UNIVERSITY
OF WOLLONGONG
AUSTRALIA

UOW WHS Unit

STORAGE, TRANSPORT & HANDLING OF CRYOGENS GUIDELINES

Contents

Hardcopies of this document are considered uncontrolled. Please refer to UOW website or intranet for latest version.

1 Purpose

This document provides advice on the safe storage, handling and transportation of cryogens such as liquid nitrogen, liquid helium and Dry Ice.

2 Scope

These guidelines apply to all authorised users who have contact with cryogens both at the University of Wollongong and on field trips.

3 Definitions

Asphyxiation	Breathing difficulties (suffocation), loss of consciousness and eventual death caused by an inadequate supply of oxygen to the body.
Boil off	The excessive release of vapours created due to the ambient heat input (while maintaining constant pressure in the storage vessel).
Cryogen	A liquid or solid substance that is extremely cold and generates a large volume of gas at atmospheric temperature and pressure.
Dewar	A purpose-built insulated, vacuum-jacketed pressure vessel used for storing / transporting cryogenic liquids.
Dry Ice	Solid carbon dioxide (CO_2).
Room Temperature	A temperature range between 15° to 25°C that is suitable for human occupancy and at which laboratory experiments are usually performed.
SDS	Safety Data Sheet.
Sublimation	The process of transition from the solid phase to the gas phase without passing through an intermediate liquid phase.
Venting	The discharge of gas vapours out of a cryogenic storage container.

4 Introduction

The word ‘cryogenic’ means ‘producing, or related to, low temperatures’. Cryogens exist in two forms - liquids (e.g. liquid nitrogen) and solids (e.g. Dry Ice). Different cryogens become liquids under different conditions of temperature and pressure, whilst others will sublimate, but all have two properties in common: they are extremely cold, and small amounts of substance can expand into very large volumes of gas. All cryogens are gases at atmospheric temperatures and pressures.

Cryogenic liquids are liquefied gases that are kept in their liquid state at very low temperatures (i.e. boiling points below -150°C). For example, liquid nitrogen is inert, colourless, odourless, non-corrosive, and non-flammable liquid with a boiling point at -195.8°C at atmospheric pressure. It has a similar appearance to water.

Cryogenic solids are solidified gases that are kept in their solid state at very low temperatures. For example, solid carbon dioxide or Dry Ice is a white solid with a melting point -56.6°C and sublimates at -78.5°C. The resultant gas is an asphyxiant that is heavier than air. Dry Ice is manufactured in the form of pellets, slices or blocks and may be supplied loose or in insulated containers.

NOTE: Use the Hierarchy of Control

When working with hazardous cryogens, such as liquid nitrogen, use the hierarchy of control to eliminate or reduce the risk if possible. If an alternative cryogen that poses a lower risk can be substituted, then it should be used.

5 Risks associated with using cryogens

Everyone who works with cryogens must be aware of their hazards and know how to work safely with them.

5.1 Asphyxiation

<u>Description</u>

Cryogens are constantly producing gas. During storage the average loss from a 5 L Dewar is approximately 0.3 L per day. Whilst filling a Dewar assume that 10% of the final volume may be spilled or lost.

Even small amounts of cryogenic solids / liquid can rapidly expand into very large volumes of gas. For example, at room temperature 1 L of liquid nitrogen expands to 695 L of nitrogen gas, whilst 1 kg of Dry Ice expands to 900 L of carbon dioxide gas.

In poorly ventilated areas these cryogenic gases can easily decrease the percentage of oxygen in air and cause asphyxiation. Entering a room with reduced oxygen (< 19.5%) is dangerous. NEVER enter a room with reduced oxygen concentration as:

- < 11% oxygen results in unconsciousness without any warning symptoms (e.g. dizziness).
- < 10% oxygen can lead to brain damage and death.

WARNING
Inhalation of pure nitrogen atmosphere produces immediate loss of consciousness; death follows unless oxygen and breathing can be quickly restored.

CAUTION
As cryogenic gases are colourless and odourless they are difficult to detect.

<u>Controls</u>

Before handling cryogens or entering an area where they are stored, always check there is adequate ventilation - refer to the Appendix for conversion ratios of cryogenic gas production.

If such ventilated areas are not available, then oxygen monitoring equipment should be fitted in line with the Air and Health Monitoring Guidelines and Local WHS Monitoring Equipment Form.

Avoid spills - refer Section 11.

Oxygen monitor

5.2 Pressure build up / Explosions

<u>Description</u>

Cryogenic liquids exhibit large volume expansion ratios which can cause rapid pressure changes (as described above).

In addition, all cryogens can condense sufficient moisture from the air to block the opening in storage vessels. This condition can result in an explosion caused by the build-up of trapped vapour in the vessel.

<u>Controls</u>

It is critical to contain these liquids in insulated Dewars with pressure relief valves - refer Section 6.1.

DO NOT put cryogens in closed vessels that cannot withstand the pressure (e.g. capped Thermos flasks).

Ensure safety valves are operational on withdrawal devices - refer Section 8.1.1.

