigerator', as described in section 4.2 on page 24.

Ferromagnetic materials close to the magnet can have positive or negative effects. On the positive side, they may be used for fine 'shimming' of the magnetic field in certain specialised magnets, or more commonly to reduce stray magnetic field to an acceptable level. On the negative side, they may make unwanted changes to the field shape in a region of high homogeneity and put extremely high forces on the magnet windings or the cryostat. The additional stress on the windings may even prevent the magnet from functioning correctly. For these reasons, any large magnetic items have to be positioned carefully, so that they neither affect the field shape nor cause damage to the magnet. If shielding is required, the effects on the magnet must be analysed carefully by computer simulation, and this is such a specialised field that it should only be undertaken by an expert.

6.4 Homogeneity of the field

The homogeneity of the magnetic field is often specified over a 10 mm diameter spherical volume (or d.s.v.). In a solenoid type magnet, a homogeneity of 1 in 10^3 can easily be achieved, and this is sufficiently high for the majority of experiments. This can be improved to 1 in 10^5 by using series shims (sufficient for low resolution NMR). However, high resolution NMR and similar experiments are usually carried out in magnets with homogeneity 1 in 10^7 (or better) over a 10 mm d.s.v., which can only be achieved using independent shims. It is much more difficult to obtain high homogeneity in a split pair magnet, and 1 in 10^2 to 10^4 over a 10 mm d.s.v. is typical. It is possible to achieve homogeneity of 1 in 10^6 (or better) if the magnet geometry is correct and independent shims are added.

6.5 Persistent mode operation

One of the main advantages of the superconducting magnet is the 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 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 fitted to the magnet in parallel with the main windings (see Figure 14). When the magnet is energised, the switch is held 'normal' (that is, not superconducting) by applying heat with an electrical heater. In this state, although the impedance of the switch is typically only a few ohms, it is so much higher that that of the magnet that almost all of the current flows through the magnet. Higher resistance switches are available for magnets which sweep rapidly. The typical energisation procedure is described further in section 6.9, and this will illustrate the operating principle of the superconducting switch.

6.6 Quenches

The magnet will only function properly if all 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). 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, evaporating the liquid helium very quickly and often warming the magnet to 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. It is said that the amount of energy released by a pin head falling through 1 mm is typically sufficient to start the quench, and the amount of energy released may be enough to put 250 g of butter into orbit! This is one of the reasons why the technology of superconducting magnets is so challenging. Even microscopic movements of the wires in the coils may be sufficient to quench the magnet.

After a magnet has quenched it will often be found that the quench has helped 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, and the magnet is always given a thermal cycle to room temperature before it is re-energised, (to ensure that it will not quench again).

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.

6.7 Protection circuit

The implications of a quench in an unprotected magnet are generally very severe. Consider the example of a magnet with an inductance of 100 H, in which the current decays from 100 A to zero in 1 second. An induced voltage of 10 kV could be produced, as the stored energy of ½ MJ is dissipated. Therefore we usually fit the magnet with a 'protection circuit' to assist in the dissipation of energy and to prevent damage to the windings. The high voltage induced during the quench still occurs inside the magnet, but the electrical insulation is sufficient to withstand it. The copper in the coil helps to dissipate the energy in the windings. The protection circuit prevents the energy stored in the other parts of the magnet from being dissipated in the quenched section by diverting the current around it.

Two main types of protection circuit are employed for Oxford Instruments magnets: resistor and diode/resistor. The design of protection circuits is a specialised field, and should not be undertaken lightly. The consequences of the use of an unsuitable design may be disastrous!

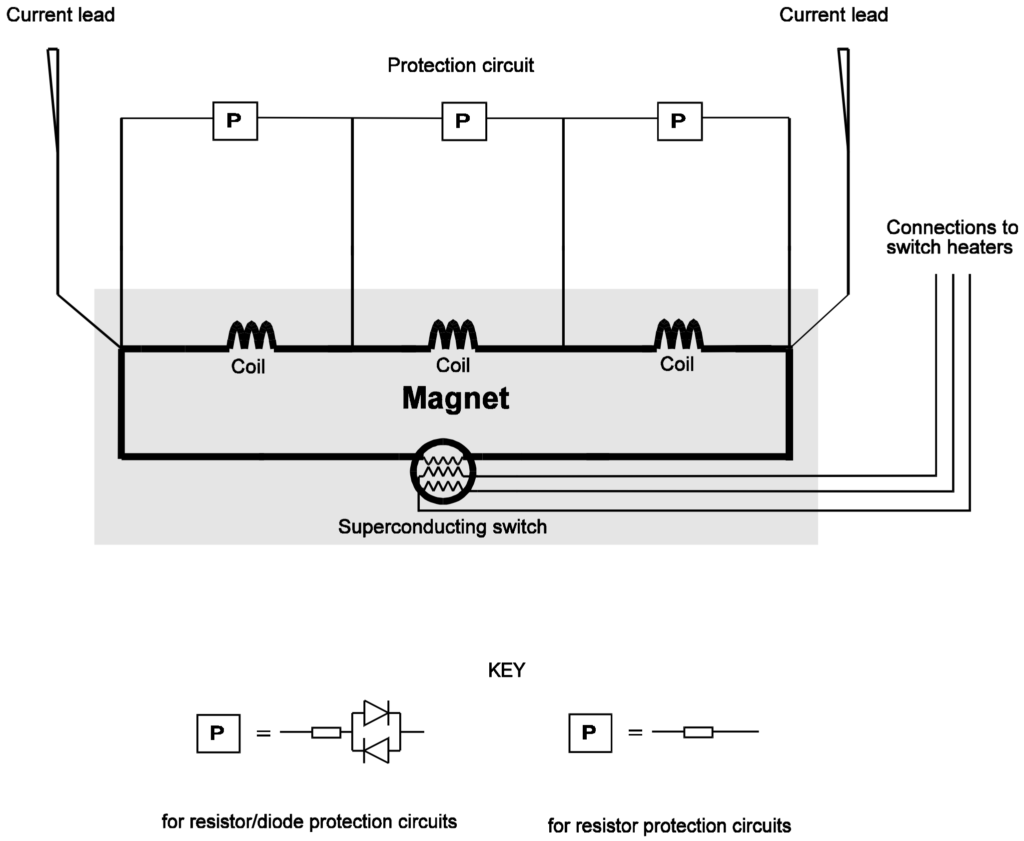


Figure 14 Typical resistor and resistor diode type protection circuit (see the key at the bottom of the diagram)

6.7.1 Resistor protection circuit

Resistor protection circuits are simpler and slightly cheaper than the diode/resistor type, but they have several disadvantages. The resistors are mounted in parallel with the windings, (see Figure 14) but this arrangement allows current to flow through the resistors when the magnet is swept (because of the induced voltage). Therefore if the magnet will be swept regularly it is necessary to mount the resistors on the baffles high in the neck of the helium reservoir, where the heat dissipated by the flowing current has little effect on the liquid helium evaporation rate. The wires from the magnet to the protection circuit conduct heat into the liquid helium and increase the evaporation rate of the system. Sometimes resistor protection circuits are mounted in the liquid bath if it is expected that the magnet will remain in persistent mode most of the time. This reduces the static boil off at the expense of higher boil off when the magnet is sweeping.

The other main disadvantage is that there is a time constant associated with this type of inductive/resistive circuit. It is sometimes necessary to wait for a few tens of seconds after a field sweep, for the current in the magnet to match the current from the power supply (or 'in the leads'). During this time, the induced voltage decays slowly (with time constant L/R).

6.7.2 Resistor diode protection

Refer again to Figure 14. The only modification to the resistor type is to put a set of diodes in series with each protection resistor as shown in the key to the diagram.

These special diodes have properties that are suitable for operation at low temperatures and high currents with high reliability. The arrangement of the diodes allows the current to flow in either direction in the event of a quench. The number of diodes is chosen so that the voltage normally induced during a field sweep is insufficient to 'open' them and let current pass through the resistor. During a quench the voltage rises until the diodes 'open', and the maximum voltage is then determined by the current flowing through the protection resistors.

Since no current normally flows through the resistors and diodes, they may be mounted low in the helium reservoir without affecting the helium evaporation rate during normal operation. Fewer heavy current leads into the liquid helium are needed, reducing the system boil-off. The other main advantage is that the magnet current follows the power supply current much more accurately, and a very short time constant is associated with the circuit. If no switch is fitted to the magnet, the time constant may be negligible. The induced voltage drops away quickly when the sweep is stopped.

6.8 Magnet power supplies

Although in principle it is possible to run a superconducting magnet with a very simple power supply, it is best to purchase an instrument that has been designed for the purpose. The power supply is required to supply a high current to a load which has low resistance and high inductance; it should be able to control the sweep rate and the superconducting switch heater, and in many cases it allows the polarity of the output to be changed so that the magnet can be energised in the opposite direction. It may also allow the magnet to be controlled by computer, using an RS232 or IEEE interface.

Modern power supplies include firmware to prevent most of the common mistakes made by inexperienced users. For example, if the magnet has been put 'persistent' the power supply remembers the set current for that field and prevents you from opening the switch at a different current by accident.

Power supplies using 'switched mode' technology provide the advantages of reduced volume and weight, and reduced power dissipation within the power supply. They are usually run in constant current mode (where the sweep rate is defined in A/min or T/min). Older designs of power supply could only be used in constant voltage mode (where the induced voltage is defined, and the magnet runs to a preset current) but it is more difficult to control the sweep rate in this way.

In any power supply that is controlled digitally, the current is changed in a series of discrete steps. The resolution of the power supply defines the size of these steps. Although they are small they may be significant in some applications.

6.9 Typical operating procedure

The operating technique for a typical superconducting magnet is presented here as a summary of the phenomena described above. This should reinforce your understanding of the fundamental principles.

6.9.1 Energisation to persistent field

Initially the power supply is connected to the magnet with the current and voltage at zero. The switch heater is energised using a small current (typically 50 mA). After a few seconds, the switch will be 'normal', and typically has a resistance between 10 and 100 ohms. This 'normal' resistance is now so much higher than that of the main windings that the current tends to flow through the magnet, not through the switch. The time constant for the magnet/switch alone is of the order of 1 second, and that this will be unaffected by a diode/resistor protection circuit, but may be increased if a resistor protection circuit is used. As the power supply increases the current, it is possible to check that the switch is open by observing the induced voltage (which is proportional to the sweep rate). As the current rises, the voltage will be seen to increase slowly as the resistive component (due to the current leads) becomes noticeable. Typically the resistance of the leads is 0.01 Ω. The total observed voltage is:

$$V = Ir + L\frac{dI}{dt}$$

where r is the resistance of the leads in series with the magnet windings.

The sweep rate should be adjusted according to the instructions in the manual, and it may be necessary to reduce the sweep rate at higher fields. If the sweep is stopped, the voltage drops to the resistive value (Ir); if resistive protection is used, the time constant may be a few tens of seconds, so the voltage will slowly decay to this value. When the desired field has been reached the sweep is stopped, and the voltage is allowed to settle. The current should be noted carefully to ensure that the magnet can be run down easily! The switch is closed by turning off the heater, and after about 10 or 20 seconds the current in the magnet leads is slowly reduced by 'running down' the power supply. (This process is referred to as '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.

6.9.2 Switch quenching

Occasionally the switch may quench as the magnet is put into persistent mode. In most stable magnets this is unlikely to quench the rest of the magnet. The magnet is likely to start to run down at the negative voltage set by the power supply, and it is possible to 'catch' it and run it back up manually using older power supplies. Modern power supplies are designed to detect a switch quench and 'catch' the magnet automatically. They switch on the switch heater and run the magnet back up to the set field automatically.

6.9.3 De-energisation or changing the persistent field

The opposite procedure is followed to de-energise the magnet. The leads are 'run up' to the value noted previously. The switch heater is then turned on, and the voltage usually changes slightly because the current in the leads does not precisely match the current in the magnet. The current is then reduced at the recommended rate and a reduced voltage may now be observed because the induced voltage is now opposite to the resistive voltage.

If the magnet is swept down the observed voltage may become negative. The magnet may now be run to the desired field and the switch closed again after a few time constants.

6.10 Magnets for special applications

Many superconducting magnets are built for special applications where the magnetic field shape may be more complex than that available from a solenoid or a simple split pair.

Some neutron beam applications require the use of an asymmetric split pair magnet, because a symmetric magnet has a zero field annulus within the clear access between the coils. As the neutron beam passes through the field reversal region it becomes de-polarised. This can be avoided by making one of the coils significantly larger than the other, moving the zero field region into the windings of the smaller coil.

Dipole and quadrupole magnets are commonly used for applications where a magnetic field is employed to bend a beam of charged particles (requiring a field perpendicular to the beam line). This type of coil is used in the Oxford Instruments superconducting synchrotron 'Helios'.

More complex designs sometimes use a large number of separate coaxial coils to produce a specific field shape. This type of magnet is often used to provide the field for high power radio frequency vacuum tubes, for example gyrotrons. These tubes require the field shape to be known precisely, and it can be adjusted while the tube is running.

6.11 Interfacing superconducting magnets to dilution refrigerator systems

Many dilution refrigerator systems (operating in the temperature range from a few mK to approximately 1 K) are fitted with superconducting magnets. In most cases the magnet is required because a wide range of physical effects can only be observed in a magnetic field. However it is also possible to produce even lower temperatures by adiabatic demagnetisation techniques. The detailed requirements of the system have to be tailored to suit the specific experiment and the following notes should help to summarise the factors that need to be taken into account.

The magnet has several effects on the performance of the dilution refrigerator system.

Eddy current heating A sweeping field, or vibration of a conducting component within a region where there is a field gradient, induces eddy currents in a conductor which are then dissipated as heat. The performance of these refrigerators may be affected by a heat load as low as 0.1μW!

Thermometry Many thermometers are affected by the presence of static or sweeping fields, making it extremely difficult to measure the temperature in the millikelvin range. Often, a cancelled field region is set up close to the field centre, so that the thermometer can be placed as close as possible to the experiment.

Effect on experiment Some of the experimental apparatus may be affected badly by the presence of the field.

If the magnet is fitted with a cancellation coil, the position of the cancelled region must be chosen carefully as described below. In general, the closer the cancelled field region is to the centre of field the more difficult it is to design the magnet because of the high forces between the coils. It is also difficult to give general guidelines about the distances required, because of the wide range of different magnets available.

In some dilution refrigerators the sample is mounted in the liquid ^{3}He/^{4}He mixture, which is contained in a non-metallic tube (for example, Oxford Instruments Kelvinox^{TLM} systems). In this case, the cancellation region is normally required only to reduce the amount of eddy current heating in the metallic parts of the mixing chamber above the magnet when the field is swept. Other systems are completely non-metallic below the 1 K pot (for example, Kelvinox ^{NT}). In either case the level of cancellation is not usually critical and it is not normally necessary to cancel the field in the vacuum space outside the mixing chamber. Field sensitive thermometers may be mounted inside the mixing chamber within the cancelled region.

Other systems use a large amount of sintered copper or sintered silver to improve the thermal contact between the sample and the ^{3}He/^{4}He mixture. If this material is used in a high field region the eddy currents induced in the sinter may dominate any heating effects from a sweeping field. If a cancellation coil is fitted to the magnet in this type of system it is for a different reason. The position is chosen only to allow a thermometer or the sensitive part of the experimental apparatus to be mounted conveniently in a low field region. The level of cancellation has to be chosen to suit the requirement, but the field is normally cancelled to 100 gauss (or sometimes 50 gauss). In general, the larger the cancelled region the more difficult and expensive it is to fit the cancellation coil.

One or two stage adiabatic demagnetisation systems may require several independent magnets. One (or two) are used for demagnetisation of the copper bundle(s) or the paramagnetic salt in order to achieve the required low temperature; another smaller coil is often used for a superconducting heat switch; a further magnet may be required to produce a field over the experimental apparatus. Clearly, all these coils have to be supported rigidly from each other. Complex cancelled regions may be required between the main magnets to allow for the superconducting switches, to reduce mutual inductive effects, and to reduce the eddy current heating effects within the conductors.

