



Science and  
Technology  
Facilities Council

# Safe Use of Cryogenic Materials

STFC Safety Code No 3

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## Revisions

|     |   |               |
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# Safe Use of Cryogenic Materials

## 1 Purpose

Across the STFC cryogenics are widely used to cool equipment, for example, magnets and detectors, and to cool or store samples and materials. The hazards associated with the use of cryogenic liquids include:

- causing contact burns (by the liquid), frostbite or cold exposure (by the vapour);
- the ability to wick in woven materials, due to their low viscosity, making contact with the skin and entrapping cryogenic liquids within clothing;
- the potential for the liquid to rapidly convert to a large quantity of gas, which, especially in a confined space, can present a suffocation/asphyxiation or over pressurisation hazards; and
- they may be flammable and/or explosive.

This code is aimed at those using cryogenic materials and provides information regarding hazards, safe methods of work and provision of suitable personal protective equipment to reduce the risk of occupational exposure to cryogenics. Many of the safety precautions observed for compressed gases also apply to cryogenic liquids with the addition of extremely low temperatures and vapourisation.

While there is no specific cryogenic safety legislation it is addressed through related general safety requirements under:

- The Health and Safety at Work etc. Act, 1974
- The Management of Health and Safety at Work Regulations 1999
- The Provision and Use of Work Equipment Regulations 1998
- The Control of Substances Hazardous to Health (COSHH) Regulations 2002 (as amended)
- The Dangerous Substances and Explosive Atmospheres Regulations 2002 (DSEAR)
- Pressure Equipment Directive (PED) and the Pressure Systems (Safety) Regulations, 2000 (PSSR 2000);
- The Confined Spaces Regulations 1997; and
- Personal Protective Equipment at Work Regulations 1992.

## 2 Scope

This code applies to the use of all cryogenic materials such as; helium, nitrogen, hydrogen, methane, oxygen, neon and solid carbon dioxide. See Appendix 1 for hazard information.

Additional hazards arise when using flammable cryogenic materials such as hydrogen and methane where more stringent safety precautions are required to prevent fire and explosion; see STFC SHE Code 20 Controlling Explosive and Flammable Gases and Dusts.

The introduction of the Pressure Equipment Directive (PED) and the Pressure Systems (Safety) Regulations, 2000 (PSSR 2000) details the safety and certification requirements for of pressurised cryogenic storage vessels, see STFC [SHE Code 33: Safety of pressure and vacuum systems](#)

This code does not address the design of containers for liquefied gases or transfer lines and pipe work but does include the siting of standard commercially manufactured storage facilities, vessels

and equipment. However, in-house designed systems which are required to have suitable pressure relief systems must have all pressure relief valves registered for statutory inspection. Reference to STFC SHE Code 33 Safety of pressure and vacuum systems, where appropriate.

This Code addresses the specific hazards associated with cryogenic materials but does not address the many related hazards **especially the treatment of cryogenic cold burns (see Appendix 8 of Code 36 – Management and provision of first aid)**. These are addressed by separate STFC SHE Codes which include:

STFC SHE Code 1 Lone Working (under development);  
STFC SHE Code 11 Work in Confined Spaces;  
STFC SHE Code 12 Manual Handling;  
STFC SHE Code 20 Controlling explosive and flammable gases and dusts;  
STFC SHE Code 33 Safety of pressure and vacuum systems;  
STFC SHE Code 36 Management and provision of first aid; and  
STFC SHE Code 37 COSHH: safe use of chemicals / hazardous substances.

## 3 Definitions

### 3.1 Cryogenic

The temperature at which refrigeration ends and cryogenics begins is not well defined. The National Institute of Standards and Technology (NIST) in the US have suggested that cryogenic temperatures are defined as those below 93.15K (-180°C). However, in the context for STFC we should consider all low temperature liquified gases. This will include the most commonly used cryogenics within STFC: liquid helium and liquid nitrogen, and also cover liquified natural gas which is produced at a temperature of approximately -162°C, as well as solid carbon dioxide.

### 3.2 Cryogenic Dewar

Cryogenic storage Dewars are specialised flasks and can take several different forms e.g. open flasks with loose-fitting stoppers and self-pressurising storage tanks. All Dewars have walls constructed of two or more layers with either a high vacuum maintained between them to provide good thermal insulation, or where there is a need to avoid a vacuum barrier the space is filled with a material such as perlite or similar insulating material.

### 3.3 Expansion Ratio

This is the ratio of the volume of the cryogenic liquid from the boiling point to normal ambient temperature and atmospheric pressure. Example expansion ratios (gas volume from liquid phase) and relative densities of the gas compared to air at normal temperature and pressure are given below:

|                      | <b>Gas volume from liquid</b> | <b>Relative Density</b> |
|----------------------|-------------------------------|-------------------------|
| Liquid methane       | 1:627                         | 0.555                   |
| Liquid nitrogen      | 1:696                         | 0.967                   |
| Liquid helium        | 1:757                         | 0.138                   |
| Liquid hydrogen      | 1:851                         | 0.069                   |
| Liquid oxygen        | 1:860                         | 1.105                   |
| Liquid neon          | 1:1438                        | 0.697                   |
| Solid carbon dioxide | 1:554                         | 1.520                   |

It should be noted that the conditions of comparison are standard temperature and pressure. Freshly evaporated gaseous cryogens can stay below ambient temperature for a considerable

time. Nitrogen in particular will fill a room from the bottom up and displace the air upwards assuming there is minimal turbulence to the atmosphere in the environment of the room.

### **3.4 Cryogenic storage tank**

This is an assembly constructed of an inner vessel and an outer jacket for insulation purposes. The insulation space is normally under vacuum.

## **4 Responsibilities**

### **4.1 Directors shall:**

- 4.1.1 be aware of all cryogenic operations requiring oxygen depletion alarms.
- 4.1.2 ensure that their staff have sufficient competent resources to manage this hazard such that any identified control measures are implemented in line with this SHE code.