5.3 Toxic hazards

<u>Description</u>

The gases released from cryogens can cause specific adverse health effects. For example, a release of carbon monoxide gas can cause death almost immediately.

<u>Controls</u>

Refer to the SDS for information about the toxic hazards of a particular cryogen.

5.4 Eye injury

Description

Brief exposures to cryogenic substances that would not affect the skin on the face or hands can damage delicate tissues such as the eyes.

Liquid cryogens can splash (possibly in eyes) while being poured. Additionally, rapid splashing and boiling of cryogens occurs when dispensing into a warm container (e.g. at room temperature) or when inserting warm objects into the cryogenic liquid.

Flying chunks of frozen debris or cryogen could also cause eye injury.

Controls

To protect the eyes when handling cryogens, always wear safety goggles. Where possible or when transferring large volumes (>4 L), use a face shield - refer Section 10.2.

Always perform operations slowly to minimise boiling and splashing.

5.5 Cold burns / Frostbite

Description

Cryogenic substances and their associated cold vapours and gases can rapidly freeze human tissue producing effects on the skin similar to a thermal burn. Prolonged exposure of the skin or contact with cold surfaces can cause frostbite. The skin appears waxy yellow. There is no initial pain, but as the frozen tissue thaws intense pain might be experienced.

Metals and liquids below -20°C can cause pain, blistering, tissue loss and shock, especially when trapped against the skin by loose-fitting gloves or apparel. The hazard level is comparable to that of handling boiling water.

Unprotected skin can stick to metal that is cooled by cryogenic substances. The skin can then tear when pulled away. Even non-metallic materials are dangerous to touch at low temperatures.

Prolonged breathing of extremely cold air may damage the lungs, while short exposure may provoke an asthma attack in susceptible people.

Cryogenic burn (*source University of NSW)*

Controls

Avoid direct contact with a cryogen or objects frozen using a cryogen. DO NOT grasp cold metal surfaces with unprotected skin. Use cryogenic gloves with tongs and dippers to withdraw or immerse objects - refer to Section 10.1 for further information.

When working with cryogens remove watches, wrist-bands or bracelets which may trap liquid cryogens close to the skin.

For information on treatment - refer Section 11.2.

Cryogenic gloves

5.6 Thermal stress to materials

Description

Materials frozen in cryogens may change characteristics (e.g. soft materials such as rubber and plastics become brittle and may shatter unexpectedly).

Be aware of the potential risk of explosion when immersing objects in liquid cryogens or as the objects thaw.

Damage can be caused to cryogenic storage and transport vessels because of large, rapid changes of temperature.

Controls

Cryogenic storage and transport vessels should be filled slowly to minimise thermal shock - refer Section 8.2.

Pour liquid cryogens only into suitable storage and transport vessels (i.e. not glassware) – refer Section 6.1.

Always place materials slowly into cryogens - refer to Section 0.

5.7 Increased flammability from oxygen enrichment

Description

Oxygen is not flammable, but it vigorously accelerates and supports combustion. Substances that burn in air, will burn more vigorously in oxygen.

Cryogenic liquids with a boiling point below that of liquid oxygen (i.e. nitrogen and helium) have the ability to condense oxygen out of the air if exposed to the atmosphere.

If the atmosphere is enriched with oxygen then the likelihood and potential intensity of fires is increased. Combustible materials not usually combustible in air will burn fiercely in an enriched atmosphere. Clothing saturated with oxygen will burn vigorously with potentially fatal results. Violent reactions (e.g. rapid combustion or explosions) may also occur if the system or process is not compatible with liquid oxygen.

Controls

DO NOT permit liquid oxygen or oxygen rich atmosphere to come into contact with organic materials or flammable substances of any kind.

It is recommended that whenever possible some coolant should be used in preference to cryogens (e.g. baths containing isopropanol or glycols).

When liquid cryogens or Dry Ice are used to cool traps attached to vacuum pumps, these traps must be emptied immediately after use.

DO NOT leave cold traps immersed in the cryogen.

DO NOT release the vacuum of any evacuated vessel while liquid nitrogen is present.

6 Storage

6.1 Cryogenic storage vessels

Liquid cryogenic storage vessels (also known as Dewars) are specialised vacuum flasks that may take several different forms including open buckets, flasks with loose-fitting stoppers and self-pressurising tanks. They are designed to reduce the risk associated with storing liquid cryogens.

All Dewars have walls constructed from two or more layers, with a high vacuum maintained between the layers. This provides very good thermal insulation between the interior and exterior of the Dewar, which reduces the rate at which the contents boil off. Precautions are taken in the design of Dewars to safely manage the gas which is released as the liquid slowly boils.

The simplest Dewars (non-pressurised) allow the gas to escape either through an open top or past a loose-fitting stopper to prevent the risk of explosion. More sophisticated Dewars (pressurised) trap the gas above the liquid and hold it at high pressure. This increases the boiling point of the liquid and allows it to be stored for extended periods. Excessive vapour pressure is released automatically through safety valves. The method of decanting liquid from a Dewar depends upon its design - refer Section 0. Simple Dewars may be tilted, to pour liquid from the neck. Self-pressurising designs use the gas pressure in the top of the Dewar to force the liquid upward through a pipe (spear) leading to the neck (known as dispensing).

