Handling, Transport and Storage of Cryogens

1. Purpose

This guideline details the procedures to follow in handling, transporting and storing of cryogens in the laboratory in order to minimise risks associated with the disposal of laboratory waste. It primarily discusses liquid nitrogen and dry ice, which are commonly used in the Department of Chemistry laboratories.

2. Scope

This procedure applies to all laboratory personnel within the Department of Chemistry who use dry ice, liquid nitrogen or other cryogens within the workplace.

3. References

AS 1894-1997: The storage and handling of non-flammable cryogenic and refrigerated liquids

AS 1894-1997/Amdt No. 1-1999: The storage and handling of non-flammable cryogenic and refrigerated liquids

OHS048.2 Laboratory Safety Guidelines - http://staff.uow.edu.au/ohs/

BOC guidelines - http://www.bocgases.ca/newsite_eng/aboutboc/safety/cryogenic/cryogeniclig.html

4. Definitions

Dewar - Purpose built cryogenic liquid holding/transporting vessels which are insulated, vacuum-jacketed pressure vessels. Larger volume models come equipped with safety relief valves and/or rupture discs to protect the cylinders from pressure build-up. These containers operate at pressures up to 350 psig and have capacities between 80 and 450 litres of liquid. A cryogen may be withdrawn as a gas by passing liquid through an internal vaporizer or as a liquid under its own vapour pressure.

Dry Ice - Solid Carbon Dioxide

5. Procedure

5.1. Physical Properties of commonly used cryogens

Liquid nitrogen is inert, colourless, odourless, non-corrosive, non-flammable liquid and extremely cold (Boiling Point @ 1 atm: -195.8°C, 77°K). It has a similar appearance to water. Nitrogen gas makes up the major portion of the atmosphere (78.03% by volume). Nitrogen is inert and will not combust, however, it is not life supporting. Volume of expansion liquid to gas (at 15°C, 1 atm.) is 682 , i.e. very small amounts of liquid convert to very large amounts of gas.

Dry Ice is a white solid with a Melting Point -56.6°C, Sublimation Point -78.5°C. Volume of expansion from solid to gas ~900.

5.2. Hazards, Risks and Controls

Apart from being unable to condense oxygen, hazards associated with solid carbon dioxide are similar to those described for liquid nitrogen *i.e*. temperature related and vapour related. In operation, similar precautions should be taken against cold burns and asphyxiation.

Hazard	Risk	Control *
Cold Burns	Liquid nitrogen can cause severe frostbite and cold burns if there is direct skin contact. The hazard level is comparable to that of handling boiling water. The low temperature of the vapour can cause damage to	Avoid direct contact with liquid. Never touch an uninsulated pipe or vessel. Never put any part of your body in

	softer tissues, eyes and lungs after short exposure times such as during splashing. Unprotected skin can freeze and adhere to liquid nitrogen cooled surfaces causing tearing on removal.	front of a liquid nitrogen supply.
Asphyxiation	Liquid N2 can rapidly be converted into gas (1 litre of liquid nitrogen evaporated to form 687 litres of nitrogen gas). In poorly ventilated areas this nitrogen gas can easily decrease the percentage of oxygen in air leading to the danger of asphyxiation. When the oxygen concentration in the air is sufficiently low, a person can become unconscious without sensing any warning symptoms, such as dizziness. %oxygen in atmosphere <10% brain damage and death <18% dangerous <20% entry not recommended	**Ventilation**. Always handle liquid N2 in well-ventilated areas to prevent excessive concentrations of gas. If such are not available then oxygen monitoring should be fitted and great care exercised to avoid spills. Note: The cloudy vapour that appears when liquid N2 is exposed to the air is condensed atmospheric moisture. The gas itself is invisible.
Pressure.	Liquid Nitrogen boils off very quickly. Pressure can quickly build up in a sealed unit with risk of explosion.	Do not put liquid Nitrogen in closed vessels that cannot withstand the pressure. No closed thermos flasks.
Thermal stress caused by freezing of materials	Materials frozen in liquid Nitrogen may change characteristics e.g. soft materials such as rubber and plastics become brittle and may shatter unexpectedly. Damage can be caused to containers because of large, rapid changes of temperature. Be aware of the potential risk of exploding vials when immersed in liquid Nitrogen, or thawed.	Pour liquid nitrogen only into containers suitable for holding liquid nitrogen (i.e. not glassware). Always place materials slowly into liquid nitrogen. Containers should be filled slowly to minimise thermal shock.
Condensing Oxygen	Liquid Nitrogen can condense oxygen from the atmosphere in containers of liquid nitrogen or vessels cooled by liquid nitrogen. This can be extremely hazardous because of the pressure rise on the slightest degree of warming above the boiling point of oxygen (-183°C) and the possibility of explosive reaction with oxidisable (organic) material.	When liquid cryogens or dry ice are used to cool traps attached to vacuum pumps, these traps must be emptied immediately after use. Never leave cold traps immersed in the cryogen. It is recommended that whenever possible some other coolant is used *e.g*. solid carbon dioxide/liquid traps or baths - the preferred liquids for such baths are isopropanol or glycols. It is recommended that such baths be used in preference to liquid nitrogen when long term storage is envisaged.
Rapid Boiling /	Rapid boiling and splashing of	Always perform these operations

