University College Dublin

Guide To The Safe Use Of Liquid Nitrogen

Liquid Nitrogen (N_2) is nitrogen gas held at an extremely low temperature (around -195^{o}C) where it forms a liquid. It is part of a group of low temperature liquefied gases known as cryogenic liquids. The purpose of this document is to provide users of liquid nitrogen with some guidance on how to utilise this material safely.

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What Are The Hazards From N_2?

Frostbite / Cold Burns

Liquid nitrogen can cause significant damage to living tissue on extended contact. Whilst a single brief contact may not cause skin damage prolonged contact certainly will. Cold burns and frostbite can lead to serious permanent disfigurement including amputation of the digits. Liquid nitrogen burns can be a particular problem if the material is spilled onto clothing without the wearer being aware or if porous gloves are worn and become contaminated. Also if liquid nitrogen is inhaled then it can cause damage to the lungs and respiratory tract. Again if splashed into the eyes irreparable eye damage can occur.

Asphyxiation

Liquid nitrogen is held in a cooled state. As it begins to warm (above around -195^{o}C) it starts to change from a liquid into a gas. One litre of liquid nitrogen if allowed to warm will rapidly generate 682 litres of nitrogen gas. Nitrogen gas is a colourless non toxic odourless gas. It is what is know as a ‘simple asphyxiant’. This means that if present in a high enough concentration the nitrogen gas can suffocate a person by virtue of the fact that its presence has driven down the relative percentage of oxygen in the air being inhaled by the individual. This is a significant risk in areas where liquid nitrogen is being stored and used.

System Pressurisation

Liquid nitrogen as outlined about will expand rapidly as it begins to warm generating nitrogen gas. This can cause the build up of extreme pressure if the material is held in a sealed or poorly vented system. This can give rise to vessel explosion / rupture.

Oxygen Condensation

In some scenarios it is possible for containers holding liquid nitrogen to become sufficiently cooled so that the oxygen in the atmosphere condenses and forms liquid oxygen on the cooled surfaces. This can occur when vessels that are open to the atmosphere are cooled on the outside by liquid nitrogen thus allowing liquid oxygen to form on the inside of the vessel. Similarly pipe work cooled internally by liquid nitrogen can allow liquid oxygen to condense on the outside. It is also a risk where continuously cooled liquid nitrogen flasks have been sitting for a long time. Liquid oxygen is an explosive material, especially in the presence of organics. It has a light blue colour and often appears as a light blue hue within cooled vessels.

How Do I Protect Myself From The Hazards Of N_2?

Use Personal Protective Equipment

Users of liquid nitrogen must always consider the wearing of personal protective equipment appropriate to the task in hand so that in so far as is possible they are physically protected from the material. Users should always ensure that they keep liquid nitrogen off their bodies / clothing and out of their lungs and eyes.

The types of personal protective equipment required will depend on the type of usage. The larger the volume of material handled and the greater the potential for body contact then the greater the degree of protection that is required.

Gloves

Protective gloves should be worn if there is a risk of material spillage onto the hands. Protective gloves must conform to BS EN 511 (Cold Protection). The gloves should be specifically designed for cryogenic handling with ribbed cuffs to prevent splashing into the glove or be loose fitting gauntlets that can easily be removed. The glove material should be rough to give good grip while handling cooled vessels and not increase the chance of spillage. Glove material should be non porous to prevent liquid nitrogen entering the space between the glove material and the skin.

Protective Clothing

Standard lab safety clothing is suitable for handling small volumes of liquid nitrogen in a university / hospital type environment. This consists of a closed lab coat and if necessary an apron. The wearing of clothing that leaves the legs uncovered should be avoided when working with large volumes.

Eye Protection

When working with small volumes of liquid nitrogen safety glasses with integral side protection should be worn. When handling larger volumes of liquid nitrogen or decanting material consideration should always be given to the wearing of goggles or a face shield.

Footwear

Feet should be covered when working with larger volumes or when decanting material. The wearing of sandals or open toed shoes and similar should be avoided.