There are several types of storage vessels for cryogenic liquids commonly used on campus and in the field –see following examples.

Bulk Storage Container

(A pressurised large volume storage vessel)

Hardcopies of this document are considered uncontrolled. Please refer to UOW website or intranet for latest version.

Non-Pressurised

Pressurised

Carrier Vessel

NEVER store or transport liquid cryogens in sealed containers. For example, Thermos flasks with tight fitting lids are not suitable as this does not allow the gas produced from the liquid boiling off at room temperature to escape.

Cryogenic storage vessels are generally designed to operate with little or no internal pressure. Ice can form on the inside of the Dewar if it is left open to the air for extended periods. This can be extremely dangerous as the openings of the Dewar can become blocked, leading to a pressure build-up, and the risk of explosion. Therefore any malfunction, such as an ice plug, that prevents the vessel from venting may build up an internal pressure that may damage or burst the vessel.

Working Vessels
(A small stainless steel Dewar / flasks which have a carrying handle / loose fitting lid or vent)

6.2 Care of cryogenic storage vessels

Cryogenic storage vessels require careful maintenance to ensure they are in good working order. They should always be handled with care and never be used if they have suffered damage or impact.

In an incident in 2006 at Texas A&M University, the pressure-relief devices of a tank of liquid nitrogen were sealed with brass plugs. As a result, the tank failed catastrophically and exploded.

Damaged tank

Always

- Adequately label vessels.
- Inspect vessels for damage prior to use (if any found, tag 'Out of service').
- Check the pressure relief valve. The pressure relief value should be replaced every 5 years. The pressure gauge should be checked by the supplier or qualified pressure testing organisation every 5 years to ensure its functionality. The O-ring seal should be checked regularly and replaced if any sign of damage is evident.
- **For testing - contact the supplier or qualified pressure testing organisation (NOTE:** As per *AS/NZS 3788:2006* all pressure relief valves on cryogenic storage vessels should be tested at intervals not exceeding 5 years, as soft seats tend to harden and stick.**)**

Never

- Cover or plug the entrance openings of depressurised vessels.
- Drop or allow any vessel to fall over.
- Subject the vessel to sharp impact or severe vibration.
- Use any non-standard fitting, stopper, connection or adaptor.
- Store vessels in unventilated, high traffic or congested areas.
- Store vessels near ignition sources.
- Store cryogens, greater than 1 Litre, in areas other than approved storage facilities.

Incorrect storage of Dewars

- Walk, roll or drag a cryogenic vessel across the floor or rough/rocky surfaces
- Plug, remove, or tamper with any pressure relief device. Vents should be regularly checked to prevent ice build-up caused by water vapour. Under normal conditions, these containers will periodically vent product.

6.3 Storage of Dry Ice

Many factors can affect the rate at which Dry Ice sublimes from solid state into gaseous carbon dioxide. These include:

- The ambient temperature and humidity.
- The quality of the storage container.
- The number of times the container is opened and closed.

The better the insulation, the slower the sublimation rate and the longer the quality of the product will be maintained.

A minus 80^{o} C freezer is appropriate for the storage of Dry Ice.

Dry Ice in storage freezer

<u>Always</u>

- Store Dry Ice in an insulated container.
- Keep the container lid closed when not in use.
- Keep proper air ventilation wherever Dry Ice is stored. DO NOT store Dry Ice in unventilated rooms, cellars, automobiles or boat holds. The sublimated carbon dioxide gas will sink to low areas and replace oxygenated air. This could cause suffocation if breathed exclusively.
- Avoid working with Dry Ice for extended periods of time as continued exposed higher than normal concentrations of CO_2 can cause adverse health effects.
  NOTE: *The maximum exposure of CO_2 over an 8 hour period in a 5 day week is 5000 parts per million or 0.5%.*

<u>Never</u>

- Put Dry Ice in sealed containers or bottles. Injury may result when the container bursts from the build-up of pressure as the CO_2 converts from solid to gas form.
- Expose Dry Ice to high ambient temperatures unnecessarily as this increases the sublimation rate and thereby the risk of creating a carbon dioxide-rich atmosphere.
- Store Dry Ice in a household refrigerator freezer. The extremely cold temperature will cause your thermostat to turn off the freezer.

7 Transportation

Transporting cryogens in an enclosed space such as a lift or vehicle cabin is extremely hazardous. Under no circumstance should a person accompany cryogens in a confined space.

Secure storage vessels (such as Dewars) firmly to limit movement when transporting to the field or refilling.

When using depressurised vessels, tape the cap down loosely to prevent it from falling off whilst still allowing ventilation.

Dewar with cap taped in place

7.1 Packaging cryogens for transport

7.1.1 Liquid cryogens

For transport small volumes of liquid cryogens (4 L and less) inside buildings use carrier and working vessels - refer Section 6.1.

