Cryogen Safety

University of Lethbridge

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1.0 Introduction

Cryogenic liquids (cryogens) are compressed liquefied gases that are kept in their liquid state at very low temperatures. All cryogenic liquids are gases at normal temperatures and pressures and have boiling points below -150°C(- 238°F) (Carbon dioxide – dry ice – has a higher boiling point and is sometimes included in this category).

Different cryogens become liquids under different conditions of temperature and pressure, but all have two properties in common:

- they are extremely cold,
- and small amounts of liquid can expand into very large volumes of gas.

The vapours and gases released from cryogens also remain very cold. They often condense the moisture in air, creating a highly visible fog. Under certain conditions, some cryogens may condense the surrounding air forming a liquid air mixture.

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

1.1 Types Of Cryogens

There are two types of cryogenic liquids used at the U of L:

- Liquid nitrogen
- Liquid helium

Nitrogen and helium are inert gases that do not react chemically to any great extent. They do not burn or support combustion. Liquid nitrogen is used for preservation of tissue samples and lab experiments requiring cold temperatures. Liquid helium and nitrogen are used to cool the superconducting electromagnet in Nuclear Magnetic Resonance (NMR) Spectrometers. Smaller amounts of liquid helium are sometimes used in other instruments.

Table 1 outlines the specific properties of several cryogens.

Table 1. Physical properties of various cryogens.

Properties	Cryogen							
	Ar	He	H_2	Ne	N_2	O_2	CH_4	CO_2
Boiling point (1 atm)								
°C	-186	-269	-253	-246	-196	-183	-161	-78
°F	-303	-452	-423	-411	-321	-297	-256	-108
Critical temperature								
°C	-122	-268		-229	-147	-118	-82	31
°F	-188	-450		-379	-232	-181	-116	88
Liquid density, g/l	1402	125	71	1206	808	1410	425	1560
Gas density (27°C), g/l	1.63	0.16	0.082	0.82	2.25	1.4	0.72	2.0
Liquid-to-gas expansion ratio	860	780	865	1470	710	875	650	790
Flammable	No	No	Yes	No	No	No [a]	Yes	No

[a] Although oxygen does not burn, it will support combustion. Oxygen-enriched atmospheres may lead to violent reactions, such as rapid combustion or explosions with incompatible materials.

2.0 Storage and Containment

Sources of cryogenic liquids on the U of L campus:

1. A bulk liquid nitrogen storage tank is located within an enclosure outside of the fourth level of University Hall. The dispensing station is located at B422.
2. Liquid helium is delivered by the supplier in portable pressurized tanks.

2.1 Types Of Storage Containers For Cryogenic Liquids

Cryogenic liquids are shipped and used in thermally insulated containers that are specifically designed to withstand rapid temperature changes and extreme differences in temperature. These vessels, called Dewars are usually insulated by vacuum jacketing and may have pressure relief valves to protect against over-pressurization.

2.1.1 Liquid Dewar Flasks. Liquid Dewar flasks are non-pressurized, vacuum-jacketed vessels, similar to a "Thermos bottle". They should have a loose fitting cap or plug that prevents air and moisture from entering, yet allows excess pressure to vent. Flasks containing helium, hydrogen and other low- boiling liquids may also have an outer vessel of liquid nitrogen for insulation.

Figure 1. Dewar flasks.

2.1.2 Laboratory Liquid Dewar Flasks. Laboratory liquid Dewars have wide-mouthed openings and do not have lids or covers. These small containers are primarily used in laboratories for temporary storage and convenient transfer.

2.1.3 Pressurized Liquid Cylinders and Dewars. Liquid cylinders are pressurized containers specifically designed for cryogenic liquids. This type of container has valves for filling and dispensing the cryogenic liquid, and a pressure-control valve with a frangible (bursting) disk as backup protection.

Figure 2. Pressurized Dewar flask (left); valve system (right).

2.2 Storing Cryogens

Keep non-pressurized Dewar flasks covered with a loose fitting cap. This method prevents air or moisture from entering the container yet allows pressure to escape. Use only the stopper or plug supplied with the container.

Ensure that ice does not form in the neck of flasks. Liquids such as helium can freeze the water vapour in the surrounding air, including the air itself, creating an ice block. Pressure may slowly build up and may cause a violent rupture. Moving parts, such as valves or pressure relief devices, can malfunction due to external ice formation.

Inspect all incoming containers before storing to ensure they are not damaged and are properly labeled. Ensure that valves designed for pressure relief are in the open position. Store containers in well-ventilated areas, away from sources of heat and moisture.