### **4.2 Line Managers (Group Leaders) and Supervisors shall:**

- 4.2.1 ensure that their staff and others within their area of responsibility are aware of the requirements of this SHE Code and consider these issues when using and storing cryogenic liquids.
- 4.2.2 ensure that a suitable and sufficient documented risk assessment is undertaken as required by SHE Code 6 Risk Management, is stored in Evotix Assure, and that all control measures identified are implemented through the establishment of documented operating instructions / procedures for handling cryogenic materials, including their movement and transport, see Appendices 1 and 2.
- 4.2.3 ensure that risk assessments include calculations for oxygen depletion, see Appendix 3, and decisions with respect to the installation of oxygen depletion monitors are recorded. Installed oxygen depletion monitors shall be regularly tested and maintained, according to the maintenance regime, and records kept.
- 4.2.4 with reference to the above, ensure that adequate ventilation is provided in areas where cryogenic liquids are used or stored.
- 4.2.5 ensure that the cryogenic storage tanks/equipment are inspected, tested and maintained according to a schedule based on risk assessment.
- 4.2.6 ensure that their staff and others using cryogenic materials in areas where they are responsible have been given information, instruction and training as to the hazards associated with these cryogens. This shall include the specific equipment to be employed, the precautions required, appropriate Personal Protective Equipment (PPE) and actions to take in the event of an emergency, specific guidance on responses to cryogenic emergencies can be found in Appendix 4.
- 4.2.7 ensure that the operator has suitable and sufficient supervision appropriate to his / her level of competence.
- 4.2.8 ensure a sufficient number of personnel, typically first aiders, are trained in the treatment of asphyxia and cold burns in the event of an incident, specific guidance on cryogenic first aid can be found in Appendix 4.
- 4.2.9 ensure that documented emergency procedures are in place in the event of a cryogenic liquid spillage.
- 4.2.10 ensure that all pressurised Dewars purchased, and valves used for pressure release on cryogenic systems, are registered for statutory inspection with the specific on-site group responsible for pressure systems.

**4.2.11** ensure that all monitors and alarms are calibrated and maintained in accordance with a planned schedule and records of these activities maintained.

**4.2.12** ensure that any vessel containing a cryogenic material is clearly marked with the name of the materials and the vessel shall only be used for that material, and that suitable hazard warning signs are affixed to the vessel.

### **4.3 Cryogen Users shall:**

**4.3.1** perform all work with cryogenic liquids in accordance with this code, local procedures / standard operating instructions for the equipment and good laboratory practices.

**4.3.2** not store cryogenic storage vessels in confined or restricted spaces for example corridors or stairwells or accompany vessels in lifts.

**4.3.3** only undertake work involving cryogenic materials after completing defined training for the safe use of cryogenic materials and instruction in the use of the equipment employed, see Appendix 5.

**4.3.4** adhere to the controls and employ the PPE, defined in the risk assessment for the work they undertake with cryogenic materials, and ensure that warning devices, such as portable oxygen monitors, where required, are used properly.

**4.3.5** only use Dewars for the specific liquids for which they were designed.

### **4.4 SHE Group shall:**

**4.4.1** provide advice and guidance on the implementation of this code and maintain a list of competent advisors for Cryogenic Risk Assessment.

## **5 References**

British Compressed Gas Association (BCGA) Codes of Practice:

CP4 Industrial Gas Cylinder Manifolds and Gas Distribution Pipework (excluding acetylene) Revision 3: 2005.

CP23 Application of the Pressure Systems Safety Regulations 2000 to Industrial and Medical Pressure Systems Installed at User Premises Revision 1: 2002.

CP25 Revalidation of Cryogenic Static Storage Tanks Revision 2: 2004.

CP27 Transportable Vacuum Insulated Containers of not more than 1,000 litres volume. Revision 1. 2004.

CP30 rev 1 2008 The safe use of liquid nitrogen Dewars up to 50 litres.

CP36 Bulk Cryogenic liquid storage at user's premises Revision 1. 2011.

Guidance Note GN11 Reduced oxygen atmospheres. The management of risks associated with reduced oxygen atmospheres resulting from the use of gases in the workplace 2007.

Cryogenics Safety Manual, a Guide to Good Practice. Safety Panel, British Cryogenics Council, 1982.

MRC Standards for Liquid Nitrogen Supply.

European Industrial Gases Association (EIGA) publications.

Useful list of websites relating to cryogenic materials:

<http://www.cryox.co.uk/links.html>

# Appendix 1. Cryogen and cryogenic hazard information for consideration when undertaking risk assessments

## A1.1 Cryogenic Liquids

The low viscosity of cryogenic liquids means that they will penetrate woven or other porous materials much faster than for example water.

## A1.2 Solid Carbon Dioxide

Solid carbon dioxide, either dry or in a solvent, is used as a refrigerant for trapping water vapour, oil vapour etc, in systems under vacuum and for other purposes such as the cooling of metal parts to cause contraction where its vaporising temperature, 194.5 K (-78.50 °C), is sufficiently low.

## A1.3 Liquid Nitrogen

Liquid nitrogen is used as a refrigerant for trapping water vapour, oil vapour etc, in systems under vacuum, and for other purposes such as the cooling of metal parts to cause contraction. Its normal boiling point is 77K (-196°C). Ice which is formed from frozen atmospheric water vapour can form on exposed liquid nitrogen systems.

The vacuum-jacketed vessels commonly used to store and transport liquid nitrogen, can typically lose upwards of 0.5% per day by evaporation.

The evaporation of one litre of liquid nitrogen produces some 696 litres of gas at standard temperature and pressure.

## A1.4 Liquid Helium

Liquid helium is used for cooling superconducting materials and for experiments involving very low temperatures. Its normal boiling point is 4.2K (-269°C). An additional common danger associated with using liquid helium can be the formation of liquid oxygen in localised areas, see Section 5.9.

The vacuum-jacketed low-pressure vessels commonly used to store and transport liquid helium lose upwards of 1% per day by evaporation.

