Liquid Nitrogen and other Cryogens safety

Introduction

The use of cryogens in the department is only authorised after proper training. University policy statement S4/03 covering the use of liquid nitrogen is in most parts also relevant to the use of liquid helium and other liquid cryogens.

Within Physics there are a range of vessels in use which are generically called Dewars by users. The policy statement S4/03 refers to Dewars as operating at less than 0.5 bar gauge, those above this value are classified as pressure vessels, usually referred to as transportable liquid cylinders, requiring more stringent safety procedures when filling and decanting, however the general precautions for handling and using liquid cryogens are similar.

Risk assessment

A written risk assessment must be prepared wherever liquid nitrogen and/or other cryogens are used or stored, describing any control measures needed to minimise the risk. The assessment must consider all relevant risks including the risk of asphyxiation.

Properties

Liquid nitrogen is a colourless, odourless liquid of boiling point -196°C, density 0.8 kg/litre, and very low viscosity. As the liquid changes to gas at ambient temperature and pressure, the expansion ratio (the gas factor) is approximately 700. The resulting cold gas is heavier than air, so it accumulates at low level. Liquid helium being lighter than air disperses much quicker than liquid nitrogen.

Hazards

The hazards of liquid nitrogen are largely related to the large volume of gas produced on evaporation and to the liquid's low temperature. Having a very low viscosity means that it rapidly and completely covers surfaces on which it is spilt and it easily penetrates cracks and voids. This means that any spillage on clothing will penetrate much more readily than, say, water. Large spillages on other surfaces may affect areas beneath the surface, by damaging materials or even by causing oxygen depletion in areas remote from the spill.

Asphyxiation

On boiling, liquid nitrogen produces approximately 700 times its volume of gas. The resulting displacement of oxygen from the atmosphere may be sufficient to cause asphyxiation. Other liquid cryogens produce similar volumes of gas with variable effects on oxygen concentration. Always adopt the worst case scenario when carrying out an oxygen depletion calculation. See the Appendix Oxygen Depletion a Brief Guide.

Cold burns and frostbite

Skin contact with liquid nitrogen or cold nitrogen gas may cause severe cold burns, comparable with those caused by boiling water. Unprotected skin may freeze onto surfaces cooled by the liquid, causing severe damage on removal. Prolonged skin exposure to cold may result in frostbite, while prolonged inhalation of cold vapour or gas may cause serious lung damage.

The eyes are particularly susceptible – even small splashes of liquid nitrogen, or short exposures to cold vapour or gas, may cause instant freezing of eye tissues and permanent damage.

These injuries can be avoided by ensuring that users always wear appropriate personal protective equipment (PPE).

Explosions due to trapped, expanding gas

If a liquid cryogen is trapped inside a container that is sealed, then expansion on warming may cause an explosion, giving rise to danger from contamination by the vessel's contents as well as injury from fragments of the vessel itself.

Glass domestic vacuum flasks must not be used for liquid cryogens.

Condensation of liquid oxygen

The boiling point of oxygen is -183°C; therefore liquid oxygen may condense in open containers of liquid cryogens or in open vessels cooled by liquid cryogens that have boiling points below that of oxygen. The unsuspected presence of liquid oxygen may give rise to explosions caused by increased pressure if the vessels are subsequently sealed and allowed to warm up. If oxidisable material is present, then liquid oxygen may react explosively with it.

Effects on materials

Many materials become brittle when cooled by liquid nitrogen and may be irreparably damaged. Other materials (e.g. glass Dewars) may fail due to temperature stresses. Glass Dewars should be enclosed in a metal can or wrapped in tape to give protection against flying glass fragments in the event of such failure.

Use only articles or materials designed for use with liquid nitrogen. Glass domestic vacuum flasks must not be used as they may fail due to thermal shock on filling.

Ventilation and oxygen monitoring

Rooms should be sufficiently well ventilated or sufficiently large, to ensure that the oxygen concentration does not fall below 19.5 vol % due to the routine conditions of use, i.e. due to:

- the normal evaporation losses from all liquid nitrogen containers in the room
- losses caused by filling the largest container from a warm condition.

In addition, the loss of the contents of the largest container immediately after filling from a warm condition should not cause the oxygen concentration to fall below 18 %.

Where the oxygen depletion calculation under the worst-case scenario would reduce the oxygen content to below 18 %, then fixed oxygen monitoring equipment must be used.

Filling

Only those who have been suitably trained may fill Dewars using a hose from a transportable container or bulk tank. This is a potentially dangerous operation and appropriate PPE must be used.

Handling

Dewars should be handled with care and not ‘walked’, rolled or dragged along the floor - rough handling may damage them, as may severe impacts. Manual handling assessments (UPS S7/99) will be needed for larger Dewars (> 20 litres).

Lifts

Liquid cryogens must not be transported in occupied lifts because of the danger of asphyxiation in the event of a leak or spill, especially in the event of a lift breakdown.

Transport

Liquid nitrogen must not be transported in a closed vehicle such as a car or van.

Information, instruction and training

All users of liquid nitrogen must have received enough information, instruction and training to enable them to understand the dangers associated with liquid nitrogen and how this relates to their own work. The Physics Department provides an introductory talk on cryogenic safety and a practical session on the filling and use of cryogenic containment vessels. The University Safety Office offers a regular comprehensive course on the Safe Use of Cryogenic Liquids: https://safety.admin.ox.ac.uk/training-a-z

Protective clothing for use with liquid cryogens

(a) Eye protection: as a minimum, safety spectacles with side shields must be worn whenever handling liquid cryogens. A face shield to EN 166 must be worn where there is a risk of splashing the face or eyes, e.g. during filling operations. Models with brow guard and chin guard offer the best protection.

