Safety & Environmental Protection Services Guidance Note

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Liquid Nitrogen

Introduction

Liquid nitrogen is a substance that is used widely in laboratories and elsewhere both as a refrigerant and to produce an inert atmosphere. Although it is so common, its associated hazards may not be fully appreciated. This note informs users of the properties of liquid nitrogen, the hazards associated with its use and the basic safety precautions associated with this substance. It covers storage in vacuum Dewars and pressurised vacuum insulated cylinders but does not extend to advice on design and operation of bulk storage tanks. Departments that operate large-scale facilities of this nature should seek specific advice from SEPS.

PART I

Characteristics and hazards of nitrogen

Nitrogen is a normal constituent of the atmosphere making up about 80% of the air we breathe. It is odourless, colourless and tasteless and therefore not detectable by human senses. It is non-flammable, non-toxic, inert to most chemicals and will not support life.
When cooled to below its boiling point (-1960C) gaseous nitrogen can be condensed to a liquid. It will remain in this form provided it remains below this temperature. On rapid warming the vapour may be briefly visible as a white mist.
Although liquid nitrogen is not toxic, it has two major life threatening hazardous properties. Firstly, because the liquid can evaporate very quickly, it can effectively displace air to create an atmosphere that is unable to support life. Secondly, it can cause injury from the intense cold of the liquid.

1. Asphyxiation (oxygen deficiency):

On evaporation liquid nitrogen produces about 680 times its own volume of gas. If this occurs in an enclosed area that is inadequately ventilated, atmospheric oxygen will be displaced by the nitrogen. Eventually the oxygen concentration may become so low that the atmosphere will not sustain life. Anyone in such an environment will become unconscious and will quickly die unless remedial action is taken.

Persons working in an atmosphere that is becoming oxygen deficient are unlikely to be aware of the increasing danger, as there are few warning signs. Those signs that may be present (e.g. increased frequency and depth of breathing and raised pulse rate) are masked by impaired perception and judgement resulting from the reduced oxygen levels. Often the first indication that anything is wrong comes too late to allow any response to the situation. Also when persons enter an area in which the oxygen concentration is severely reduced they may become immediately unconscious. It is therefore vital to ensure that systems of work do not create circumstances where these risks may be manifest.

Multiple fatalities have occurred when rescuers have themselves been overcome while attempting to assist an unconscious colleague. The temptation to enter an area to effect a rescue is strong. However, the risks of doing so are extremely high. It is essential that emergency action is planned in advance and that staff are trained to understand the action to be taken in such circumstances. Rescuers MUST NOT attempt to enter an oxygen deficient atmosphere unless they are wearing suitable breathing apparatus and are trained in its use.

2. Cryogenic (cold) burns:

Liquid nitrogen is kept at 'cryogenic' or very low temperatures –1960C below. Contact with liquid or vapour at such temperatures can produce damage to the skin and other tissues. The effect is similar to a burn. Delicate tissue, such as that of the eye, is damaged more easily and prolonged exposure can lead to frostbite.

Contact with non-insulated parts of equipment or vessels containing liquid nitrogen can produce similar damage. Moreover, unprotected parts of the skin may stick to low temperature surfaces as the moisture of the skin becomes frozen. Thus extreme care must be taken as the flesh can be torn away whilst trying to break free.

Inhalation of cold vapour can cause damage to the lungs and may trigger an asthma attack in susceptible individuals, whilst prolonged exposure to low temperatures can induce in generalised hypothermia.

To minimise the risk of injury appropriate working techniques and protective clothing are necessary.

First aid for cryogenic burns.

1. Flush the area of skin with tepid water.
2. Do not apply direct heat or use hot water.
3. Do not use a forceful flow of water as this could cause tissue damage.
4. Move the casualty to a warm place and seek medical attention.
5. If the burn is severe, call an ambulance.
6. While waiting for medical attention, continue to flush the areas with tepid water, remove any tight jewellery.
7. Do not allow the patient to smoke and do not offer hot beverages.

3. Oxygen enrichmeant:

The low temperature of liquid nitrogen may cause gaseous oxygen to condense from the atmosphere and accumulate as liquid oxygen. The presence of this bluish liquid significantly increases the risk of fire or explosion. This can occur if Dewars or insulated flasks of liquid nitrogen are left open, or where incorrect lids are used. If exposure to the atmosphere continues over an extended period oxygen can condense into the liquid nitrogen making the mixture as hazardous to handle as liquid oxygen. This is a particular problem with wide-necked, shallow containers. [Note: Remember, to prevent dangerous pressure build-up containers must never be fully sealed.]

