The Business of Science®

Safety Matters

Creating safe environments for science

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Our Committment

Internationally recognised world leaders in the design, manufacture and seamless integration of superconducting magnet and ultra low temperature environments, Oxford Instruments NanoScience enables real advances in scientific research. The company's superconducting magnet and cryogenic technologies are at the heart of instrumentation used in nanotechnology and condensed matter physics research, with specific applications including optical spectroscopy, quantum fluids, superconductivity, single electron transport, spintronics and quantum electronics.

We also provide critical system elements for analytical techniques, such as Nuclear Magnetic Resonance Spectroscopy, Fourier Transform-Mass Spectroscopy and X-ray Crystallography, which play a vital role in drug discovery and life science applications.

Combining original thinking, technical expertise and a commitment to exceeding customer needs, Oxford Instruments NanoScience works with its customers to understand and meet the most demanding experimental requirements.

With over 50 years experience, Oxford Instruments NanoScience has been setting new standards in superconducting magnet and cryogenic technology. We aim to build on our track record on innovation by increasing our understanding of customers' needs. By meeting challenging technological requirements, our products create the environments that enable scientists to make real advances in many diverse research and commercial applications.

Providing an extensive range of products, from standard packages to tailored solutions developed in close collaboration with our customers, we place the needs of our customers at the heart of our business strategy. Promoting high standards of safety in the operation of our equipment is an essential element of our customer support activity.

This guide has been produced to provide essential safety guidelines for personnel working within the cryogenic laboratory environment. These guidelines should be used to complement the training that all personnel must receive before working with cryogenic equipment and high magnetic fields. In addition, we are able to provide many items required to achieve best safety practice in the laboratory, through our comprehensive Oxford Instruments Direct Cryospares® spares and accessories service.

We are committed not only to creating the right environment for science but also to facilitating a safe environment for experimental scientific research, in partnership with the scientific community.

This Handbook contains important safety information relating to the installation, use and servicing of Oxford Instruments cryogenic and high-field magnet systems. Use of these systems without observing this information is dangerous and could result in death or serious injury.

- Do not use or allow others to use these systems unless they have first read this handbook.
- Do not use or allow others to use these systems without proper training; this Handbook is not a substitute for training.
- This Handbook is written for users who are technically trained to work in a laboratory environment and with equipment of this nature; persons who are not so trained should not use the equipment.
- This Handbook is written in the English language. You must ensure that the persons using these systems understand the English language or alternatively ensure that they receive the information contained in this Handbook in a language which they understand.

- Ce manuel est écrit en Anglais. Vous devez vous assurer que les personnes utilisant ces systèmes comprennent l'Anglais ou alternativement vous assurer qu'ils reçoivent l'information contenue dans ce manuel dans une langue qu'ils comprennent.
- Dieses Handbuch ist auf Englisch geschrieben worden. Sie müssen sicher sein, dass die Anwender der Systeme die englische Sprache beherrschen. An sonsten müssen Sie dafür sorgen, dass den Anwendern die im Handbuch enthaltenen Informationen auf eine ihnen bekannten Sprache zur Verfügung gestellt wird.
- Questo manuale é in lingua Inglese. É necessario assicurarsi che chiunque utilizzi questi sistemi comprenda l'inglese o, in alternativa, riceva le informazioni contenute nel manuale in un linguaggio conosciuto.
- Este manual está escrito en Inglés por lo que deben asegurarse de que las personas que usen estos sistemas entiendan el idioma Inglés o por otra parte asegurarse de que reciban toda la información contenida en este manual en el idioma que ellos entiendan.
- このハンドブックは英語で記載されています。このシステムを使用する人は英語がわかる、もしくは理解できる言語で本書に記載されている情報を把握していることを確認してください。
- 本手册以英文撰写。您必须确保使用这些系统的人员理解英语，或者确保他们以他们理解的语言接收本手册中包含的信息。

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Warning: This booklet contains essential safety information and warnings which could help you to reduce the risks when you are using a cryogenic or high magnetic field system supplied by Oxford Instruments. Make sure that no one is allowed to use the system without reading the relevant parts of this booklet.

Scope: The booklet covers the hazards that you could commonly encounter with liquid helium and liquid nitrogen cryostats, the vacuum systems associated with them and superconducting magnets supplied by Oxford Instruments. It does not describe the law for any country, does not cover cryogens other than liquid nitrogen and helium and is not intended for large scale installations.

Training: Proper training by a competent person with local knowledge is essential because all cryogens are potentially hazardous; this booklet is not a replacement for such training.

Note: Please note and observe any warnings and instructions in this booklet, and the Operator's Handbook (which may contain specific warnings and procedures for your system). These documents are essential parts of the system and should be kept with the system for the whole of its life (even if you sell or give it to someone else).

Disclaimer: Although every effort has been made to ensure that the information in this booklet is accurate and up to date, errors may occur. Oxford Instruments shall have no liability arising from the use of or reliance by any party on the contents of this booklet and, to the fullest extent permitted by law, excludes all liability for loss or damages howsoever caused. However, if you find any errors or omissions or have any other suggestions please tell us about your experiences so that we can continue to improve this booklet. Your experiences may help others.

Contents

1 Introduction

Oxford Instruments designs and builds cryogenic systems that are safe to use. However, it is important that when using our equipment you are aware of the potential hazards posed by liquid nitrogen, liquid helium, vacuum systems and high magnetic fields. All users working on or with the Oxford Instruments equipment must be technically competent in a laboratory or test environment. Users accustomed to working in a laboratory environment need to know the reasons for the recommended procedures, this booklet outlines the most common hazards in appropriate technical terms.

This edition of *Safety Matters* has been expanded to give additional guidance to users of "dry" (sometimes referred to as Cryofree®) systems, where liquid ("wet") cryogens are replaced by a cryo-cooler. In some instances the cryo-cooler may be used to recondense a volume of liquid cryogens - in this configuration the guidance for both "wet" and "dry" systems should be followed.

Everybody who operates the system should read and understand this booklet. Keep it close to your cryogenic system and use it to remind yourself about the correct procedures. There are other very good books on the subject of safety and these should be used. One example is *Cryogenics Safety Manual - a guide to good practice*.[1]

All cryogens are potentially hazardous but knowledge of their properties will help you to understand why precautions are necessary. Liquid helium and liquid nitrogen are less dangerous than some other cryogens because they are neither poisonous nor flammable.

It is important that you pay attention to the information contained within this document. For example, even small cryogenic burns are extremely painful and can take a long time to heal. If care is not taken severe injuries or even death could be caused through the incorrect use of cryogenic systems.

Please note:

- If the equipment is not used in the manner specified by Oxford Instruments then the protection provided by the equipment may be impaired.
- The equipment is not suitable for use with explosive or flammable gases.
- The equipment is not suitable for use in explosive, flammable or other hazardous environments.
- Maintenance: only qualified and authorised persons should carry out servicing and repair work on this equipment.
- Only use genuine Oxford Instruments spare parts. Contact OI Direct or Oxford Instruments Customer Support to obtain these.[2]

All content in this booklet is for information and guidance only. It does not remove the requirement for the user and installer to carry out their own risk assessment and put in place their own hazard management plan in line with the local regulations and best practice. Oxford Instruments would consider the guidance given as a minimum standard.

1.1 Conventions used throughout this booklet

The following general symbols are used throughout this booklet to draw special attention to the most important messages. Other, more specific, symbols are used in certain cases. However, the paragraphs which are not marked with a symbol should still be read carefully as they either describe additional hazards or give further explanations.

Warning Symbol

The yellow warning triangle highlights dangers which may cause injury or, in extreme circumstances, death. The text explains the hazard and the correct procedure. The warning triangle may be followed by specific symbols and instructions.

Caution Symbol

A white symbol in a blue circle indicates something that you must do. The general caution symbol highlights actions that you must take to prevent damage to the equipment.

PPE Symbols

These symbols indicate that Personal Protection Equipment (PPE) should be used e.g. loose fitting, insulating gloves and a protective face shield should be worn, suitable for protection against splashes of liquid helium and nitrogen.

Functional Earth Symbol

This is the symbol for functional earth.

1.2 The sections you should read

It may not be necessary for you to read the whole of this booklet, since it is designed to cover a wide range of products. This section should help you to find out the parts that are relevant to you. Before you start to use any cryogenic or vacuum equipment you should be properly trained by a competent person. Contact your Safety Officer, arrange for the necessary training and then use this booklet as a summary of the hazards you are most likely to encounter. Remember that it is your responsibility to ensure the safety of all personnel, equipment, services or data links in or near your laboratory.

1.2.1 If you are setting up a laboratory

If you are designing a new laboratory or planning to install new equipment in an existing laboratory, the please read:

- Sec. 1.5: Setting up your laboratory

1.2.2 If you are using a cryogenic system (without a magnet)

If you are using a cryogenic system (without a magnet) it is important that you read the following sections which give you advice on the essential safety procedures:

- Sec. 1.3: Common hazards in cryomagnetic laboratories
- Sec. 2: Using vacuum and high pressure systems
- Sec. 3: Operating cryogenic systems

- Sec. 5: Working with electrical equipment

1.2.3 If you are using a system that contains a cryo-cooler (without a magnet)

If you are using a cryogenic system that contains a cryo-cooler it is important that you the following sections which give you advice on essential safety procedures:

- Sec. 1.3: Common hazards in cryomagnetic laboratories
- Sec. 2: Using vacuum and high pressure systems
- Sec. 3: Operating cryogenic systems
  - sec. 3.19: Notes on Operating Dry Cryogenic Systems
- Sec. 5: Working with electrical equipment

1.2.4 If you are using a system that is too heavy to lift by hand

If you are using a system that is too heavy to lift by hand it is important that you read the following sections which give you advice on the essential safety procedures:

Sec. 6 explains how to lift heavy systems safely.

1.2.5 If you are using a system fitted with a superconducting magnet

If the system you are using contains a magnet it is important that you read all sections listed previously and:

- Sec. 4: Superconducting magnet systems

1.2.6 If you are using a nuclear orientation thermometer

The procedures for using a low activity cobalt-60 (^{60}Co) gamma ray source are given in

- Sec. 7: Poisons and Hazardous Substances

1.3 Common hazards in cryomagnetic laboratories

The following list shows the range of hazards that you may encounter when you are using laboratory scale cryostats. You can protect yourself against all of these hazards by following the procedures described in this booklet, supplemented by any material in your Operator's Handbook.

- Extreme cold and the consequent risk of cold burns or frostbite;
- Asphyxiation (if the atmospheric oxygen is displaced);
- Fire and explosion hazards (through oxygen enrichment);
- Magnetic fields effects on medical implants;
- Large attractive forces between the magnet and other magnetic objects;
- Electrical hazards (including high voltage hazards);
- Vacuum hazards;
- High pressure hazards;
- Radioactive contamination from nuclear orientation sources used as thermometers;
- Damage or injury caused by lifting heavy equipment using incorrect procedures.

Signs that a hazard might be developing:

- Unusually high (or low) cryogen evaporation rate;

- Unusual condensation of atmospheric moisture on to any part of the cryostat;
- Unexpected patches of ice on any part of the cryostat;
- Difficulty in opening or closing any of the valves.