3.0 Hazards

If not handled correctly, cryogens may be hazardous to personnel. There are four major hazards associated with cryogens: extremely cold temperatures, asphyxiation, oxygen enrichment, and over pressurization of containers and systems.

3.1 Extremely Cold Temperatures

Cryogens are extremely cold and can cause instant, severe frostbite. A jet of cryogen vapor can freeze the skin or eyes faster than liquid contact. Tissue damage may be extensive, particularly if the eyes are involved; permanent damage may result. Contact with cryogens can freeze tissue and produce a cryogenic burn or frostbite. Unprotected skin may adhere to metal that is cooled by cryogens. The skin can tear when pulled away.

Even non-metallic materials are dangerous to touch at low temperatures. At cryogenic temperatures, many materials, such as rubber, plastic and carbon steel can become so brittle that they shatter. Only materials approved for use with cryogens should be used.

3.2 Asphyxiation

Air consists of 78% nitrogen, 21% oxygen, and trace gases making up the remaining 1%. Cryogens can create oxygen deficiency because they have large liquid-to-gas expansion ratios (generally >700). For example, one litre of liquid nitrogen vapourizes to 696 litres of nitrogen gas when warmed to room temperature. A small liquid spill produces a large volume of gas that can displace the air in a confined space, thus creating a serious oxygen deficiency (<19.5% oxygen) that can asphyxiate occupants of the area. Although nitrogen is non-toxic, it will displace air and can create a hazardous work environment. Simple asphyxiants, such as nitrogen do no have good warning properties and one may simply pass out without any warning and may die without regaining consciousness. Oxygen sensors should be used in confined areas to indicate oxygen deficiency. The NMR labs at CCBN and in University Hall have oxygen sensor systems.

3.3. Oxygen Enrichment.

Liquid helium is so cold that it can liquefy air upon contact. When transferring nitrogen through uninsulated metal pipes, the air surrounding the containment system may condense. Nitrogen, which has a lower boiling point than oxygen, will evaporate first. This can leave an oxygen-enriched condensate on the surface that may increase the flammability (combustibility) of materials near the system, presenting all of the same hazards as liquid oxygen. Many materials considered non-combustible could burn in the presence of liquid oxygen.

3.4 Over-pressurization of Containers and Systems

Without adequate venting or pressure-relief devices on the containers, enormous pressures can build up. Cryogens boil as they sit in their storage vessels by absorbing heat energy from their much warmer surroundings. The gas boiling out of the liquid must escape or the pressure will increase. The pressure can cause an explosion called a "boiling liquid expanding vapour explosion" (BLEVE). Users must ensure that cryogens are never contained in a closed system. Use a pressure relief vessel or a venting lid to protect against pressure build up. Unusual or accidental conditions such as an external fire, or a break in the vacuum which provides thermal insulation, may cause a very rapid pressure rise. Since the pressure relief valve may not be able to handle this increased pressure, the containers must also have another backup device such as a frangible (bursting) disc.

4.0 Personal Protective Equipment

4.1 Avoid Skin Contact

Cover all exposed skin by wearing long sleeve shirts, long pants, a long sleeved lab coat, and shoes that fully cover the feet. Gloves should be loose-fitting, lightweight, flexible and insulated so that they can be quickly remove dif cryogenic fluids are spilled on them.

Remove watches and metal jewelry from your hands before working with cryogens. If exposed to cryogenic liquids or boil-off gases, the jewelry can freeze to the skin.

4.2 Protect Your Eyes and Face

Wear safety glasses whenever you are near a cryogenic liquid. Face shields shall be used in the following situations: when a cryogen is poured; for open transfer; if fluid in an open container is likely to bubble.

5.0 Handling and Use of Cryogens

Only handle cryogens if you are trained and fully aware of the properties of the materials and the equipment to be used.

5.1 Ventilation Requirements

To reduce the risk asphyxiation, ensure proper ventilation where cryogens are stored or used. When dispensing liquid nitrogen from bulk storage, ensure adequate ventilation by opening the overhead door. An oxygen sensor must be installed whenever cryogens are used in confined areas, such as NMR labs. In the event of a quench, large volumes of liquid nitrogen or liquid helium would be released, displacing the air from the room.

5.2 Transporting Cryogenics

Move cryogenic liquid containers carefully. Always use a hand truck, flat deck trolley, or other proper handling device and use a strap to secure the container. Keep cryogenic liquid containers upright at all times except for the minor tilting on the cart during transport.