Oxygen enrichment is also a potential problem in closed systems containing liquid nitrogen (e.g. vacuum systems). If air is able to leak into the system the oxygen may condense and accumulate in traps or low points and can pose a hazard if it comes into contact with other chemicals or equipment such as pumps or ignition sources. Designers of such systems must thus address such hazards.

4. Over-pressurisation of storage containers:

Ice plugs may form in the neck of Dewar flasks that are left open. These can block the outlet and cause a build up of internal pressure which may result in either the plug being explosively ejected, or possibly the rupture of the vessel. If an ice plug is found extreme caution is needed.

The area should be vacated and those dealing with the incident equipped with appropriate protective clothing. This should include a full face-shield and appropriate gloves. Any pressure within the Dewar is relieved by piercing a hole through the ice-plug with a thin, hot L-shaped wire. While doing so do not lean over the Dewar outlet! Having released any internal pressure the plug may then be thawed and removed safely. The formation of ice plugs can be prevented by the diligent use of the correct Dewar stoppers.

Sealed sample containers (e.g. Eppendorf tubes) stored in liquid nitrogen may suffer a build-up of internal pressure as they return to room temperature after removal from storage. This occasionally results in explosive rupture of a tube. Eye protection must therefore be worn when removing samples from cryogenic storage. The hazard from a bursting tube can be minimised by enclosing samples in a protective outer container (e.g. plastic secondary container, or ice bath) or by covering immediately on their removal from the storage vessel and leaving them enclosed (but not sealed) until they have reached the desired temperature.

5. Embrittlement of materials:

Many materials become brittle and prone to fracture when they are cooled to low temperature. This is true of both metals and plastics and has obvious implications for the design and selection of systems and vessels used to contain or convey liquid nitrogen. Stainless steel, nickel steel, copper and aluminium alloy have reasonably good low temperature characteristics. PTFE is commonly used for sealing of liquid nitrogen systems. Carbon steel becomes brittle and is normally unsuitable for low temperature applications. If in doubt, seek specialist advice.

Where liquid nitrogen is to be stored within the laboratory only containers known to be suitable for use at very low temperature should be used. This will entail careful initial selection of equipment and effective training of laboratory staff to meet such obligations.

Some types of floor covering (e.g. vinyl) are likely to become brittle and prone to damage if liquid nitrogen is spilt on them. This can create a tripping hazard and is unsightly. Consideration of this factor at the laboratory design stage can be cost effective in avoiding a subsequent need for replacement of damaged coverings.

PART II

Safe Use of liquid nitrogen in Small-Scale Facilities

1. Managerial control of liquid nitrogen use:

As in all aspects of safety, an effective system of management is required to control the risks that may arise from work that is undertaken. This will include a suitable system of risk assessment to identify and control potential risks. These elements are basic legal requirements. Under the terms of the University Safety Policy Statement it is the responsibility of Heads of Department to establish systems to ensure that these requirements are met. The following features would be included in a suitable management system.

- Arrangements to check that locations where liquid nitrogen is stored or used are suitable for this purpose. (i.e. adequately ventilated)
- A system to ensure that only the minimum volume of liquid nitrogen consistent with the practical and technical requirements of the work is held in stock.
- Arrangements to provide staff with adequate training in the hazards of liquid nitrogen and safe working techniques.
- Provision of appropriate protective equipment and supervisory arrangements to ensure that this is properly used.
- Arrangements for periodic maintenance of cryogenic equipment.
- An effective emergency procedure that is known and understood by staff.

Some of these are discussed further in this note. Other issues that are not included above may also be relevant under local circumstances.

2. Training:

All people who work with liquid nitrogen should be given adequate instruction about the risk of asphyxiation, cold burns and the other associated hazards outlined in Part I. Particular attention should be drawn to the insidious nature of the asphyxiation risk and to emergency procedures to be followed in the event of spillage. Effective practical training in the correct use of protective clothing and in the equipment and methods that should be used in the handling of liquid nitrogen during normal operational work should also be provided. All personnel must be made aware of the correct emergency action. This training might be included as part of the departmental induction program.