The Operator's Handbook for your system may give additional information about hazards not covered by this booklet, and specific procedures to follow.

If you are in doubt about any aspect of the operation of the system you should contact a local expert or your supplier. If you sell or give your system to someone else you are obliged to warn them in writing about the potential hazards and you may like to use this booklet to do so.

If you suspect that there is a fault with your system (perhaps indicated by one of the signs listed above) cease your experiment and use of the equipment immediately. Take immediate action to repair the fault (if you know how to do this) or close down the equipment and have it attended by an appropriately qualified person.

1.4 Working alone

Lone working can be dangerous. If you have an accident there will be no one there to help you. Do not work alone.

If despite this warning you have decided to take the risk of lone working you must ensure that there is a risk assessed and approved lone working policy in place for the laboratory. It is your responsibility to ensure that such a policy is in place to ensure your safety.

1.5 Setting up your laboratory

When you are setting up your laboratory you should:

- Design the laboratory with safety in mind.
- Consult an expert who has experience of setting up other similar laboratories.
- Set up procedures to be followed by anyone using the equipment.
- Make sure that the correct procedures and local regulations are always followed.
- Train all personnel and supervise them properly.
- Display clear notices to warn people that they are entering a potentially hazardous area. Remember that even if the door is locked, some other people have keys. For example, cleaners and security staff are often working when there is no one else around; they are at risk too.
- Tell the local Safety Officer about your system and ask them to make local emergency services aware of the hazards, as this may affect the procedures they follow when they are dealing with fires or other incidents.
- Consult your local fire authority about the equipment you should install in case of a fire. They may require that portable fire fighting equipment is non-magnetic. Ask them to check whether your smoke detectors will be set off by helium gas (as some are).
- Consider carefully whether the floor in your laboratory is strong enough to take the weight of the system. Seek professional advice if necessary[3].
- If you are using a superconducting magnet system, consider whether there are any large magnetic items close to the system (such as magnetic beams in the floor). Check whether the stray field will affect other equipment or people in your laboratory or in other

rooms nearby (even on other floors)[3]. Use the information in your stray field map and the guidelines herein.
- Install an overhead crane (or other lifting equipment) capable of lifting your equipment safely.
- Make sure that the material of the floor will not be damaged (or become hazardous) if cryogens are often spilt on it.
- Position the system so that it is not necessary to pass the cryostat in order to reach a safe exit point.
- Provide suitable venting for exhaust gases.
- Make sure that the laboratory is sufficiently well ventilated. If there is any doubt, install sensors which will warn you if the oxygen level becomes dangerously low (less than 18%). Fixed and portable oxygen monitors are readily available[2]. Helium gas tends to collect near the ceiling whilst cold nitrogen gas tends to collect near the floor; you are therefore advised to fit one monitor at a high position and one low.
- Provide an earthing point to be used to connect an earth cable to the cryostat; this is especially important for systems with superconducting magnets that are not earthed via the magnet power supply.
- Provide appropriate first aid equipment.
- Refer to relevant local health and safety publications.

If you require advice or want a formal site survey you should contact the Oxford Instruments help desk (ServiceNSUK@oxinst.com).

2 Using vacuum and high pressure systems

2.1 Vacuum pump operation

Operating rotary pumps without appropriate oil mist/dust filters complying with the manufacturers instructions or in line with local regulations will emit fume/dust, which represents a health hazard. Do not operate rotary pumps without mist filters.

Do not breathe these fumes/dust. If a suitable filter is not available the exhaust from the pump must be piped outside the building.

2.2 Vacuum vessels and overpressure

If a fault occurs, vacuum spaces in cryogenic systems can become overpressurised and rupture, potentially causing death or injury. All vacuum spaces must be protected with an overpressure relief valve.

If a system is operating for an extended period a small air leak may go unnoticed. The air that leaks into the vacuum space will freeze on to any cold surface or be absorbed on to a sorption pump, if fitted. Only when the system is warmed up does it become apparent that a large quantity has been collected; this may expand to fill the vacuum space to a pressure higher than it can safely withstand.

A hazardous situation can occurs if one of the vessels filled with cryogenic fluid becomes damaged. At this point the cryogenic fluid may be released into the vacuum space, where it breaks (softens) the vacuum, rapidly warming and vapourising the cryogenic liquid.

To protect against both eventualities suitable relief valves are required on the liquid and vacuum spaces to vent the gas generated; all Oxford Instruments systems are protected in this way. See Fig. 2.1.

2.3 Vacuum vessels and collapse

Do not evacuate vessels unless they are designed to work under vacuum. Vent evacuated vessels slowly.

Some systems may be damaged if you allow the pressure in a vacuum space to rise too quickly. The shock of the sudden pressure increase may cause an otherwise safe tube to collapse. Some systems must be vented slowly to allow the pressure to equalise in different parts of the system. Consult the system user manuals for details.

2.4 High pressure cylinders

You must determine local laws and regulations relating to high pressure cylinders and follow them.

High pressure cylinders are commonly used to store gases, at pressures up to 200 bar. Most countries have laws regarding their use. Helium and nitrogen gas are the most common requirements for cryogenic laboratories.

- Store upright and secure at all times to prevent them falling. They must never be left free-standing.
- Store cylinders where they are not at risk from accidental damage.
- Move cylinders by means of a purpose-designed trolley in the upright position with the valve closed and the regulator and other equipment removed. Cylinders must never be rolled along the ground on their side.
- Only use a regulator that is designed and labelled for use with the gas in the cylinder.
- Cylinders must be labelled with the contents, pressure, size and safety information.
- Store cylinders where the magnetic field is below 10 gauss.

If high pressure cylinders are ruptured or the valve is knocked off, they can become dangerous projectiles which can break through thick walls or travel hundreds of metres by rocket propulsion. Always handle high pressure cylinders with extreme care.

2.5 High pressures - cryo-coolers

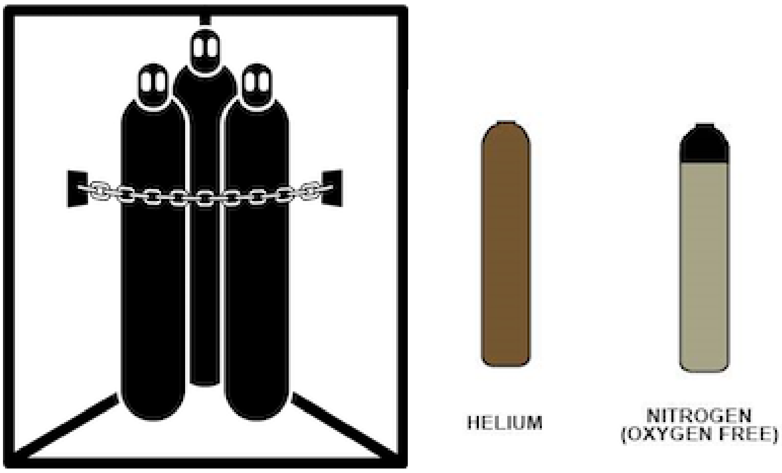
Most cryo-coolers, interconnecting lines and compressors will contain a charge of high pressure gas. The presence of these high pressures should be taken into account when decommissioning a system containing a cryo-cooler.

Oxford Instruments should be contacted for advice on how best to dispose of the system. sec. 3.19 contains additional guidance on working with systems containing cryo-coolers.

Decoupling high pressure lines can be dangerous. Always use 2 spanners when decoupling the lines to ensure the fittings do not rotate and work loose.

(a) (b) (c)

Fig. 2.1: Examples of Oxford Instruments (red) over-pressure relief mechanisms.



(a) (b)

Fig. 2.2: Cylinder security and EU colour codes for helium and nitrogen cylinders.

3 Operating cryogenic systems

Cryogen handling is hazardous because of issues associated with extreme cold, rapid expansion leading to dangerous pressure increase or pressurised jets of very cold vapour, or rapid expansion leading to oxygen displacement which could result in asphyxiation. It is your responsibility to ensure that you protect not only yourself but also others from the potential hazards. Make sure that you understand the properties of the cryogens in use and handle them appropriately. You can protect yourself from the extreme cold by wearing suitable clothing and personal protective equipment (PPE) but you can only protect yourself against the other hazards by making sure that they do not occur.

In particular, you need to protect yourself from:

- Extreme cold;
- Asphyxiation;
- Fire and explosion hazards;
- Hazards associated with vacuum systems used to contain cryogens.

Fit clear labels to cryogenic vessels to indicate their contents. This helps other people to determine what precautions they need to take to ensure their own safety. Remember that it is your responsibility to keep the working environment safe for other people and they may hold you liable if you do not.

> The system will be fitted with one or more safety devices for pressure relief. Do not modify or obstruct (even partially) these devices.

> If a safety device operates (opens) it may vent quantities of cold gas. Laboratory activities must be regulated to allow for this possibility.

3.1 Personal protective equipment

Personal protective equipment is readily available and should be used, see Fig. 3.1.

- Wear a face shield to protect your face and, in particular, eyes. Eyes rarely heal well.
- Use cryogenic gloves if there is any danger of touching cold metal; these should be loose fitting so that you can remove them easily. If you are transferring small quantities of liquid nitrogen and there is no danger of touching cold metal then it is usually safer not to wear gloves.
- Wear overalls or similar clothes without pockets or turn-ups.
- Wear a cryogenic apron to protect against spills. Cryogens have low viscosity and penetrate clothing much faster than water.
- Wear sensible shoes (not sandals) and make sure that your trousers cover the top of your shoes to prevent spilt cryogens running into them.

In the event of liquid nitrogen being splashed onto your bare hands accidentally, spread your fingers as you move your hand away. This will help to prevent cryogenic burns caused by droplets of the liquid being trapped between your fingers.

Even if you try to push or throw away a very cold object in contact with your bare skin it could stick to you (by rapid freezing of local moisture) and tear away the skin. Do not underestimate

Fig. 3.1: A full set of PPE including cryogenic apron and gloves and face shield. The operator is protected from cold helium gas by gloves and face shield.

Fig. 3.2: Stainless steel transfer lines for liquid nitrogen

the speed with which this freezing process can occur, it is effectively instantaneous compared to an operators reaction response.

3.2 Storage, transport and handling of cryogens

Store and transport cryogens using only the containers for which they were specifically designed. See for example fig. 3.3.

Store and transport cryogenic vessels in an upright position. Internal support is generally restricted to the region at the top of the neck tubes and the system may be easily damaged by tipping the vessel away from vertical.

- Storage and transport vessels must be made of suitable materials. Many materials (even some common steels) become dangerously brittle at low temperatures.
- Bungs (or stoppers) should be tied to the top of the container. This prevents them from being lost and ensures that they cannot be blown out by high pressure and become dangerous projectiles.
- If you are using a magnet system, the cryostat and transport vessel should be constructed of non-magnetic materials.
- Never use hollow tubes as dipsticks. If a warm tube is inserted into liquid nitrogen, liquid will spout from the top of the tube due to vapourisation and rapid expansion of liquid inside the tube.

3.3 Spillage of cryogens

Spilt cryogens will dramatically reduce the temperature of the item on which they are spilt; this may affect it in many ways.