6.12 Summary

In summary, superconducting magnets are used for a very wide range of applications from laboratory scale equipment to large energy storage systems, and they may even be used in the propulsion mechanisms for large ships in the near future. Oxford Instruments is always interested in designing special superconducting magnets for novel applications.

7 Transferring cryogens

Before you attempt to transfer any cryogenic fluids, you must make yourself aware of the potential hazards. There is no substitute for training from a competent person, but the potential hazards are summarised in the Oxford Instruments booklet "*Safety Matters*".

7.1 Liquid nitrogen

Liquid nitrogen has a high latent heat of evaporation, and it is easy to transfer the liquid over short distances using uninsulated tubing. Most materials become very brittle when they are cooled to 77 K, and there is a risk of them shattering while the liquid is being transferred. If this happens it is likely that liquid will be sprayed over the surrounding equipment (and people). You should therefore use a material that is known to be safe, for example stainless steel tubing. It is only necessary to use a vacuum insulated line if liquid nitrogen is to be transferred over a long distance (say > 10 metres). Some laboratories have liquid nitrogen 'on tap', using such lines for connection to the storage vessel.

In most cases, the nitrogen supply is provided from a storage/transport vessel. These are usually of the self-pressurising type, with a pressure regulator fitted. In any case, the nitrogen is transferred by pressurising the vessel.

Some superconducting magnets and large cryostats may need to be cooled down slowly in order to avoid damage from thermal shock or unequal cooling rates in the different parts of the system. Refer to the instruction manual for the system.

7.2 Liquid helium

Liquid helium is notoriously difficult to handle. If you do not use the correct equipment or techniques you can easily lose all the liquid. The following information explains a suitable procedure and to points out some of the problems commonly encountered. Since the liquid is expensive, it is worth going to some lengths to avoid wasting it.

Liquid helium has a very low latent heat of vaporisation: that is, only a very small amount of heat is required to evaporate it. (For example, a heat load of 1 W will evaporate 1.4 litres of liquid per hour.) However, helium gas has a very high enthalpy. In other words, it is very easy to generate gas at 4.2 K, but it is much more difficult to warm that gas up.

Remember! Not much cooling power is obtained by evaporating the liquid helium but the cold gas can provide a high cooling power. See the table in section 10.4 on page 69 to estimate the amount of liquid helium required to cool a system to 4.2 K.

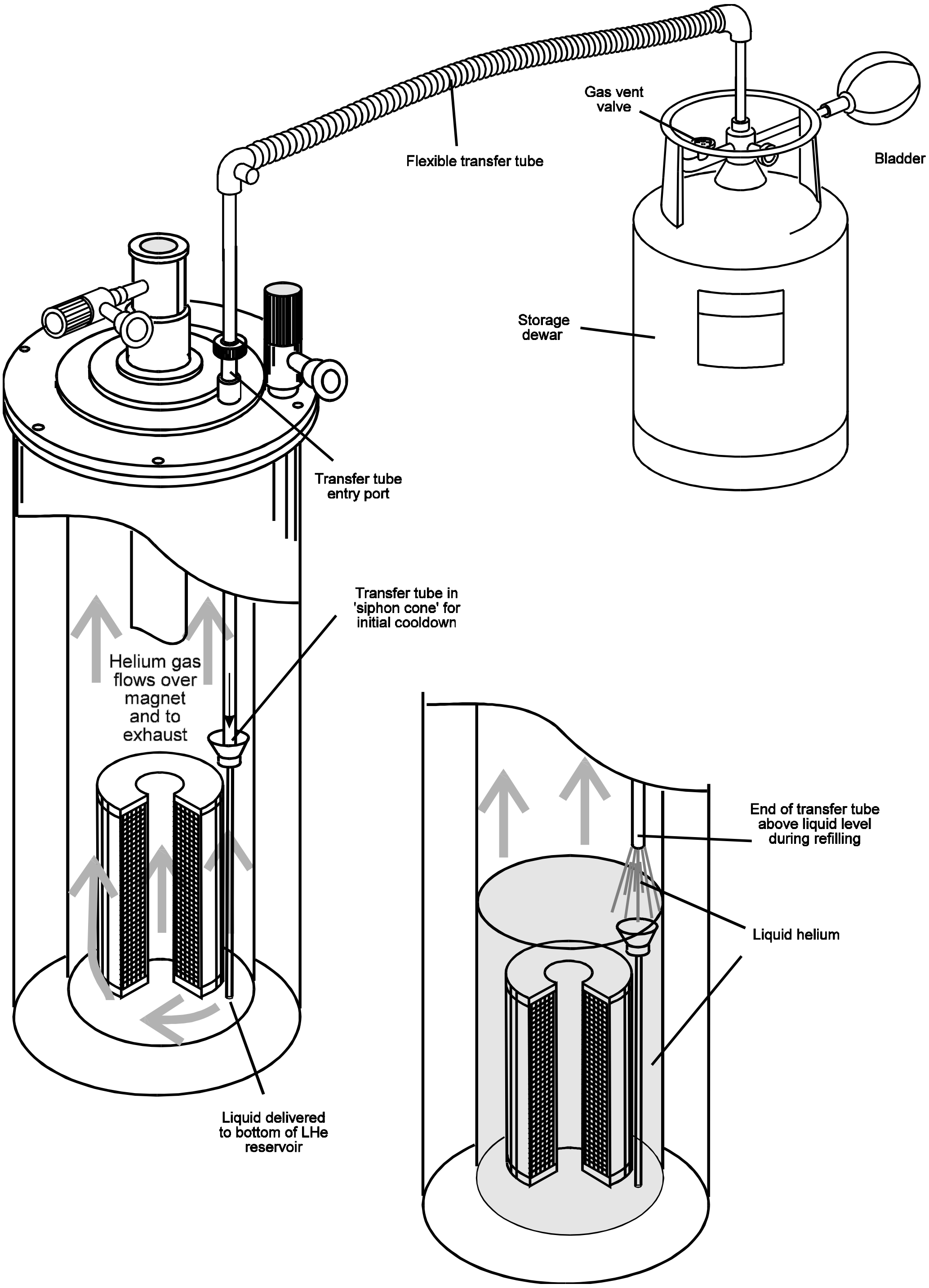


Figure 15 Transferring liquid helium into a typical laboratory cryostat

7.3 Using liquid helium efficiently

Transfer tubes

Liquid must be transferred through a vacuum insulated tube, so that it is thoroughly isolated from the room temperature surroundings. This tube is often referred to as a 'siphon'. Many different designs of transfer tube are available. The simplest and cheapest are of rigid construction. It is easier to use tubes with a flexible horizontal section because the two vessels do not have to be positioned as carefully. One end of the tube may be put into the storage vessel first, and when it has been pre-cooled, the other end can be put into the cryostat. However, rigid transfer tubes usually have lower loss rates.

Sensitive pressure regulation

It is important to be able to adjust the pressure in the storage dewar to set the liquid transfer rate. Dewars can be pressurised with the rubber lining (or 'bladder') of a soccer ball because of the low latent heat of liquid helium. When the bladder is squeezed gently warm gas enters the dewar and evaporates some of the liquid. This increases the pressure in the vessel. When the bladder is released, it expands to a larger size than before. Enough pressure can usually be generated by this technique to promote a transfer. It is especially useful during the slow transfer required to cool down the system. The pressure can be controlled with great sensitivity using a bladder.

Precooling

All but the smallest cryostats must be precooled to 77 K with liquid nitrogen, and the liquid nitrogen must all be removed before the helium transfer is started.

Deliver liquid to the lowest possible point

If you are cooling a system to 4.2 K you must deliver the liquid to the bottom of the helium reservoir. Initially, the liquid evaporates as soon as it leaves the transfer tube, but the gas then has to flow up past all the warm components in the helium reservoir, and this flow of gas cools them. In a complicated system it may not be possible for the transfer tube to reach the bottom, and a siphon cone is fitted higher in the reservoir. A small tube passes from the cone to the bottom of the reservoir; the transfer tube fits into the cone. See Figure 15 on page 51.

Slow initial transfer

During the initial cool down, it is important to transfer the helium very slowly. If the transfer is too fast the cold gas will pass out of the cryostat too quickly to make use of all of the available cooling power (enthalpy). A faster transfer may cool the system slightly more quickly, but only at the expense of considerable extra use of liquid. As a rough guide for a laboratory scale cryostat, set the flow rate so that the helium recovery line is only covered with ice for a few metres at most. If you see liquid air running from the recovery line the transfer rate is much too high.

When liquid collects

When the helium reservoir reaches a low enough temperature liquid starts to collect. You should see the ice on the recovery line start to melt quite quickly. You can then increase the pressure in the storage dewar to increase the transfer rate. You will not be able to generate a very high pressure by squeezing a bladder, but you can supply helium gas from a high pressure cylinder fitted with a suitable pressure regulator. A pressure of 300 mbar is typically suitable. If you transfer the liquid too slowly it takes longer to fill the cryostat and the losses in the transfer tube are proportional to the time taken, (for example 2 litres per hour).

Re-filling with liquid

When you have to refill the liquid helium reservoir you should pre-cool the transfer tube. Otherwise the warm gas from the tube will evaporate a large amount of liquid from the reservoir. Do not transfer the liquid to the bottom of the reservoir and do not push the transfer tube into the 'siphon cone' when refilling. Sometimes a 'phase separator' is fitted to the cryostat end of the transfer tube, to direct the warm gas up and away from the liquid surface, while the heavier liquid falls into the reservoir. It is not necessary to use this if care is taken to pre-cool the transfer tube. See Figure 15 on page 51.

7.4 Avoiding helium transfer problems

It is difficult to transfer liquid helium efficiently. You have to look for all of the available signs whenever you transfer it, otherwise you might transfer all of the liquid out of the storage dewar without collecting any in the cryostat. The following notes may help to see signs that the transfer is not working properly, or explain what went wrong so that you do not have the same problem again. Always look out for these signs.

'Touches' Touches between the inner and outer tube of the transfer tube are normally visible as icy patches. If there is a touch, some of the liquid will be lost in the transfer tube.

Poor vacuum If the vacuum in the transfer tube is poor, you will see condensation on most of the tube. The heat load on the inner tube will be high enough to evaporate most of the liquid. Sometimes the inner tube will cool enough to cryopump the gas in the vacuum space, and the transfer may then proceed normally, but you should pump the transfer tube thoroughly before you attempt another transfer.

Leak tight seals If the seal on the transfer tube port is not air tight, air will be cryopumped into the cryostat. It freezes between the transfer tube and the tube which guides it through the baffles. If you cannot pull the transfer tube out of the cryostat at the end of the transfer you may have to warm the whole system up to 77 K or higher.

Correct transfer rate Try to predict how much ice you expect to see on the recovery (or exhaust) line. If you get more (or less) than you expect, try to work out the reason.

7.5 Common problems

7.5.1 Recovery line covered with ice for more than a few metres

If the recovery line is cold over a long distance, it indicates that the transfer is too fast and that the cooling power of the gas is being wasted. Reduce the pressure in the storage vessel.

7.5.2 Very little ice on the recovery line

This sign usually indicates that the transfer rate is too low, usually for one of the following reasons:

a) The transfer tube may be blocked - remove it, warm it up and blow warm helium gas through the tube to clear the blockage.

b) The siphon cone or tube below it may be blocked - warm up the system to clear it.

c) The pressure in the storage dewar may be too low. In some laboratories the pressure of the recovery line is surprisingly high - increase the pressure to suit. Check (or non-return) valves in the recovery line can also affect the back pressure on the system.

d) The storage dewar may be empty. If so the pressure in the dewar usually drops again quickly after it has been re-pressurised.

e) The transfer tube may not be long enough to reach the liquid in the storage dewar. The symptoms would then be similar to those seen when the dewar is empty - if a longer transfer tube is not available you may be able to use an extension screwed to the end of the siphon. Transfer losses will be increased, and the extension should be no longer than 30 cm. Even if you use an extension tube, it is possible that no liquid will be transferred at all when the liquid level falls below the end of the insulated section of the transfer tube.

7.5.3 Cryostat shows no signs of cooling, or cooling too slowly

There are several possible causes for problems of this type:

a) Liquid may not be reaching the bottom of the reservoir - check that the transfer tube is long enough or that it fits into the siphon cone properly.

b) The transfer may be too slow - increase the pressure slightly or see section 7.5.2.

c) The system may not have been pre-cooled sufficiently well. For example, it is harder to cool the system from 100 K to 70 K than from 70 K to 40 K because the specific heat capacity of most materials changes quickly with temperature. Cryostats with vapour cooled shields need to be pre-cooled carefully, to ensure that the shields are cold enough.

d) The vacuum in the cryostat may have failed (or 'gone soft'). If the heat load due to conduction through the gas in the vacuum space is too high, the system will not cool. You can usually see condensation or ice on the outside of the cryostat if this happens.

e) The liquid nitrogen may not have been removed thoroughly enough after pre-cooling. A very large amount of helium is required to freeze nitrogen and cool it to 4.2 K - if this is suspected the system has to be warmed up. It can take a long time for frozen nitrogen to thaw and evaporate.

f) If the transfer tube has long extension pieces screwed on to it (even if they are 'vacuum insulated'), the liquid may be evaporated before it reaches the cryostat. Normally the cold gas will cool the system down, but liquid may not collect.

7.5.4 Cryostat cannot be filled to the expected maximum level

On some systems the liquid may start collecting, and the level starts to rise in the normal way, but suddenly the evaporation rate increases.

a) This may indicate that the cryostat has started to leak (check the OVC), but it is more likely to be that the liquid level has reached a heavy flange which has not been cooled sufficiently. If this is consistent with the drawings of the system, it may be best to stop the transfer for a few minutes (or hours) to allow the flange to cool. Then continue to fill.

b) Check that the level probe is working properly.

c) Very occasionally, thermal oscillations may be set up in the recovery line. This can introduce a very large amount of heat. Oscillations can usually be heard quite clearly. It sounds as if the cryostat is breathing. Change the configuration of the recovery line as much as possible. A buffer volume in the line may help to damp the oscillations.

d) Sometimes a gas meter in the recovery line can initiate oscillations. If you can by-pass the meter (even temporarily) it may help the oscillation to settle down.

e) Sometimes this sign indicates that there is a problem with the transfer tube. (I don't have a complete explanation for this observation!) If possible try using a different transfer tube. It does not need to reach the siphon cone providing that there is still liquid in the cryostat.

f) Finally, if the recovery system is running at a high pressure it sometimes causes problems of this type.

7.5.5 Problems with refilling cold systems

a) If the boil off is initially very high when the transfer tube is introduced try to pre-cool it more thoroughly - put the tube into the storage dewar first, and the pressurise the dewar slightly. You will hear oscillations as the tube cools. When cold liquid starts to emerge from the tube push it into the cryostat. In some laboratories, there are very strict rules on the recovery of helium gas. Consult the person who runs the recovery system for advice and training.

b) Many of the other problems described above may be encountered.

8 Cryostat wiring

A range of materials is used for wiring in low temperature experiments and it is worth considering which is the most appropriate for your experiment. Optimised wiring for a cryostat is often the result of a compromise between the thermal and electrical requirements of the system. A few simple techniques can be applied to the majority of situations. This chapter is intended to introduce you to these techniques. Other books give further details of more specialised techniques, and in particular, the book by Richardson and Smith (see section 12) contains much interesting information.

8.1 Thermal requirements

A limited amount of cooling power is available, and it is important to minimise the heat load on the system. Heat conducted along the wires (to the experiment or thermometers) therefore affects the temperature of the system. Materials with high thermal conductivity clearly affect the temperature of the system more than those with lower conductivity.