The evaporation of 1 litre of liquid helium produces some 757 litres of gas at normal temperature and pressure.

The low temperature of liquid helium can solidify any other gas. Solidified gases and liquids allowed to form and collect can plug pressure-relief passages and foul relief valves. Users should endeavour to store and handle liquid helium under positive pressure and in closed systems to prevent the infiltration and solidification of air or other gases. Where this is not possible there are measures which can be taken e.g. use of a helium bottle or installation of a small heater in the helium, to maintain a positive pressure. The pressure relief systems installed should be positioned in such a way that they will not be blocked by freezing of any other gases leaking into the system. The pressure relief systems must be well maintained.

## A1.5 Liquid Hydrogen

Liquid hydrogen at 20K is used as one of the moderators for the ISIS neutron spallation source. Its normal boiling point is -252.7°C.

Condensed air could result in oxygen enrichment and explosive conditions near a liquid hydrogen storage system. Further guidance on explosives atmospheres can be found in the [Controlling Explosive and Flammable Gases and Dusts](#) code (Safety Code 20).

Although it is non-corrosive hydrogen can cause some metals to undergo a process of hydrogen embrittlement. This is a process by which various metals become brittle and fracture following exposure to hydrogen, and particularly important where high-strength steels have been used because of their susceptibility to hydrogen.

If a liquid hydrogen leak or spill occurs, a hydrogen cloud will rise and could flow horizontally for some distance or even downward, depending on the terrain and weather conditions. Hydrogen is a very small molecule with low viscosity, and therefore prone to leakage. In a confined space, leaking hydrogen can accumulate and reach a flammable concentration. Hydrogen's flammability range is between 4% and 75% in air, which is very wide compared to other gases/vapours and the energy required to initiate combustion is much lower than for other common fuels. Hydrogen combustion is more rapid than combustion of other fuels but produces little overpressure in an open environment. In confined spaces hydrogen ignition can result in flame acceleration and generation of high pressures capable of exploding buildings and ejecting shrapnel.

### **A1.6 Liquid Oxygen**

Liquid oxygen is not currently used on STFC facilities for any cryogenic purposes. Its normal boiling point is 90K (-183°C).

Organic materials such as oil, grease and hydrocarbons can react explosively, while other materials that are usually considered non-combustible may burn in contact with liquid oxygen.

### **A1.7 Liquid Methane**

Liquid methane at 100K is used as one of the moderators for the ISIS neutron spallation source. Its normal boiling point is -161.5°C.

It has no odour and is therefore difficult to detect a leak. An odorant could be added but at cryogenic temperatures it is extremely difficult or impossible to add an odorant. However, it may also be undesirable to add the odorant impurity to the system.

The flammability range for liquefied methane is between 5% and 15%.

### **A1.8 Ice Formation**

Accidental air leakage into a liquid cryogen storage vessel (e.g., from inadequate purging) will result in the introduction of moisture. The water may form ice plugs in the neck of open Dewars and cause a build-up of pressure. As the pressure rises within the Dewar, the ice plug may be expelled at high velocity or in extreme cases the pressure may build up sufficiently to rupture the vessel.

Should an ice plug be found, extreme caution should be exercised and the area immediately vacated since the pressure built up within the system is unknown. Clearing ice-blockages is a specialist job and under normal circumstances only carried out by someone with the appropriate knowledge and training. Personnel dealing with the incident should do so from a safe position. Methods of clearing ice-blockages can vary but a hole can be pierced through the ice with a heated metal tube to release the pressure, since this will allow the pressure to be released without the tube becoming a projectile.

Ensure that the Dewar is examined by the manufacturer or a competent person before returning it to service.

Ice plugs can be prevented by diligent use of the correct Dewar stopper.

### **A1.9 Pressure Build-up**

Continuous evaporation generates a gaseous atmosphere and an increase in pressure inside a liquid cryogen storage vessel, which, if not properly controlled and released by suitable measures, can result in significant build-up of pressure. A pressure relief valve (PRV) of suitable specification, which has been registered for statutory inspection, should be used to prevent over pressurisation of the vessel or system.

### **A1.10 Oxygen Enrichment**

Operators should be trained to recognise that the low temperatures of liquid nitrogen and helium, having a lower boiling point than oxygen, can cause oxygen to condense out of atmospheric air. This can occur around cold pipework, valves and in open Dewars. This oxygen enrichment may result in increased flammability and explosion risk. Oxygen enriched liquid must not be allowed to come into contact with oils or grease, or flammable materials as spontaneous combustion can occur. These contact areas should be cleaned to oxygen clean standards. The selection of appropriate insulating materials should be made based on the possibility of oxygen enrichment.

### **A1.11 Manual Handling / Ergonomic Issues**

The manual movement of storage Dewars, storage tanks and equipment can give rise to manual handling injuries to back, neck, hands and fingers and proper lifting and moving procedures should be adopted.

The layout of the work area should be ergonomically designed to ensure a safe system of working and to minimise or eliminate manual handling requirements as far as is reasonably practicable. Consideration should be given to factors such as:

- Static storage tanks/equipment and piped liquid/gas supplies should be used in preference to mobile storage tanks/equipment wherever possible;
- Where mobile storage tanks/equipment are used, then it is essential that the handles and/or wheels on the storage tanks/equipment/trolleys are secure, and that all wheels turn freely. Wheels and axles employed on such equipment should, be subject to routine inspection and maintenance;
- Where large Dewars are used and maneuvered into position using the mobility handles, it is important that these handles should NOT be used for lifting the Dewar for which they are specifically NOT designed for this purpose.
- Wheels or platforms should be of adequate construction, and tested to withstand the weight of a full tank;
- To avoid impact damage to the tank, the base should be extended where possible to provide bumpers or similar protection; and
- If any manual handling is required, this must be subject to an appropriate risk assessment to ensure it is carried out safely without any risk of personal injury (e.g. back strains, hernias, sprains, cuts or fractures).
- Some Dewars should not be tipped or stored on their sides since they are not sufficiently robust to withstand this without damage being caused. There may be no indication of this on the Dewar.