Larger volumes of liquid cryogens (greater than 4 L) should be transported using Dewars which are on wheels and have pressure relief valves or pressure venting lids.

Lifting and carrying full liquid cryogen Dewars (>10 L) is a two-person task and should not be carried out alone. (1 L of liquid nitrogen weighs approximately 0.8 kg. Therefore 10 L Dewar is 15 kg).

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

7.1.2 Dry Ice

The most significant risk incurred when transporting Dry Ice is an unsafe atmosphere due to sublimation and spillage. The form of Dry Ice will affect the rate of sublimation, with pellets sublimating faster than blocks. Approximately 10% of the Dry Ice will sublimate every 24 hours to produce carbon dioxide gas. Carry Dry Ice in a well-insulated container such as a Styrofoam esky (with a loose fitting lid to allow pressure release).

7.2 Transporting cryogens in vehicles

When transporting cryogens in vehicles, safety precautions need to be followed.

7.2.1 Liquid cryogens

ONLY transport liquid cryogen storage vessels on an open utility back, utilities with separate compartment or a trailer. ALWAYS carry a SDS in the vehicle.

7.2.2 Dry Ice

AVOID transporting Dry Ice in the cab of a truck or the passenger compartment of a car. Preferably transport Dry Ice in vehicles where the driver's cab is isolated from the load compartment. If it is transported inside a car or van, then consider the following:

- Minimise the quantity of Dry Ice being transported.
- Use a well-insulated container such as a Styrofoam esky.
- Plan to pick up the Dry Ice as close to the time it is needed as possible and minimise the length of time the product is held in an enclosed space.
- Reduce the temperature of the load compartment to as low as comfortable to minimise sublimation.
- ALWAYS ensure that the heating / vehicle air supply is switched to draw in 'fresh air' from outside the vehicle or open the window.
- Place the container in the boot.
- ALWAYS carry a Dry Ice SDS in the vehicle.
- ALWAYS unload product as soon as possible at the end of the journey and move to a suitable storage location.

7.3 Transporting cryogens in a lift

A minimum of two persons should work together to transport these substances via a lift, as outlined below:

- The first person places the storage vessel in the lift. A Do Not Enter Lift Sign should also be placed in the lift to inform people what the substance is and that they should not to travel in the lift whilst the substance is being transported.
- The first person then selects the floor/level and exits the lift before the doors close. **(The cryogen must travel unaccompanied to the destination floor).**
- The second person must be stationed on the destination floor to receive the storage vessel.
- The second person removes the storage vessel and signage when the lift arrives.

Transporting cryogens in lift

NOTE: NEVER accompany cryogens in a lift as there is an asphyxiation risk from boil off in an enclosed space, especially if the lift breaks down.

8 Handling

8.1 General requirements

Only custom-made cryogenic equipment should ever be used to handle cryogenic products.

- Always use cryogens in a well-ventilated area, especially when filling a warm container / transfer tube or inserting a warm object, as large volumes of cryogenic gas are evolved.
- Always fill warm Dewars slowly to reduce temperature shock effects and to minimise splashing.
- Use only containers or fittings (pipes, tongs etc.) that have been designed specifically for use with cryogenic liquids as non-specialised equipment may crack or fail. In particular, DO NOT use common vacuum flasks designed for food/drink as they can implode resulting in flying glass fragments.

Protect all glass Dewars against the possibility of flying glass fragments, arising from failure by mechanical or temperature stress damage, by covering or sealing all exposed glass either in an insulated metal can or by wrapping with adhesive tape.

8.1.1 Pressuring Dewars

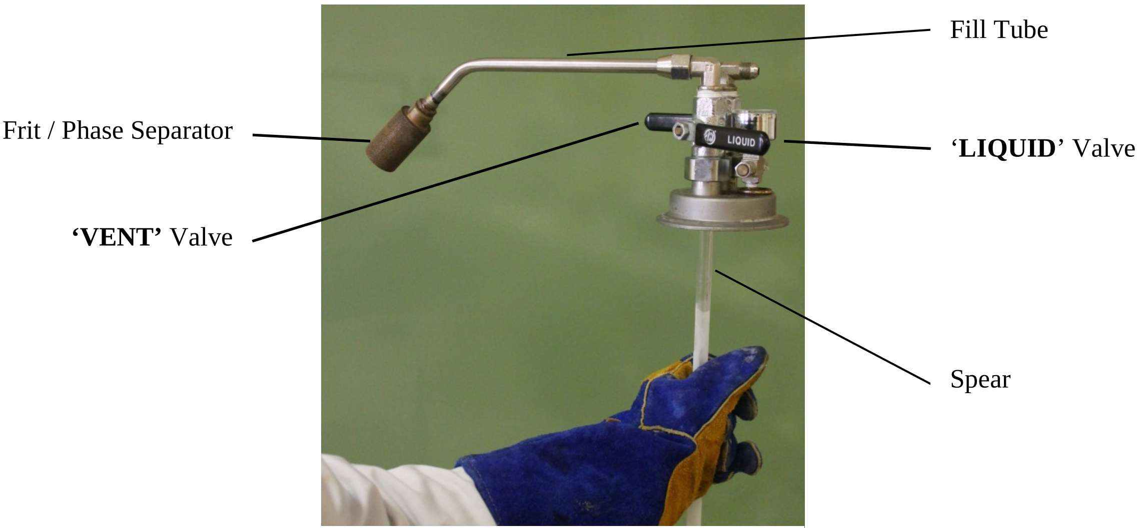
NOTE: This procedure should ONLY be performed by or under the supervision of trained and competent persons.