Unfortunately, most materials with high electrical conductivity also have a high thermal conductivity. This means that wires with low electrical resistance are likely to introduce more heat, (and affect the temperature more) than wires with high resistance.

However, superconducting materials have no electrical resistance, and if they are significantly below their superconducting transition point they have negligible thermal conductivity. Conventional superconductors can be useful at temperatures below approximately 8 K, and new high T_c materials are now being used above this temperature in some systems. Multi-filamentary superconducting wire with a low conductivity matrix (for example CuNi) is therefore especially useful in ultra low temperature systems.

If high heat loads from the wiring are inevitable because of the electrical requirements, it is often possible to use the exhaust gas from the cryostat to cool the wires. This is especially important in liquid helium systems as the enthalpy of the gas is many times higher than the latent heat of evaporation of the liquid. If the exhaust gas is allowed to flow freely over the wires, the conducted heat load may be reduced by a factor of 20 or more.

The low temperature ends of the wires usually have to be thermally anchored (or 'heat sunk') to ensure that they are at the required temperature. Details of the most common heat sinking techniques are given in section 8.3.3. Effective heat sinking of the wiring reduces the amount of heat conducted into the experiment. If the wires are connected to a thermometer without heat sinking the heat conducted along the wires will certainly warm the thermometer, so that it indicates an artificially high temperature.

8.2 Electrical requirements

If you find that your cryostat does not work properly after you have changed the wiring you may have to search for the cause of the problem, and replace your new wiring. For example, if you use thick copper wires for an application which only needs thin constantan wires, you are introducing more heat to the system, and affecting its temperature more than necessary. The following table shows a range of possible solutions for common wiring requirements. Use it to choose the best material before you start to wire the cryostat.

Electrical requirement	**Typical application**	**Suggested solution**
Current << 0.1 A Voltage < 50 V	Resistance thermometers, (4 wire)	0.1 mm hard enamelled constantan, manganin or phosphor bronze wires
Current < 2 A Voltage < 50 V	Low power heaters	Hard enamelled copper, 0.1 to 0.2 mm diameter. Below 8 K multi-filamentary superconducting wire with CuNi matrix
Current 2 to 150 A Voltage < 50 V	Superconducting magnet current leads	Many strands of hard enamelled copper wire for high temperature parts, multi-filamentary superconducting wires below 8 K. Gas cooling essential!
Current << 1 A Voltage 50 to 500 V	Piezo-electric drive	PTFE insulated copper wires (for low electrical resistance), or flexible stainless steel coaxial cables (for low heat load)
Low noise pick up	Sensitive low temperature measurements	Twisted pairs or flexible stainless steel coaxial cables
High frequency and Low loss	RF signals to/from experiment	Strip-line, twisted pairs, flexible or semi-rigid coaxial cables, stainless steel waveguides.

Table 3 Choosing appropriate wiring

8.3 Practical techniques

8.3.1 Wiring looms

Individual wires are small and fragile, and it is difficult to handle them. It is often best to stick them together with GE 7031 varnish, so that they form a ribbon. This can be done conveniently by stretching the wires between some nails arranged as shown in Figure 16. The inner nails are used to guide the wires, which are then wrapped around the outer nails. The wires should be arranged so that they are touching, and they should then be painted with GE varnish between the inner nails. Second and third coats can be applied, and they should be left to dry for at least 2 hours. The wires may be cut at the outer nails, so that the ends are ready to be soldered to an electrical connector.

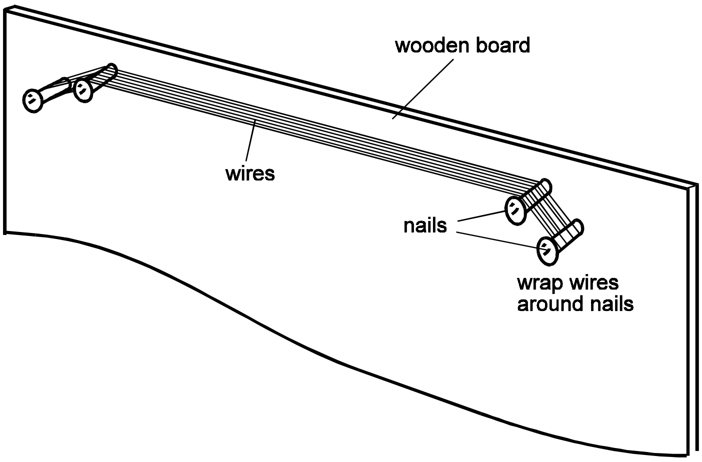


Figure 16 Making a wiring loom

8.3.2 Twisted pairs

Electrical noise is often picked up by an electrical circuit, and if sensitive measurements are being made the noise may make it difficult to detect a signal. The noise can also contribute to radio frequency heating of the sensor in ultra low temperature systems. One of the popular and simple ways of reducing the electrical noise pick up is to arrange the wires in twisted pairs. The wires are twisted together for their whole length, so that the currents induced by flux passing between the wires in each twist is cancelled by that in the next twist.

Some experimentalists report that the pitch of the twist is important. Others claim that it is only important to maximise the number of twists per unit length. However it is often difficult to eliminate interference effectively, and a range of techniques is used to reduce noise pick up. The book by Richardson and Smith (see section 12) describes these techniques in some detail.

8.3.3 Heat sinking

Effective heat sinking, or thermal anchoring is one of the most important features of good cryogenic wiring. A variety of techniques is used to ensure that the wires are fixed at the required temperature. Wires are often heat sunk at several points, and each heat sink helps to reduce the amount of heat conducted to lower temperatures. These techniques can be so effective that on some systems it is possible to run wires and coaxial cables from room temperature to an experiment at < 10 mK without introducing too much heat.

The easiest systems to consider are those where the wires are in gas or liquid. For example, if the experiment is carried out in liquid helium or helium gas in a variable temperature insert, the gas flows over the wires before it leaves the cryostat. This cools them very effectively, and it is only necessary to make sure that sufficient length of wire is in contact with the cold gas. Allow the wires to spiral around some convenient mechanical support, such as a pumping line or support leg.

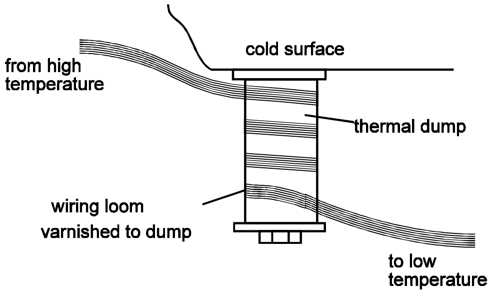


Figure 17 Heat sinking wires

However, in many systems the experiment is carried out in vacuum, so no cooling is available from the gas. The temperature of the wires therefore has to be fixed in some other way. The simplest way is to wrap them around a copper post (sometimes known as a 'thermal dump') which is held at a known temperature. GE varnish is used to make sure that they are in good thermal contact with the post. Although its thermal conductivity is only moderately good, it gives a large area of contact. See Figure 17.

If it is important that the capacitance between the wires and ground is very low (for example, less than 100 pF), alternative methods of heat sinking have to be considered. One method is to clamp the wires firmly, and another is to encapsulate them in epoxy resin.

8.3.4 Hermetic feedthroughs

A wide range of hermetic feedthroughs is available from the manufacturers of electrical connectors. These are suitable for use at room temperature, but they are not guaranteed for cryogenic temperatures. It is possible that some of them could be used, but before relying on them they should be tested by repeated thermal cycling to the working temperature.

Some glass to metal seals can be used reliably in situations where their temperature is changed slowly. They can be cooled using exchange gas, but if they are immersed directly in liquid nitrogen the thermal stresses sometimes cause them to leak after a few cycles. Special feedthroughs can be made using epoxy resins, but it is important that these are designed correctly. See the reference to epoxy to metal joints in Richardson and Smith's book (Section 12 gives details of the book).

8.3.5 Thermo-electric voltages

If two dissimilar metals are joined together they tend to act as a thermocouple, and small voltages (typically microvolts) can be generated. If very low voltage signals are being measured steps have to be taken to reduce the thermal voltages, so that they do not affect the readings. The best way to do this is to ensure that there are no joints in the wires. If there have to be some joints, it is important to ensure that the joints in both wires are at exactly the same temperature. It is possible to buy special feedthroughs for thermocouples, which allow the wire to pass through a metal tube, and these can be used for other similar applications.

8.3.6 Four wire measurements

If only two wires are connected to the sensor they must be used to supply the excitation current and measure the voltage across the sensor. This current causes a voltage drop along the wires because of their resistance. This voltage is added to the voltage across the sensor, and although it is possible to estimate the fraction of the voltage caused by the wires, the accuracy is limited.

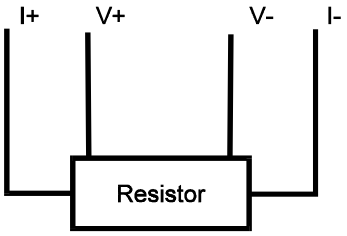


Figure 18 Four wire measurement

However, if four wires are connected to the sensor it is possible to make accurate measurements of electrical resistance even if the resistance of the wires is quite large. Two of them are used to supply the excitation current. The voltage drop across these wires is not measured, so it does not matter how high it is. The other two wires are used to measure the voltage across the sensor, using a high input impedance voltmeter. Since there is no current flowing in these wires there is no voltage drop along them, and their resistance can also be neglected.

This is especially useful to allow resistance thermometers to be measured through thin constantan wires. A sensor with a resistance of a few ohms can be measured accurately through leads with a resistances of several hundred ohms. Many four wire sensors have their terminals labelled (V+, I+, V-, I-), and it is important to make connections to the correct terminals. V+ and I+ may not be interchangeable, because they are connected to the sensor at different positions, so that the contact resistance is not measured. Similar techniques can be used for capacitance measurements.

8.3.7 Coaxial cables

A range of cryogenic coaxial cables is available, made from stainless steel and/or beryllium copper. If they are sufficiently long, and they are heat sunk effectively, they can be used for systems operating at the lowest temperatures. Some of these cables are magnetic; if this is likely to affect your experiment check them carefully.

S1 coax is suitable for signals with frequencies up to a few kilo-hertz. At higher frequencies the insertion loss of the coax rises very rapidly. Although this cable has a characteristic impedance of 40 Ω, (rather than the usual 50 Ω) this has little effect on the signal provided that the cable length is much less than one wavelength. At low frequencies this is likely to be true.

Semi-rigid cables have much better high frequency performance. They are suitable for frequencies up to about 20 GHz. They can be heat sunk in several different ways. If the cable is in gas, heat is conducted away from the inner conductor through the dielectric material to the outer, and then from the outer to the gas. It may not be necessary to make any other arrangements for heat sinking.

8.4 Ultra low temperatures

The same techniques can be applied to systems working at temperatures well below 1 K. These systems are capable of reaching temperatures of a few millikelvin, but heat load of 0.1 μW is enough to produce noticeable warming at these temperatures.

The experimental wiring is typically fixed at the following temperatures to reduce the heat load to an acceptable level:

- 4.2 K, cooled by the liquid helium bath, where the majority of the heat is absorbed
- 1.2 K, on the 1 K pot
- 0.6 K, on the still
- 50 mK, on the cold plate
- on the mixing chamber, to cool the wires to the same temperature as the experiment

8.5 UHV systems

Ultra high vacuum systems require special wiring techniques outside the scope of this booklet. However:

- The materials must be suitable for UHV (having a low vapour pressure at the working temperature)
- The materials often have to be suitable for baking to high temperatures
- There must be no trapped volumes (for example in threads)

It is relatively easy to wire a system if the whole of the UHV chamber is always kept at a very low temperature, because the vapour pressure of most materials is negligible.

9 Properties of materials

Many reference books are available, listing the properties of materials at low temperatures. Some of the most commonly used information is included in this section.

9.1 Physical properties of helium and nitrogen

Property	Nitrogen	Helium
Normal boiling point (NBP) (K)	77.3	4.22
Latent heat of vaporisation at NBP (Joules/gram)	198	20.9
Amount of liquid evaporated by 1 Watt at NBP (litre/hour)	0.024	1.38
Liquid density at NBP (g/cm^3)	0.808	0.125
Gas density at NTP (g/cm^3)	1.16×10^{-3}	1.66×10^{-4}
Gas at NTP to liquid at NBP volume ratio	694:1	750:1
Enthalpy change of gas (J/g). 4.2 K - 77 K	-	384
(at 1 atmosphere) 77 K - 273 K	234	1018

Table 4 Physical properties of helium and nitrogen

Note that helium has a very low latent heat of evaporation compared with nitrogen, but that it has a very high enthalpy. This means that the techniques used to store or transfer these two liquids are quite different.

The vapour pressures of ^{3}He, ^{4}He and N_2 and the enthalpy of ^{4}He (for the useful temperature ranges), are shown in the graphs on the following pages.

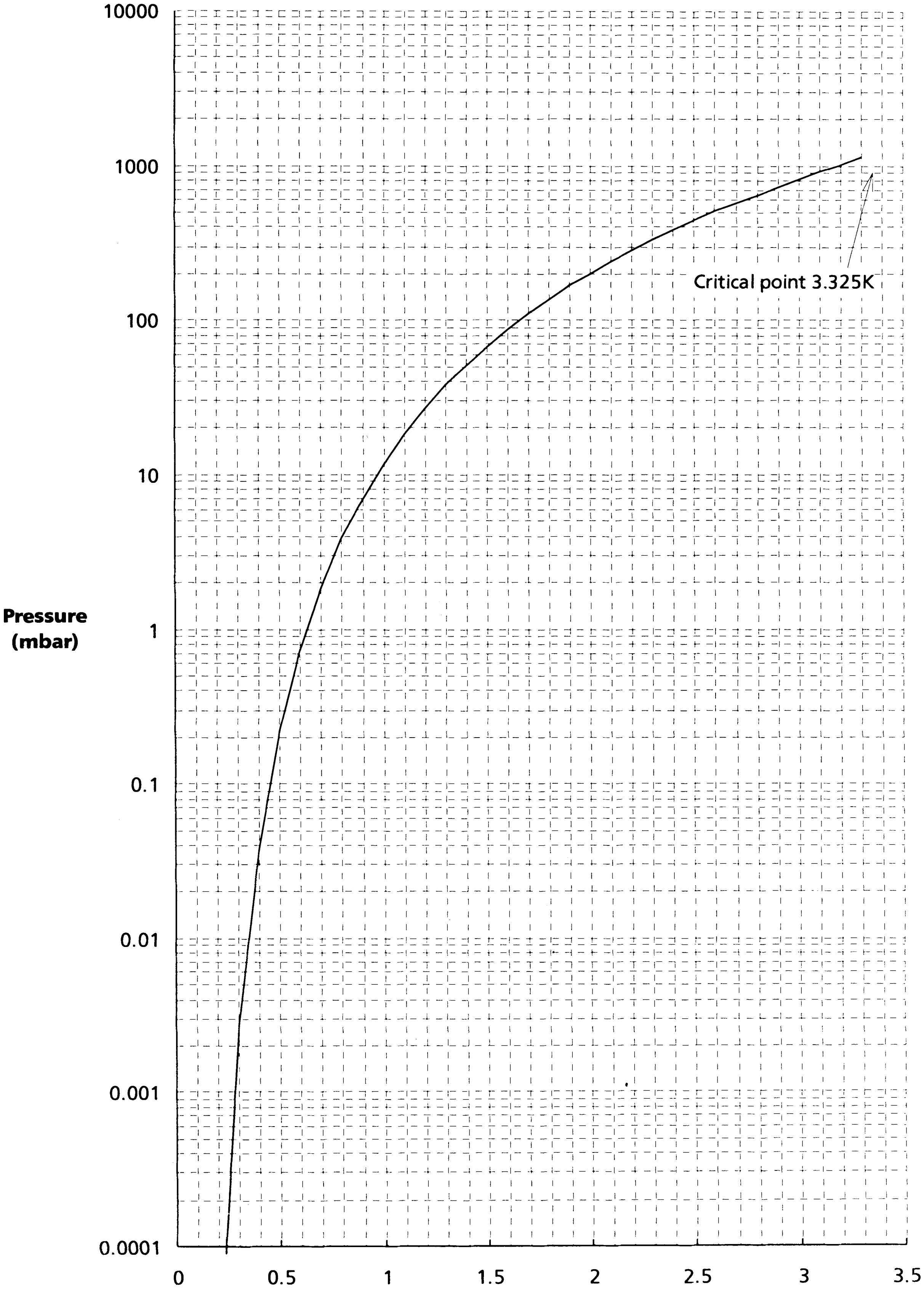
9.2 Thermal conductivity integrals

The following table of thermal conductivity integrals shows values for some of the common materials used in a cryostat. The figures can be used to calculate the effect of a component on the cryostat's evaporation rate. Refer to the guidelines in section 10.1.