### **A1.12 Cold Contact Burns**

Liquid or low-temperature gas from any of the specified cryogenic materials will produce effects on the skin similar to a burn. This will vary with temperature and exposure time. Cryogenic fluids that are allowed to come into contact with human skin can cause severe damage to living tissue. Similarly, contact with uninsulated pipes etc. will cause contact burns and may result in the skin freezing to the uninsulated pipework etc.

Similarly the gases released as cryogenic liquids vapourise can permanently damage delicate tissues e.g. the eyes can be damaged by an exposure to cold gases too brief to affect the skin.

Never allow any unprotected part of your body to touch un-insulated pipes or vessels that contain liquefied gases since the extreme cold may cause the point of contact to stick to the metal by virtue of the freezing of the available moisture and tear the flesh when you attempt to separate the contact.

### **A1.13 Frostbite and Exposure**

Individuals not suitably protected against low ambient temperatures often associated with cryogenic handling may suffer cold exposure, slowing their reactions and capabilities and could lead to hypothermia. Continued exposure is likely to result in frostbite.

### **A1.14 Physiological Effects**

#### Effect of Cold on Lungs

Short exposure may produce discomfort, whereas prolonged exposure and inhalation of vapour or cold gas, whether respirable or not, can produce serious lung effects.

### **A1.15 Toxicity**

Most liquefied gases have low toxicity; however, in high concentration may cause nausea or dizziness. Even prolonged breathing of pure oxygen can result in harmful physiological effects.

### **A1.16 Asphyxiation**

Liquid nitrogen is colourless, odourless, exists at  $-196^{\circ}\text{C}$  (at atmospheric pressure), and is widely employed in cold storage applications. Virtually all common liquefied gases have no odour, and so cannot be detected by smell, exceptions being ethane and ethylene.

An increase in temperature through spillage, release or even simple exposure to surrounding air causes the liquid to boil and the cryogen to return to the gaseous phase. A large expansion in volume accompanies this change in physical state; one litre of liquid producing hundreds of litres of gas (see Section 3.3 for specific gases). This can result in a problem of asphyxiation through displacement of atmospheric oxygen, and will be applicable to cryogenic liquids in general.

Any reduction in the normal content of the oxygen in the breathing atmosphere must be considered a hazard. In sudden asphyxia, such as that from inhalation of pure nitrogen, unconsciousness is immediate. Other degrees of asphyxia will occur when the oxygen content of the working environment is less than 20.9% by volume. Effects from oxygen deficiency become noticeable at levels below 18% and sudden death may occur at 6% oxygen content by volume. This decrease in oxygen content can be caused by a failure / leak of the cryogenic vessel or transfer line and subsequent vaporisation of the cryogen.

Asphyxia symptoms for low oxygen levels:

|             |   |
|-------------|---|
| 18% - 19.5% | May affect physical and intellectual performance without person's knowledge.  |
| 15% - 18%   | Decreased ability to work strenuously. May impair co-ordination and may induce symptoms in persons with coronary, pulmonary, or circulatory problems. |
| 12% - 15%   | Respiration deeper, increased pulse rate, and impaired co-ordination, perception and judgement.   |
| 10% - 12%   | Further increase in rate and depth of respiration, further increase in pulse rate, performance failure, giddiness, poor judgement, blue lips.         |
| 8% - 10%    | Mental failure, nausea, vomiting, fainting, ashen face, blue lips.  |
| 6% - 8%     | Loss of consciousness within a few minutes, resuscitation possible if carried out immediately.  |
| 0% - 6%     | Loss of consciousness almost immediate, death ensues, brain damage, even if rescued.  |

Oxygen depletion sensors are typically set to register an alarm at 19.5% oxygen.

### **A1.17 Explosion - Pressure**

Heat flux into the cryogen from the environment will vaporise the liquid and potentially cause pressure build up in cryogenic containment vessels and transfer lines. Adequate pressure relief must be provided to all parts of a system to permit this routine out-gassing and prevent an explosion.

Liquified gases are usually stored at or near their boiling points, and hence there is always some gas present in the container. Due to the large expansion ratio of a cryogenic liquid when vapourised a build up of high pressure can occur when the liquid evaporates, and as such there should be sufficient pressure relief valves in all lines between valves and between shut-off valves and downstream equipment. The evaporation rate will depend on the fluid, storage container design and environmental conditions, but the container capacity must include an allowance for the evaporation of the liquid into the gaseous state. The pressure relief devices should be maintained and checked regularly for leaks or damage.

Containers of cryogenic liquids must never be closed so that they cannot vent. Where a special vented stopper or venting tube is used the vent must be checked regularly to ensure it has not plugged with ice formed from water vapour condensed from the air.

Pressure relief valves must be installed on all vessels and piping which contain cryogenic liquids or might under some failure conditions contain the cryogenic liquid e.g. cryostat vacuum vessels. The maximum safe working pressure for all piping and vessels should be determined and used to identify the flow requirements for the relief valve in a worst-case failure scenario e.g. failure of a cryostat insulating vacuum to atmosphere; trapping of a cryogenic liquid due to valve failure or operator error etc. Selection of a pressure relief valve should not be just on the pressure value but also allow adequate flow rate to prevent further pressure build up occurring.

All system vents must be directed away from personnel or designated working areas.

### **A1.18 Explosion - Chemical**

Cryogenic fluids with a boiling point below that of liquid oxygen are able to condense oxygen from the atmosphere. Repeated replenishment of the system can thereby cause oxygen to accumulate as an unwanted contaminant. Similar oxygen enrichment may occur where condensed air accumulates on the exterior of cryogenic piping. Violent reactions, e.g. rapid combustion or explosion, may occur if the materials which make contact with the oxygen are combustible.