When pressuring Dewars the steps below should be followed:

- Ensure the spear is suitable for use (i.e. not blocked or damaged) before pressuring a Dewar.
- Ensure the vent valve is open and pointing away from the operator before placing in the Dewar.
- Insert the spear to minimise vapour generation.
- Ensure the band clamp is properly placed over the vessel and decanting spear.
- Ensure that the clamp tightening knob is secured and close the vent valve.
- Allow Dewar to slowly pressurise before dispensing.

Manual pressurisation of Dewars should ONLY be carried out if the cryogen is required immediately after change over:

- Nitrogen supply from taps can be used to pressurise Dewars if required.
- Only use a low flow of gas from the tap.
- Turn the gas tap off after dispensing the desired amount of the liquid cryogen.



Liquid drawing device

8.1.2 De-pressurising Dewars

When de-pressuring Dewars the steps below should be followed:

- Ensure the vent valve is pointing away from the operator and any fittings are disconnected.
- Slowly open the vent valve on the decanting spear.
- DO NOT remove the band clamp until the pressure gauge shows below 2 psi.

Place a cap on the Dewar in preparation for re-filling.

De-pressuring a Dewar

8.2 Transfer from a bulk storage Dewar

This procedure should always be carried out in a well-ventilated area.

- Two competent people are required to perform this task at all times – one filler and one observer.
- Persons filling must be in constant attendance to the filling operation.
- The correct PPE should always be worn; face mask, solid cover footwear, lab coat OR long sleeves and pants, insulated gloves - refer Section 10.
- Always keep a 5 L bucket of cold water on hand to defrost any components that may ice up during the decanting procedure.
- Take the transporting Dewar to the bulk Dewar - refer Sections 7.1 and 7.3.
- If the bulk Dewar is not easily accessible or needs more ventilation whilst transferring, ensure that the bulk Dewar decanting valve is closed and then move the bulk Dewar to a more accessible or well ventilated position.
- Check the decanting hose for any sign of damage (e.g. kinks in hose, damage to braiding, broken/cracked connections). If damage is evident, DO NOT decant any liquid cryogen from the vessel with the damaged hose as any perforations will leak cryogen under pressure. Tag the Dewar 'Out of service' and report the incident in SafetyNet. Contact an authorised supplier, e.g. BOC or Taylor Wharton, to purchase a new decanting hose. Have a competent/authorised individual replace the decanting hose and remove 'Out of Service' tag before the Dewar can be used again. Close off the incident in SafetyNet.
- Whilst holding the decanting hose firmly, point it in a safe area and slowly open the decanting valve to purge the hose of any residual moisture. Close the decanting valve again.
- Place the decanting hose in the mouth of the transport Dewar (i.e. DO NOT allow the liquid cryogen to fall through a distance) and position it so that it won't fly out.
- Keep firm pressure down on the filling hose and slowly open the decanting valve on the bulk Dewar till liquid is flowing.
- A suitable flow is evident by a slow vapour trail coming from the mouth of the Dewar been filled.
- DO NOT open the decanting valve fully; this will create high pressure in the fill line. A slow flow is all that is required for safe transfer.
- Once the decanting process has begun, you can step back from the decanting hose.

Trolley for bulk storage Dewar

Decanting and vent valves

Check vapour trail

- Listen for the change in sound as the Dewar fills – a higher pitch indicates the Dewar is getting full. Reduce the flow rate at this point.
- Turn off the decanting valve from the bulk Dewar when the transport Dewar is full (i.e. when the liquid overflows out the top).
- Place the cap back on the transport Dewar.
- Ensure the decanting valve is closed and securely store the dispensing hose. Allow the hose and valve to defrost normally.
- If the decanting valve will not close fully, defrost the area using cold water and then turn the decanting valve to the closed position. If problems still occur, immediately contact the supplier (e.g. BOC).
- Wheel the bulk Dewar back into its storage position and remove PPE.
- Return the transport Dewar to its location - refer Sections 7.1 and 7.3.

Monitoring dispensing

8.3 Dispensing / Decanting liquid cryogens from a Dewar

ALWAYS:

- Be in constant attendance during the dispensing / decanting operation.
- Keep the door fully open if dispensing indoors. Use an appropriate vessel fitted with loose cap.
- Wear appropriate PPE - refer Section 10.
- Ensure receiving vessel is clean and dry.
- Allow time for boil off to recede during dispensing / decanting.