(b) Hand protection: For filling operations, non-absorbent, insulated gloves to EN 511, or loose fitting leather gloves (for ease of removal in case of spillage) must be worn and coat sleeves should cover the ends of the gloves. Gauntlets are not recommended as liquid may run down inside them. These gloves will also protect the skin from contact with objects that have been cooled by liquid nitrogen. ***Gloves are not intended to protect the hands against immersion in liquid cryogens.***

(c) Foot protection: open toed shoes must not be worn.

(d) Clothing: as a minimum, a lab coat or overall should be worn. If boots are worn then trousers should be worn outside of them, not tucked into them. A splash resistant apron will give added protection where dewars are being lifted or carried, or wherever there is a high risk of splashing, e.g. during filling operations.

(e) Ear protection: ear plugs or ear defenders may be required where excessive noise occurs when filling or venting liquid nitrogen systems (a noise assessment may need to be carried out – see UPS S1/06).

Appendix: Oxygen Depletion Calculations

Four cases are considered here:
(a) normal evaporative losses, (b) filling losses, (c) spillage of the vessel's contents, and (d) loss of the entire contents of the vessel immediately after filling. The British Compressed Gases Association (BCGA) recommends that, for the purpose of risk assessment, the worst case possibility (d) is considered.

The oxygen concentration, C_{oxygen} in a room may be calculated using the formula

$C_{oxygen} = 100 \times V_{oxygen} / V_{room}$

where V_{oxygen} is calculated as in b), c), or d) below, and V_{room} is the room volume in m^3.

(a) Normal evaporative losses

Over a long enough period, the percentage decrease in oxygen concentration due to normal evaporation losses from a vessel is approximately

$0.21 \times 100 \times Ct$, where

$Ct = L \div (V_{room} \times N)$

- 0.21 represents the normal concentration of oxygen in air (21%)
- Ct is the increase in nitrogen concentration
- L is the gas evaporation rate in $m^3\ h^{-1}$
- N is the number of air changes per hour.

Manufacturers quote evaporation losses for their vessels (normally as a volume of liquid nitrogen lost per day). Allowance should be made, by doubling these figures, for the deterioration of insulation performance over the lifetime of the vessel

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$L = 2 \times 700 \times$ liquid nitrogen loss in litres per day $\div (24 \times 1000)$, where

• 2 allows for the deterioration of insulation
• 700 represents the gas factor for nitrogen (1 litre of liquid nitrogen produces about 700 litres of gas)

For example, a basement room 4x3x3 m (36 m3) contains five 10L Dewars, whose evaporative losses are quoted by the manufacturer as 0.15L liquid nitrogen per day per Dewar. A basement room has about 0.4 air changes per hour.

$L = (2 \times 700 \times 5 \times 0.15) \div 24 \times 1000 = 0.044\ m^3\ h^{-1}$

$Ct = 0.044 \div (36 \times 0.4) = 0.003$ and the oxygen depletion is 0.06%.

In this case, the normal evaporation losses have an insignificant effect on the oxygen content of the room. However, where Ct = 0.07 or higher, then the oxygen depletion becomes 1.5% or more, and extra ventilation and/or oxygen monitoring will be required.

(b) Filling losses
When a vessel is filled, some loss always occurs as it is cooled to liquid nitrogen temperature. The BCGA recommends that a loss of 10 % of the vessel's capacity should be assumed in order to assess the risk from filling losses.

$V_{oxygen}\ (m^3) = 0.21\ [V_{room} - (0.1 \times V_{vessel} \times 700 \times 10^{-3})]$, where

• 0.21 represents the normal concentration of oxygen in air (21%)
• represents the loss of 10 % of the vessel's capacity
• V_{vessel} is the vessel's capacity in litres
•700 represents the gas factor for nitrogen (1 litre of liquid nitrogen produces about 700 litres of gas)

With the same room and Dewars as in (a),

$V_{oxygen} = 0.21\ [36 - (0.1 \times 10 \times 700 \times 10^{-3})] = 7.413\ m^3$

$C_{oxygen} = 100 \times V_{oxygen} / V_{room} = 20.6\%$

Once again, there is no significant effect on oxygen concentration in normal use.

(c) Spillage
For the spillage of the entire contents of a vessel

$V_{oxygen}\ (m^3) = 0.21\ [V_{room} - (V_{vessel} \times 700 \times 10^{-3})]$

If the entire contents of a 10L Dewar were spilt in this 36 m^3 room, then

$V_{oxygen} = 0.21\ [36 - (10 \times 700 \times 10^{-3})] = 6.09\ m^3$

$C_{oxygen} = 100 \times V_{oxygen} / V_{room} = 16.9\ \%$

This spillage would significantly deplete the oxygen concentration.

(d) Filling of a vessel followed by the spillage of its entire contents
$V_{oxygen}\ (m^3) = 0.21\ [Vroom - (1.1 \times V_{vessel} \times 700 \times 10^{-3})]$, where

1.1 represents 10% filling loss + 100% loss of the vessel's contents by spillage

This is the worst case that should be considered in the risk assessment - both (b) and (c) are taken into account.

$V_{oxygen} = 0.21\ [36 - (1.1 \times 10 \times 700 \times 10^{-3})] = 5.943\ m^3$

$C_{oxygen} = 100 \times V_{oxygen} / V_{room} = 16.5\ \%$

The risk assessment shows that alternative storage arrangements must be considered or oxygen monitoring must be installed. Alternative arrangements may include:

- *siting the vessels elsewhere*
- *using smaller vessels*
- *arranging for any pressure relief devices to vent to a safe place outside of the room*
- *installing mechanical ventilation, possibly linked to the low oxygen alarm*
- *requiring staff to wear personal oxygen monitors*