If cryogens must be transported by elevator, use the elevator key lock to send the filled Dewar to the desired floor. The greatest risk of spillage occurs when moving the Dewar in or out of the elevator (e.g. the elevator does not stop level with the floor or if the transport cart wheel becomes lodged in the space between the elevator and the floor). Use extra caution when loading and unloading the elevator to prevent accidental spillage. Always enter the elevator last when loading a Dewar into the elevator; always exit the elevator first when unloading a Dewar from the elevator.

Do not place a filled container in your vehicle. Contact Shipping/Receiving at 329-2615 to arrange for transport of liquid nitrogen from University Hall to other locations on campus.

5.3 Selecting Materials for Use with Cryogens

Only use materials approved for use with cryogens. Many common materials such as carbon steel, plastics and rubber become brittle and can crack if exposed to the extremely low temperatures of cryogenic liquids. Many materials also shrink at cryogenic temperatures, potentially causing leaks at hose connections.

5.4 Cooling Operations

When using cryogenic liquids to cool an object, insert the object SLOWLY using tongs. This procedure minimizes any boiling and splashing which occurs when warm objects are added rapidly.

5.5 Transferring Cryogenic Liquids

When transferring cryogenic liquids from one container to another, cool the receiving Dewar flask before filling it. Always start filling slowly to allow the vaporization to chill the receiving container. After the vaporization and liquid boiling has decreased, fill the container at the normal rate. A diffuser should be attached to the transfer hose to reduce turbulence and the release of gas while filling. Never fill containers higher than the indicated level. Fill containers only with liquids they are designed to hold.

Transfer or pour cryogens slowly to minimize boiling and splashing. Use a phase separator or special filling funnel (the top of the funnel should be partly covered to reduce splashing). When it is not safe or convenient to tilt the container, use a transfer tube to remove the liquid. Insert the transfer tube through the neck of the container and well down into the liquid. The packing material or stopper on the transfer tube should form a seal in the neck of the container. Normal evaporation usually produces enough pressure to push liquid out. If necessary, the container may be pressurized with the same gas as the liquid or with an oil-free inert gas. Use just enough pressure to force liquid out.

Figure 3. Dewar and transfer tube.

Figure 4. Transfer tube.

6.0 Equipment Maintenance

The liquid nitrogen supplier will inspect the bulk storage tank and dispensing system on a bi-annual basis and provide an inspection report for follow-up. Problems with the dispensing station, PPE, Dewars, or any other approved storage containers must be reported immediately to OHS and the lab supervisor.

Avoid forcing connections, using homemade adaptors, or tampering with containers in any way. When doing maintenance work on oxygen handling systems, cleanliness is extremely important. Grease or oil must not be allowed to contaminate any parts.

7.0 Disposal

Never dispose of liquid cryogens down the drain. Piping in laboratory sinks may not be able to withstand cryogenic temperatures. Allow cryogenic liquids to evaporate in a fume hood.

8.0 First Aid Measures

Avoid contact with cryogenic liquids, their vapours and any cooled surfaces. You should obtain medical assistance as soon as possible if cryogens contact your skin or eyes. Immediately upon exposure, the frozen skin appears waxy and yellow and the burn is usually not painful; however, the affected area will painfully swell and blister while the skin defrosts. If contact does occur, immediately flush the area with large quantities of warm (not hot) water. If the skin is blistered or the eyes have been exposed, obtain medical attention immediately. Locate emergency eyewash stations and safety showers wherever there may be accidental exposures to cryogens. Report all injuries to OHS (submit an online Accident/Incident Report).

9.0 Emergency Procedures For The Liquid Nitrogen Dispensing Station

1. In the event of an emergency, **press the red emergency stop button** located to the right of the dispensing station. This will stop the flow of liquid nitrogen from the storage tank.
2. Call **2345.**
3. If the situation is deemed to be immediately dangerous to life or health, call 911. Your location is 4th level entrance to University Hall and can be accessed by traveling east on Valley Road and turning right on Coulee Trial.
4. Ensure the area is well ventilated by opening the overhead door.
5. Restrict access to the area.

If you are unable to stop the release by pressing the emergency stop button:

1. Retrieve the key to the outdoor storage tank enclosure (key is located in the Utilities office; Security also has a key.)

2. Put on the insulated gloves and face shield.

3. Open the door and turn off the main supply valve HCV-19 (see photo; valve is painted red).

4. Remove any casualties from immediate danger. Avoid all contact with liquid nitrogen. Ensure space is well ventilated before entering.

If you are unable to turn off the main supply valve, call the Praxair Emergency Number 1-800-363-0042.

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