Reduce The Risk Of Asphyxiation

Liquid nitrogen must be used and stored safely so as to minimise the risk of user asphyxiation. Asphyxiation can occur of nitrogen gas is allowed to reach an unsafe level in the atmosphere. Remember that one litre of liquid nitrogen can generate 682 litres of nitrogen gas. This gas can drive down the relative concentration of oxygen in the atmosphere (especially in confined and poorly vented areas) leading to potential health affects – see Table 1 below.

Table 1. Asphyxia – Effect of O_2 Concentration

% O_2 In Atmosphere	Effects and Symptoms
18-21	No discernible symptoms can be detected by the individual.
11-18	Reduction of physical and intellectual performance without the sufferer being aware.
8-11	Possibility of fainting within a few minutes without prior warning. **Risk of death below 11 vol%.**
6-8	Fainting occurs after a short time. Resuscitation possible if carried out immediately.
0-6	Fainting almost immediate. Brain damage may occur, even if rescued.

Storage vessels for liquid nitrogen are designed so that nitrogen gas can boil off. In larger systems this vented nitrogen gas is often collected and piped off safely. However in most storage vessels the gas vents to atmosphere. This can be a significant safety issue in bulk storage area.

When storing and using liquid nitrogen there are a number of rules must be followed to minimise the risk of asphyxiation:

- Only those vessels composed of suitable material may be used for storing liquid nitrogen. Some materials e.g. glass and some plastics, may fracture at low temperatures releasing nitrogen gas.
- Liquid nitrogen should not be transported through heavily populated areas of buildings in case of spillages. Stairs should be avoided unless the volumes being transported are small. Persons involved in the transport of liquid nitrogen should wear appropriate personal protective equipment.
- When using lifts to transport liquid nitrogen the following should be adhered to:
    - Dewars must not be accompanied in lift. A lift is a confined space and should leakages occur asphyxiation is possible.
    - One person should place the Dewar in the lift whilst another waits to receive the Dewar from the lift once the journey is complete

  - There should be a clearly visible sign on the Dewar warning others not to enter the lift with the Dewar
  - Where possible a goods lifts should be use
  - If transporting Dewars in vehicles then they should be held in a well ventilated area in a separate compartment to the driver.
- Lone working with liquid nitrogen should be avoided wherever possible. If required a Lone Working Risk assessment should be carried out.
- Bulk storage (>25 litres) areas for liquid nitrogen must:
  a. Display hazard warning signage
  b. Be restricted to authorised personnel only
  c. Be continuously ventilated if possible
  d. Have more than one escape route if possible
- Storage areas and areas in which liquid nitrogen is in use should be subjected to an assessment of potential oxygen levels during spillages, storage and topping up / refilling activities. If deemed necessary by this exercise then atmospheric oxygen depletion sensors must be installed in the relevant areas or the ventilation must be improved.
- Avoid the use of wide-necked, shallow vessels to prevent excessive evaporation and the possibility of oxygen depletion.
- When disposing of liquid nitrogen do not pour it down the sink or allow it to vaporise into enclosed areas such as laboratories, fridges, freezers, cold rooms, etc. Liquid nitrogen to be disposed of through vaporisation must be left in well ventilated area e.g. a fume hood.
- Do not store liquid nitrogen in walk in freezers / cold rooms.

Reduce The Risk Of Pressurisation

As outline above once liquid nitrogen begins to warm it generates relatively large quantities of nitrogen gas. In sealed or poorly vented systems this can be problem. To overcome this the following must be adhered to:

- Purpose designed non pressurised vacuum flasks must be used to store volumes of liquid nitrogen of between 1-50 litres, i.e. Dewar Flasks. Dewar Flasks must have non sealed stoppers to allow boiling material to vent. Any glass component of a Dewar flask must be covered in tape to prevent shattering in the event of an explosion.
- Always make sure that containers of liquid nitrogen are suitably vented and unlikely to block due to ice formation.

- Do not use liquid nitrogen within sealed systems unless precautions have been taken to prevent warming. In such cases the use of a pressure release value should be considered.

Prevent Oxygen Condensation

Measures should be taken to prevent the generation of the explosive liquid oxygen. These include:

- Use liquid nitrogen to cool sealed or evacuated systems only in order to prevent oxygen condensation on the inside of cooled vessels.
- The ability of liquid nitrogen to cause condensation of liquid oxygen from the air onto cooled pipe work must be considered when designing processes involving liquid nitrogen.
- Storage vessels should be emptied and allowed to dry on a regular basis wherever possible.
- Users should always be vigilant for the presence of liquid oxygen which emits a blue hue.