Loose fitting cap

NEVER:

- Overfill containers or allow spillage as spillage could damage flooring and cause injury - refer Section 5.5. (Only fill vessels to 75% to help contain boil off from spilling out).
- Pour liquid cryogens from a height above eye level.
- Allow the liquid cryogen to fall through a distance to reach the receiving vessel. (Pour directly into the mouth of the receiving vessel).

Incorrect method of decanting

8.3.1 Decanting from pressurised Dewars

- DO NOT stand in front of the outlet of a Dewar while decanting it.
- Ensure that the transfer vessel is over the metal frit before commencing decanting from a pressurised Dewar.
- Always open the ‘LIQUID’ valve slowly.

Decanting from pressurised Dewar

8.3.2 Decanting from un-pressurised Dewars (>10 L) with a tipping stand

- DO NOT stand in front of the outlet of a Dewar while decanting it.
- Ensure the transfer vessel is close to the Dewar lip.
- Slowly tip the Dewar over to ensure controlled transfer.

Decanting using a tipping stand

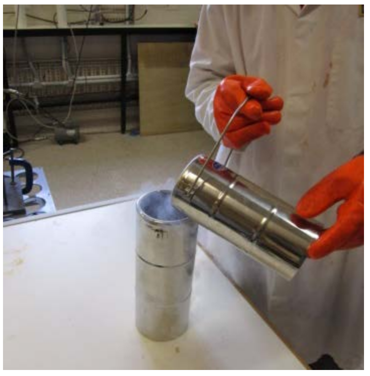
8.3.3 Decanting from small un-pressurised Dewars (<10 L)

- Ensure a firm grip is maintained on the Dewar.
- Place transfer vessel at a suitable height.
- Slowly tip the Dewar to ensure controlled transfer.

Decanting from small un-pressurised Dewar

8.3.4 Vessel to vessel transfer

- Applicable when transferring from a carrier vessel to a working vessel or vessels used for long term storage of samples.
- Make small slow additions to minimise boil off.
- Excess should be returned to an un-pressurised Dewar OR left in a clear and well-ventilated area to boil off and identified clearly to inform others of possible hazards.
- DO NOT pour liquid cryogens from a height above eye level.



Vessel to vessel transfer

8.4 Decanting using a funnel

A filling funnel should be used when pouring liquid from a small vessel into a small container.

- Ensure that vessel is below eye level, ideally shoulder height maximum.
- Use a funnel with specially formed base to allow boil off to escape freely.
- Ensure that the vapour from the boil off is not directed at the operator.
- Add in small slow additions so that boil off does not spill over.
- Allow vessel temperature to equilibrate before filling to top in order to minimise over flow.

Decanting using a funnel

Dispose of any excess liquid cryogen as per directions in Section 9.

8.5 Vacuum lines

Consider the following if you are using vacuum lines:

- Only half fill vessels before immersing glassware in them.
- If vessels require a cover ensure the cover allows ventilation. Check regularly for ice build-up.
- When topping up vessels make slow additions to minimise boil off.

Special considerations when using liquid nitrogen on vacuum lines:

- Vessels are to be supported on stands if they need to be elevated.
- DO NOT allow oxygen to condense in the vacuum line:
    - Ensure vacuum line is not leaking.
    - Remove liquid nitrogen vessel before releasing vacuum.
    - Isolate vacuum pump after vacuum line has reached room temperature.
    - Slowly bleed air into the system to minimise rapid pressure build up.

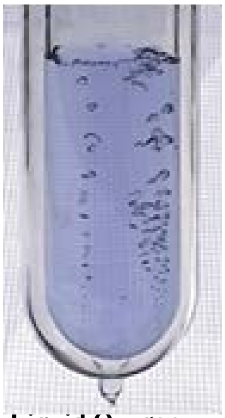
Vacuum line

8.6 Using liquid cryogens in the laboratory

Beware of the formation of liquid oxygen in cold-traps that are open to the air or the increase of liquid oxygen content in a flask of liquid nitrogen that has been cold for a long period. (Liquid oxygen has a blue water-like appearance). However, most liquid cryogen containers are closed except for a small neck area and the vapour issuing from the surface forms a barrier which keeps air away from the liquid thus preventing oxygen contamination.

Some instrumentation or laboratories may have local rules for the use of cryogens which should be followed.

NOTE: An explosion at UMIST was apparently caused by overcooling of Rotaflo taps which leaked and allowed oxygen to condense into a sample tube. Subsequent warming resealed the Rotaflo but blew the tube apart as the oxygen evaporated.



Liquid Oxygen

8.7 Using liquid cryogens in the field

As field applications are very similar to laboratory application the same handling requirements apply; however there could be additional hazards for your field location. Ensure that these risks are considered in your fieldwork risk assessments.