Helium, Hydrogen and Nitrogen will all condense air creating conditions for a potential explosion hazard by causing oxygen entrapment in unsuspected areas. In addition, extremely cold surfaces are also capable of condensing oxygen from the atmosphere.

An atmosphere that contains more than 21% of oxygen by volume creates a dangerous fire hazard. Higher concentrations of oxygen will increase both the chance of a fire and its intensity and can involve materials normally regarded as being non-flammable. There should be no sources of ignition in areas where oxygen enrichment is likely to occur.

If there is any use of cryogenic oxygen itself this would fall within the scope of DSEAR and further guidance can be found in STFC [SHE Code 20: Controlling explosive and flammable gases and dusts](#).

### **A1.19 Explosion - Radiation**

Under irradiation conditions, such as in a proton beam, liquid nitrogen can explode when the cryogen is warmed. This is thought to be due to reactions with ozone formed from trace amounts of oxygen in the cryogen.

### **A1.20 Embrittlement of Materials**

When materials are cooled the Young's modulus of the material will typically increase by around 20% down to liquid helium temperatures. This will increase the material's strength and stiffness, but also the brittleness which could also cause failure of parts due to a change in this property.

A wide variety of materials become brittle at very low temperatures associated with cryogenic liquids, e.g. plastics, rubber, Teflon and carbon steels; and as such are unsuitable for use in such environments. However, there are a number of suitable metals and these include austenitic stainless steels, 9% nickel steel, copper and its alloys and aluminium alloys. Suitable plastics are reinforced plastics such as G-10 and G-11 (glass-epoxy composites).

### **A1.21 Fire**

Think about the method of extinguishing fires in relation to the cryogen etc. e.g. CO<sub>2</sub> extinguishers can cause static discharge of sufficient magnitude to ignite some gas mixtures.

### **A1.22 Combustibles Storage**

Flammable substances and combustible materials should not be stored or allowed to accumulate in the vicinity of cryogenic liquid installations, and is particularly important where cryogenic oxygen is being stored due to issues of spontaneous combustion.

### **A1.23 Material Stresses**

Not all materials will respond well to contact with liquid cryogenics or accidental cryogen spillages resulting in subsequent failure of the component's integrity. The sudden contraction of materials that have become exposed to the cryogenics could be sufficiently large to cause high internal stresses resulting in the failure of critical parts.

## Appendix 2. Risk Assessment

A risk assessment will be required for the initial design of facilities, processes and equipment handling cryogenic materials. A subsequent risk assessment will then be required for their use. General guidance on the Risk Assessment process can be found in STFC SHE Code 6 Risk Management.

A risk assessment will be required for handling cryogenic materials at the initial design and for their subsequent use. General guidance on the Risk Assessment process can be found in STFC SHE Code 6 Risk Management. Factors that should be taken into account include:

- The cryogen being used.
- Maximum volume of cryogen required to be held at point of use.
- Maximum volume of cryogen required to be held in storage vessel.
- Maximum number of samples to be stored.
- Type and quantity of sample storage vessels to be used (including back-up systems).
- Operations to be performed at point of use.
- Lone working.
- Location or options for location (if there is more than one possibility).
- Manual handling and ergonomic considerations.
- Maintenance of plant.
- Confined spaces.
- Restricted access.

Suitable control measures should be identified as part of this risk assessment and should include controls for:

- Design and layout of the room and equipment.
- Ventilation requirements.
- Oxygen depletion monitors and alarms.
- Safe systems of work.

### A2.1 Experimental Design (Large Scale)

The siting and installation of any storage tank, associated transfer lines and equipment should be carried out by a trained and competent specialist contractor, taking into account the findings of the risk assessment. It is the responsibility of the person carrying out the siting and installation of the storage tank to ensure that the work is carried out in accordance with the appropriate regulations / codes of practice, and that all parts / components used are safe and suitable for the intended application.

The British Compressed Gases Association (BCGA) Codes of Practice provide guidance and a framework to assist with fulfilling the legal duties and achieving best practice (see references in Section 5).

External storage tanks must have sufficient clearance on all sides to comply with BCGA CP36.

The design of piped gas delivery systems for cryogenic liquids needs to conform to the requirements of the Pressure Systems Safety Regulations 2000 (as amended) and internal STFC guidance and is dealt with in Safety Code 33.

## **A2.2 Experimental Design (Small Scale)**

Small scale laboratory use of cryogenics should only be conducted in well-ventilated areas and / or where oxygen depletion sensors are installed. The control unit for the detector should be installed outside the area where the cryogen(s) and detector are situated such that it can be checked before entering the area. Only the minimum amount of cryogen should be used, taking into account the calculations in Appendix 3 which determine the maximum cryogen quantities permitted such that the percentage content of oxygen of the room would not be sufficiently reduced. Excess quantities should not be stored in the laboratory.

Ensure ergonomic design of the work area to minimise or eliminate movement of the tanks/equipment and manual handling requirements.

## **A2.3 Installation**

Ensure a safe location for the cryogenic storage tanks/equipment is provided, and where possible site storage tanks/cylinders outside buildings and avoiding confined spaces and the liquid cryogen piped into the workplace.

STFC recommendation is that nitrogen storage tanks of 500 litres capacity and above must be located outside, in a location acceptable to the supplier, and designated a “No Parking” area.

Where necessary, the storage tanks/cylinders should be secured against accidental impact damage by vehicles using the installation of suitable safety rails or bollards.

Careful consideration should be given to external venting of all gas exhausts/pressure relief lines from the cryogenic storage tanks/equipment; for example where the gases are flammable they should not be vented near ignition sources, or where the relative density is greater than air venting in proximity to confined spaces should be avoided.

The specification, installation, use, inspection and maintenance of the cryogenic storage tanks/equipment should be correct for the usage.

## **A2.4 Ventilation**

The requirement to provide adequate ventilation to avoid or minimise the accumulation of asphyxiant gases and atmospheres in the workplace is addressed in section 4.2.3. This should take into account the following:

For rooms above ground level with no special ventilation openings, natural ventilation will provide typically one air change per hour. With well sealed windows (e.g. double glazing), this will be less. Basement rooms only average 0.4 air changes per hour.