Additional Safety Precautions

- Do not allow untrained persons to use or handle liquid nitrogen.
- A Material Safety Data Sheet for liquid nitrogen must be readily available.
- All metallic jewellery should be removed when handling liquid nitrogen as metal items will quickly spread the cold from any contact with the cryogenic material.
- When pouring liquid nitrogen do so slowly and carefully to minimise splashing and rapid cooling of the receiving container.
- Always use thongs when placing or removing items from liquid nitrogen.
- Never overfill Dewars.
- Use dip sticks to check liquid depth in Dewars. Do not use fingers.
- Pregnant females and asthmatic workers must seek medical approval prior to working with liquid nitrogen.
- Low temperature damage to the insulation on electrical cables can lead to electrocution and equipment damage. Liquid nitrogen users must ensure that cables are not placed where they can be affected by spillages.
- Carry out a risk assessment for the use of liquid nitrogen (refer to www.ucd.ie/safety)
- Only use purpose built trolleys and cantilevered units for the movement and decanting If large cylinders. Avoid the manhandling of large cylinders for decanting.

What To Do If Something Goes Wrong?

- In the event of a cold burn from liquid nitrogen:
    a. Remove any restrictive clothing - but not any that is frozen to the tissue
    b. Flush the affected area with tepid water (not above 40°C) to return tissue to normal body temperature
    c. Do not apply any direct heat or rub affected area
    d. Cover with a loose, sterile dressing and keep patient warm
    e. Obtain medical assistance immediately

- All users of liquid nitrogen should be aware of the symptoms of anoxia (physiological oxygen depletion). These include dizziness, a narcotic type affect; nausea, confusion, etc. Persons experiencing such symptoms should remove themselves to fresh air. Persons observing such symptoms in co-workers should remove them to fresh air. In the event that breathing stops inform the local first aider and give artificial respiration.

- Do not attempt to rescue anyone from a confined space if they were working with liquid nitrogen and they have lost consciousness - open the door if possible and raise the alarm on internal telephone number 7999.

- For minor spillages (<1 litre) of liquid nitrogen the following protocol should be followed:
    a. Evacuate the immediate area.
    b. Allow liquid to evaporate, ensuring adequate ventilation
    c. If present ensure that the oxygen depletion sensor has been reset before re-entering the room.
    d. Following return to room temperature inspect area where spillage has occurred
    e. If there is any damage to the floors, benches or walls inform the buildings office on internal telephone 1111
    f. If any laboratory equipment has been damaged following the spillage inform the laboratory manager / supervisor

- For major spillages (>1 litre) of liquid nitrogen the following protocol should be followed:
    a. Evacuate the immediate area
    b. Inform emergency services on internal telephone 7999
    c. If present ensure that the oxygen depletion sensor has been reset before re-entering the room.
    d. In the event that an oxygen depletion sensor is not present do not return to the area until it has been declared safe

Liquid Nitrogen Spillage - Oxygen Depletion Assessment

The most significant risk from liquid nitrogen is death by asphyxiation where a spill or leakage depletes the atmospheric oxygen locally. If the oxygen concentration falls below 18% adverse effects will occur resulting in loss of mental alertness and performance combined with distortion of judgement. In atmospheres containing less than 10% oxygen death by asphyxiation is rapid: just two breaths of oxygen-free air kills.

The resulting oxygen concentration following a spill can be determined using the equation:

$$\frac{100 \times V_o}{V_r}$$

Where:

$\mathbf{V_0}$ = $0.209(V_r - V_g)$

$\mathbf{V_r}$ is the volume of the room in m^3

$\mathbf{V_g}$ is the maximum gas release upon the expansion of the cryogenic liquid (volume of liquid in m^3 x expansion coefficient - 682)

To calculate the oxygen concentration when oxygen is the cryogenic liquid, V_o in the formula becomes $0.209(V_r - V_g) + V_g$.