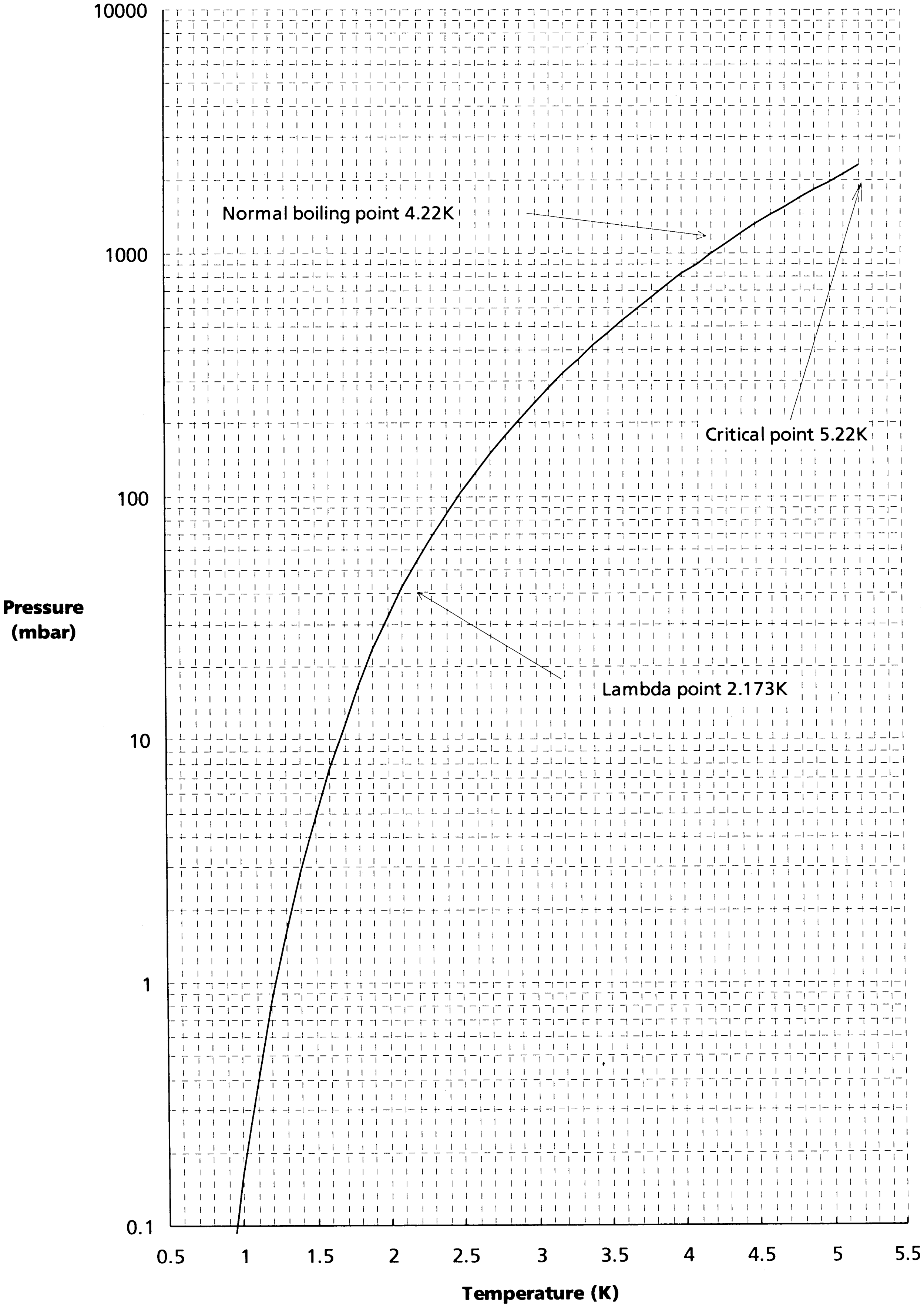
Material	300 K to 0 K	77 K to 0 K
Copper (electrolytic)	1620	690
Aluminium (99% pure)	730	220
Copper (phosphor deoxidised)	460	54
Brass (typical)	170	20
Constantan	52	8.8
Stainless steel	30	3.2
G10 fibreglass (typical)	1.5	0.17
Nylon	0.8	0.13

Table 5 Typical thermal conductivity integrals (in convenient units, W/cm)

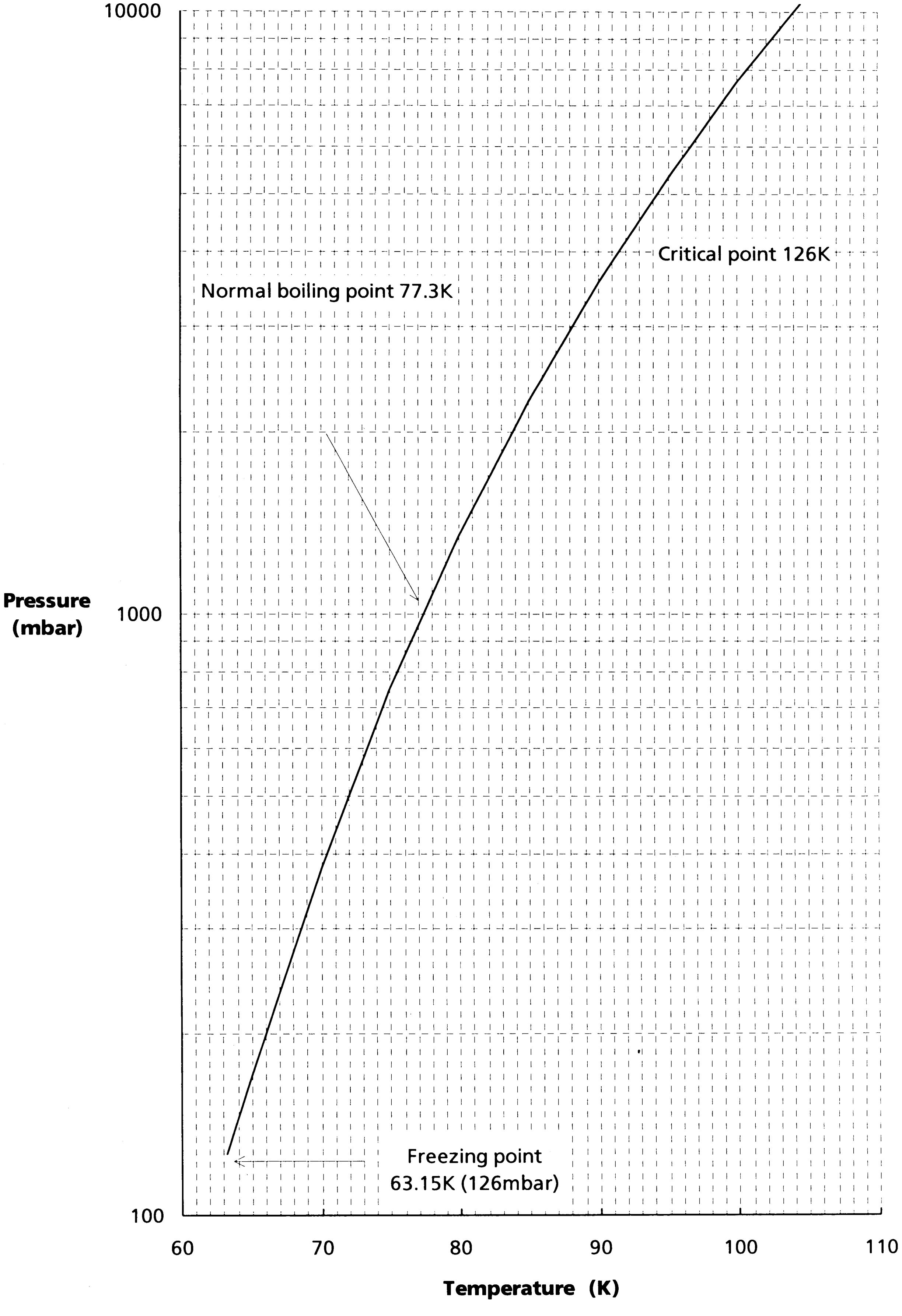
Vapour Pressure of helium 3



Vapour pressure of helium 4

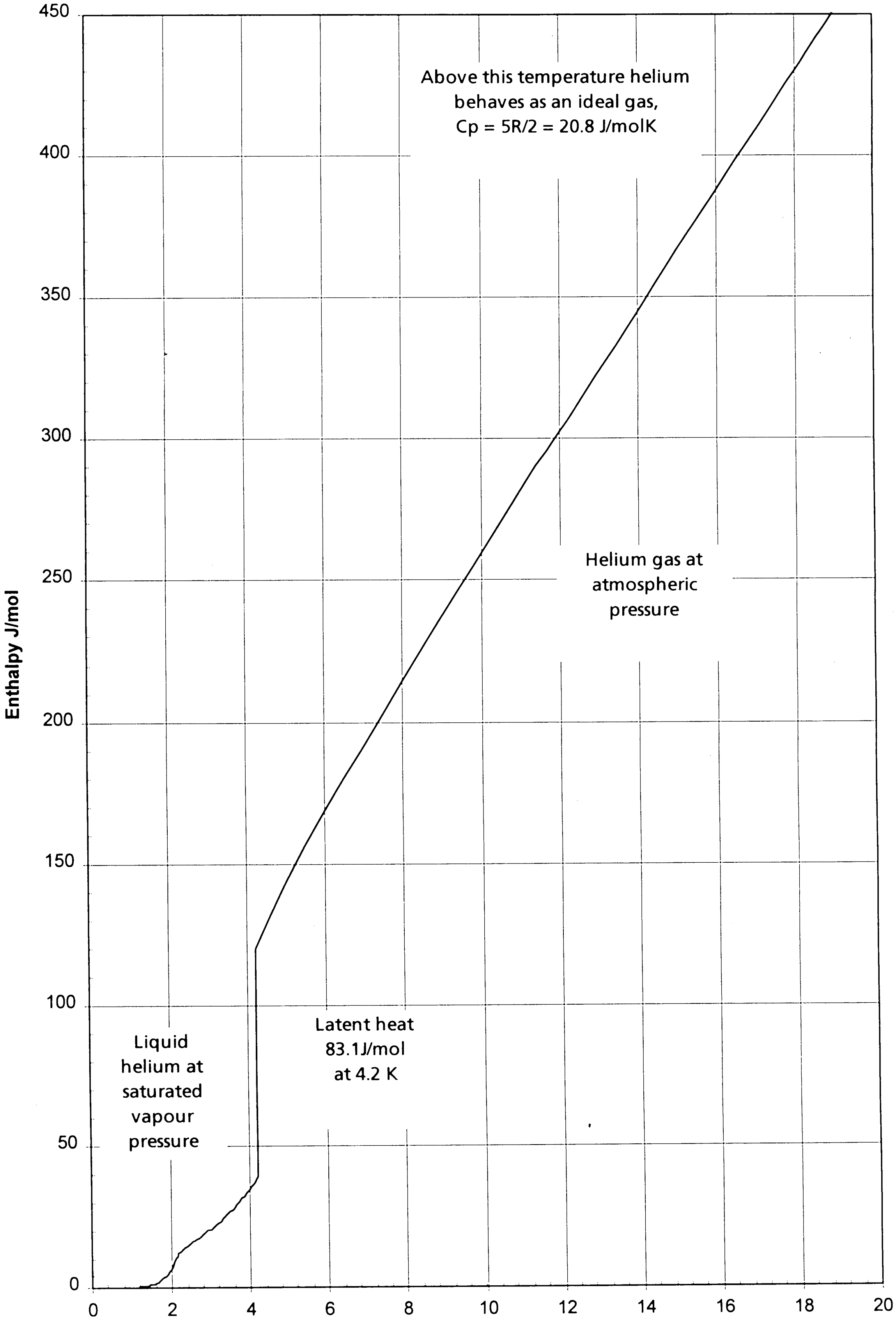


Vapour pressure of nitrogen



Enthalpy of helium 4

(at atmospheric pressure for T > 4.2K, at saturated vapour pressure for T < 4.2K)



10 Useful formulae and information

You can use the following information to estimate the heat load on a cryostat. However, remember that it is difficult to predict the boil off of a cryostat accurately because very many factors affect the result. Some of them are not known accurately.

10.1 Thermal conductivity and gas cooling

The heat conducted from temperature T_2 to T_1 is given by the formula:

$$\dot{Q} = \frac{A}{l} \int_{T_1}^{T_2} K dT$$

The figure for the thermal conductivity integral ($\int_{T_1}^{T_2} K dT$, as given in Table 5 on page 62) is substituted into this equation, with the cross sectional area A, and the length l.

The two columns in Table 5 indicate the value of the integral with the warm end at 300 K or at 77 K, and the cold end at 0 K. For all calculations of this type where the cold end is at a temperature of 20 K or below, it is reasonable to take the integral shown. If you want to calculate the conducted heat load from 300 K to 77 K, subtract the figure in the right hand column from that in the left column. This is equal to the integral from 300 K to 77 K.

If the material is cooled by cold helium gas flowing over it (evaporated by the heat load calculated), the amount of heat conducted through it is reduced by the following factors.

Temperature range	300 K to 4.2 K	77 K to 4.2 K
Theoretical gas cooling factor	32	10

Table 6 Gas cooling factors

However, in most large systems this may even be an underestimate, because there are other significant heat loads on the liquid helium vessel. The gas generated by these other loads all has to pass up through the neck tubes, and it is possible for the contribution due to the conduction of heat down the neck tubes to be reduced to zero.

Some cryostats have multiple neck tubes, with different services passing down the different tubes. It is not usually necessary to balance the heat loads down the different necks, because the strong convection currents in the helium gas tend to increase the flow up the tube with the highest contribution to the heat load. This means that the heavy current leads for a superconducting magnet may be fitted in one neck tube, and the lighter wiring for other services in another.

10.2 Thermal radiation

The amount of heat radiated from one body to another at a different temperature is proportional to the difference between the fourth power of their temperatures. In particular, for two long concentric cylinders, the following equation applies.

$$\frac{\dot{Q}}{A_1} = \sigma \frac{1}{\frac{1}{\varepsilon_1} + \frac{A_1}{A_2}\left(\frac{1}{\varepsilon_2} - 1\right)} \left(T_2^4 - T_1^4\right)$$

where $\sigma = 5.7 \times 10^{-8}$ W m^{-2} K^{-4} (Stefan's Constant)

ε_1 and ε_2 are the emissivities of the surfaces (see below).

A_1 is the area of the inner surface (m^2)

A_2 is the area of the outer surface (m^2)

T_1 is the absolute temperature of the inner surface (K)

T_2 is the absolute temperature of the outer surface (K)

Note that for two black bodies, the maximum value of this heat load is 46 mW/cm^2 for radiation from 300 K to 20 K or below, and 200 μW/cm^2 from 77 K to 20 K or below. The emissivity of a surface depends on many factors, including its cleanliness. The following figures should be used only as a guideline.