3. Protective clothing:

Eyes should be protected by goggles or by a full face-shield. Where there is an obvious risk of splashing a face-shield is more appropriate as it will protect the skin of the face as well as the eyes.

Overalls or side-fastening lab coats (preferably without pockets) should be worn. Sleeves and trousers should be worn outside gloves or boots. Metallic jewellery should be removed. Open-toed shoes should never be worn and bare skin must be covered.

4. Handling liquid nitrogen:

Pouring liquid nitrogen or immersion items should always be performed slowly to minimise the thermal shock. This also minimises the boiling and splashing that inevitably occurs. Partly cover wide necked, or shallow containers to reduce splashing and loss of liquid. Use tongs or instruments when inserting or withdrawing objects from liquid.

When inserting open-ended pipes into a cryogenic liquid always block off the warm end until the cold end has cooled to the temperature of the liquid. If this is not done, a stream of liquid and cold gas may issue from the warm end.

When liquid nitrogen evaporates rapidly initially it forms a cloud of white vapour. This cloud is cold and very dense and will tend to accumulate at low level. Care should therefore be taken when working with liquid nitrogen close to open manholes, trenches or other sunken areas in which people are working. When warmed to room temperature nitrogen gas is very slightly lighter than air but will mix with it readily.

When transporting liquid nitrogen by lift do not travel in the lift at the same time.

5. Cryogenic equipment:

Dewars and other insulated containers should be treated with care as even minor impacts may lead to slow, or even catastrophic loss of vacuum. Regular inspection of containers for damage is valuable. Frost patches on the exterior of cryogenic plant are indicative of poor insulation or of a leak. If such evidence is found specialist advice should be sought from the equipment supplier. Maintaining a record of the volumes used to top-up Dewars can also help to identify deterioration in container insulation by highlighting an increased evaporation rate.

Vacuum insulated liquid cylinders

Vacuum insulated liquid cylinders are commonly used as a central storage facility for cryogenic fluids within departments. Often they are used to top-up individual atmospheric pressure vacuum Dewars. This process is usually accompanied by an unavoidable discharge of nitrogen vapour. In addition, pressurised containers have the potential to release large volumes of liquid or vapour through discharge of the pressure relief valve or failure of a bursting disc. A careful assessment is necessary of the likely effect on the oxygen concentration in the area where pressurised Dewars are stored and used. In particular, the effect of extended or uncontrolled discharge and the need for emergency procedures should be considered. Containers should only be filled, or liquid discharged from them, in areas that are well ventilated.

Vacuum insulated liquid cylinders are pressure vessels and, as such, are subject to legal requirements affecting this class of equipment.1 More detailed information on this is available from SEPS. In summary such equipment must be constructed to a recognised standard and must have safe operating limits set for its use. After purchase the user department is responsible for ensuring that the equipment is properly maintained and that it receives a "thorough examination" at periodic intervals according to a written scheme of examination. Users may expect liquid nitrogen suppliers to ask for evidence that these requirements have been met and to decline to fill any container where such evidence is lacking. Gas or cryogenic equipment suppliers will usually offer maintenance contract that incorporates the necessary "thorough examinations" and maintenance work. Departments are likely to find that this service is the most practical and cost-effective way of meeting the legal requirements.

6. Ventilation requirements:

Whenever liquid nitrogen is stored or used there will always be some evaporation of nitrogen vapour. This will occur passively from storage vessels and may also be an active process as a result of the work. Given that this will occur, the level of ventilation available has to be sufficient to prevent oxygen depletion of the area if a hazardous situation is to be avoided. Often passive room ventilation is sufficient but sometimes this may need to be supplemented by provision of additional measures. This could be in the form of additional openings. (These should be situated at high and low level and should, ideally, be located so as to promote a flow of air through the room.) Mechanical systems may be necessary where high ventilation rates are needed.

An assessment of the ventilation requirements should be made for all locations where liquid nitrogen is stored or used. The degree of ventilation necessary is dependant on the rate and volume of liquid evaporation, the volume of air into which it is evaporated and the effectiveness with which the vapour mixes with this air. As a general rule, the ‘worst case’ scenario should normally be assumed. Working practices that minimise evaporation rates and volumes should be followed so far as practicable.

Small-scale storage (e.g. one or two Dewars)
Although some evaporation will occur from Dewars, the rate of release is low. For small numbers of Dewars, natural room ventilation will normally be adequate.