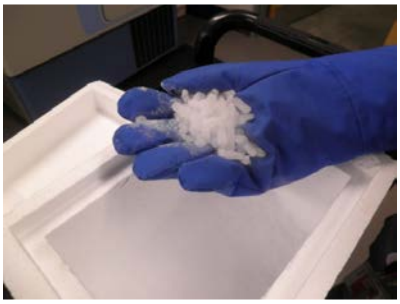
8.8 Handling Dry Ice

Dry Ice is extreme cold which makes the solid dangerous to handle without protection. If touched briefly it is harmless, but prolonged contact with the skin will freeze cells and cause injury similar to a burn (frostbite). Always handle Dry Ice with care and wear protective cryogen gloves whenever touching it.

While generally non-toxic, the release of gas from it can cause asphyxiation due to displacement of oxygen in confined locations.

Obtain Dry Ice in the form and size in which it will be used. NEVER cut or use a hammer to break a block of Dry Ice into smaller pieces.

DO NOT place on tile or laminated counter top, as the extreme cold will break the bonding agent holding the tile or laminated material in place. Use a wooden cutting board or piece of plywood is best.



Handling Dry Ice pellets

8.9 Working alone

Clearly, the risks of working with cryogens are greater when someone is working alone, especially outside normal working hours when it might be difficult to summon help. Staff should not handle liquid cryogens alone unless this is absolutely essential; whenever possible they should work in pairs.

Faculties should put special procedures in place if any staff are required to work out of hours, including instructions to be followed in case of emergencies. Inexperienced staff and students are especially at risk, and faculties should make it clear whether they are allowed to work alone or out of hours and, if so, what they may and may not do whilst unsupervised.

In all cases, a risk assessment is required for lone working.

NOTE: No medium, to high risk activities should be performed outside normal working hours i.e. 8 am – 6pm.

9 Disposal

Precautions should be taken in disposing of cryogens:

- Never dispose of cryogens in a confined area or pour it down the sink.
- Larger volumes should be vented outside. Small amounts can be vented in a fume-hood.
- DO NOT pour over the floor as this can damage property.
- DO NOT dispose of Dry Ice in areas accessible to the general public.

10 Personal Protective Equipment

Personal protective equipment is only designed to protect from incidental contact with a cryogenic substance. It does not provide adequate protection for long term exposure and must never be considered adequate enough to allow direct contact with or immersion in a cryogenic liquid. Protective clothing suitable for handling cryogens shall be provided with particular attention given to cryogenic gloves and eye/face protection.

10.1 Hand protection

Wear gloves made for cryogenic work (blue cryogenic gloves) or smooth leather welding type gloves without gauntlets (refer *AS 2161.5:2008 Occupational protective gloves – Protection against cold*).

Gloves should be loose fitting to allow for easy removal in the event any liquid is spilled inside the glove. Ensure that the sleeves of a laboratory coat are pulled down over the gauntlet of the glove to prevent any liquid from entering the glove.

Rubber gloves should not be used because they will harden instantly. If your hand is bent when the glove hardens, you may not be able to remove your hand.

A thin gas barrier forms between the skin and the cryogenic liquid when it is spilled on the skin. This will protect you unless the liquid hits you under force. This gas barrier is very cold and can also burn you.

To add or remove materials from cryogenic liquids, use non-metallic tongs or a dipper.

Dipper

10.2 Eye / face protection

When filling Dewars or transferring cryogenic liquids from one container to another, *AS 1337.1-2010 Personal eye protection – Eye and face protectors for occupational applications* recommends a full-face shield be worn.

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. Safety goggles will keep liquid out of eyes but leave face exposed. Cold liquids can hit your face and run under the glasses/goggles into your eyes. Additionally, face shields protect against the failure of glass apparatus (resulting in implosion) and brittle failure of items cooled by cryogens.

10.3 Clothing

Lab coat or overalls are advisable to minimise skin contact, also, wear trousers over shoe/boot tops to prevent shoes filling in the event of a spillage.

Wearing an apron made of leather or some other non-absorbent material is ideal, but not essential, when decanting large volumes of liquid cryogens from bulk Dewars into smaller Dewars. Arms and legs should not be exposed – a minimum requirement is that long sleeves and long pants be worn. Most clothing material will absorb spilled liquid cryogens, bringing the liquid close to the skin. Best practice is to also wear a laboratory coat or overalls.

10.4 Footwear

Enclosed footwear, preferably safety shoes/boots, must be worn whilst handling liquid cryogens. Leather will shed the spilled liquid. Ensure that pants are placed over boot/shoe tops to prevent any liquid entering the boots/shoes. Sneakers are typically made with absorbent materials which could draw liquid toward your skin.

Decanting using PPE

11 First aid and emergency procedures

Refer to the applicable SDS for First Aid information.

11.1 Asphyxiation

If you suspect that someone is suffering from asphyxiation, DO NOT enter the affected area alone. Call for help. If the victim is unconscious, call **University Security ext 4900** first and then call for a First Aider. Remove the casualty to the fresh air only if it is safe to do so. 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, a rescue should only be made by those trained in the use of breathing apparatus and confined space entry procedures.

11.2 Cold burns

For brief or 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 – refer Section 5.5. 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.