Splashing of Liquid nitrogen	liquid nitrogen always occurs when charging a container at room temperature or when inserting objects into the liquid.	slowly to minimise boiling and splashing. Use tongs to withdraw objects immersed in the liquid, and take care where both tongs and object are then placed. (Note that objects soft and pliable at room temperature usually become very hard and brittle at cryogenic temperatures).

* Personal Protective Equipment should be worn at all times when handling cryogens.

5.3. Personal Protection

The following personal protection is recommended when handling liquid nitrogen or other cryogens.

- **Face Shield:** The eyes and face are best protected from splashing liquid by a full face shield. Safety glasses without side shields do not give adequate protection.
- **Gloves:** At the very least always wear loose-fitting leather gloves. The gloves should fit loosely, so they can easily be removed if liquid nitrogen spills or splashes into them. Thermal protective gloves specifically designed for cryogenic use with close fitting ribbed cuffs will prevent this from happening. Also always wear suitable protective gloves when handling anything that has been in contact with liquid nitrogen.

  Do not put gloved hands directly into liquid nitrogen. Insulated gloves are not made to permit the hands to be put into a cryogenic liquid. They will only provide short-term protection from accidental contact with the liquid.
- **Shirt:** Arms should not be exposed - wear long-sleeved shirt or laboratory coat.
- **Trousers:** These are to be worn outside the shoes to prevent the possibility of liquid spilling into shoes. Preferably without cuffs.
- **Shoes:** Enclosed footwear, preferably safety shoes/boots, must be worn whilst handling liquid nitrogen.

5.4. Training

New users of liquid nitrogen should receive instruction in its use from experienced members of the academic or technical staff to ensure they have been shown the correct way to handle and transport liquid nitrogen.

5.5. Handling

5.5.1. Dispensing Liquid Nitrogen from a Dewar

- The door MUST be kept FULLY OPEN while dispensing liquid nitrogen from a Dewar and persons filling must be in constant attendance to the filling operation.
- Do not hold the vessel with unprotected hands while filling.
- Nitrogen is to be dispensed only into smaller containers which have carrying handles.
- Never overfill containers. Spillage damages flooring and could cause injury.
- Have the height of the container so that the delivery is immediately at the mouth of the receiving vessel. (I.e., do not allow the liquid nitrogen to fall through a distance to reach the receiving vessel.)
- Use a filling funnel when pouring liquid nitrogen into a Dewar or small container.
- Use only the stopper supplied with the Dewar. Inadequate venting can result in excessive gas pressure, which can damage or burst a container.
- Never plug small containers of liquid nitrogen; cover them loosely when not in use to prevent accumulation of moisture and formation of ice.

5.5.2. Using Liquid Nitrogen in the Laboratory

- Liquid nitrogen will condense oxygen from the air. This is most alarmingly demonstrated if a person leaves his/her vacuum pump's coldfinger in a Dewar of liquid nitrogen overnight. In the morning the coldfinger will contain **LIQUID OXYGEN** up to the level of the nitrogen in the Dewar.
- Never dispose of liquid nitrogen in a confined area or pour it down the sink.
- Some instrumentation or laboratories may have local rules for the use of cryogens which should be followed.

5.6. Transport

5.6.1. Small Volumes

Inside buildings, from room to room, the best transport of liquid nitrogen (4 L and less) is to use a small Dewar which has carrying handles and a loose fitting lid or vent. This will allow the gas produced from the liquid boiling off at room temperature to escape.