Material	**Typical emissivity**
Matt black paint	0.5 to 0.8
Clean metal	0.01 to 0.15
Superinsulation	0.05 or less

Table 7 Emissivity of materials

The design of cryogenic systems with optical windows is very complex, and the necessary information is not included here.

10.3 Convection

Convection in a narrow cryostat neck has a small effect and may safely be neglected. It is very difficult to predict the effect of convection on the performance of a large cryostat, and the simplest solution is to put as many baffles in the neck as possible. However, if the gas is forced through very small apertures it may introduce turbulence which can transmit heat down the neck of a cryostat.

10.4 Cooling materials to 4.2 K using liquid helium

The volume of liquid helium required to cool a mass of metal from one temperature to another using the full enthalpy of the gas or using the latent heat alone was reported by J B Jacobs, Advances in Cryogenic Engineering, Volume 8, 1963, p. 529, as follows.

Cryogen		**^{4}He**	**^{4}He**	**N_2**
Initial temperature of metal		300 K	77 K	300 K
Final temperature of metal		4.2 K	4.2 K	77 K
Using the latent heat	Al	66.6	3.20	1.01
of vaporisation only *	St. Steel	33.3	1.43	0.53
	Cu	31.1	2.16	0.46
Using the enthalpy	Al	1.61	0.22	0.64
of the gas **	St. Steel	0.79	0.11	0.33
	Cu	0.79	0.15	0.29

Table 8 Amount of cryogenic fluid required to cool metals (litres/kg)

Notes:

* corresponds to a rapid and inefficient transfer of liquid.

** corresponds to a slow and efficient transfer of liquid.

10.5 Superconducting transitions of common materials

Material	**Critical temperature**	**Typical critical field**
Pb	7.2 K	80 mT at 0 K
Nb	9.5 K	0.25 T at 0 K, 0.2 T at 4.2 K
Al	1.14 K	10.5 mT at 0 K
NbTi	10.6 K	12 T at 4.2 K
Nb_3Sn	18.3 K	25 T at 4.2K
Soft solder (PbSn)	7 K (approximately)	?

Table 9 Typical superconducting transitions of some common materials

11 Glossary of terms

1 K pot	A vessel filled with liquid ^{4}He which is pumped to a low pressure to reduce its temperature. It may be filled continuously through a variable flow needle valve, or a fixed impedance set for the required flow rate. Often used to condense ^{3}He gas in a *^{3}He refrigerator* or ^{3}He/^{4}He *dilution refrigerator* which works at lower temperatures.
18B4	A four stage vapour *booster pump* produced by Edwards High Vacuum Ltd.
20 K transfer	A technique for pre-cooling a *dilution refrigerator* system. Liquid helium is transferred into the helium reservoir until the inner vacuum chamber is cooled to approximately 20 K. The *exchange gas,* is then pumped away. This can be done much more quickly at 20 K than at 4.2 K. The *helium transfer* is then continued until liquid collects, and the fridge is cooled further by condensing the *mixture* into the *dilution unit.* Although the principle is simple, this technique requires some practice.
40 K shield	On some low loss liquid helium *cryostats,* the radiated heat from the liquid nitrogen vessel may constitute a large fraction of the total heat load on the liquid helium vessel. In this case, the helium evaporation rate can be reduced considerably by fitting a *gas cooled shield* between the liquid helium and liquid nitrogen vessels. This shield is cooled by the exhaust gas from the liquid helium reservoir and it is usually designed to float at a temperature of approximately 40 K.
9B3	A three stage vapour *booster pump* made by Edwards High Vacuum, and sometimes used on Oxford Instruments *dilution refrigerator* systems to circulate of the ^{3}He/^{4}He *mixture.*
A.C. bridge	An 'a.c. resistance bridge' is used to measure the resistance of a thermometer whilst minimising the *self heating* in the sensor. Most often used in the temperature range below 4.2 K.
Air mount	A pneumatic vibration isolation device.
Allen Bradley ™	Carbon resistor often used as an economical thermometer in the range from approximately 1 K to near room temperature, manufactured by the Allen-Bradley Company.
Araldite ™	Trade name for a two part epoxy resin, supplied by Ciba Geigy ™. Sometimes used to make vacuum tight joints.
Auto GFS	An automatic version of the GFS transfer tube for efficient operation of a CF cryostat using an 'Intelligent Temperature Controller' (for example, ITC503). Oxford Instruments GFS700 series transfer tubes are the automated versions of the manual GFS600 series.
Auto needle valve	A needle valve which allows the flow of a cryogen to be adjusted to the optimum rate automatically, using an Intelligent Temperature Controller (for example, ITC503).
Auto PID	ITC503 has an 'auto PID' button on the front panel, and an internal look up table for the PID values. When this function is enabled, the temperature controller goes to the look up table to find the best PID values for the set temperature.

Auto tuning	ITC503 is supplied with Oxford Instruments ObjectBench software which allows it to determine the optimum PID values for a system at a given temperature. It tunes the proportional band and integral time constant, and sets the derivative control to zero.
AVS 47 bridge	An a.c. resistance bridge suitable for ultra low temperature resistance thermometers. Made by RV Elektroniikka in Finland and available through Oxford Instruments. Replaces the earlier models, AVS 45 and AVS 46.
Backing pump	A pump used to obtain the *medium vacuum* required for the operation of most *high vacuum* pumps (such as *diffusion pumps, booster pumps,* and *turbomolecular pumps).* Usually a *rotary vane pump* is used as the backing pump.
Backstreaming	The motion of a fluid or vapour in the direction opposite to that of the desired flow (for example, a small flow of pump oil from the pump towards the cryostat).
Baffle	Either - a thin sheet of metal in the neck of the *helium can* of a *cryostat,* in a pumping line, or on a sample rod, to act as a *radiation shield* and to reduce convection. Or - a screen in a pumping line to prevent or reduce *backstreaming* of pump oil or to reduce vibrations transmitted through the gas.
Ballast resistor	See *dump resistor.*
Band	Reinforcing ring fitting the outside of a tube closely in order to strengthen it. A close fitting band on the outside of the tube helps it to withstand the collapsing force from an internal vacuum, even if the band is not glued or soldered to it. It does this by keeping the tube circular and reducing the tendency to buckle.
Belly	The wider part of the helium vessel, usually used to allow a large volume of liquid to be stored in a cryostat with a narrow *neck.* Most low evaporation rate *cryostats* have a narrow *neck* and a wide belly.
Bladder	The rubber lining for a 'soccer' ball, used to pressurise liquid helium *storage vessels* to promote the low pressure required to transfer liquid. If you squeeze the bladder with your hands warm gas is forced into the cold region of the helium vessel. This warm gas evaporates some of the liquid in the reservoir (because of its low latent heat). The gas that is released increases the pressure, and the bladder expands to larger than the original diameter. See *helium transfer* and section 7.2. Also sometimes used as a closed volume for a gas to expand into safely.
Blow off valve	Pressure relief valve.
Blow out tube	Piece of stainless steel tube (often with a thread fitting on one end), used to transfer liquid nitrogen into and out of the helium vessel during the *pre-cooling* process. The thread screws into the *siphon cone.*
Boil off	Evaporation from a cryostat.
Booster (pump)	A pump used to create a higher vacuum than a *rotary pump*, but designed to operate at a high flow rate. Generally used to increase the volume flow rate; for example, *Roots pumps,* or multi-stage *diffusion pumps* which are referred to as vapour *booster pumps.*

Breaking a connection	A demountable connection is said to be 'broken' when the two parts are separated.
Bung	A stopper or plug.

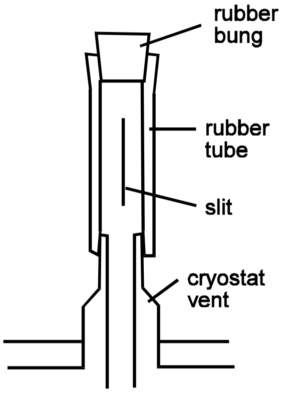


Figure 19 Bunsen valve

Bunsen valve	A simple non-return valve made from a piece of rubber tubing with a rubber bung in the end, and a slit cut in the side (see diagram). However it is not safe to rely on a Bunsen valve to protect the system and they should only be used if the system has a proper relief valve elsewhere.
Bursting disc	A safety device which breaks to relieve a high pressure in a pipe or vessel. Once broken, it has to be replaced with a new one.
Bush	In cryogenics this usually refers to a short piece of thick walled tube, used to reinforce the joint between a thin walled tube and a flange.
Calcium fluoride	A colourless material sometimes used as a window in a cryostat, allowing visible to far infra-red radiation to pass through. Insoluble in water, susceptible to mechanical shock and easily scratched. Not available as a cold leak tight window. *ZnSe* is generally preferred because it is easier to use.
Cancellation coils	A set of coils attached to the end of a magnet to produce a low field region close to the magnet. (For example, it is possible to reduce the field from a 10 T magnet, at a point 30 cm from the field centre, to less than 10 mT.)
CF	See *Continuous flow cryostat.*
CGR	Carbon glass resistor, a *resistance thermometer* for the temperature range from 1.5 to 300 K, with low magnetic field dependence.
Check valve	Usually a one way valve, which allows flow in only one direction.
Christmas tree fitting	Fitting to connect a rubber tube to a port.
Closed cycle cooler	A mechanical refrigerator used to achieve low temperatures by circulating helium gas.
CLTS	Cryogenic linear temperature sensor. A *resistance thermometer* with a linear change of resistance with temperature. They have very high magnetic field dependence.

CMN	Cerium magnesium nitrate. This material is used as a thermometer in the temperature range from a few mK to approximately 1 K because of the temperature dependence of its magnetic susceptibility.
Cold plate	On an Oxford Instruments *dilution refrigerator,* this is a plate situated between the coil heat exchanger and the top sintered silver heat exchanger. When the system is running at base temperature, the temperature of this plate is approximately 50 mK. It is sometimes used for *heat sinking* of experimental services. See Figure 12 on page 36.
Cold trap	Either - A cold trap is fitted to a high vacuum system to condense vapours that have a high freezing point, and thus improve the effectiveness of the pumping system. This is usually done by fitting a reservoir of liquid nitrogen in the line above the diffusion pump. For most cryogenic systems, it is not essential to have such a trap in the pumping system, since the cryostat acts as its own cold trap as soon as it is filled with cryogens. However, in low *boil off* systems, it is possible that the thin layer of ice frozen onto the *superinsulation* will increase the *emissivity* of the surface enough to have an effect on the *boil off*. Also used to prevent the *backstreaming* of pump fluids into the vacuum space. Or - A cold filter used to condense contaminants out of a flow of gas, perhaps to prevent the blockage of narrow tubes in a cryostat by ice.
Compensation coils	Coils designed to improve the field *homogeneity* of solenoid magnets.
Concentrated phase	See *phase boundary.*
Cone seal	A reliable low temperature vacuum seal can be made by making the joint between two components a closely matched taper. A thin layer of *vacuum grease* between the surfaces makes the seal. These can be used as an quicker alternative to indium seals.
Conflat ™	*UHV*/high temperature joint using a knife edge and a *copper gasket* to create a vacuum tight seal.
Constantan	Alloy of nickel and copper, used for wiring in *cryostats* because of its low thermal conductivity and its relatively constant resistivity with varying temperature. It has a high electrical resistance but accurate resistance measurements may be made using *four wire measurements.* Also used as a resistance wire for the winding of low power heaters.
Constriction	A small diameter orifice fitted in the pumping line of a vessel which is intended to reach a temperature below the *lambda point,* to restrict the *film flow* up the pumping line and prevent *thermal oscillations* in the pumping line.
Continuous flow cryostat	A type of cryostat that does not have its own cryogen reservoir, and so needs to be connected to a storage dewar whenever it is in use. See section 4.3.2. Oxford Instruments Optistat, Microstat and Ultrastat are examples of CF cryostats.
Copper gasket	A copper ring used to make the seal between two *Conflat flanges.* The gasket has to be replaced every time the seal is re-made.
Crack (a valve)	When referring to a valve, 'cracking' is the process of opening it by only a very small amount.

Cracked oil	If the oil in a *diffusion pump* or a vapour *booster pump* is overheated or exposed to gas at high pressure it tends to break down. It is then said to have 'cracked'.
Cressal ™	Power resistor, used sometimes for *magnet protection.*
Critical current, I_c	The maximum current that a superconducting component can stand without turning *normal.* This is usually expressed as a critical current density, J_c for the material. See *critical field* and *critical temperature.*
Critical field, H_c	The maximum field that a *superconductor* can stand before it turns *normal.* Note that this transition is affected by the current density in the conductor, and the temperature.
Critical temperature, T_c	In a *superconductor,* this is the maximum temperature that the material can stand before it turns *normal.* See *critical field* and *critical current.* In a *normal* fluid this is the temperature above which the gas cannot be condensed by pressure alone. Below this temperature it is referred to as a *vapour,* and above it as a gas. In a *superfluid,* this is *lambda point*.
Cryocon ™	A copper loaded thermal contact grease supplied by Air Products. See also *'N' grease.*
Cryocooler	See *Closed cycle cooler.*
Cryofree ™	A range of Oxford Instruments superconducting magnet systems designed for operation without liquid cryogens. They are cooled by *closed cycle coolers.*
Cryogen	A liquid whose normal boiling point is significantly below room temperature, used to provide refrigeration by its latent heat of evaporation.
Cryopump	In general, an entrapment pump which uses a cold surface to freeze the gases in a vacuum space. It retains them at a sufficiently low temperature to keep their vapour pressure below the required pressure in the vacuum chamber. The *sorption pump* which is used to remove ^{3}He from an ultra low temperature system and store it at high pressure may also be referred to as a cryopump.
Cryostat	The terms *cryostat* and *dewar* tend to be used interchangeably to describe the vessel used to contain *cryogens,* usually to cool a *superconducting magnet* or other experimental apparatus.
D.A.M.	Double aluminised mylar; sometimes used as *superinsulation.*
D.C. bridge	A 'd.c. resistance bridge' is used to measure the resistance of a thermometer using direct current. It is usually possible to reduce the excitation current to a value low enough to prevent noticeable *self heating* in the sensor. However, at the lowest temperatures an *a.c. bridge* is usually used.

D.C. SQUID	The D.C. SQUID is the most sensitive detector of magnetic flux. The introduction of the second Josephson junction (biased by a direct current) causes a finite, time averaged direct voltage across the Josephson junctions. The system can be used to provide an output signal that is linear with flux. Oxford Instruments Φ_0 D.C. SQUIDs are examples of these devices.
Dead volume	A volume which has to be filled with fluid that is not useful for operation. For example, the annulus around a magnet has to be filled with liquid to cool the magnet. Since the magnet may need to be covered with liquid, this volume does not extend the running time of the system between liquid *helium transfers*.
De-gas	Deliberate *outgassing,* usually promoted by heating a component while it is subjected to a *high vacuum.*
Derivative control	One of the controls on a *three term controller.* Derivative control is of limited use on small systems, and it is usually set to zero. However, it may be used to reduce overshoot resulting from a step change in temperature.
Dewar	See *cryostat*. Named after Sir James Dewar, 1842 - 1923.
Diaphragm pump	A pump which uses a reciprocating diaphragm to reduce the pressure. Generally used because it is oil free, but the *ultimate pressure* for a single stage pump is typically limited to approximately 100 mbar.
Diffusion pump	A *high vacuum* pump which works by entraining gas molecules with a flow of oil vapour which is then condensed and re-cycled. It requires a *backing pump* to produce a *medium vacuum* at the outlet of the pump. The pressure in the vessel to be evacuated must already have been reduced to an acceptable level by a *roughing pump.*
Dilute phase	See *phase boundary.*
Dilution refrigerator	A refrigerator which uses the two isotopes of helium to produce temperatures in the millikelvin range (usually continuously). See *Kelvinox* and section 5.2.
Dilution unit	The part of a *dilution refrigerator* below the *1 K pot.* (Usually demountable.)
Diode protection	Some *protection circuits* (designed to be used with magnets which will be swept rapidly), have diodes wired in series with the protection resistors. These diodes only allow current to pass when the voltage rises sufficiently to open them. The circuit is designed so that this only happens during a *quench.* The benefit of this type of protection is that no power is dissipated in the protection circuit during normal operation so it can be mounted in the helium reservoir. See section 6.7 on page 44.
Dipstick	Narrow tube with thin membrane over a housing at the top end, used as a simple level probe for liquid helium. The thermal gradient set up in the tube leads to *thermal oscillations* which are felt by the vibration of the membrane. The frequency of the oscillation with the lower end in liquid helium is noticeably lower than that when it is in cold gas, allowing the liquid level to be determined easily (in most cases). The dipstick should not be left in the cryostat when it is not in use because it introduces a large amount of heat.