Treat Dry Ice burns the same as a regular heat burns. See a doctor if the skin blisters or comes off. Otherwise, if only red it will heal in time as any other burn. Apply antibiotic ointment to prevent infection and bandage only if the burned skin area needs to be protected.

11.3 Cryogen spill

If there is a large spillage of liquid cryogen (> 10 L):

- Evacuate the area and call for help.
- Provide additional ventilation if it is safe to do so.
- Quarantine the area.
- Follow the recommended Emergency Procedure for major spills of toxic material.
- DO NOT attempt to re-enter the area until it is declared safe to do so.

12 Training

Training in the Safe Storage, Transport and Handling of Cryogens is available through the Professional & Organisational Development Services (PODS). This course gives the participant knowledge of the risks associated with cryogens and how to manage those risks both generally and task specific. Theory and practical assessments are used to determine competency.

13 Related documents and references

- AS 1337.1-2010 Personal eye protection – Eye and face protectors for occupational applications
- AS 1894-1997 The storage and handling of non-flammable cryogenic and refrigerated liquids
- AS 2161.5:2008 Occupational protective gloves – Protection against cold
- AS 2030.4:1985 The verification, filling, inspection, testing and maintenance of cylinders for the storage & transport of compressed gases.
- AS/NZS 3788:2006 Pressure equipment – In-service Inspection
- BOC Guide to Safe Handling of Cryogenic Liquefied Gases.
- SDS Liquid Nitrogen
- Care with Cryogenics – Inert Gases – Nitrogen Liquid & Gaseous Laboratories, Myron Sawka, BOC Trainer
- Safety with Liquid Gaseous Nitrogen & Dewar Decanting, Myron Sawka, BOC Trainer

14 Version control table

Version Control	Date Released	Approved By	Amendment
1	June 2013	WHS Manager	New document
2		WHS Manager	Rebrand. Added references to Air and Health Monitoring Guidelines, Local WHS Monitoring Equipment Form, and Do Not Enter Lift Sign. Removed Taylor-Wharton contact details and referred to “qualified pressure testing organisation”. Restructured section 7. Replaced picture in 8.3.4 and moved original to 8.4. Reformatted/ rebranded document.

15 Appendix

As a guide to calculate the safe volume of a cryogen that can be stored or handled in an unventilated space, calculate the ratio of cryogenic gas volume to room volume (using Equation 1 and Equation 2). If the ratio is >0.015, then special precautions or ventilation are required.

Equation 1: Amount of cryogenic gas produced from the volume of cryogen present

Gas Release (V_g, in m^3) = Cryogen volume (L) x Expansion Factor / 1000

Equation 2: Ratio of cryogenic gas volume to room volume

Percentage Oxygen ($\%O_2$) = $21 (V_r – V_g) / V_r$

Where:

V_g = gas release (in m^3)

V_r = room volume (in m^3)

Gas	Expansion Factor	Boiling Point Centigrade	Boiling Point Kelvin
Acetylene	.	-84.0	189.1
Argon	847 to 1	-185.7	87.4
Boron trifluoride	.	-100.3	172.7
Carbon dioxide	553 to 1	-78 5(b)	194.6
Carbon monoxide	..	-192.0	81.1
Chlorotrifluoromethane	.	-81.4	191.6
Deuterium	..	-249.5	23.6
1,1-Difluoroethylene	.	-83.0	190.0
Ethane	...	-88.3	184.8
Ethylene	. .	-103.8	169.3
Fluorine	888 to 1	-187.0	86.0
Fluoroform	..	-84.0	189.1
Helium-3	757 to 1	-269.9	3.2
Helium-4	757 to 1	-268.9	4.2
Hydrogen	851 to 1	-252.7	20.4
Hydrogen chloride	..	-85.0	188.0
Krypton	700 to 1	-151.8	121.3
Methane	578 to 1	-161.4	111.7
Neon	1438 to 1	-245.9	27.2
Nitrogen	696 to 1	-195.8	77.3
Nitrous oxide	666 to 1	-89.5	183.6
Oxygen	860 to 1	-183.0	90.1
Ozone	.	-111.9	161.3
Tetrafluoromethane	. .	-128	145
Tritium	..	-248.0	25.1
Xenon	573 to 1	-109.1	164.0

15.1 Special Helium precautions

The most critical safety issue in dealing with liquid helium is its temperature. It is so cold that it will FREEZE ALL GASES except Helium. This includes not only H_2O, but also N_2 and O_2; all of these can freeze inside a liquid helium Dewar or delivery lines, forming an "ice" plug which can potentially close up the neck and create a bomb.

For this reason, it is imperative that procedures be in place and followed exactly to prevent air or other gases from entering the liquid delivery lines at any time. Should a blockage be suspected remove the Dewar to a safe location and contact the supplier immediately. Attempting to transfer liquid helium in non-vacuum jacketed piping can cause air surrounding the outside of the transfer pipe to condense and liquefy. The nitrogen in this liquid will evaporate first, leaving an enriched oxygen liquid behind. The area where this liquid collects should be insulated and oxygen-compatible.