- Cryogens spilt on vacuum equipment may freeze rubber vacuum seals (such as O-rings) and cause loss of insulating vacuum. Route transfer lines away from vacuum seals and use a hot air blower if ice starts to collect.
- If you spill cryogens on electrical cables they may freeze and fracture the insulating layer causing electrical hazards. Keep all cables above the level where cryogens may be spilt on them, not on the floor.
- Spilt cryogens can also condense the moisture from the air to form a thick mist which can obscure your vision. If you are enveloped in a cloud of cold gas you may lose your balance and fall. This is particularly dangerous if you are standing on a platform.
- The floor of your laboratory may be damaged if cryogens are spilt over it. In particular, plastic tiles may become very brittle or may be cracked by rapid cooling.

3.4 Liquid nitrogen – specific techniques

Liquid nitrogen should not be stored in open topped dewars as it would then be exposed to air. Since liquid oxygen has a higher boiling point than liquid nitrogen, oxygen from the air will then condense into the nitrogen. If this is allowed to continue, the oxygen concentration may become so high that the liquid becomes as dangerous to handle as liquid oxygen. This applies particularly to wide-necked dewars, and is a potential fire or explosion hazard.

Liquid nitrogen can be poured from small vessels using a funnel or tipping trolley. If the storage dewar is too big to be tipped over, or if it might be damaged, the liquid can be transferred by

(a)

(b) (c)

Fig. 3.3: Examples of (a) open, (b) self-pressurising liquid nitrogen storage vessels.[2] Also shown in (c) are examples of tipping trolleys.

Fig. 3.4: A typical liquid helium storage vessel.

Fig. 3.5: A typical portable oxygen monitor.

pressurising the storage dewar using helium or nitrogen gas but not air.

Only use suitable metal tubing to transfer cryogens. Do not use rubber, silicone rubber or plastic tubing as it can shatter with the thermal shock. Although polythene and nylon are sometimes used this practice is not recommended. Any materials you decide to use should be carefully tested in safe conditions, and only used if approved by the manufacturer. Uninsulated transfer lines can be used but losses should be expected and there are potential fire hazards associated with oxygen enrichment around the tube.

Make the following daily checks on liquid nitrogen vessels:

- Check the boil-off and investigate if it is higher or lower than expected.
- Check the liquid level and refill if necessary.
- Check that the relief valves have not been tampered with.
- For each valve visually inspect that the vent mechanism is seated squarely in the valve.
- After a liquid helium transfer into a liquid nitrogen shielded helium vessel, check that the nitrogen vents are still clear.

Remember that inserting a warm tube into a dewar of liquid nitrogen will cause cold gas (and possibly liquid) to be ejected from the open end.

Even vessels containing an inert cryogen (e.g. liquid nitrogen) can be a potential fire or explosion hazard. Do not leave cryogen vessels open to the atmosphere. Dewars should be covered with the lid provided to minimise contact with the air.

3.5 Liquid helium – specific techniques

Liquid helium is the coldest of all cryogenic liquids. It will therefore condense and solidify any other gas coming into contact with it. Any surfaces cold enough to condense air in normal operation could also increase the oxygen concentration to a dangerous level. This condensation of liquid oxygen is why liquid helium containers often have labels saying “Flammable liquid” even though the liquid helium itself is not flammable.

Liquid helium must be kept in specially designed storage or transport vessels (dewars) to prevent high boil-off rates, high vessel pressures and external icing of storage vessels (see for example Fig. 3.4). Dewars should have a non-return valve fitted in the helium exhaust line at all times or be connected to a helium recovery system, so that air does not enter the neck and block it with ice.

If possible, liquid helium should be kept at a slight positive pressure, so that if there is a leak, helium may leak out but air does not leak in.

Make the following daily checks on liquid helium vessels:

- Ensure that the non-return valve (or recovery system) is still fitted to the exhaust.
- Check that all other ports on the top plate are sealed properly.
- For each valve visually inspect that the vent mechanism is seated squarely in the valve.
- Check the boil-off and investigate if it is higher or lower than expected.
- Check the liquid level and re-fill if necessary.

Liquid helium has a small latent heat of vapourisation, therefore transferring it from vessel to vessel will result in high boil-off and potentially dangerous pressures unless a specifically designed transfer tube, with vacuum insulation, is used. Techniques for using these will be described in your Operator's handbook. Check the vacuum of the transfer tube prior to use.

Inserting warm objects into liquid helium will result in high boil-off. Insert warm objects into liquid helium vessels very slowly. This makes sure that they are well cooled by the cold gas before they reach the liquid and:

- Reduces the hazard from rapid boiling which produces a jet of cold gas.
- Considerably reduces the consumption of liquid helium, saving money.

Liquid helium may cause blocked vents or oxygen enrichment.

Only transfer liquid helium using a specifically designed transfer tube with vacuum insulation. Check the vacuum of the transfer tube prior to use.

Clean to "oxygen clean" standards any surface cold enough to condense air in normal operation.

Inserting a warm tube into a dewar of liquid helium will cause cold gas (and possibly liquid) to be ejected from the open end.

3.5.1 Cryogenic systems with windows

Some systems may be supplied with optical windows, intended to provide optical access to the sample position. These windows can form part of the outer vacuum chamber (OVC). The windows can be especially vulnerable to damage and unused windows should be protected from damage. Damage to the optical windows can result in a softening of the OVC vacuum, potentially leading to a rapid boil-off of cryogens in the system.

3.6 Protection against asphyxiation/anoxia

Atmospheres containing less than 18% oxygen content are potentially dangerous. Entry into atmospheres containing less than 20% oxygen content is not recommended.

Asphyxia due to oxygen deficiency is often rapid with no prior warning to the victim. *There is no sensation of breathlessness to warn you that you are being asphyxiated* as breathlessness is a symptom of a high concentration of CO_2 and not of low concentration of oxygen. A general indication of what is liable to happen in oxygen deficient atmospheres is given in the next table although the reactions of individuals can vary widely.

Tbl. 3.1: Guide to the effects of an oxygen deficient atmosphere on the human body.

O_2 content (% volume)	**Effects (at atmospheric pressure)**
20.95	Normal level
19–21	Normal operation

O_2 content (% volume)	Effects (at atmospheric pressure)
17–19	Should be OK for fit and healthy individual but not advisable for operators with a known respiratory or heart medical condition.
14–17	Unsafe even for fit and healthy individuals
11–14	Diminution of physical and intellectual performance without the person's knowledge.
8–11	Possibility of fainting after a short period without warning.
6–8	Fainting within a few minutes; resuscitation possible if carried out immediately.
0–6	Fainting almost immediate; death ensues; brain damage even if rescued.

3.7 Guide to the stages of asphyxia

The victim may not be aware of the onset of asphyxia. If any of the following symptoms appear in situations where asphyxia is possible and breathing apparatus is not in use, immediately move the affected person to the open air, following up with artificial respiration if necessary:

- Rapid and gasping breathing
- Rapid fatigue
- Nausea
- Vomiting
- Collapse or incapacity to move
- Unusual behaviour

It is important to make the relevant preparations before you put yourself at risk. For a slowly reducing oxygen level the first symptoms will be increased pulse and breathing rate with impaired judgement, however these symptoms may go unnoticed. The next stage is the inability to stand up or even to crawl. At this point it may already be too late for you to help yourself.

Before conducting work you must conduct an assessment for the risk of asphyxiation. Mitigations that can protect against that risk are as follows:

- Ensure that there is sufficient ventilation in your own laboratory and in other rooms nearby.
- Install sensors which will sound an alarm if the oxygen level is too low unless you are sure about the effectiveness of the ventilation in a room. Inexpensive, portable oxygen monitors are readily available[2] - see Fig. 3.5.
- Leave the room immediately if a large amount of cold gas is released quickly (for example after a superconducting magnet has suddenly become resistive or "quenched", releasing its energy into the liquid helium).
- Ventilate the room well if you are precooling a large system with liquid nitrogen.
- Leave the room immediately if a large amount of liquid is spilt. Consider sounding the fire alarm if there is likely to be a fire hazard, or to clear the area quickly.
- If there is a possibility of a lack of oxygen in the room, hold your breath, to remind yourself of the urgency of leaving the area.
- Do not accompany storage or transport vessels in confined spaces (especially in lifts, elevators or enclosed vehicles).
- Use a suitable exhaust system to pipe exhaust gases away from the cryostat to the atmosphere or into a recovery system. This needs to be designed by a competent engineer.

- If you store cryogenic liquid vessels in a room that is not well-ventilated, put warning signs on the doors so that no one enters the room until it is well ventilated. The room should be locked and the oxygen concentration must be checked before anyone enters the room.
- Remember that cold nitrogen gas tends to collect near the floor, and helium gas near the ceiling.

Ensure that oxygen monitors are calibrated regularly according to the manufacturer's instructions.

3.8 Calculating possible oxygen depletion following a cryogen spillage

Part of the risk assessment process should consider the worst case scenario when the entire contents of a cryogenic storage vessel are lost to the room. For example, a dewar of volume $V_d(m^3)$ is filled with a cryogenic liquid with gas to liquid ratio f (see Sec. 8.1). If you assume 10% filling losses then the total volume of gas lost to the room is:

$$1.1 f V_d \quad [\mathrm{m}^3]$$

If the volume of the room is V_r then the oxygen percentage may be reduced from 21% to

$$\frac{21}{V_r}\left(V_r - 1.1 V_d f\right) \quad [\%]$$

Example: The room is 7 x 8 x 2.5 metres (140 m^3). A 25 litre dewar of liquid nitrogen has just been filled. If the entire contents are spilled the oxygen concentration is reduced to

$$\frac{21}{140}\left(140 - (1.1 \times 25 \times 694)/1000\right) = 18.1 \quad [\%]$$

Based on Tbl. 3.1 the room is just big enough to allow a fit and healthy and healthy individual to work.

This calculation assumes uniform dispersion of nitrogen gas throughout the room, however particular consideration should be given to an operator working in a pit as the cold nitrogen gas will sink into the pit and cause higher levels of oxygen depletion than suggested above.

3.9 Ventilation

The type of ventilation depends on a multitude of factors such as type of location, gas type, possible leaks etc. Ventilation can be natural or forced. The design criterion is the number of air changes per hour.

In locations above ground level with no special ventilation openings natural ventilation will provide typically one change per hour. This is not the case in buildings with windows that are tightly sealed. For underground rooms with small windows 0.4 changes per hour can be considered as an average value. To achieve more than two air changes per hour a forced ventilation system is necessary. Different regulations may recommend or require for different situations a specific number of air changes per hour.

Natural ventilation is generally sufficient for handling transportable cryogenic vessels above ground level, provided that the room is large enough or that any outdoor area is not enclosed by walls. An indoor area should have ventilation openings with a total area of 1% of the ground area. The openings should be positioned diagonally across the room. The density of the gas should also be taken into consideration with the main opening at the highest point for gases lighter than air (e.g. helium) and at ground level for gases heavier than air (e.g. cold nitrogen).