Larger-scale storage
Where larger numbers of Dewars are concentrated together or where storage is in pressurised containers a more detailed assessment will be needed. The provision of additional permanent ventilation openings may be necessary. If provided, these should be regularly checked to make sure they are clear. (There is a tendency for people to block them up to avoid draughts!) A low-oxygen alarm may be appropriate in some locations. Consult SEPS for advice on such areas.

Processes involving deliberate evaporation of liquid
Where liquid nitrogen is deliberately evaporated as part of a work process a written assessment of the consequences is necessary. Often the rate of nitrogen evaporation will be known, or can be calculated, allowing the likely effect on the oxygen concentration within the room to be considered quantitatively. Activities involving deliberate release of liquid nitrogen must not be carried out if they are likely to result in oxygen concentration in the area being reduced below 20% during normal operations. If such a risk is identified, modification or relocation of the process to avoid this likelihood must be considered. Provision of additional natural or mechanical ventilation may be needed. Provision of a low-oxygen alarm should be considered in locations where a significant risk of oxygen depletion is identified by the risk assessment.

7. Spillage and releases:

In all locations the effect of accidental spillage or release of liquid nitrogen must be considered. As a general rule the effect of spillage of the largest container present or the combined volume of containers connected by a manifold system should be assumed. The effect of this on the oxygen content of the area should be calculated.* SEPS can provide assistance with this if required. It is suggested that spillages of liquid nitrogen capable of releasing a volume of nitrogen gas in excess of 5% of the room volume should be considered sufficient to require evacaution and ventilation of the area. It should not be assumed that this will always be a "safe" limit and it may be necessary to evacuate for lesser spillages depending upon local circumstances. Staff should be provided with training and instructions about the action to take in the event of spillage and about the risks of remaining in the room and of re-entry before it has ventilated thoroughly.

Usually spillages will evaporate and disperse fairly quickly if the room is well ventilated. This may mean opening windows or doors outside the area of immediate risk to promote an airflow. Departments which store large volumes of cryogenic liquids should consider how to achieve effective ventilation without having to re-enter the area in the event of a large-scale spill. Heads of Department are responsible for ensuring that these arrangements are incorporated into a formal departmental emergency plan. Where it is necessary to leave the room unattended (e.g. overnight) it should be secured to prevent inadvertent access to what might be a hazardous area. Warning signs should be posted. Everyone who works in the area (including ancillary personnel) should be instructed in the part they may play in a real emergency. (e.g. by "dummy run"). Do not assume that supervisory staff will be on hand to take charge.

* [To calculate the volume of nitrogen gas produced by a known volume of liquid nitrogen, multiply the volume of liquid released by the liquid/gas expansion factor (x 680 for nitrogen) Example: The contents of a standard 25 litre dewar will produce 25 x 680 = 17000 litres (or 17 m3) of nitrogen gas. If this is released in a room with a volume of 170m3, the oxygen content will be reduced to approximately 19%. In a smaller room the oxygen depletion will be greater.]

8. Alarm systems:

Alarm systems capable of alerting occupants to falling oxygen levels in an area are commercially available. Whilst they are very useful warning devices they do have a significant running cost and cannot prevent an area from becoming oxygen deficient in the first place. They should therefore be regarded only as a second line of defence. Alarm systems are unlikely to be reasonably practicable or necessary for most small-scale storage but might be appropriate for the larger facility or for some activities involving evaporation of large quantities of liquid nitrogen within enclosed areas.

When an alarm system is provided it is important that all staff are trained in the actions they should take if the alarm sounds and are explicitly instructed NOT to enter the area covered by the alarm while it is sounding.

Footnotes:

SEPS are equipped to make measurements of atmospheric oxygen levels using a portable meter and can assist departments with this on request.

1. The Pressure Systems Safety Regulations 2000 and their associated Approved Code of Practice currently contain the principal legal requirements concerning the physical integrity of pressure vessels.

References

1. British Compressed Gases Association Code of Practice CP27 " Transportable vacuum insulated containers of not more than 1000 litres volume." ISSN 0260-4809

2. British Standard BS 5429 : 1976 "Safe operation of small-scale storage facilities for cryogenic liquids."

3. "Safety of pressure system" - Pressure Systems Safety Regulations 2000 and Approved Code of Practice. HSE Books Ref: L122 ISBN 0 7176 1767 X

Suppliers of cryogenic liquids and manufacturers of specialist equipment also have a range of useful safety literature available. This is normally free of charge.