For general handling of transportable cryogenic vessels in locations at or above ground level, natural ventilation is generally sufficient, provided the room is large enough, or the outdoor area

is not enclosed by walls.

An indoor location should have ventilation openings, e.g. door and wall vents, with a total area of at least 1% of the floor area. The openings should be at low level (e.g., 0.5 to 1.0m from floor) and be positioned diagonally across the room.

Cold nitrogen gas is heavier than air and will accumulate at low level. Where possible liquid nitrogen should not be handled in basement rooms, rooms with ventilation at high level only, and rooms where the gas can be trapped in pits or gullies.

Helium is lighter than air and as such will rise and fill a room from the ceiling downwards. Providing an enclosed space is not completely full of helium a person suffering from helium asphyxiation will tend to collapse to the floor where oxygen levels will be higher and the likelihood of survival greater.

Either natural ventilation (air vents, windows etc.) or forced ventilation (mechanical extraction system) may be used depending upon the number of air changes needed per hour; a forced ventilation system is usually necessary for more than 2 air changes per hour.

Consideration must be given to the use of oxygen level meters and alarm systems in areas where the ventilation is poor.

## **A2.5 Fixed Oxygen Monitors/Alarms**

Additional control measures may also be necessary to warn of the development of an unsafe atmosphere and / or assist in ensuring people do not remain in an unsafe area where an asphyxiating atmosphere may exist. These may include any of the following:

- Oxygen depletion monitors must be fitted wherever the risk assessment indicates that oxygen levels may be depleted to less than 19.5%.
- The monitors are installed to test the atmosphere in the workplace e.g. laboratory before entry, and give clear warning during occupancy of that area.
- The alarm should activate at 19.5% and produce a visible and audible alarm signal.
- Any activation of the alarm should give rise to concern and immediate action and any person inside the room should isolate the source of asphyxiant if safe to do so and leave immediately.
- Where the risk assessment indicates that oxygen levels could be depleted to below 18.5%, there should be two levels of alarm, at 19.5% and 18.5%.
- The second alarm should produce a distinct visual and audible alarm signal.
- Fixed monitors are preferable to personal monitors as they protect all personnel in the workplace, rather than just an individual person, and the monitors should normally be positioned near to the potential asphyxiant exposure points, at a height in the order of 1m above the ground.
- External alarm repeaters linked to the fixed monitors should also be installed where necessary, and sited so that they are clearly visible and audible to personnel before entering the workplace.
- The oxygen depletion monitors may also be used to initiate any of the following actions

on reaching the second action level of 18.5%:

- To activate a solenoid operated safety shut-off valve to isolate the liquid nitrogen supply from the storage tank.
- To raise the air extraction rate (typically from 10 - 15 air changes per hour to 20 - 30 air changes per hour).
- To activate a door interlock system to prevent access (but allow escape). For rescue purposes override facilities should be provided.
- Where a mechanical extraction system is used and if the ventilation fails for any reason, the system can also be linked to a solenoid operated safety shut-off valve system to isolate the liquid supply from the storage tank.

Restoration of normal operation following an emergency shut-down, following oxygen depletion to below 18.5%, should only be carried out by a competent person.

## **A2.6 Safe Systems of Work**

The following safe working practices should be considered and incorporated into a suitable written safe system of work for use with cryogenics:

Safe working practices:

- Any vessel containing a cryogenic material must be clearly marked with the name of the materials and the vessel shall only be used for that material.
- If cryogenic vessels are to be transported to another area the route should be assessed prior to movement; and the assessment should include: obstructions and clutter; floor surfaces, lifts, stairs, kerbs, ramps and steep inclines, moving through populated areas, suitable rest stops for those moving vessels etc.
- Wherever possible avoid transporting liquid cryogen containers up or down slopes, steps or stairways;
- Wherever possible avoid transporting liquid cryogen containers through densely populated areas;
- Wherever possible avoid transporting liquid cryogen containers over uneven ground or in areas where there is poor lighting or any risk of slips, trips or falls;
- Wherever possible avoid transporting liquid cryogen containers through restrictive access ways;
- Liquid cryogen containers must be regularly inspected and maintained to ensure they are in good condition, in particular the wheels, bearings and axles;
- If the use of a stairway to move a liquid cryogen container is unavoidable, then the following additional safety precautions should be considered:
  - If the container is large or bulky, it is recommended that two people carry the container;
  - Ensure that access to the stairway is restricted to only the people carrying the container;
  - Consider wearing additional personal protection against spillage;
  - Consider the installation of a stair lift where practicable.

- Open transfers of cryogenic liquids and venting or purging operations should be carried out in well ventilated areas.
- Care should be taken when handling cryogenic liquid storage Dewars. Avoid mechanical shock and damage to the vessel's vacuum insulating jacket. Breakdown of the insulation will cause rapid boil-off of the liquid contents, producing large quantities of gas and a possible increase in pressure within the vessel.
- Do **NOT** store vessels in confined or restricted spaces for example corridors or stairwells.
- Transport of vessels and Dewars in lifts must be covered by a risk assessment and operating instructions / procedures. Vessels and Dewars should only be transported in service lifts or lifts for which there is manual key control to prevent passengers entering the lift on intermediate floors if possible. However, if this is not possible alternate controls must be employed e.g. multiple persons, barriers and signage to prevent lift use during the movement of cryogenics; the vessels and Dewars must **NOT** be accompanied in the lift.
- Specific care should be taken when open ended hollow dipsticks for determining cryogen depth in non-pressurised containers are used because they will produce a spout of liquid cryogen due to the rapid expansion of the liquid and gasification inside the tube.
- Ensure that there are suitable control measures in place if there is a potential for any lone working directly with cryogenic materials.

### Liquid Helium

- Liquid helium Dewars should be of an approved multi-layered insulation and wide necked type.
- Liquid helium Dewars should not be completely drained before returning to the helium plant.
- Liquid helium Dewars should not be tilted.