Any laboratory area below the normal floor level (such as a pit) can easily become enriched with nitrogen; such an area must be regarded as a special hazard and restricted accordingly or appropriate sensors and procedures put in place. The area must be covered by local risk assessment and hazard management plan. Oxford Instruments do not take responsibility for injuries or damage sustained by a failure of system operators to take appropriate precautions.

3.10 Protection against fire hazards

Oxygen enrichment may cause spontaneous combustion.

In the event of a fire, sound the fire alarm and make sure everyone leaves the area. After the fire has been extinguished make sure that the system is safe.

Most of the fire hazards encountered in normal laboratory scale cryogenic systems are caused by oxygen enrichment. Liquid oxygen can condense from the air on to surfaces which are at temperatures below 90 K. You can often see liquid air running from a cold helium recovery line if a helium transfer is carried out too quickly or if a magnet has quenched. This liquid can promote fires, even with materials that normally might not be regarded as combustible.

Liquid nitrogen in an open bucket tends to condense atmospheric oxygen into solution because the boiling point of oxygen is above that of nitrogen. In equilibrium, the liquid may have an oxygen concentration higher than 50%. Any combustible materials exposed to this liquid can burst into flames spontaneously. A foam plastic bucket containing liquid oxygen presents a serious hazard.

Liquid air and liquid oxygen can be handled safely, but only by those who have had the necessary training.

If you transfer liquid nitrogen through a tube that is not vacuum insulated, remember that oxygen enrichment will occur around the outside of the tube. Any flammable lagging material that comes into contact with this liquid (e.g. cloth or polystyrene) could burn fiercely if it is accidentally ignited. Some materials even ignite spontaneously in liquid oxygen.

Avoid the risk of fire by taking the following precautions:

- Make sure that there is no oil or grease in a position where it may be exposed to liquid air (even if it is liquefied by accident).
- Do not use compressed air to blow liquid nitrogen out of a cryostat because the oxygen in the compressed air will condense into the liquid very easily.
- Forbid smoking in the areas where cryogens are handled.

- Make sure that suitable fire extinguishers are available; CO2 extinguishers are normally recommended.
- Train people to use the fire extinguishers properly.

Special expertise is required to put out these fires safely so if you have not been trained how to do it find someone who has. If you choose the wrong type of fire extinguisher or you do not use it properly you may block the exhaust vents of the cryostat with ice. Blockages are discussed in the next section.

3.11 Protection against blockages – return valves

These guidelines are intended to apply only to preventing rupture of a cryogenic vessel caused by an accidental blockage of the exhaust or from warming up a cryostat which has accidentally condensed contamination from the atmosphere on to cold surfaces. These instructions should be sufficient for most laboratory systems. Flammable or explosive cryogens which are likely to cause explosions because they have ignited are not covered by this booklet.

Symptoms of a partial or total blockage include:

- Abnormally low or high cryogen boil-off.
- Abnormally low or high pressure readings.
- Difficulties in removing ancillary items such as demountable current leads, measurement probes or transfer syphon.
- Inefficient helium transfer.

Blockages are frequently caused by leaks past cracked or badly seated O-rings or by poor cryogenic practice such as leaving cryogenic vessel necks open to the atmosphere.

Check the system boil-off regularly. If there is no boil-off and you know that the system is not empty check whether a blockage is preventing the natural boil-off. The pressure inside the cryostat may rise until it reaches a dangerously high level. Always fit a non-return valve to at least one of the liquid nitrogen vessel ports (see for example Figs. 3.7, 3.6).

Even after all the liquid has evaporated there should still be a perceptible flow of exhaust gas as the gas in the system warms up. In other words, even if the system contains no liquid it is not “empty” or safe until it reaches ambient temperature.

If the exhaust ports are connected to pumping lines that are too small in diameter, or to a helium recovery system of inadequate volume, this will create an impedance to helium flow in the event of the magnet quench which could cause a dangerous pressure rise in the system. Make sure that the lines have a large enough diameter for the expected gas flow. In general, you should assume that the diameter of the tube must never be smaller than the diameter of the exhaust port.

The helium reservoir exhaust must be fitted with a non-return valve or connected to a helium recovery system (see for example Fig. 3.6). The recovery system must include adequately sized flap valves to prevent back-flow from the recovery system into the cryostat.

This prevents ambient air leaking back into the cryostat. The valve should be at least large enough to handle the normal gas flow during a liquid helium

Fig. 3.6: Quench relief valve and non-return valve fitted to the helium reservoir exhaust of an Oxford Instruments cryostat.

Fig. 3.7: Non-return/pressure relief valve for nitrogen vent ports on standard Oxford Instruments cryostats

transfer.

Systems must be fitted with pressure relief valves to allow helium and nitrogen gas to leave the system quickly. If your system was fitted with these valves when it was supplied, it must never be cooled down without them.

All relief valves on the system should be large enough to handle the maximum possible gas flow, caused by all possible failure modes happening together (e.g. magnet quench and vacuum jacket failure). In the event of a magnet quench or major vacuum failure the evaporated helium will be vented safely through the valve(s). Various pressure relief valves are illustrated in this guide.

Rapid failure of the insulating vacuum can cause all the liquid in the cryostat to evaporate very quickly.

If the system exhaust vents become blocked, dangerous pressures can quickly build up in the system causing the vessel to rupture. Make sure that all exhaust vents are kept clear of ice. Check for blockages regularly and often.

Make sure the system vents are at a temperature higher than 0°C, so that water does not freeze there. Fitting a short length (say 20 cm) of plastic pipe over the vent tubes can help; they will be covered with ice but if they are long enough the open end will be warm enough to stay dry. Make sure that systems are not stored where they might be exposed to rain or moisture, unless they are properly protected.

3.12 Clearing blocked tubes

If you find that all the vents of a vessel are blocked, you should quickly and calmly evacuate the area and then find an experienced cryogenic technician to help you clear it. The expert will be better qualified to decide how much time to spend trying to clear the blockage before it is too dangerous to be near the cryostat. If only one vent is blocked and the vessel is still venting safely through another vent, it may not need urgent attention but you should still clear the blockage as soon as possible.

If you cannot find an expert you are advised to contact Oxford Instruments Customer Support before trying to clear it yourself. They will be able to provide detailed advice. For instance, if the magnet is in persistent mode at field you will have to balance the risks associated with running the magnet to zero (which increases the helium boil-off during the ramping process) with the risk of damaging the magnet if it warms sufficiently to quench.

Unblocking procedures require a supply of helium gas at room temperature or a warmed copper or stainless steel tube or a combination of both. Remember that the process of unblocking a tube or vent is unlikely to entirely remove any frozen water or air from the system. It will simply move it to a different place in the system.

Check for nitrogen vessel blockages regularly and often (see Fig. 3.8) and clear them as soon as possible. It is preferable to use warm gas lines and blow warm helium (or nitrogen) down the blocked tube to melt the blockage. If this is not possible a stiff rod can be used but great care is required to avoid damaging the thin-walled tubes of the cryostat.

If the vent has been blocked for an unknown length of time or if you cannot clear the blockage, evacuate everyone from the area, and do what you can to reduce the secondary risks from the effects of possible rupture of the vessel. If it is not possible to make the area safe, consider moving the vessel to a safe place but this requires a careful risk assessment as the act of moving the system will increase the internal boil-off which will increase the rate of pressure rise.

When clearing blocked tubes, when the blockage clears there may be a sudden jet of very cold gas or even liquid resulting in a risk of cold burns to exposed skin.

Care should be taken to ensure that appropriate protective clothing is worn.

3.13 Blocked variable temperature inserts, frozen-in probes / ultra low temperature inserts

If you find that the system's variable temperature insert is blocked and a measurement probe or ultra low temperature insert is frozen in, there is a risk of the probe becoming a projectile. Special care must be taken to safely remove it.

Oxford Instruments variable temperature inserts (VTI) come in two forms:

Dynamic: The sample is mounted on a top loading probe which is inserted into the central tube of the VTI via a flange on the cryostat top plate. The liquid helium is drawn from the main reservoir through a needle valve and pumped away from the top of the central tube. The sample space forms part of the VTI gas circuit.

Static: The sample is mounted on a top loading probe which is inserted into the central tube of the VTI via an NW flange on the cryostat top plate. The central tube of the VTI is usually referred to as the sample space and is normally filled with a *static* charge of 4He exchange gas to give good thermal contact between the sample and the VTI heat exchanger. The exchange gas in the sample space is completely separate from the helium which flows through the VTI heat exchanger.

More detail can be found in the Operator's Manual.

As the VTI is a relatively small volume, very-high pressures can build quickly if a blockage occurs and the VTI is warmed up. This is particularly true for a static VTI where the central chamber, containing the exchange gas and probe/ULT insert, is a sealed tube.

Under normal operation when running the VTI at low temperatures the pressure in the VTI

Fig. 3.8: Checking for nitrogen vessel blockages using a metal probe.

sample space is below atmospheric pressure. Leaks can therefore result in air continuously entering the VTI space. These leaks can occur at the coupling flange between the probe/ULT insert and the VTI space or on the room temperature fittings of the probe/ULT insert. In this instance nitrogen and oxygen in the air can condense in the lower parts of the VTI space. At the same time, water vapour in the air can condense and freeze, to form ice higher up in the space. This ice can potentially block the path of gas from the lower to the upper parts of the VTI sample space. Subsequent warming of the VTI can then cause a fast and dangerous buildup of pressure in the lower half of the VTI space. The pressure below the ice blockage can increase to a level where the resulting force exceeds the shear strength of the ice blockage and the shear strength of the probe/ULT insert coupling flange clamp. This can result in the hazardous situation where the probe/ULT insert is ejected from the top of the VTI.

This situation is understood to only be possible in the condition where there is a gross leak into the VTI sample space of order > 10^{-3} mbar·lts/sec when running for more than 1 month. A dynamic VTI is considered to be less prone to this failure mode as in normal operation the VTI sample space is continuously pumped, so any air ingress through a leak will tend to be extracted from the VTI by the pump.

3.14 Prevention of VTI sample space blockages - best practice

A few best practice steps should be taken to avoid the possibility of a VTI sample space blockage. Particular care should be taken for long-term experiments where the measurement probe/ULT insert is expected to be kept continuously cold for many weeks. The most likely source of leak is the O-ring (or co-seal) at the top of the VTI where it interfaces with the measurement probe/ULT insert.

Each time the measurement probe/ULT insert is inserted into the VTI the following checks should be made:

- The O-ring should be very carefully inspected. Look closely for:
  - Small pieces of grit or swarf which may have become stuck or partially embedded in the O-ring. If necessary clean the O-ring with isopropanol, or if in doubt replace it.
  - Cracks, cuts or gouges. If in doubt replace it.
  - Dents or eccentricity which will prevent it seating correctly. If in doubt replace it.
- Carefully inspect the O-ring seating surface of the VTI flange. It should be free from scratches, dents and gouges. If in doubt seek advice from the system / laboratory supervisor or photograph the flange surface and send it to Oxford Instruments NanoScience customer support (ServiceNSUK@oxinst.com) with a description of the system.
- Carefully inspect the O-ring seating surface of the measurement probe/ULT insert. It should be free from scratches, dents and gouges. If in any doubt seek advice from the system / laboratory supervisor or photograph the flange surface and send it to Oxford Instruments NanoScience customer support (ServiceNSUK@oxinst.com) with a description of the insert and system it is intended to be used with.