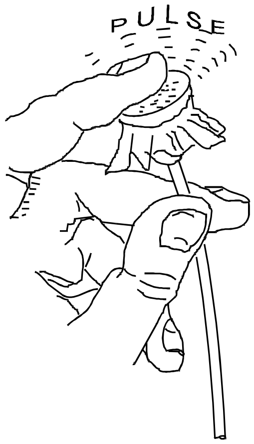


Figure 20 Using a dipstick as a helium level probe

Displacer	Object used to fill a *dead volume.*
Double vacuum space dewar or cryostat	A *cryostat* which has two independent vacuum spaces. One insulates the liquid nitrogen vessel from the room temperature surroundings, and the other insulates the liquid helium vessel from the liquid nitrogen.
Drop off plate	A safety device which is held in place by a vacuum, but which is allowed to fall away in the event of a high pressure building up in the vacuum space. Thus the pressure is released. The drop off plate must be deliberately replaced before the vacuum space can be evacuated again.
Dry pump	A fluid free vacuum pump, (for example, a *diaphragm pump*, claw pump, scroll pump or (sometimes) a *turbomolecular pump*). These pumps are useful if you want to avoid contaminating the vacuum system with pump fluids.
d.s.v.	Diameter spherical volume. (For example, the *homogeneity* of the field of a magnet may be specified over a 10 mm d.s.v.)
Dump	See *dump vessel* or *dumping bobbin.*
Dumping bobbin	Copper post around which wiring is wrapped, in order to give good *heat sinking.* See Figure 17 on page 59.
Dump vessel	Vessel used to store gas at low pressure, usually used for valuable gases, like ^{3}He.
Dump resistor	A power resistor used to dissipate energy, for example, in a magnet *protection circuit.*
Dynamic VTI	A type of variable temperature insert *(VTI)* in which gas flows over the sample continuously. The gas temperature is controlled by a heat exchanger at the bottom of the sample space. Good levels of temperature control can be achieved, with rapid temperature changes. See *static VTI* and section 4.3.1 on page 26.
Ears	Lifting points welded to the side of a *cryostat* for attachment of a winch.

Eddy currents	Currents induced in a conducting material when the magnetic field changes. These currents can cause mechanical damage because of the large Lorentz forces between the current and the remaining field. They also dissipate heat in the conductor, and this may cause a problem if the cooling power of the system is severely limited; for example, at ultra low temperatures. The eddy currents may be induced by vibration of a conductor within a region where the magnetic field is varying, and the heat dissipated in this way may affect the base temperature of a *dilution refrigerator*/ high field magnet system.
Emissivity	A factor used to compare the power radiated per unit area of a surface with that emitted by a perfect black body at the same temperature.
Energisation	A superconducting magnet is said to be 'energised' when its current is being increased.
EPR / ESR.	Electron paramagnetic resonance / electron spin resonance (which are synonymous).
Exchange gas	A gas which is deliberately introduced to transfer heat from one body to another. Often used to *pre-cool* parts of the *cryostat* within the *IVC* to the temperature of the helium reservoir, and then pumped out before running to a lower temperature. Also used in *static VTI's* to make thermal contact to the sample.
Eye bolts	Lifting points which are screwed into the top plate of the *cryostat.*
Faraday shield	An electrostatic shield constructed of many separate electrical conductors bonded together to prevent the flow of eddy currents in conductors within the shield.
Feed-through	Access from one volume into another (for example, for wiring, coax cables or optical fibres). Often leak tight (or *hermetic*).
Field decay	The field produced by a *superconducting magnet* drops very slowly with time when it is in *persistent mode*. This happens because the joints between the sections of wire used to make the magnet are very slightly resistive, and some of the energy stored in the magnet is dissipated at the joints.
Film burner	On a *dilution refrigerator,* this device is fitted to the mouth of the *still*, to inhibit flow of the film of *superfluid* ^{4}*He* up the pumping line. It is desirable to minimise the amount of ^{4}He circulated with the ^{3}He. However, modern film burners are often passive devices which do not need power to be supplied electrically.
Film flow	*Superfluid* helium forms a film which coats the inner surface of the vessel in which it is contained. This film is very mobile, and it creeps up the pumping line very quickly, reaching the point on the line which is warm enough to evaporate the film. The resulting large flow of helium affects the ability of the pump to reach a very low pressure. Wherever possible, a *constriction* is fitted in the pumping line to reduce the perimeter of the tube. This effectively decreases the film flow, (and so the amount of liquid lost without giving useful cooling) is minimised, and the base temperature of the vessel can be improved very significantly. Other techniques are also used, for example a knife edge on the end of the pumping line, or polishing the inside of the pumping line.
Film heater	A heater made up by laying a resistive track on a thin film of heat resistant plastic. These heaters can be glued to the point which is to be heated.

FIR	Far infra-red.
Flux jumping	The uncontrolled movement of a flux line through a superconductor, which dissipates energy. This effect can be effectively eliminated by fine sub-division of the *superconductor,* as in, *multi-filamentary wire.*
Fore-line trap	A container filled with an adsorbent material (for example, *molecular sieve*) which is fitted in the pumping line above the inlet of a *rotary pump.* It is used to prevent oil mist from the pump *backstreaming* into the vacuum chamber.
Former	Some coils are wound on a 'former' to maintain their shape and to give a mechanical fixing point.
Fountain effect	A phenomenon in a *superfluid* which demonstrates the two fluid properties. A fountain pump (made from an electrical heater and a *superleak*) can generate a flow of *superfluid helium II.* Also known as the *thermo-mechanical effect.*
Four wire measurement	Standard type of measurement made with high resistance wiring, to determine the resistance of a sensor accurately. Two of the wires are used to supply the current to the sensor, and the other two are used to measure the voltage across the sensor. If the impedance of the voltmeter is high, the current flowing through the voltage sensing leads is negligible, so there is no significant voltage drop in the sensing leads to affect the measurement. See section 8.3.6.
Fridge	Short form of "refrigerator".
FTIR	Fourier transform infra-red spectroscopy.
G.A.	General arrangement or general assembly drawing, showing how the sub assemblies fit together.
GaAs diode	A gallium arsenide semiconductor diode sometimes used as a cryogenic thermometer. The voltage across the diode varies with temperature at a constant current.
Gas ballast	Most *rotary pumps* are fitted with a 'gas ballast', which is a small valve designed to let a flow of air into the high vacuum side of the pump during the compression part of the cycle. The flow of air carries away condensable vapours from the oil, allowing water vapour to be pumped more effectively. The *ultimate pressure* of the pump is affected slightly.
Gas cooled shield	A *radiation shield* in a *cryostat,* cooled using some of the enthalpy from the helium gas that has evaporated from the helium reservoir. Sometimes used between a liquid nitrogen jacket and a liquid helium reservoir as a *40 K shield.* More often used in liquid nitrogen free or *vapour shielded cryostats.*
Gas cooled siphon	A helium transfer tube which uses the cold exhaust gas from the cryostat to cool a radiation shield within the tube, reducing the liquid helium losses during the transfer. Most often used for *continuous flow cryostats* which have a low flow rate.

German silver	Also called nickel silver. An alloy of copper, nickel and zinc combining strength, malleability, ductility and good resistance to corrosion; widely used in cryogenics until suitable stainless steels became available. (47% Cu, 41% Zn, 9% Ni, 2% Pb.)
G.E. varnish	G.E.™ 7031 varnish, a thermally conducting varnish used to stick wiring onto *heat sinks.*
G.E. varnish solvent	Approximately 50% methylated spirit or ethanol and 50% toluene and rather toxic.
GFS	Gas flow shielded transfer tube. See *gas cooled siphon.*
GF2	Now replaced by the *GF3.*
GF3	A small *diaphragm pump* usually used to promote a flow of helium or nitrogen through a cryostat by reducing the pressure at the outlet.
GHS	Gas handling system.
Grease seal	See *cone seal.*
Guard ring	Some cryostats are fitted with a liquid nitrogen cooled heat exchanger (called a 'guard ring') in the neck of the helium reservoir to reduce the amount of heat conducted into the liquid helium.
^{3}He	The lighter isotope of helium, which is used in *^{3}He refrigerators* and ^{3}He/^{4}He *dilution refrigerators* to obtain temperatures below 1 K. This gas is very expensive. Most liquid helium contains very small quantities of ^{3}He, but it is not commercially viable to extract it.
^{4}He	The common isotope of helium.
He I and He II	See helium I and helium II.
Heliox ™	A range of *^{3}He refrigerators* produced by Oxford Instruments, (not to be confused with 'Helios' ™, the superconducting synchrotron, also produced by the Company).
^{3}He refrigerator	A type of cryostat which uses the evaporation of ^{3}He to provide cooling for an experiment. Temperatures below 0.3 K may be achieved by this type of system. Some ^{3}He refrigerators are *single shot sorption pumped* systems, whilst others circulate the gas continuously, using a room temperature pumping system.
Heat leak	An unwanted flow of heat into the system.
Heat sink	Either - A point where heat is removed, so that the *heat leak* on the system is minimised. (For example, wiring may be wrapped around a *dumping bobbin.)* Or - a finned panel in electronic equipment to dissipate the heat generated.
Heat switch	Either a mechanical or a superconducting device used to make or break thermal contact.
Helium I	The *normal* phase of liquid ^{4}He.
Helium II	The *superfluid* phase of liquid ^{4}He. (That is, liquid ^{4}He below the lambda transition.)

Helium can	The reservoir that contains liquid helium, usually at the normal boiling point of 4.2 K and 1 atmosphere.
Helium sealed pump	A vacuum pump which is completely sealed from the atmosphere, allowing a gas to be circulated in a closed circuit without the gas escaping and without any air entering the circuit. These pumps normally have a special shaft seal and the *gas ballast* valve is removed. They are used to circulate the ^{3}He/^{4}He mixture in a *dilution refrigerator.*
Helium transfer	Liquid helium must be transferred from one vessel to another very carefully because of its low latent heat of evaporation. Refer to the description of the *helium transfer,* and the list possible causes of failure in section 7.2 on page 50.
Hermetic	Completely sealed.
High vacuum	Usually defined as the pressure range from 10^{-3} to 10^{-7} mbar.
Homogeneity	Uniformity. For example, the homogeneity of a the field of a typical *superconducting magnet* may be defined as 1 part in 10^{4} over a 10 mm *d.s.v.*
ICR	Ion cyclotron resonance.
IGH	Intelligent gas handling system (KelvinoxIGH is used to automate the operation of *Kelvinox dilution refrigerators).*
Impedance	A restriction, put in a tube to limit the flow of a fluid or to reduce the pressure in a flowing fluid (and sometimes to cool it).
Impregnation	The spaces between wires in the coils of a *superconducting magnet* are usually filled to prevent movement of the wires when the magnet is energised. A range of different materials is used. Often the impregnation is forced into the windings by a process known as 'vacuum impregnation'.
Inconel	A nickel/chromium/iron alloy, (72% Ni, 14-17% Cr, 6-10% Fe, 0.1% C).

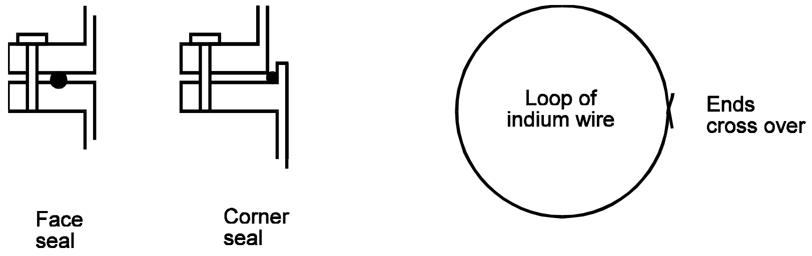


Figure 21 Schematic diagram of indium seals

Indium seals	A joint which can be removed and replaced easily, using a thin indium wire to form the seal. The wire is compressed into intimate contact with the two surfaces that are to be sealed together. See Figure 21 and section 4.9. Similar joints may be made using lead wire, but for low temperature operation, it is easier to use indium because it is softer. Indium also 'wets' glass at room temperature when a small amount of pressure is applied.

Insert	The part of the cryostat that is loaded into the helium reservoir down the *neck*. See *VTI*. Sometimes it is used to refer to the probe on which the sample is mounted.
Integral control	One of the controls on a *three term controller,* which eliminates the residual error from *proportional band* control.
ITC4	The predecessor of the ITC500 series of temperature controllers*.*
ITC501	A instrument designed to monitor up to ten thermometers. It may be interfaced to a computer.
ITC502	A *three term controller* made by Oxford Instruments. It has the capability of monitoring up to three thermometers at the same time, and supplying heat to control the temperature of one part of the *cryostat.* The sensor interface can be configured to suit a wide range of thermometers, (typically within the temperature range 0.25 to 500 K).
ITC503	A temperature controller similar to the *ITC502*, but with automatic tuning for the *three term controller,* and the ability to use a look up table to set the optimum *PID* values for the set temperature automatically.
IVC	Inner vacuum chamber. Normally this refers to the vacuum chamber in the *insert* which is surrounded by liquid helium.
Jacking screws	Threaded holes are usually machined into one of the flanges of an *indium seal.* Jacking screws are inserted into these holes to help prise the flanges apart, since the indium holds them together firmly.
Johnson coupling	A vacuum insulated joint used in cryogen transfer lines.
K	Kelvin, the SI unit of absolute temperature.
Kapitza resistance	A thermal boundary resistance between liquid helium and solids. This increases rapidly as the temperature drops, and can limit heat exchange significantly at temperatures below 100 mK.
Kapton ™	A plastic material (made by Dupont) used in sheet form for cryostat windows or for electrical insulation. Capable of withstanding quite high temperatures.
Kelvinox ™	A range of *dilution refrigerators*, made by Oxford Instruments.
KF fittings	Klein flange fittings, which use a simple clamp to hold the flanges together. Often referred to as "*NW* fittings". Also referred to as "ISO type DN fittings". Suitable for low, *medium* and *high vacuum.*
KRS5	A synthetic mixed crystal of thallous bromide and iodide often used as a window material suitable for the range from the red end of the visible band to the extreme infra-red. It is very delicate and toxic. Wherever possible it is recommended that *ZnSe* is used instead.
Lambda plate	See *lambda point fridge.*

Lambda point	The temperature at which ^{4}He becomes *superfluid,* 2.17 K. So called because of the shape of the specific heat capacity versus temperature curve.
Lambda point fridge	Device used to cool the lower part of the helium reservoir (around the magnet) to a temperature close to the *lambda point* to allow the magnet to be energised to a higher field. This is done by drawing liquid from the bath into a tube or chamber, and reducing the pressure of the vapour above this liquid with a *rotary pump* to cool it. It relies on the strong convection currents in the liquid; the cold liquid cools all the components below the lambda point refrigerator. The poor thermal conductivity of the liquid above the lambda point refrigerator gives thermal isolation from the surface of the liquid which remains at 4.2 K and at atmospheric pressure; therefore the helium reservoir can be re-filled without stopping the operation of the refrigerator. Sometimes referred to as a *lambda plate* or *pumped plate.* See section 4.2 on page 24.
LCMN	Lanthanum cerium magnesium nitrate; this material can be used as a thermometer in the temperature range below 1 K, like *CMN*.
Leak detector	Usually a *mass spectrometer,* tuned to be sensitive to helium, allowing very small vacuum leaks to be detected and often accurately located using helium gas.
Lifting ears	See *ears.*
Lifting eye	See *eye bolts.*
LHe	Liquid helium.
LN$_2$	Liquid nitrogen.
LOx	Liquid oxygen.
LPF	*Lambda point fridge* (that is 'refrigerator').
MagLab	A range of turnkey measurement systems made by Oxford Instruments. They are used for thermal, electrical and magnetic characterisation of materials.
Magnabond ™	The trade name for a range of methods used for the *impregnation* of the windings in Oxford Instruments superconducting magnets.
Magneto-resistance	A change in the electrical resistance of a material when a magnetic field is applied. Sometimes used to measure magnetic field.
Magnet protection	See *protection circuit.*
Magnet support system	The structure used to support the magnet and the *protection circuit.* Usually it includes other services for the main helium bath and instrumentation for the magnet.
Main bath	Liquid helium reservoir.
Manganin	Alloy used for high resistance electrical wires, which has relatively constant electrical resistivity with varying temperature. (84% Cu, 12% Mn, 4% Ni).