### A2.7 Personal Protective Equipment (PPE)

Exposure to the hazards arising from cryogenic materials is often attributable to the wearing of inadequate PPE. The following guidance should aid selection of items of PPE to be used when handling cryogenic materials.

- Suitable eye protection, such as safety glasses or a full face safety visor, is essential whenever handling or transferring cryogenics to protect against unexpected liquid splashes. Full face shields should be used when transferring liquids to an open container.
- Appropriate hand protection should be worn when working with liquid cryogenics to protect against possible liquid splashes, or touching equipment that has been in direct contact with the liquid. Gloves should be non-absorbent insulated safety gloves which are designed to prevent cryogenics from flowing into the gloves. Tongs should be used to remove objects immersed in cryogenic liquids. Non-absorbent gloves should always be worn when handling solid carbon dioxide or anything that is or may have been in contact with cryogenic liquids or vapours. Gloves are particularly useful in preventing burns from associated cold pipe work and valves.
- Where possible, the body should be covered to protect against possible liquid cryogen splashes, or equipment that has been in contact with the liquid such as un-insulated pipes or vessels. The clothing should be made without pockets or turn-ups where liquid can collect. Trousers, which should be cuffless, should be worn outside the footwear. Shorts should not be worn.
- Where liquid nitrogen is being used above floor level (e.g. on a bench or being carried up

and down steps at chest height), consideration should also be given to wearing additional splash protection such as a splash-resistant apron.

- Suitable footwear to prevent the ingress of cryogenic liquids in the event of a spillage should be worn. Avoid open-toed shoes or sandals and those made of porous materials e.g. suede, which would allow the liquid to penetrate the surface.
- If there is a likely requirement for an oxygen depletion monitor and there is no fixed installation present then a portable oxygen depletion alarm may be appropriate.

### **Note**

Metal objects e.g. watches, jewellery, rings etc. should not be worn as metals can become frozen to the skin. Objects that are soft and pliable at room temperature usually become very hard and brittle at the temperatures of these liquids and can be prone to being broken easily producing sharp edges etc.

### **A2.8 Alarms**

Any confined area where cryogenic liquids are to be used must have appropriate oxygen depletion systems. They must be maintained and suitable to warn all personnel either working in the area or before they enter an area where oxygen depletion is possible.

## Appendix 3. Calculations of Oxygen Levels Following Sudden Release and Normal Evaporation of Nitrogen

The asphyxiation risk level can be calculated by assuming the immediate and uniform introduction of gas from the vessel of largest storage capacity within the workplace.

$$\text{Oxygen Concentration} = \frac{V_{ox}}{V_w} \times 100\%$$

Where:

$V_{ox}$  = volume of oxygen ( $m^3$ )  
 $V_w$  = volume of available air ( $m^3$ )

$$V_{ox} = 0.21 \times \{V_w - (V_t \times 696)\}$$

Where:

$V_t$  = net storage tank capacity ( $m^3$ )  
696 = expansion ratio of nitrogen (liquid to gas)

$$V_w = V_r - V_i$$

Where:

$V_r$  = volume of workplace (obtained from room/area dimensions: length, width and height). If the height is greater than 2m, then the height should be taken as 2m for the purpose of the calculation since this is a little greater than a 'typical' person's height.

$V_i$  = volume of objects/items/equipment within the workplace (e.g. sample storage vessels).

If the calculation shows the oxygen concentration in the workplace will be less than 19.5%, then an action plan will need to be developed to formulate suitable preventative and risk control measures.

### Worked Example

This is the standard example used for this type of calculation. A Dewar containing 100 litres of liquid nitrogen in a room 10m by 10m by 3m. Calculate the resulting oxygen concentration if all the nitrogen was released into the room immediately and uniformly.

To calculate  $V_w$ , we will use 2m instead of 3m as the maximum height. We will also assume that the equipment will occupy 10% of the residual space in the room.

$$\text{Thus, } V_w = (200 - 20) = 180 \text{ m}^3$$

$$V_{ox} = 0.21 \times \{180 - (0.1 \times 696)\} = 23.18$$

(Where 100 litres = 0.1  $m^3$ )

$$\text{Oxygen Concentration} = \frac{23.18}{180} \times 100\% = 12.9\%$$

This would indicate that if a sudden release was to occur, there would be a serious potential for major / fatal injury to persons within the room and further precautions would be required. The principal control measures to be considered would be to increase ventilation rates.

If we also consider a more typical situation where the storage and use of liquid cryogenics, e.g. liquid nitrogen, are used in a laboratory, the potential for oxygen depletion can be estimated. There will be a continual evaporation of the nitrogen and the concentration of this gas may accumulate in a room over a period of time (assuming a certain evaporation rate from vessels, Dewars and / or pipework) may be calculated using the following equation:

$$\text{Gas Concentration, } C = \frac{L}{Vn}$$

L = gas release (m<sup>3</sup> / h)

V = room volume (m<sup>3</sup>)

n = air changes per hour

For rooms at or above ground level, natural ventilation will typically provide 1 air change per hour. However, if the rooms are windowless or have windows which are tightly sealed, the number of air changes will be less than 1 per hour. For underground rooms with small windows, 0.4 changes per hour maybe a typical value.