When using a static VTI, there is an additional pump out valve and pressure relief valve on the system sample space. This pump out valve is used to add a charge of helium exchange gas to the static VTI sample space (or alternatively to evacuate this space). The relief valve is designed to protect the space against an excessively high pressures being generated in the VTI space in a fault condition. If either of these valves fail to close fully they are also a potential source of an air leak. It is therefore recommended that:

- A blank be fitted to the flange of the pump out valve port when it is not in use so that even if the valve fails to close fully the port will still be sealed.
- To stop the relief valve venting when adding the charge of helium gas before the probe load, care should be taken to vent the sample space with flowing helium gas at a pressure below the opening pressure of the relief valve (typically <0.1 bar gauge).
- If the pressure relief valve ever vents gas, then the O-ring should be leak tested using a mass spectrometer leak detector to make sure it has re-sealed correctly.

In addition to the points previously mentioned it is important to ensure that no leaks are present on the probe/ULT insert. Oxford Instruments NanoScience sells a test sock for room temperature leak testing of measurement probes/ULT inserts. Use of this test sock combined with a leak detector (mass spectrometer) is recommended prior to every measurement probe/ULT insert load into a VTI. Use of this sock will leak test every joint on the measurement probe/ULT insert such that the only joints untested will be:

- The exchange gas admittance port (static VTI only) which should be blanked off as described above.
- The pressure relief valve which should be re-leak tested if it ever vents gas.
- The O-ring at the top of the VTI which should be inspected as outlined previously.

Following the checks listed above will help minimise the risk of any leaks resulting in sample space blockages. An alternative to the test sock and the multiple checks above, is that the user leak tests the probe inside the sample space using a leak detector (mass spectrometer) after each load of the probe. This method can be used to check all the joints listed above, however additional time in the process is then required to pump the background helium level in the sample space down to 10^{-4} mbar·lts/sec range required in order to detect gross leaks of order 10^{-3} mbar·lts/sec.

3.15 Detection of a vti sample space blockage

⚠ A frozen in probe or ultra low temperature (ULT) insert can become a projectile resulting in a risk of death or injury. Never put any part of your body directly above the probe/ULT insert.

Indicators of a blockage include:

- The VTI failing to reach normal base temperature. There maybe other reasons for this so it should be considered with the VTI sample space pressure readings and VTI gas flow/VTI pumping line pressure
- VTI space pressure higher than expected at low temperature
- Probes/inserts with height and/or rotation adjustment that cannot be adjusted in-situ
- Rotating sample stage drive rods frozen and unable to move (sample-in-gas probes only)

If any such indicators are seen that cannot be explained by other causes the operator is advised to see Sec. 3.16 on warming up a system with a frozen in probe/ULT insert.

A VTI sample space blockage may also be detected during the following standard VTI operations:

1. **High temperature operation of the VTI**

In an unrestricted VTI sample space, there will be a clear scaling relationship between the temperature of the VTI and the pressure at the top of the VTI sample space (which is typically monitored using a mechanical pressure gauge at the top of the VTI). It is recommended that this expected change in pressure is monitored when warming from base temperature to ~ 65 K before setting higher VTI temperatures. If the pressure does not rise as normal this could be an indication of a blockage in the VTI sample space and the operator is advised to see Sec. 3.16 on warming up a system with a frozen in probe/ULT insert.

2. **Unloading the probe/insert**

   The details of the unloading procedure will depend on whether or not the probe/insert is to be unloaded from a cold or warm VTI, and on whether or not the probe/insert is designed so that its height and/or angular position can be altered in situ. Remember that if air has blocked the VTI sample space and the VTI temperature is raised above 77 K, the probe/insert could suddenly be ejected from the VTI if the mounting flange clamp fails or as the clamp is undone.

3.15.1 Unloading from a cold (temperature below 77 K) VTI

Prior to unloading the probe/insert, when venting the VTI sample space to atmospheric pressure with room temperature helium gas the VTI temperature should increase as normal due to the heat load from the warm gas. If this does not happen this could be an indication of a blockage in the VTI sample space and the operator is advised to see Sec. 3.16 on warming up a system with a frozen in probe/ULT insert.

If the VTI temperature increase is normal then (with the VTI temperature below 77 K):

1. For probes/inserts with height and/or rotate adjustment (including sliding seals), check that the probe/insert is free to move without undoing the mounting flange clamp. If it cannot be moved this could be an indication of a blockage in the VTI space and the operator is advised to see Sec. 3.16 on warming up a system with a frozen in probe/ULT insert.

2. For probes/inserts without height and/or rotate adjustment, undo the clamp retaining the measurement probe/ULT insert and gently try lifting the probe/ULT insert. If it is stuck and cannot be pulled up as normal, this is an indication of a blockage in the VTI space and the operator is advised to see Sec. 3.16 on warming up a system with a frozen in probe/ULT insert.

3.15.2 Unloading from a warm (temperature above 77 K) VTI

If air has blocked the VTI sample space and the VTI temperature is raised above 77 K, the pressure in the sample space will rise. This increase in pressure may cause the probe/insert to suddenly be ejected from the VTI should the mounting flange clamp fail or as the clamp is undone.

If you suspect that air has blocked the VTI sample space it is therefore recommended to first check the probe/insert for free movement while the VTI is at a temperature below 77 K by following the procedure outlined above in “Unloading from a cold VTI (temperature below 77 K)”. If no problems are observed during this procedure, then the clamp retaining the measurement probe/ULT insert should be re-fitted if it was undone and the helium gas supply to the VTI sample space should be shut off. The VTI can then be warmed above 77 K to the desired temperature before re-opening the helium gas supply to the VTI sample space and unloading the probe/insert.

3.16 Warming up the system with a frozen in probe / ultra low temperature insert

If the sample probe is frozen within the sample space it may become a projectile when warming the system up. Ask nearby personnel to evacuate the area until further notice and cordon off the system with barriers.

Notify the laboratory or cryogenic systems supervisor of the potential hazard on warm up.

For Cryofree® systems turn off the pulse tube/GM refrigerator compressor and allow the system to warm up naturally.

For systems with cryogen reservoirs ("wet" systems) leave the system to warm up naturally (if this is an excessive warm up period, conduct a risk assessment before considering accelerating the warm up process).

Stay out of the area until all the system temperature sensor readings are above 0°C.

3.17 Warming up a system

Take care when you warm a system to room temperature; read the Operator's Handbook and follow the instructions carefully.

All the chambers in the cryostat must be free to vent safely, even if you think that they are empty. If the system has been cold for a very long time, or there is a leak into the cryostat, air may have been cryopumped on to the cold surfaces inside the system - there is no way to detect whether this has happened. As the air evaporates its volume may increase very quickly and the vessel may not be strong enough to take the resulting high pressure.

Remember:

- 1 L of liquid helium expands to 750 L of gas at room temperature and atmospheric pressure (NTP), or becomes 1 L of gas at 750 bar if it is not free to expand
- 1 L of gas at 4.2 K expands to 70 L at NTP, or becomes 1 L of gas at 70 bar if it is not free to expand
- Even if all of the liquid and gas has been removed, it is possible that some air is frozen in the vessel or the pumping line, and this will expand in a similar way
- Until you warm up the system, you cannot tell whether there is any frozen air in one of the chambers. For your own safety, assume that it is there

3.18 Dangerous cryogens

You need additional training before handling cryogens other than helium and nitrogen.

The correct handling procedures for the less common cryogens which may be toxic, flammable or explosive are not included in this booklet. If you are using cryogens other than liquid nitrogen or liquid helium it is important that you obtain the necessary training.

3.19 Notes on operating dry cryogenic systems

Cryogen free devices use no cryogens but it is important not to become complacent about safety issues when using Cryofree® systems. Always be vigilant to the safety issues and implications surrounding cryogen free equipment. The risk of significant injury or death is just as real whilst using these devices. The danger of blockages and resultant explosions are the same as for liquid cryogen based devices. Also other risks exist with the use of cryogen free devices which are detailed below.

The cooling “engine” for Cryofree® systems are usually mechanical coolers using either a Pulse Tube or Gifford McMahon process. These coolers rely on large pressure differentials generated by electrically driven compressors consuming high power and typically running from the 3-phase supply. Compressor systems introduce a number of risks into a laboratory and users must be aware of these risks, which exist in parallel to the usual risks around cryogenic systems. Follow the manufacturer’s guide lines for safe operation of the cryo-cooler equipment.

Most cryo-coolers operate with high pressure lines (up to 30 bar) between the compressor and the cold head. It is important to regularly check these lines for damage or deterioration as release of high pressure gas can be dangerous. High pressure lines can also move irregularly as pressure changes occur. Ensure compressor gas lines are anchored and cannot cause injury to personnel or damage to other systems in case of movement. Follow the manufacture’s guidelines when coupling or decoupling high pressure lines.

Most compressor systems are located some distance from the cryo-cooler and normally in nearby store rooms or basements to reduce noise transmission. Keep all areas surrounding compressor systems free of other equipment. Do not store flammable materials, wood, paper or other combustible material near to compressors as this may present a fire risk. Ensure there is adequate room for airflow around the compressor.

Compressor based systems all operate with high power loadings and normally use a supply of chilled water to keep the compressor cool during operation. Check electrical safety circuits and cabling regularly on these systems. Ensure no water leaks occur and any spilt water is cleaned up immediately. Failure to check these items can result in electrocution or fire risk presenting significant risk to life.

Decoupling high pressure lines can be dangerous. Ensure that manufacturer guidelines are followed. Always use 2 spanners when decoupling the lines to ensure the fittings do not rotate and that the fittings are not accidentally dismantled as the lines are decoupled. If a fitting is accidentally dismantled while the line is under pressure, internal components of the fitting can be ejected at high velocity resulting in a risk of serious injury or even death.

3.20 First aid treatment for cold burns

Flush the affected areas of skin with copious quantities of tepid water but do not use any form of direct heat such as hot water or a room heater. Move the casualty to a warm place (about 22°C). If medical attention is not immediately available arrange for the casualty to be transferred to hospital without delay. While waiting for transport:

- Loosen any restrictive clothing.
- Continue to flush the affected areas of skin with copious quantities of tepid water.

- Remove any metallic straps, bracelets etc. from the affected area of skin.
- Protect frozen parts with bulky, dry, sterile dressings. Do not apply so tightly as to cause restriction of blood circulation.
- Keep the patient warm and at rest.
- Ensure ambulance crew or hospital is advised of details of the accident and first aid treatment already administered.
- Smoking and alcoholic beverages reduce the blood supply to the affected part and should be avoided.

If the patient's lungs have been exposed to enough cold gas to cause distress, or if in doubt, take him or her to hospital immediately. Even transient exposure to cold gas can produce discomfort in breathing and can provoke an attack of asthma in susceptible people. If the victim is suffering from dizziness or loss of consciousness due to asphyxiation:

- Make sure that you are safe first (and in some cases this means that you should not enter the area without breathing apparatus)
- Summon medical help immediately
- Move the victim to a well-ventilated area if it is safe to do so
- Apply artificial ventilation or resuscitation if necessary.