Mash	See *mixture.*
Mass spectrometer	A machine that is able to distinguish between materials of different molecular mass, usually used in cryogenics as a helium sensitive vacuum *leak detector.*
Matsushita ™ resistor	Carbon resistor used as a substitute for *Speer*™ resistors.
MCD	Magnetic circular dichroism.
MD	Modular dewar. A liquid helium or liquid nitrogen *cryostat,* usually with a cryogen reservoir, and usually with removable base flanges or tails.
Medium vacuum	Generally defined as the pressure range from 1 to 10^{-3} mbar.
Melting curve	The ^{3}He melting curve thermometer is a primary thermometer that is usually used in the temperature range from 1 to 250 mK.
Mixing chamber	The coldest part of a *dilution refrigerator,* where the ^{3}He is diluted using ^{4}He. This dilution process is the source of the cooling power.
Mixture	The charge of ^{3}He/^{4}He gas required for a *dilution refrigerator.* Sometimes referred to in the USA as *mash.*
Molecular sieve	A material with a well defined pore size (for example, zeolite) often used in *sorption pumps* or *foreline traps.*
Monel	A nickel/copper/iron alloy. (66% Ni, 2% Fe, 2% Mn, 30% Cu).
MRI	Magnetic resonance imaging, a technique which uses nuclear magnetic resonance in imaging mode. Often used for clinical diagnosis 'in vivo'.
MSS	*Magnet support system.*
Multi-filamentary wire	Most superconducting wire is made up as a bundle of filaments of superconductor, embedded within a copper (or copper alloy) matrix. This type of construction helps to make the flow of current through the wire more stable, by preventing *flux jumping.*
Mumetal	High magnetic permeability alloy used for magnetic shielding. (75% Ni, 2% Cr, 5% Cu, 18% Fe).
Mylar ™	A semi transparent plastic material (made by Dupont ™) and often used in cryostats as a window material or for electrical insulation.
'N' grease ™	Thermal contact grease, supplied by Apiezon ™. See also *Cryocon.*

Nb_3Sn	A superconducting intermetallic compound of niobium and tin which has a very high *critical field* and *critical temperature,* and is used for the inner coils of many very high field *superconducting magnets.* Fields higher than 20 T have been achieved by Oxford Instruments, using this type of conductor. The superconductor has to be formed by a solid state reaction in the wire after the coil has been wound, because it is extremely brittle. The reaction takes place over a period of several days in a carefully controlled atmosphere at a high temperature.
NbTi	A superconducting alloy of niobium and titanium which is used for *superconducting magnets* with fields up to a maximum of approximately 9 T at 4.2 K (11 T at 2.2 K). It has the advantage of being cheaper and easier to use than Nb_3Sn.
Neck	The part of the helium vessel between room temperature and the liquid reservoir.
Needle valve	A valve for fine control of fluid flow.
Nextel ™	A matt black paint sometimes used in *cryostats* to increase the *emissivity* of a surface.
Nitrogen free dewar	See *vapour shielded dewar.*
NMR	Nuclear magnetic resonance.
NO	See *nuclear orientation.*
Normal	Either- a fluid which is not *superfluid.* Or- a material which is not *superconducting.*
NQR	Nuclear quadrupole resonance.
NRC2 ™	A commercially available aluminised mylar, used as superinsulation in many cryostats.
NTP	Normal temperature and pressure; although it is not universally agreed, many authors take this to mean 20°C and 1 atmosphere (or 1013 mbar). However it is often confused with *S.T.P*, meaning 0°C and 1 atmosphere - take care!
Nuclear orientation	The anisotropic emission of gamma rays from certain specialised sources, which can be used as a thermometer in the mK temperature range.
NW fittings	Nominal width. See *KF fittings.* Sometimes referred to as 'ISO type DN' fittings.
ObjectBench	Software used to control Oxford Instruments systems.
ODMR	Optically detected magnetic resonance.
'O' ring	Usually a rubber ring which is clamped between two surfaces to make a vacuum seal.
Olive	A ring of soft material used to make a vacuum or high pressure seal between a tube and a fitting. Nylon olives are usually used with plastic tubes, and copper olives are used for metal tubes.
OMF	Oil mist filter.

Oscillations	See *thermal oscillations.*
Outgassing	Desorption of gas from a surface under vacuum.
OVC	Outer vacuum chamber, the insulating vacuum space around the liquid helium (and liquid nitrogen) reservoir(s).
Oxygen clean	Equipment that is used with oxygen (gas or liquid) has to be cleaned to a high standard. Many materials (including common oils and greases) can be spontaneously ignited in contact with oxygen.
PCD	Pitch circle diameter, the diameter of the circle on which a ring of holes is located.
PCR	Pitch circle radius. (Equal to *PCD* / 2.)
Peltier effect	If current is passed through the junction between two dissimilar metals which exhibits the properties of a thermocouple, cooling can be obtained. This is sometimes used to cool baffles in pumping lines, (to reduce the *backstreaming* of oil vapour from a vacuum pump).
Penning gauge	*High vacuum* gauge which measures the ionisation current produced by a cold cathode in a low pressure gas.
Persistent mode	A *superconducting magnet* is put into persistent mode by closing the *superconducting switch* that is fitted in parallel with the windings to complete the superconducting circuit. The power supply may then be removed, and the rate at which the field decays is very low (typically 1 part in 10^4 to 1 part in 10^7 per hour, depending on the complexity of the design, and the type of superconducting joints.
Phase boundary	In a *dilution refrigerator,* the phase boundary is the boundary between the *concentrated phase* and the *dilute phase.* The concentrated phase floats on top of the dilute phase, and it is arranged that the boundary is located inside the *mixing chamber.* See *phase diagram of* $^3He/^4He$*.*
Phase diagram (of $^3He/^4He$)	See Figure 11 on page 35. The special properties of a *mixture* of ^3He and ^4He are used to provide the cooling process in *dilution refrigerators.* If such a mixture of the two liquids is cooled below a certain *critical temperature,* 0.86 K, it separates into two layers. The concentration in these phases corresponds to the two points on the coexistence curve for the temperature of the liquid. The lighter *concentrated phase* (mostly ^3He), floats on top of the *dilute phase* (a few % ^3He in ^4He). The cooling power of the refrigerator is provided by the diffusion of ^3He across the phase boundary, from the concentrated to the dilute phase.
Phase separator	Device used to separate liquid from gas, for example, it may be fitted on the end of a *siphon* to prevent the jet of warm gas from reaching the surface of the liquid at the beginning of a *helium transfer.*
Pick up tube	Usually a tube used to collect liquid helium from the reservoir, for use by a *VTI* or *1 K pot.* Vacuum insulation is often used.
PID	*Proportional band, integral* and *derivative* controls. See *three term controller.*

Pirani	Vacuum gauge for the *medium vacuum* range, which works by measuring the thermal conductivity of the low pressure gas.
Piston seal	An '*O*' *ring* seal which allows relative movement between a tube and the housing which surrounds it. Used to prevent damage due to differential thermal contraction, or to load a *top loading probe* into an *insert.*
Potting	Material used to impregnate the windings of a coil to prevent mechanical movement of the wires. See *impregnation*.
Pre-cooling	Before a system is cooled to 4.2 K using liquid helium, it is usually pre-cooled to 77 K using liquid nitrogen. This very considerably reduces the amount of liquid helium required to cool the system to 4.2 K, reducing the cost of the cool-down. With care, the temperature of the liquid nitrogen may be reduced to about 65 K by reducing its *vapour pressure* with a *rotary pump.* This helps to pre-cool the system further, but there is a risk of freezing the nitrogen if it is pumped to too low a pressure. It is generally only worth the risk if you are cooling a very large mass.
Pre-cool loop	Some complex *cryostats* are fitted with a liquid nitrogen cooled heat exchanger which is used to pre-cool the system. This technique has the advantage of reducing the cool-down rate (and thus reducing the stresses induced by differential thermal contraction) and eliminates the need to fill the liquid helium vessel with nitrogen (which can sometimes only be removed with difficulty). It has the disadvantage that the cool-down may be very slow.
Pressed contacts	One method of transferring heat from one component to another is by pressing them together firmly. Perhaps surprisingly, it is found that the efficiency of heat transfer is dependent on the force applied to the joint, and not the area of contact.
Probe	Either - see *top loading* probe. Or - the sensor for a liquid level meter (for example).
Proportional band	One of the settings on a *three term controller.* The proportional band is the band of input signals over which the output is proportional to the input, (usually expressed as a percentage of the span of the input). Outside that range, the output is either fully on or fully off. Proportional band varies as the reciprocal of the gain.
Protection circuit	A circuit connected in parallel with the windings of a *superconducting magnet,* to dump the energy stored in the magnet in the event of a *quench.* It protects the windings against damage if the magnet quenches. See section 6.7.
Pumped field	The field that can be achieved by a superconducting magnet when its temperature is reduced to 2.2 K and it is energised to a higher current. This is typically up to 25% higher than the 4.2 K field. If the magnet is designed for this type of operation it will be described in the manual. Otherwise the magnet may be damaged because it cannot withstand the increased mechanical forces generated at higher fields. Refer to your magnet manual for details.
Pumped plate	See *lambda point refrigerator.*

'Q' compound ™	A black compound made by Apiezon ™, for temporarily sealing vacuum components. Useful for leak testing of sub-assemblies of a system.
QSB	Quarter swing baffle. A high throughput valve, sometimes referred to as a butterfly valve.
QHE	Quantum Hall Effect.
Quench	When part of the windings of a *superconducting magnet* goes *normal* (that is non-superconducting or resistive) the energy dissipated by the current flowing through this resistive part of the coil generates heat. The heat usually causes the normal region to propagate rapidly through the whole magnet (unless it is cryogenically stabilised). The stored energy of the magnet (½LI^2) is dumped into the helium reservoir as heat. Note that a magnet with an inductance of 100 H and current requirement of 100 A is not unusual, and that the stored energy of such a magnet is 500 kJ. The energy is dissipated within a few tens of seconds, so the liquid helium is usually evaporated very quickly. See section 6.6 on page 43.
Quench valve	(Perhaps) a relief valve which opens to release helium gas from the cryostat if a magnet quenches.
°R	Degree Rankine, the absolute scale of temperature with the same size unit as the Fahrenheit scale. 0°F = 459.69°R.
Radiation	One of the processes of heat transfer from a warm body to a cooler body. The amount of heat transmitted is proportional to the difference between the fourth power of the temperatures of the surfaces, the surface area, and the *emissivities*. Note that the maximum amount of radiation transmitted from a room temperature surface to a surface below 20 K is 46 mW/cm^2, and from a 77 K surface to 20 K or below, 200 μW/cm^2.
Radiation shields	Radiation shields are usually fitted around the colder parts of the system to minimise the heat load radiated from high temperatures. (For example, a liquid nitrogen cooled or *vapour cooled shield* is fitted around a liquid helium vessel.)
Recovery (system)	A low pressure system designed to collect and store helium gas that evaporates from the *cryostat* for re-use as gas or for liquefaction.
Reference junction	A *thermocouple* is usually made with two junctions between the dissimilar metals. One is used as the sensor, and the other (the reference junction) is held at a known temperature (for example, 0°C or 77 K), so that the size of the thermoelectric voltage is much less dependent upon the temperature of the instrument used to measure it.
Relief valve	A valve which opens to prevent or relieve a high pressure.
Remnant field	When a *superconducting magnet* is energised and then de-energised, the field is rarely reduced to zero. Because of the properties of superconducting wire, a small field remains (typically 10 to 100 gauss), and this can usually be cancelled by energising the magnet slightly. The remnant field is usually in the same direction as the original field because of diamagnetic effects.

Resistance thermometer	A sensor whose electrical resistance varies with temperature in a known way can be used as a thermometer. Many different types are available, including *RhFe,* platinum, carbon and carbon glass (*CGR*), germanium, *CLTS,* and *RuO_2.*
R.F. SQUID	A highly sensitive detector of magnetic flux. The single Josephson junction is biased by a radio frequency signal. The system can be used to provide a d.c. output signal that is linear with flux. Oxford Instruments Φ_0 R.F. SQUIDs are examples of these devices.
RhFe sensor	Rhodium iron resistance thermometer.
Roots pump	A mechanical *booster pump.* See standard vacuum equipment catalogues for details.
Rotary (vane) pump	A mechanical vacuum pump used to obtain a *medium vacuum, (*or *high vacuum)* and often used as a *backing pump.*
Roughing pump	A pump used to reduce the pressure in a vacuum system to a level where a different higher vacuum pump can start to work.
RPM	Revolutions per minute.
RuO_2 resistor	Ruthenium oxide thick film resistor, which can be used as a *resistance thermometer* at temperatures between about 15 mK and 10 K.
Scotchbrite ™	Trade name for a plastic based scouring pad, which may be used, with care, for polishing metal or removing old indium wire from a metal surface.
Sealed rotary pump	See *helium sealed pump*.
Self heating	If the excitation current used to measure the resistance of a sensor is too high, it is possible for Joule heating to warm the sensor to a temperature higher than the surroundings. The effect can often be seen if a standard multi-meter is used to measure a low temperature sensor.
Shielded room	Room built around sensitive equipment to reduce the amount of electrical noise on signals from the experiment.
Shield (electrical)	A grounded electrical conductor surrounding a sensitive electrical circuit to reduce pick-up of electrical fields.
Shim coil	A special coil wound in such a way that it can introduce field gradients to cancel inhomogeneities in the field from the main coils of a *superconducting magnet.* The shims may be wired in series with the main coils or energised independently. Very high *homogeneity* magnets may have room temperature shims as well as superconducting shims. See section 6.4 on page 42.
Short sample	The *critical current* versus field characteristics of a short length of superconducting wire are checked by the manufacturers. The results from this short length characterise the batch of wire, and are used by the magnet designer as one of the design variables.