### Worked Example

Consider a room 10m by 10m by 3m that houses 6 x 25 litre capacity non-pressurised liquid nitrogen vessels. The rate of evaporation from the vessels is 0.625 litres / 24 hours. This information should be provided by the manufacturer of the vessel when purchased. It is typically around 2.5% of the liquid capacity of the vessel per 24 hours for a new vessel. Since there will be a deterioration in the vacuum insulation with time we will assume that the rate of evaporation is double the stated value. The room is above ground but has no windows and is estimated to have 0.5 air changes per hour by natural ventilation. The gas expansion factor for nitrogen is 696.

$$\text{Gas released, } L = \frac{\text{Number of Dewars} \times \text{Volume released per Dewar} \times \text{Expansion Ratio}}{\text{Time Period} \times 1000^\dagger}$$

(<sup>†</sup>The factor of 1000 should only be used if the volume of release is expressed in litres.)

$$L = \frac{6 \times (0.625 \times 2) \times 696}{24 \times 1000} = 0.2175 \text{ m}^3 / \text{h}$$

$$V = 3.0 \times 10.0 \times 10.0 = 300 \text{ m}^3$$

$$n = 0.5$$

We will assume that 10% of the room is occupied by items e.g. furniture etc.

$$V_w = V_r - V_i = 300 - 30 = 270 \text{ m}^3$$

Thus:

$$C = \frac{0.2175}{270 \times 0.5} \times 100\% = 0.161\%$$

i.e. the nitrogen concentration of this room is increased by 0.161%. Since the normal oxygen content is approximately 21%, the reduction in oxygen after this gradual release over time is calculated as:

$$\text{Oxygen Concentration} = \frac{21 \times 100}{(100 + 0.161)} = 20.97\%$$

The reduction in oxygen content is negligible for a room of this size storing 6 x 25 litre Dewars. Obviously while the liquid nitrogen is being transferred there will be a small increase in the amount of gas evolved but this should be a short term operation and have no significant effect.

## Appendix 4. Emergency Procedures

Before entering an incident area ensure that it is safe to do so since it is possible for an oxygen deficient environment in the vicinity of a major cryogenic accident to be present. Cold gas will displace ambient air including oxygen.

Remove victims to a known safe zone before administering first aid. Report the incident immediately by calling 3333 at DL and 2222 at RAL and Swindon and 222 at UK ATC to initiate the First Aider response. Severe cryogenic "burns" are similar to burns from fire.

Beware that liquid helium can liquefy oxygen in the air so all sources of ignition should be removed from the area.

### Cryogenics Accident Checklist

During Cryogenic Accident:

- Do not rush into the area where somebody has physical collapsed through probable asphyxiation, since you are also likely to become a casualty.
- Evacuate the area, and deploy warning signs if necessary.
- Thoroughly ventilate the area, opening doors and windows or using active forced ventilation systems.
- In case of Dewar rupture, evacuate area immediately.
- Close open valves if possible.
- Do not re-enter the area without self-contained breathing apparatus unless it is proved safe to do so. The presence of oxygen deficiency monitors will indicate the oxygen levels in the vicinity.
- If liquid helium is present, remove direct sources of ignition because of the potential for oxygen enrichment.
- Prevent liquid cryogenics from entering drains, basements, pits or any confined space where accumulation may be dangerous.

### Immediate Treatment:

- **Do not** pull clothing away from burned or frozen skin.  
Warm the contact area by immersion in warm water (approximately 38°C), with body heat, or warm air.
- **Do not** use an open flame or other significant heat source to warm burned or frozen skin.

**NOTE:** This procedure is the opposite of the procedure for a burn injury which is to rapidly cool the contact area.

- **Do not** rub or massage the affected area.
- **Do not** permit smoking or alcohol consumption.
- **Do not** give analgesics (e.g. paracetamol, aspirin).
- Prevent infection by cleansing with mild soap and water.

### After Cryogenic Accident:

Remove injured personnel: Do not use intense heat on frozen skin; warm very slowly with warm water; resuscitate if necessary; get immediate medical help.

Ventilate the area.

## Appendix 5. Training

| Role   | Initial Training    | Refresher | Frequency | Comments   |
|--|---------------------|-----------|-----------|--|
| Staff & tenants likely to work with cryogenic materials.<br><br><b>And</b><br><br>Managers and supervisors of staff using cryogenic materials. | ½ day               | ½ day     | 5 years   | Generic cryogenic safety training will need to be supplemented by 1:1 practical instruction in the use of specific cryogenic systems, PPE and alarms, by experienced cryogenics users or Original Equipment Manufacturers (OEMs) or suppliers. |
| Staff and tenants likely to design cryogenic systems   | 1 day               | 1 day     | 5 years   | Full cryogenic safety course with worked practical examples.   |
| Courses Available:   | Internal SHE Group. |           |           |  |
| Contractors  | n/a                 | n/a       | n/a       | Contractors who maintain cryogenic storage vessels and delivery systems should be competent to do so.  |

## Appendix 6. Audit Checklist

| Ref   | Item  | Rating | Comments |
|---|---|--------|----------|
| 1<br>(Section 4.2.2)                                    | Have Group Leaders ensured that all areas within their area of responsibility using cryogenic liquids have been subjected to risk assessments?  |        |          |
| 2   | Have cryogenic liquid user risk assessments been conducted for all activities in the area involving the use of cryogenic liquids, or are such assessments incorporated into the generic risk assessments for the areas? |        |          |
| 3   | Are these risk assessments documented in the Evotix Assure?   |        |          |
| 4   | Have actions arising from the cryogenic liquids assessments been completed to plan?   |        |          |
| 5<br>(Section 4.2.6)<br>(Section 4.3.3)<br>(Appendix 5) | a) Have all supervisors / managers / Group leaders and users been trained in this cryogenics code, risk assessment and the STFC Risk Assessment database?<br><br>b) Has refresher training been undertaken?             |        |          |
| 6   | Have all cryogenics risk assessments been reviewed, at least on a 2 yearly basis?   |        |          |

## Appendix 7. Document Retention Policy

| Records Established                                     | Minimum Retention Period | Responsible Record keeper | Location of records                | Comments / Justification  |
|---|--------------------------|---------------------------|------------------------------------|---|
| Gas monitoring systems testing and maintenance (4.2.11) | Current + 3 Years        | Line Management           | Local record systems/Evotix Assure | SHE Group Maintain on Evotix Assure at DL, Local management at RAL? |
| Cryogenic risk assessment (4.2.2)                       | Current + 5 Years        | Line Management           | Evotix Assure                      | SHE Group maintain Evotix Assure                                    |