4 Superconducting magnet systems

Many cryogenic systems include superconducting magnets which operate at very high fields. Extensive studies continue into the direct effects of magnetic fields on health (especially in relation to clinical Magnetic Resonance Imaging (MRI), where patients are necessarily exposed to very high fields for diagnostic imaging).[4] References to recommendations on exposure limits are shown in Tbl. 4.4 and Tbl. 4.2. It is the users responsibility to ensure that all applicable regulations are followed.

The following hazards should be considered as a minimum:

- Magnetic items may move suddenly and uncontrollably towards a magnet; an example graph showing the sudden increase of force at a particular distance is given in Fig. 4.1. Please remember that most tools are magnetic.
- Someone could be trapped between a large magnetic item (such as a gas cylinder) and a cryostat, resulting in severe injury or death
- Medical electronic implants (such as pacemakers) may be affected by a magnetic field
- The magnetic field is three dimensional so the field may affect rooms not only on the same floor, but also on the floors above and below your laboratory.
- Steel reinforcement in the floors and walls could become magnetised. Use the stray field plot to assess this risk.
- Magnets contained within a bath of liquid may suddenly quench. This sudden release of energy can vapourise the liquid cryogens releasing cold gas into the room that could displace the air leading to a risk of asphyxiation as discussed in Sec. 3.6.
- Remember all the potential cryogenic hazards as outlined elsewhere in this document.
- Magnetic data on credit cards, disks and other magnetic storage media may be corrupted.

Every magnet site should be reviewed individually to determine precautions to be taken against these hazards and it is strongly recommended that a formal site survey and risk assessment be carried out.[3]

4.1 Before energising the magnet

Before you start to energise a magnet:

- Ensure that all loose ferromagnetic objects are secured or moved to a safe distance. These will normally be safe outside the 5 gauss field contour (see your stray field map).
- Check that the protection circuit is connected. Oxford Instruments magnets fitted with persistent switches are impossible to energise unless this condition is satisfied.
- Connect one ground wire between the earth points on the cryostat and the power supply and a second wire between the same cryostat earth and a laboratory earth point; refer to your installation manual for details.
- Check that there is an insulating cover fitted over the current lead terminals.
- Display warning signs (preferably illuminated) at all the laboratory doors, to remind people that the magnet is operating.
- Display warning signs giving notice of the possible presence of magnetic fields and of the potential hazards in all areas where the field may exceed 5 gauss.
- Ensure that all electronics and interfacing equipment are removed to areas where the

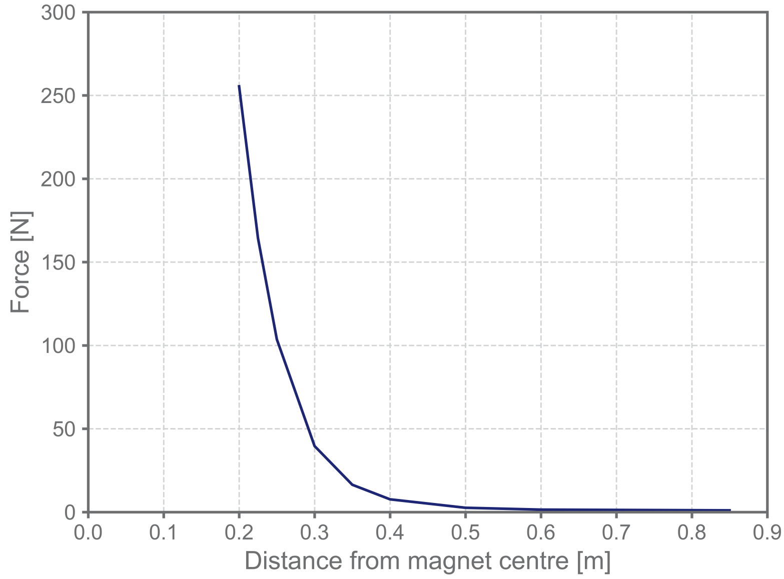


Fig. 4.1: Magnetic force example showing the force experienced by a 200 gram mild steel object as it approaches a large superconducting solenoid.

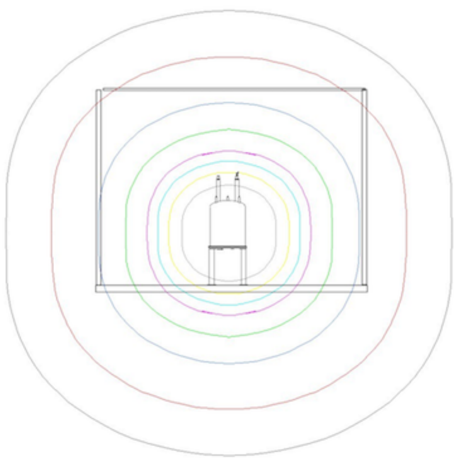


Fig. 4.2: Stray Field is three dimensional and affects areas on floors above and below your laboratory.

field level is sufficiently low; some guidelines are provided in Table 4.1 but you may need to refer to individual instruction manuals.
- Assess the safe working field level of all other equipment and take appropriate action.
- Check that the helium reservoir is protected by suitable pressure relief valves in case the magnet quenches.
- Consider carefully whether the exhaust gas or gas released by a pressure relief valve in the event of a quench could injure someone working on the system. If necessary, put guards around the hazardous regions.
- Check that there is enough liquid helium in the system.
- On systems with optical access the windows can be especially vulnerable. Unused windows should be protected. You are advised to guard "active" windows to provide a second line of defence against stray ferromagnetic objects.

4.1.1 Grounding of superconducting magnet systems

Please see Sec. 5.4 on the grounding of and the electrical hazards of superconducting magnetic systems.

4.2 While the magnet is at field

While the magnet is at field make the following checks regularly:

- Check the liquid helium and liquid nitrogen levels and refill if necessary.
- Check that the boil-off rates of helium and nitrogen gas are normal.
- Check that the warning signs are still in place: restrict access to unauthorised personnel.
- Do not bring magnetic objects close to the magnet. They should normally remain outside the 5 gauss limit. *Never attempt to check for the presence of a magnetic field with a ferromagnetic object such as a standard tool. You are risking serious injury or damage to equipment or personnel.*
- Only use non-magnetic storage/transport dewars and non-magnetic trolleys for liquid helium and liquid nitrogen.
- Use non-magnetic tools to work on a system if the magnet is energised.
- Remember that even non-magnetic electrically conductive materials may experience a force or resistance to motion due to field induced eddy currents.
- Give a verbal warning to people entering the room; remember that keys and coins are often magnetic.
- Ensure that there is sufficient ventilation.

4.3 Effects on personnel and instrumentation

Personnel must be restricted so as to prevent access to areas with fields greater than 5 G; warning signs should be erected.

Other equipment may be directly affected by the presence of large magnetic fields. The field may cause permanent damage or it may only have a temporary effect until the field is removed. Use a stray field map of your system and the Tbl. 4.1 to provide guidance. If your magnet is "Actively Shielded" there will be two stray field maps provided; normal operation (shield is active) and one showing the maximum stray field burst during a quench, in which the field contours are much extended.

Use the information in “Maximum stray field burst during quench” in cases where two stray field maps are provided.

4.4 Exposure limit values for exposure to magnetic fields

Guidelines on exposure to both static (DC) and time-varying (AC) magnetic fields are constantly evolving as new scientific and medical evidence is presented. It is therefore wise to refer to official legislation standards for a list of relevant documentation.

Tbl. 4.4 summarises static field recommendations from three sources together with limits issued by the Council of the European Union. These limits relate to both *public* and *occupational* exposure by personnel who work with magnetic fields.

In the context of magnets supplied by Oxford Instruments, AC magnetic fields of low frequency will be experienced if:

- An operator is located within the stray field region when the magnet is being ramped up or down;
- The operator moves into or out of the magnetic field.

Exposure limit values (ELV) information for these cases is summarised in the Tbl. 4.2. The International Commission on Non-Ionizing Radiation Protection (ICNIRP) limits are closer to those being considered by the Council of the European Union. As before these limits relate to occupational exposure by personnel who work with magnetic fields; they do not apply to the general public.

4.5 Medical implants

The operation of medical electronic implants, such as cardiac pacemakers, may be affected by static or changing magnetic fields.

Erect suitable notices to warn visitors and make sure that none of your staff is vulnerable.

Pacemakers do not all respond in the same way or at the same field level. You may not know that one of your visitors has a pacemaker so it is important to erect suitable signs to warn them of the danger. The stray fields caused by your magnet in other surrounding rooms may be enough to affect them.

Other medical implants, such as aneurysm clips, surgical clips or prostheses, may contain ferromagnetic materials. Therefore they may experience strong forces near the magnet which could result in injury or death. Rapidly changing fields (for example pulsed gradient fields) may induce eddy currents in any metallic implant, even if it is not magnetic, and generate heat.

4.6 Superconducting magnet quenches

There is always the risk of a quench, even in a very reliable and stable magnet. External factors can affect the stability of the magnet so you should always be prepared. There are three effects that must be considered:

- The collapse of the field
- The possible generation of high voltages

- The sudden release of cryogens as gas

During a quench, the stored energy in the magnet then evaporates most of the liquid helium very quickly. The helium recovery system is unlikely to be able to handle such a large amount of gas (perhaps 20 m^3 or more in a few seconds), and the relief valves will release the excess gas into the laboratory, displacing the air. If reasonably practicable, the cold gas should be vented in a safe area where nobody could be injured.

Use your system data to calculate the worst case oxygen depletion for the room containing the system.

If you are using an Oxford Instruments power supply for your magnet the output current will automatically be switched off safely. In the event of a magnet quench (either spontaneous or induced) the magnetic field will rapidly fall to zero. This may affect objects present in the room.

You should take the following actions after a a magnet quench:

- In poorly ventilated areas evacuate the room immediately and do not enter the room again until you know that there is sufficient oxygen in the air
- Check that pressure relief valves have re-sealed properly so that they do not let air back into the system
- Replace any broken bursting disks (if your system has any)
- Check that the nitrogen vent ports are still clear (if your system has any)
- Refill the helium vessel as described in the system Operator's manual
- Carry out a standard 4.2 K electrical and continuity breakdown check
- Re-energise the magnet according to instructions given in your Operator's handbook

In the event of a magnet quench all the cryogens may be released. The volumes of gases at room temperature will be approximately 70 m^3 for every 100 litres of cryogen (helium and nitrogen). Take precautions against asphyxiation.

If a superconducting magnet quenches releasing helium gas to the atmosphere you should evacuate the laboratory immediately and allow good ventilation until the helium gas has been dispersed. If you do not do this you may be asphyxiated.

Some large magnet systems are fitted with an Emergency Run Down Unit (ERDU); further details are provided in a dedicated ERDU document. Ensure that you understand how and in what circumstances you should activate the ERDU.

Pressing the emergency run down button will make the magnet quench. There is always the risk that the quench will damage the magnet. Only use it if the magnetic field poses a serious threat to personnel, for example if someone has been trapped by a magnetic object.

Tbl. 4.1: Guidelines for safe location of some sensitive equipment. Placement of equipment in lower stray field may be recommended for particular equipment.