Side access	For some requirements it is important that there should be a line of sight access from a vacuum chamber outside the cryostat to a cold sample without any windows in the beam line. The *windows* would normally be used to reduce the *radiation* load on the sample by absorption. However, it is possible to reduce the thermal radiation load to the level of microwatts by using long tubes which are blackened on the inside. This reduces the solid angle of access for the radiation to an acceptable level.
Si diode	A silicon semiconductor diode sometimes used as a cryogenic thermometer. The voltage across the diode varies with temperature at constant current.
Single shot	A non-continuous process. For example if you reduce the pressure over a reservoir of a liquid *cryogen*, you can obtain a lower temperature for a limited time, until all the liquid has evaporated. Also used in a *dilution refrigerator* to determine the volume and concentration of the ^{3}He/^{4}He *mixture*.
Siphon	Vacuum insulated liquid helium transfer tube. Alternative spelling "syphon". See *helium transfer,* and section 7.2 on page 50.
Siphon cone	A fitting inside the helium reservoir of the cryostat for the *siphon* to plug into. For an efficient cooldown with liquid helium the liquid must be delivered to the lowest point so that the cold gas flows over the mass that has to be cooled, using the full enthalpy of the gas. In a complicated system, it is often impossible to arrange for a line of sight hole to the lowest point in the helium vessel. In this case a siphon cone is fitted, and there is a small tube from the cone to the bottom of the vessel. See Figure 15 on page 51.
SMD	Standard magnet *dewar* (a range of bucket dewars made by Oxford Instruments).
Soft	A vacuum space is said to have 'gone soft' if the pressure is allowed to rise to an unacceptable level, so that it is no longer a good thermal insulator. This may be caused by a leak or by outgassing of dirty surfaces.
Soften	To soften a vacuum space, *exchange gas* is deliberately allowed into it, for example, the *OVC* is sometimes softened to warm up the system. Choice of exchange gas must be made with care to avoid unacceptable contamination of the *superinsulation.*
Sorb	Abbreviation for *sorption pump.*
Sorption pump	A vacuum pump which works by adsorbing gas. The adsorbent material is usually charcoal or *molecular sieve.* When the sorb is warmed, it releases gas, and when it is cooled again it pumps the gas to a pressure dependent upon the temperature. A very high (and clean) vacuum can be achieved by this type of pump. The capacity of the pump to absorb gas depends on the amount of sorb material. They are often used for *single shot* ^{3}*He refrigerator* systems.
Spectromag ™	A range of systems built by Oxford Instruments, with a *superconducting magnet* and optical windows through to the sample space. Designed to fit into an optical spectrometer for magneto-optical experiments.

Spectrosil ™	A type of man made silica, often used as a window material for visible and far infra-red radiation. It cuts out the unwanted thermal *radiation* in the near infra-red. The grades usually used are 'Spectrosil B' and 'Spectrosil WF' (water free). The pass bands are very similar, except that the B grade has additional absorption bands at wavelengths between 1 and 3 μm.
Speer ™	Carbon resistor manufactured by the Speer company (grade 1002), and used for thermometry at temperatures below 10 K. No longer being manufactured, but small supplies may still be found in low temperature laboratories.
Split pair magnet	If optical access is required perpendicular to the field direction of a *superconducting magnet,* it is necessary to wind the magnet as two separate coils with a space between them. The two coils are usually arranged approximately as a Helmholtz pair. They have to be supported very rigidly, because there may be an attractive force of several tens of tonnes between them. See Figure 13 on page 40.
SPM	Scanning probe microscopy, a general term for a variety of novel microscopy techniques. (See *STM*).
SQUID	Superconducting Quantum Interference Device, used for many sensitive measurements. See *D.C. SQUID* and *R.F. SQUID*.
Standard leak	A device which supplies helium gas at a precisely calibrated rate, used to calibrate helium sensitive *mass spectrometer leak detectors.*
Static (VTI)	A variable temperature system in which the sample is surrounded by a static *exchange gas.* The exchange gas space is surrounded by a heat exchanger whose temperature is controlled to achieve the desired temperature at the sample position. This type of insert has the advantages that extremely high temperature stability can be achieved and it is impossible to block the fine capillary tubes which supply the coolant while changing the sample. However they have the disadvantage that the sample cannot be cooled as quickly as in a *dynamic VTI.* See section 4.3 on page 26.
Still	The part of a *dilution refrigerator* where the ^{3}He is evaporated (or distilled) so that it can be pumped away. This effectively reconcentrates the ^{3}He. ^{3}He has a much higher *vapour pressure* than the ^{4}He, so it evaporates more easily from the still.
Still shield	A *radiation shield* fitted to many *dilution units,* cooled by the *still.*
Stinger	A special type of closed cycle cooler used to recondense the helium that has evaporated from the cryostat.
STM	Scanning tunnelling microscope.
Storage vessel	A *dewar* for transport and storage of cryogens.
STP	Standard temperature and pressure; that is, 0°C and 1 atmosphere (or 1013 mbar). Sometimes referred to as *NTP.*

Stycast ™	A range of epoxy resins made by Emerson and Cuming ™. Some of the products in this range are used for low temperature joints. Providing that the joint is designed correctly, very reliable seals can be made.
Style 10	A power resistor used by Oxford Instruments in *magnet protection* circuits*.* Capable of dissipating very large amounts of energy without burning out.
Superconductor	A material which loses its electrical resistance completely when cooled below its *critical temperature*. Many common metals become superconducting if their temperature is reduced sufficiently; for example, lead, tin, aluminium. However, the most useful superconductors for practical devices are alloys of niobium (Nb_3Sn and NbTi). New 'high T_c' materials are beginning to be used too.
Superconducting magnet	See section 6, starting on page 39.
Superconducting switch	Device made from superconducting wire. It is warmed to turn the wire *normal* and open the switch, and it is allowed to cool to close the switch. This type of switch is often fitted across the terminals of a *superconducting magnet* for *persistent mode* operation.
Supercooled	If the *vapour pressure* above a liquid is reduced to cool the liquid below its normal boiling point, and then the gas pressure is allowed back up to 1 atmosphere, boiling will stop until the temperature of the bulk liquid rises to the normal boiling temperature. The liquid is referred to as 'supercooled'. This technique is sometimes used to reduce the intermittent vibration caused by boiling nitrogen.
Superfluid	Liquid which has special properties including almost infinite thermal conductivity and negligible viscosity, associated with a quantum mechanical Bose-Einstein condensation (for ^{4}He). ^{4}He becomes superfluid below the *lambda point.* ^{3}He only becomes superfluid at temperatures below 3 mK.
Superinsulation	Low *emissivity* materials used in the high vacuum insulation space of a *cryostat* to reduce the heat load due to thermal *radiation.*
Superinsulated dewar	See *vapour shielded dewar.*
Superleak	A vacuum leak which is only detectable if *superfluid* liquid helium is used. It is usually very difficult to detect the source of the leak. Superleaks may be made deliberately by using firmly packed jeweller's rouge or Vycor ™ glass.
Switch	See *superconducting switch.*
Taconis oscillations	See *thermal oscillations.*
T_c	See *critical temperature.*
Tesla	The SI unit of magnetic flux density.
Teslatron	A range of turn-key cryogenic or magnetic systems built by Oxford Instruments*,* run by computer to automate experiments.

Thermal anchor	A point on a *cryostat* where the temperature of one item is fixed to the temperature of another. For example, wiring may be thermally anchored on a *dumping bobbin.*
Thermal dump	*Thermal anchor.*
Thermal link	A connection between two components to ensure that they are both held at the same temperature. (For example, in a liquid nitrogen shielded *cryostat,* the nitrogen vessel is thermally linked to the *neck* of the helium vessel to reduce the amount of heat conducted into the liquid helium.)
Thermal oscillations	An acoustic frequency oscillation set up in a narrow tube which has a large temperature gradient along its length. This phenomenon is used as a cheap but effective level probe for liquid helium; see *dipstick.* However, if unwanted oscillations are allowed in a *cryostat,* they may introduce very large amounts of heat (perhaps watts), affecting the boil off significantly. Also known as *Taconis oscillations.*
Thermocouple	When two dissimilar metals are joined together, they produce a thermoelectric voltage which varies with the temperature of the junction. This can be used as a thermometer, having the advantage that it is very small, has low thermal mass (and so responds to changes in temperature quickly), and is not affected greatly by magnetic fields. With the right choice of metals, thermocouples can be used at temperatures as low as 4 K. A *reference junction* is usually used to remove the effect of the temperature of the measuring instrument from the signal. A sensitive device is required to monitor this type of sensor.
Thermo-mechanical effect	See *fountain effect.*
Three term controller	An instrument used to enable the temperature of a sample or refrigerator to be controlled at a 'set point'. It is so called because the control is achieved by three terms; *proportional, integral* and *derivative.* The controller usually supplies heat to balance the cooling power of the system. If the three terms are correctly set, the temperature can be controlled at a constant value, or a step in temperature can be achieved with the minimum amount of overshooting, and in the minimum time.
TL	*Top loading.*
TLE	Top loading electrical, used to load samples onto the *mixing chamber* of a *dilution refrigerator,* whilst making electrical contacts to the sample, and allowing the *top loading siphon* to be removed.
TLM	Top loading into mixture in a *dilution refrigerator.* One end of the sample probe is at room temperature, and the other end may be at 20 mK or below. See *Kelvinox.*
Top hat	Short length of vertical tube which allows the helium exhaust port of a *cryostat* to be brought out horizontally, leaving space on the top plate for other services.
Top loading	Top loading is the process of changing the sample in a cryostat without having to warm the whole system to room temperature, using a *top loading probe* or *top loading siphon* to insert the sample.

Top loading probe	Sample probe which is used to put a sample into the *cryostat* without dismantling the system. Sometimes the sample is put in place and the *top loading probe* is removed from the cryostat, but usually the probe is left in place until the sample is to be changed again.
Top loading siphon	A type of *top loading probe* used to pre-cool a sample for a *TL* or *TLE dilution refrigerator* system to 4.2 K, before loading it onto the *mixing chamber.* The *precooling* is achieved by drawing liquid helium from the main bath through the siphon. When it is cold enough, the sample is left in place and the top loading siphon is removed from the cryostat.
Torr	Unit of pressure. 1 torr = 1 mm of mercury. 1 atmosphere = 760 torr. Now replaced by the 'millibar' in common usage. 1000 mbar = 1 bar.
Training	The process whereby the performance of a *superconducting magnet* improves after it has quenched. It is not unusual for a new magnet to *quench* at increasingly high fields on subsequent runs as the windings settle into their optimum position. This training is carried out in the factory, and should not be observed in new Oxford Instruments magnets. Some old magnets exhibit training after warming to room temperature and cooling again to 4.2 K. See section 6.6 on page 43.
TTL	Transfer tube for liquid helium, low loss type. Or - Transistor transistor logic, often used to describe logic levels of 0V and +5V.
TTN	Transfer tube for liquid helium, normal loss type.
Tufnol ™	Trade name for a laminated composite material used as an electrical insulator in many cryogenic systems.
Turbomolecular pump	A mechanical *high vacuum* pump that has a set of rotor blades like those in a turbine. The rotor runs at a very high speed. It requires a *backing pump* to produce a *medium vacuum* at the higher pressure end of the pump.
Twisted pairs	When two wires are twisted together for their whole length. Their sensitivity to pickup from alternating magnetic fields is reduced. Often used in conjunction with electrical shielding for sensitive electrical measurements.
Ultimate pressure	The lowest pressure that a pump can achieve in ideal conditions.
UHV	Ultra high vacuum. Usually this refers to the pressure range below 10^{-7} mbar.
Vacuum grease	Silicone based grease used to lubricate rubber *'O' rings* for vacuum seals.
Vacuum lock	A device used on many *top loading probes* to prevent air from entering a vacuum system, or another gas (for example, ^{3}He) from escaping while a sample is changed.
Vapour	A fluid which is below its 'critical pressure', (and therefore may be condensed by pressure alone).
Vapour cooled shield	A radiation shield cooled by the enthalpy of exhaust gas.

Vapour pressure	When a liquid and vapour are in thermal equilibrium, the pressure of the vapour varies with temperature (up to the *critical temperature*). It is possible to use this property as an accurate thermometer if the appropriate working fluid is chosen for the required temperature range.
Vapour shielded dewar	A *cryostat* in which the *radiation shields* are cooled by the enthalpy of the gas that evaporates from the reservoir. The number and position of the shields varies from one cryostat design to another. Often referred to as *superinsulated dewars.*
VC30/VC31	Gas flow controllers with a vacuum gauge, needle valve and flow meter, made by Oxford Instruments.
VC40/VC41	Gas flow controllers like a *VC30* but with an additional flow meter for nitrogen gas.
Vitreosil ™	A type of man made quartz often used as a window material for visible and far infra-red radiation. It cuts out most of the unwanted thermal *radiation* in the near infra-red.
VSM	Vibrating sample magnetometer, (for example Oxford Instruments MagLabVSM).
VTF	Variable temperature facility, a general expression, covering variable temperature inserts (*VTI*), *continuous flow cryostats,* and furnaces.
VTI	Variable temperature insert, used to set the temperature of a sample over a wide range, usually using a flow of liquid helium as the source of the required cooling power. Temperature range, approximately 1.2 or 1.5 K to room temperature or higher.
Windows	Windows are often fitted in *cryostats* to allow a beam of electromagnetic radiation to enter or leave the sample space. The material of the window is usually chosen to minimise the amount of thermal *radiation* from the warm surroundings, but to transmit the radiation that is wanted. Since thermal radiation is in the near infra-red part of the spectrum, windows which are designed to transmit infra-red often introduce relatively large amounts of heat, but otherwise the performance of most systems is hardly affected by properly designed windows.
Wood's metal	An alloy of bismuth, lead, tin and cadmium, that is often used as a low melting point solder. The melting point is 65 to 70 °C. It is important to avoid inhaling the fumes when using this material because of the hazard to health.
ZnSe	A material which is used as a reliable alternative to *KRS5* or *calcium fluoride* for *cryostat* windows. However the useful pass band is reduced. It is toxic and easily scratched.

12 Useful reference books

The following reference books contain useful background information about the fundamentals of cryogenic practice, and the basics of the design of cryostats. Using these books you would probably be able to design a working cryostat but it is often better to buy a well proven product. In this way you gain the benefit of many years of cryogenic experience, with the guarantee that you will be able to concentrate more on designing your experiment.

12.1 General practical techniques

Experimental Techniques in Low Temperature Physics (3rd edition).
by G K White,
Oxford University Press, 1987, ISBN 0-19-851381-X (Pbk), ISBN 0-19-851359-3

Experimental Principles and Methods below 1 K.
by O V Lounasmaa,
Academic Press, 1974, ISBN 0-12-455950-6

An Introduction to Milli-kelvin Technology.
by David S Betts,
Cambridge University Press, 1989, ISBN 0-521-34456-5

Experimental Techniques in Condensed Matter Physics at Low Temperatures.
by Robert C Richardson and Eric N Smith,
Addison Wesley Publishing Company Inc, 1988, ISBN 0-201-15002-6

Low Temperature Laboratory Techniques.
by A C Rose-Innes,
English Universities Press, 1973, ISBN 0 340 04778 X
(Probably out of print, but worth looking in the library).

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

Vacuum Technology its Foundations Formulae and Tables.
Leybold AG, Koln, 1987, Kat Nr 199 90.

Eleménts de Cryogénie.
R R Conte (in French).
Masson & Cie, Paris, 1970. (Probably out of print, but very useful).

12.2 Safety

Cryogenics Safety Manual - a guide to good practice.
The British Cryogenics Council,
Mechanical Engineering Publications Ltd, 1982, ISBN 0-85298 5010.

Safety Matters (for cryogenic and high magnetic field systems)
Oxford Instruments, 1994, 1995.

12.3 Thermometry and instrumentation

Instrumentation and Methods for Low Temperature Measurements in High Magnetic Fields.
H H Sample and L G Rubin
Cryogenics, November 1977, page 597

Introduction to Thermometry below 1 K.
(A review of available techniques)
Oxford Instruments, Ultra Low Temperature Group, 1990.

12.4 Properties of materials

Materials at Low Temperatures.
Edited by Richard P Reed and Alan F Clark,
American Society for Metals, 1983, ISBN 0-87170-146-4

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

12.5 Theoretical reference books

Vacuum Technology, (second, revised edition).
A Roth,
North Holland, 1989, ISBN 0 444 86027 4

Superconducting Magnets.
Martin N Wilson,
Clarendon Press / Oxford University Press, 1983,
ISBN 0-19-854805-2, ISBN 0-19-854-810-9 (Pbk).

Cryogenic Systems.
Randall F Barron,
Clarendon Press / Oxford University Press, 1985, ISBN 0 19 503567 4