Liquid nitrogen must never be stored or transported in sealed containers. Thermos flasks with tight fitting lids are not suitable,

5.6.2. Larger Volumes

Larger volumes of liquid nitrogen should be transported using "onion" (25 litre) Dewars and "transport" Dewars (40litre) which are on wheels and have pressure relief valves or pressure venting lids.

Lifting and carrying full liquid nitrogen Dewars, >25 litre is a two-person task, and should not be carried out alone. The weight of a full 25 litre Dewar is approximately 28kg. (One litre of liquid nitrogen weighs approximately 0.8kg)

For transport of large nitrogen Dewars outside, over pavers and walkways a specialised trolley should be used. Stay completely clear of grates, large cracks, and/or uneven portions of the pavement, and any other hazards which could catch a wheel and cause tipping.

5.6.3. Use of a Lift in the transport of cryogens

No personnel should accompany a cryogen Dewar in a lift. There is an asphyxiation risk from boil off in an enclosed space, especially if the lift breaks down. It is recommended that two people work together to transport liquid nitrogen via the lift.

- One person must be stationed on the relevant floor to receive the liquid nitrogen Dewar when the lift arrives.
- The second person places the Dewar in the lift, selects the floor/level and exits the lift before the doors close. The cryogen must travel unaccompanied.
- The first person removes the Dewar when it arrives.

5.7. Storage

Liquid nitrogen, greater than 1 litre, must never be stored in areas other than the approved Departmental storage facilities. The liquid nitrogen storage area should be constantly ventilated.

Liquid nitrogen Dewars have loose fitting lids or vents to allow the gas produced from the liquid boiling off at room temperature to escape. Dewars with the withdrawal device attached are equipped with pressure relief devices to control internal pressure. Under normal conditions, these containers will periodically vent product. Do not plug, remove, or tamper with any pressure relief device. Vents should be regularly checked to prevent ice build-up caused by water vapour.

Containers should be handled and stored in an upright position. Do not drop, tip, or roll containers on their sides.

5.8. First Aid and Emergency Procedures

5.8.1. Cold Burns

- For brief, localised contact with cold material - flush the area with tepid water for 15 minutes. Water is used because of its high heat capacity. Obtain First Aid assistance.
- Prolonged contact will cause skin to blister and will require medical treatment. Call a First Aider or medical assistance. In the case of frostbite water is sprayed on the site for at least 15 minutes and a sterile dressing is applied.
- If any liquefied gas contacts the skin or eyes, immediately flood that region of the body with large quantities of cold water, and then apply cold compresses. Seek First Aid.
- When handling liquefied gases, a supply of cold water should be available in case accidental splashing occurs.

5.8.2. Asphyxiation

If you suspect that someone is suffering from asphyxiation, do not enter the affected area alone first call for help. If the victim is unconscious, call 4555 first and then call for a First Aider. Remove the casualty to the fresh air. Keep casualty warm and rested.

If any of the following symptoms appear in situations where asphyxia is possible:

- Rapid and gasping breath
- Rapid fatigue
- Nausea
- Vomiting
- Collapse or inability to move.
- Unusual behaviour

However, if the casualty is in a confined space do not attempt to rescue affected persons. In confined spaces or where oxygen deficient atmospheres may be present rescue should only be made by those trained in the use of breathing apparatus and confined space entry procedures. This excludes all personnel within the Department of Chemistry. The Fire Brigade should be called in all instances where a trapped person requires rescue.

Liquid nitrogen should normally be used only in a well-ventilated area. However, there may be occasions eg transport of Dewars in lifts, when this may not be possible. To avoid the danger of oxygen depletion, the following taken from webpage http://www.tlchm.bris.ac.uk/safety/lnitcry.htm should noted:

- **Safe limit in an unventilated space**: Calculate the room volume in m^3 and the max volume of nitrogen in m^3 (this can be found from the volume of liquid in litres x0.7). If the volume of nitrogen amounts to >0.15 of the room volume,
- **Spillage during filling:** during filling assume that 10% of the final volume may be spilled.
- **Loss during storage:** the boil off loss from a 5l Dewar is expected to be 0.2l per day.

5.8.3. Cryogen Spill

If there is a large spillage of liquid nitrogen, evacuate the area and call for help. Follow the recommended Emergency Procedure for major spills of toxic material.

6. Reference Personnel

All changes to this document shall be referred to the Departmental Safety Committee prior to implementation.

7. Documentation

ChemSWP	Dispensing Liquid Nitrogen from 40L storage Dewar.