Safe working field (gauss)	Equipment or restriction
1	Image intensifiers
1	Electron microscopes
1	Accurate measuring scales
1	Nuclear cameras
5	Pacemakers
5	Public access without warning signs
5	Cathode ray tubes
10	Computers
10	Watches and clocks
10	Credit cards
20	Magnetic storage media
25	Magnet power supply
25	Temperature Controller
25	Shim coil power supply

Tbl. 4.2: ELVs for AC magnetic fields (1 – 8 Hz).

Activity in static field region	Frequency (Hz)	Europe[5]	USA[6]
Slow head turning	1	200 mT	60 mT
Rapid head turning	2	50 mT	30 mT
Rapid head turning	3	22 mT	20 mT
Rapid head turning	4	13 mT	15 mT
Rapid head turning	5	8 mT	12 mT
Rapid head turning	6	6 mT	10 mT
Brisk walk in or out	7	4 mT	9 mT
Running in or out	8	3 mT	8 mT

Tbl. 4.3: ELVs for different regions and regulatory bodies.

Regulatory body	Region	Whole body	Limbs
ICNIRP[7,8]	Europe	200 mT over 8 hrs	8 T (peak value)
NRPB[9]	U.K.	200 mT over 24 hrs	200 mT over 24 hrs
ACGIH[6]	U.S.A.	60 mT over 8 hrs	600 mT over 8 hrs
Council of the European Union[5]	Europe	200 mT over 8 hrs	-

Tbl. 4.4: ELVs for DC magnetic fields (0 - 1 Hz). The values are time weighted averages. The regulatory bodies are: ICES (International Committee on Electromagnetic Safety), ICNIRP (International Commission on Non-Ionizing Radiation Protection, EU), ACGIH (American Conference of Governmental Industrial Hygienists, USA) and the Council of the European Union. †: EU 2013 (the “Directive”) and UK 2016 (the “Regulations” that implement the Directive).

Regulatory body	Public limits	Occupational limits
ICES	167 mT	500 mT
ICNIRP 2009	400 mT	2 T (8 T with control of movement), Limbs: 8 T
ACGIH	400 mT	2 T (8 T with special worker training and controlled work place environment), Limbs: 20 T
EU 2013/UK 2016†		Head and trunk, sensory: 2 T, Limbs: 8 T
		Any part of body, health: 8 T, Interference with active implanted medical devices: 0.5 mT
		Attraction and projectile risk in the fringe field of high field strength sources (>100 mT): 3 mT

5 Working with electrical equipment

The following recommendations apply to electrical equipment supplied by Oxford Instruments for use with laboratory systems including:

- Superconducting magnet power supplies
- Shim power supplies
- Temperature controllers
- Liquid cryogen level meters.

Each will be supplied with an individual instruction and service manual. This will contain caution and warning statements that you must read, particularly if repair or adjustment is required.

5.1 Protective ground

Unless it is being powered from its own internal batteries, the instrument must always be connected to an electrical ground (earth) when it is being used, to reduce the risk of electric shocks. The ground wire (green/yellow) in the instrument power cable must be connected to the laboratory electrical ground. Only use extension cables if they have an earth conductor. Do not disconnect the protective ground inside or outside of the instrument and do not have external circuits connected to the instrument when its protective ground is disconnected. It may be appropriate to use a residual current trip device as additional protection.

> The instrument will not stop working if the earth wire is not connected, and there is no indication that you might be in danger. Make sure that it is checked regularly (at least annually) by a competent person.

5.2 Working environment

Do not use electrical equipment in:

- Rain or excessive moisture;
- Flammable or explosive gases.

Unless specifically stated, Oxford Instruments' equipment is not designed to be water or splash proof, or to be used in areas where there are flammable or explosive gases or fumes.

5.3 Repair and adjustment

Some internal adjustments can be made to electrical equipment supplied by Oxford Instruments. Although we do not encourage you to make these adjustments we try to supply you with enough information to allow you to do it safely.

> Lethal voltages are accessible inside the instrument. Disconnect the AC power supply before you remove the covers or fuses. It is **not sufficient**

to switch off the main power switch. Only do this type of work if you are suitably qualified, are sufficiently skilled to understand all the risks you are taking and follow any local regulations that may apply.

There may be capacitors inside the instrument and power supply filter which are still charged to a high voltage even after the AC power has been removed. Discharge all of them carefully before you start work.

Some fault finding and calibration operations can only be carried out with the power connected to the instrument. If you have to reconnect the AC Power supply with the protective covers removed you must remember that **you are putting your life at risk.**

5.4 Electrical hazards from superconducting magnets

Superconducting magnets usually have high inductance and operate at high currents. A large amount of energy is therefore stored in the magnet when it is at field.

If a magnet quenches an inductive voltage is generated of magnitude

$$E = \frac{1}{2}LI^2$$

where L is the inductance of the magnet and I is the current in the magnet. Since it is not uncommon for the current to decay at 100 A/s it is possible to generate voltages of a few kilovolts. For this and many other reasons, Oxford Instruments superconducting magnets are designed in sections and a protection circuit is applied to each magnet section. When a magnet quench occurs, this protection absorbs a significant proportion of the energy in each section and limits the voltages generated across each section. As the magnet sections are inductively coupled as well as electrically connected, the instantaneous voltages across the various sections can be positive or negative. The total voltage appearing at the magnet terminals is the sum of the section voltages which will be less than the peak section voltages. The terminal voltage is further limited by the persistent switch protection (if fitted) and voltage limiting circuitry in the Oxford Instruments superconducting magnet power supplies.

Cryomagnetic systems may be fitted with “fixed” or “demountable” current leads. Systems with fixed leads should remain connected to the (earthed) power supply at all times. You should also connect a high-current ground wire (earth cable) from the power supply earth terminal to the earth point on the cryostat. A tapped hole and bolt will be provided, identified by the international earthing symbol.

Some cryomagnetic systems are supplied with “demountable” current leads. These may be removed from the cryostat after the magnet is energised and put into persistent mode. The magnet power supply may then be disconnected. It is essential that a special “shorting plug” and “baffle stick” are then fitted in place of the demountable lead. Your Installation Manual or Operator’s Handbook will explain how to do this. The shorting plug (a) helps to protect the magnet and (b) keeps the cold electrical connectors free from ice.

In order to ground the system when the power supply is disconnected you must connect a high-current earthing cable from a suitable laboratory earth terminal to the earth point on the cryostat, identified as above. Refer to your installation manual for details. For systems with demountable leads you should always make a record of the operating current and polarity and keep this with the system for reference.

The term “demountable” is sometimes used to refer to a bolted connection between the superconducting leads and the magnet. In the context of this document these should be viewed as “fixed” leads as the demountable element of the leads cannot be accessed while the system is cold.

Superconducting magnets can contain large amounts of stored energy, which if rapidly de-energised could give rise to large voltages. It is essential for safe operation that any vessel containing a magnet must be connected to protective ground via a sufficiently low-impedance link with a current capacity suitable to carry the peak de-energisation current.

If the user decides to disconnect either the ground link between the cryostat and the magnet power supply, or the magnet power supply mains supply connection, it is then against Oxford Instruments advice to energise the magnet. Should the magnet be energised in this state it is the responsibility of the user to ensure that the cryostat has an alternative ground of suitable current capacity and low enough impedance.

5.5 Summary of electrical hazards from the magnet

- Do not run the magnet without the protection circuit connected.
- Do not modify the protection circuit.
- Ground all equipment, including the cryostat and electronics.
- If the magnet is at field and has fixed leads then do not disconnect the magnet power supply.
- Where present, fit the insulating rubber cover over the cryostat magnet current lead terminals.
- If the leads are demountable follow instructions in you operator’s handbook that describe their removal and subsequent fitting of the shorting plug and baffle stick.

5.6 Magnet current leads and connections

Typically superconducting magnets will be operating at currents of the order of hundreds of amps. As power dissipation in resistive components scales with the square of the current, the resistance of the current leads and associated joints needs some special attention.

The current leads will usually have joints at the back of the power supply and at the top of the cryostat. The condition and detail of these joints will be important in determining how much power, and therefore how much heat is generated, at the joint. For most of the magnet systems supplied by Oxford Instruments, the current lead joints at the back of the power supply (or power supply rack) will consist of crimped eye’s terminating the Hypalon insulated leads bolted to plated copper busbars, typically using brass M8 bolts.

The bolts are intended to be the mechanical clamp of the joint and are not intended to be a primary current path. It is *essential* that the current lead crimp terminal is in contact with the busbar so that current can flow between the busbar and the current lead with only a small proportion of the current flowing through the more resistive brass bolt.

Fig. 5.1 shows how the current leads should be connected. The link-bar, linking 2 power supply modules, is on the opposite side of the busbar to the current lead termination eye. This means that they are both in good direct contact to the busbar. The M8 bolt passes through the items making the joint in the correct sequence:

1. An M8 brass washer
2. The slotted link-bar
3. The power supply busbar
4. The current leads termination eye
5. A second M8 brass washer
6. The M8 earth tag eye (not shown)
7. An M8 spring washer
8. The M8 brass nut

The M8 bolt should be tightened to a torque of 9 Nm. *Always use 2 spanners or wrenches* to avoid stressing the power supply busbar.

Fig. 5.2a shows how **not** to connect the current leads. In this example there is an M8 brass nut, M8 spring washer, M8 washer and the earth tag M8 eye between the current lead termination eye and the power supply busbar. This will create a significantly resistive joint which will get very hot in normal operation. This heat will be conducted into the power supply via the copper busbar and could cause the over-temperature interlocks to trip. Alteratively damage could occur from overheating the current leads.

Fig. 5.2b shows another example of how **not** to connect the current leads. In this example there is only the M8 earth tag eye between the current lead termination eye and the power supply busbar, but even this will create an additional barrier to current flow. As a result a greater proportion of current will flow through the brass bolt and the joint will dissipate heat and get hot, even in normal operation.

5.7 Connecting multiple units (mercury ips)

Some of the rack mounted Mercury power supply systems may require connecting the current outputs of more than 2 units. It is important to consider that the link-bars are only designed for 70A. The convention, therefore, is to connect 2 units together with link-bars to form a pair. Then to connect the pairs either with short link cables pair-to-pair, or by link cables connecting each pair to the rear high-current paddles.

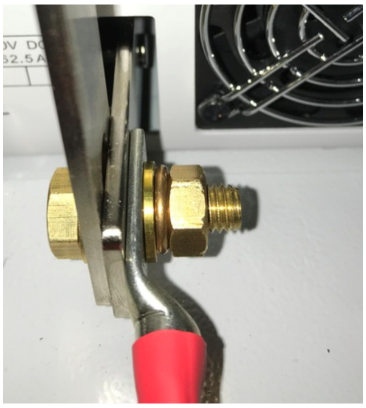


Fig. 5.1: Correct connection of magnet current leads to a power supply. The crimp connector on the magnet current lead is in direct contact with the power supply busbar, with the washer, spring washer and nut on the opposite side.

(a) (b)

Fig. 5.2: Two examples of magnet current leads **incorrectly** connected to a magnet power supply. In both instances the magnet current lead crimp connector is not in direct contact with the power supply busbar.