An example of the oxygen depletion resulting from a spill of 50 litres of liquid nitrogen in a room 5m x 10m x 3m is calculated below:

100 X 0.209(150 m^3 - (0.05 m^3 X 682))/150 m^3 = 100 x 0.209 X 115.9/150 = 16.2%

Oxygen levels will deplete to 16.2% in the immediate area following a spill of 50l of liquid nitrogen in a room of this size. This is a serious drop in oxygen levels and could lead to serious injury in the event of a spill. In this scenario forced ventilation or oxygen depletion sensors would be required.

Liquid Nitrogen Storage - Oxygen Depletion Assessment

Liquid nitrogen evaporates from Dewar flasks at known rates. Material is also released to the atmosphere during container pouring and filling operations. These factors may lead to potential depletion of oxygen levels in storage and pouring areas. If the calculated oxygen levels are likely to be ≤19% at any time then control measures are required. Other factors impact on oxygen depletion rates, including the rate of air exchange within the storage areas. The principles of assessing the level of oxygen depletion during storage and pouring operations are outlined below, along with a worked example for the storage and daily refilling of two 200 litre Dewars of liquid nitrogen held in a room 4m x 5m x 3m in size with no forced ventilation.

THEORY	WORKED EXAMPLE
1. Assume storage of ***n*** Dewars of ***w*** litres capacity.	1. 2 x 200 litre flasks of liquid nitrogen. ***n*** = 2; ***w*** = 200
2. Static losses from the flasks can be taken as ***x litres/hr.*** These data are available from the supplier of the Dewar flasks and vary from model to model. To take account of flask wear and tear the rate of loss is doubled for flasks in regular use i.e. ***2(x)***.	2. From information supplied by the manufacturer static loss rate from each flask (***x***) = 0.25 litres/hr. Double to take account of flask wear and tear (***2x***) =0.5 litres/hr per flask
3. The ***expansion coefficient*** of liquid nitrogen is 682	3. Liquid nitrogen ***expansion coefficient*** = 682
4. ***n*** times ***2(x)*** times ***expansion coefficient*** = litres of gas loss per hour	4. 2 x 0.5 x 682 = 682 litres/hr
5. Covert loss to m^3 per hour by multiplying by 10^{-3}.	5. 682 x 10^{-3} = loss rate of $0.682m^3$ per hour.
6. Estimate storage room size in m^3	6. 4m x 5m x 3m storage room = $60m^3$

7. Estimate the number of air exchanges per hour in the storage room. For rooms with no forced ventilation a figure of 0.4 is recommended.	7. No forced ventilation, air exchange value of 0.4 used.
8. (Rate of release) / (Number of air exchanges per hour x room volume) = **% change in ambient oxygen levels**. Normal ambient oxygen levels are taken as 20.9%.	8. (0.682) / (0.4 x 60) = ~0.028 or 2.8%. This is equivalent to a change in ambient oxygen content from 20.9% to 20.3%. This would give no cause for concern during storage operations.
9. When topping up flasks can assume a loss rate of 10% for each volume of liquid, ***y***, decanted. So rate of loss during topping up in m^3/hr equals (***y)***(0.1)(expansion coefficient)(10^{-3}).	9. Both Dewars are topped up daily using 24 litres of liquid nitrogen in order to take account of above static losses. Topping up loss rate therefore = (24)(0.1)(682)(10^{-3}) = 1.637 m^3 of nitrogen gas lost.

10. Add loss due to topping up activities to static losses from flasks to get overall level of cryogen release. If total rate of change is ≥5% then forced ventilation or ongoing oxygen depletion monitoring may be required. A relative change in ambient oxygen level of 5% applied to the standard ambient oxygen level of 20.9% equates to a drop of approximately 1% ambient oxygen level.	10. Change in % ambient oxygen levels equals loss during decanting plus static losses (refer to step 8 above) = (1.637 / 0• +60)+(0.682) / (0.4 x 60) = 0.055 or ~5.5%. This equates to a change in ambient oxygen content from 20.9% to 19.8%. This is getting close to a sub optimal oxygen level so during topping up activities increased ventilation and / or ongoing ambient oxygen level monitoring could be required. • We assume that the decanting procedures takes significantly less than one hour and no air exchanges take place during the decanting process, therefore the value of this parameter (number of air exchanges per hour) is taken as zero and the full room volume is used as the divisor here.