6 Lifting and transporting heavy equipment

Some cryogenic systems are so heavy that they can only be lifted by a large capacity crane, but nevertheless may contain very delicate components. If you choose the right way to lift the system you will be able to:

- Ensure your own safety
- Avoid damaging fragile equipment.

All service engineers from Oxford Instruments will require evidence of certification before any lifting equipment is used. Additionally, note that:

- You must determine local laws and regulations relating to lifting and follow them.
- Anyone lifting heavy equipment should be properly trained; these notes are not a substitute for proper training.

Systems should be lifted only using the lifting points provided. You must never lift by putting a chain or lifting strap underneath the system as special skills are required to do this safely.

> Use only lifting equipment that has been formally certified as safe, according to your local regulations.

6.1 Lifting points

All Oxford Instruments equipment that is too heavy to lift by hand will be fitted with suitable lifting points which are designed to carry the weight of the system safely. They will be positioned so that the system will stay vertical when it is lifted. The only way to lift systems is to use all the lifting points provided.

Fig. 6.1a shows lifting “lugs” that are welded directly to the outer vessel of the system. There will typically be four lugs and all should be used. Fig. 6.1b shows one (of four) “collar” eyebolts screwed into a system top plate. Smaller systems or sub-systems may have a single eyebolt located vertically above the centre of gravity.

Pairs of collar eyebolts must be oriented correctly as shown in Fig. 6.2.The plane of the eyes of a pair of collar eyebolts should be within ±5° of the plane containing the centre of gravity and the axes of the eyebolts.

Collar eyebolts have a rated SWL for axial lifting, which also applies if the angle α is less than 30°. If the angle is greater than 30° the SWL must be reduced by the appropriate factor (for details see BS EN ISO 3266:2010+A1:2015 or the local equivalent).

You should always refer to the Operator’s Handbook in case your system has special lifting requirements.

6.2 Lifting equipment with an overhead crane

Before you use the crane:

- Check that the safe working load (SWL) of the crane will not be exceeded.

- Check that all lifting equipment has been tested, that it is properly certified and that you do not exceed its SWL.
- Always use closed shackles rather than open hooks.
- Confirm that the magnet (if any) is at zero field and the system is empty of cryogens.

Lift your cryostat safely using appropriate and certified lifting equipment. When you are using a crane:

- Make sure that no one is allowed underneath an unsupported load.
- Always stand clear of the load in case the crane or lifting straps fail.
- Lift the system slightly clear of the ground and check it for balance and stability before lifting it higher; don't allow the load to swing.
- Avoid sudden movements which would impose a high shock load.
- Make sure that the lifting cable remains vertical so that the load cannot slide sideways as one side leaves the floor before the other.

Never work underneath an unsupported system hanging from a crane. If the system falls you may be killed.

6.3 Transporting systems safely

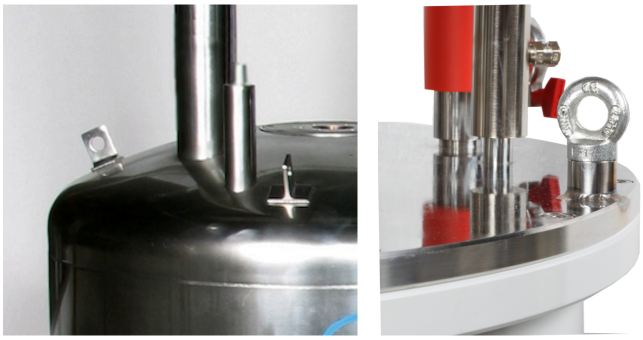
Most systems can be transported safely on a pallet using a trolley or fork lift. However, remember that:

- Some tall systems have a high centre of gravity and can fall over easily
- Systems should be moved gently because they are fragile
- Systems should always remain vertical.

Care should be taken if moving the system over longer distances (for example to another laboratory). Please contact Oxford Instruments for advice on how best to transport your system.

6.4 Maintenance

Have the eyebolts, lifting points and all other lifting equipment regularly inspected by a competent person, who will check that they are free from cracks, distortion, and any other defects.



(a) (b)

Fig. 6.1: Lifting lugs and eyebolts as fitted to Oxford Instruments systems

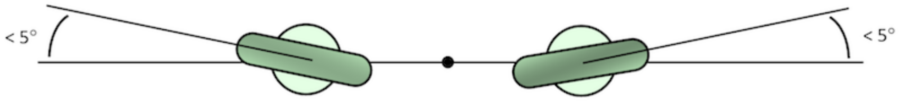


Fig. 6.2: The plane of the eyes of collar eyebolts should be within $\pm 5^{\circ}$ of the plane containing the centre of gravity and the axes of the eyebolts.

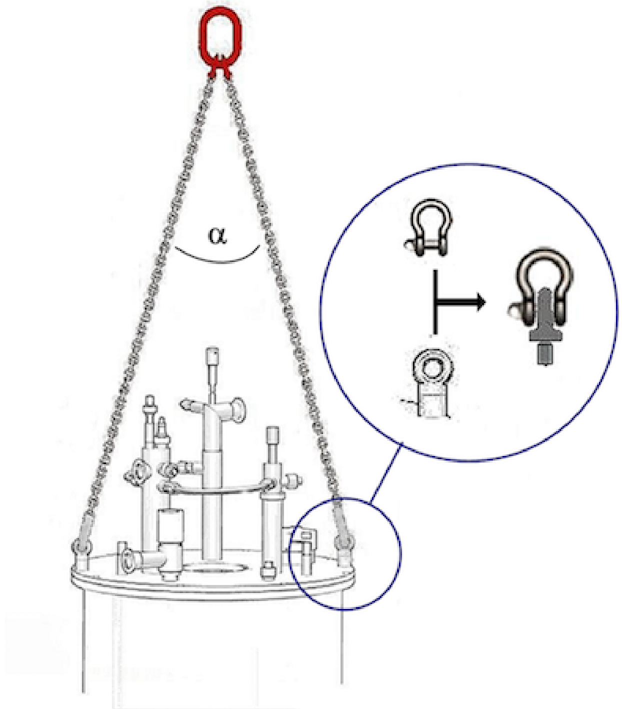


Fig. 6.3: Lift your cryostat safely using appropriate and certified lifting equipment. See Sec. 6.1 for details.

7 Poisons and hazardous substances

Certain Oxford Instruments systems contain materials that are poisonous or hazardous in other ways. For instance, some window materials are poisonous. All may be handled safely if the recommended procedures are followed carefully.

Consider identifying a special working area and monitor it for contamination regularly.

Hazardous materials will be supplied with suitable documentation to warn you of potential hazards. The (British) Control of Substances Hazardous to Health (COSHH) regulations require that this information is easily available to anyone working with these materials and the COSHH form for a material is often the best way to give you this information.

Before disposing of this equipment, it is important to check with the appropriate local organisations to obtain advice on local rules and regulations about disposal and recycling. You **must** contact Oxford Instruments Customer Support (giving full product details) before any disposal begins.

7.1 Radioactive Sources – Cobalt-60

This safety information is specific to Cobalt-60 sources supplied by Oxford Instruments. If you are using any other source then additional precautions may have to be taken.

You must determine local laws and regulations relating to Cobalt-60 sources and follow them.

Radioactive Cobalt-60 sources (^{60}Co) are sometimes used as thermometers on dilution refrigerator systems operating at temperatures below 100 mK. Sources supplied by Oxford Instruments normally have an activity of less than 185 kBq. If a source is to be supplied to you then details of the source (isotope, activity, form, emitter type etc.) and specific safety information will be sent to you.

However some general safety precautions are repeated here:

- The Cobalt-60 crystals should only be handled by trained personnel.
- The crystal should always be kept in its lead container or transport packaging until it is required. If you have a local radiation store, store it there.
- Once removed from its packaging it must not be left unattended.
- Do not handle with your bare hands. Always use plastic non-powdered gloves and stainless steel tweezers. Work smartly with the source but do not rush unnecessarily.
- Avoid direct body contact with the crystal. If contact takes place wash hands and areas of contact with soap and water immediately.
- Do not drop or cut the crystal.
- Do not stare directly at the source as far as reasonably practicable as the eyes are particularly sensitive to ionising radiation. Keep the eye to crystal separation greater than 30 cm and do not focus directly on the crystal for any longer than is necessary to mount it.

- Do not use naked flames in the vicinity of the source.
- Wash your hands thoroughly with soap and water immediately after working with the source. If you have a radioactive source, local regulations may require you to register it. You should ensure that you comply with all local, national and international rules and regulations.

7.2 Rare earth materials – cryo-coolers

In order to efficiently operate cryo-coolers require regenerator materials possessing high heat capacities. This has resulted in the use of materials containing rare earth metals in the construction of some cryo-coolers. As such care should be taken when decommissioning a system that has a cryo-cooler installed.

Oxford Instruments should be contacted for advice on how best to dispose of the system.

8 Appendices

8.1 Properties of helium and nitrogen

Properties	Helium-4	Nitrogen
Chemical formula	^4He	N_2
Molecular weight	4	28
Normal boiling point (NBP) [K]	4.22	77.3
Critical point [K]	5.2	126.6
Liquid density at NBP [$g \cdot cm^{-3}$]	0.125	0.807
Ratio of gas at 273K (0°C),1 atm. : liquid at NBP	750	694
Latent heat of vapourisation at NBP [$J \cdot g^{-1}$]	20.9	198
Liquid at NBP boiled off by 1 watt [l/hour]	1.38	0.0225
Gas density relative to dry air at 288K (15°C) and 1 bar	0.14	0.98
Gas density at 273 K (0°C) and 0.101325 MPa	0.166	1.165
Fire/explosion hazard	No	No
Air liquefaction hazard	Yes	Yes
Asphyxiation hazard	Yes	Yes

8.2 Risk assessment

A "Risk Assessment" for an installed cryomagnetic system will consider and evaluate all the risks described in this booklet and implement mitigating procedures where necessary. You are responsible for the preparation of a Risk Assessment before any maintenance, inspection, modification or repair work is carried out; this is a formal requirement by engineers from Oxford Instruments.

If you require advice or assistance in the preparation of a Risk Assessment please contact Oxford Instruments Customer Support.

References

1. Richardson, R. N. *Cryogenics safety manual: A guide to good practice*. (British Cryoengineering Society, UK, 1998).

2. Equipment described in this booklet is available from Oxford Instruments at www.cryospares.com.

3. For site guidance and service work contact Oxford Instruments Customer Support at ServiceNSUK@oxinst.com.

4. Refer to www.mrisafety.com for an in-depth discussion and extensive references.

5. Council of the European Union. Procedure 1992/0449/COD. *Official Journal of the European Communities* (1994).

6. American Conference of Governmental Industrial Hygienists. *Threshold limit values for chemical substances and physical agents and biological exposure indices*. (1995).

7. International Commission on Non-Ionizing Radiation Protection. Guidelines on limits of exposure to static magnetic fields. *Health Physics* **66**, 100–106 (1994).

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9. McKinley, A. Restrictions on human exposure to static and time varying electromagnetic fields and radiation. *Doc. Nat. Radiol. Protect. Board* **4**, 5 (1993).

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SO Number:	
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