C6. Handling and Use of Cryogenic Materials (Liquid Nitrogen and Dry Ice)

BACKGROUND

This document covers the most commonly used cryogenic gasses on the UBC Okanagan campus: liquid nitrogen and dry ice. While general safety and handling information can be used when working with other cryogenic gases, specific handling inquires should be directed to Health, Safety & Environment (hse.ok@ubc.ca).

This document reviews the following topics:

- Physical and chemical properties of nitrogen and dry ice
- Hazards of cryogenic material
- Safe handling of cryogenic material
- Liquid cylinder construction
- Filling cryogenic dewars
- Personal Protective Equipment (PPE)
- Safe moving of liquid cylinders
- Elevator transport
- Emergencies
- Trouble shooting

CHEMICAL AND PHYSICAL PROPERTIES

Nitrogen

- makes up 78% of the atmosphere
- colourless, odourless, tasteless and nontoxic
- boils at -196°C
- non-flammable
- gas state is slightly lighter than air

Dry Ice

- tasteless and odourless
- no residue as solid sublimes (transition of substance directly from solid to gas state)
- at room temperature, dry ice sublimes at 14% per hour
- germ and bacteria free
- nontoxic
- non-flammable
- gas state is heavier than air
- boils at -78.5°C

HAZARDS

Cryogenic Gases

Primary hazards:

a) asphyxiation
b) contact burns
c) shattering of receptacles on contact
d) potential for pressure rupture of containers, pipelines or systems
e) musculoskeletal lifting injuries

1. *Asphyxiation*

Liquid nitrogen and CO_2 when returned to the gaseous state, can displace oxygen from the air and can create an oxygen-deficient atmosphere under specific conditions. *It may be prudent to install oxygen monitors in areas where liquid nitrogen or dry ice is stored and ventilation is minimal.* The physical properties listed in the table below help explain how cryogenic gases can displace oxygen.

Physical Properties of Liquefied (Cryogenic) Gasses						
Gas	**N_2**	**O_2**	**Ar**	**H_2**	**He**	**CO_2**
Boiling Point °C	-196	-183	-186	-253	-268	-78.5
Volume Expansion	696	860	696	850	745	540

- 1 litre of liquid nitrogen will expand to 696 litres of 100% gaseous nitrogen at 25°C
    - The nitrogen can displace the oxygen in the area, leading to asphyxiation
    - Hence, cryogenic liquids should always be stored in well-ventilated areas

The normal concentration of oxygen in air is about 21% by volume. This provides for a safe working environment with respect to the oxygen required to support life. An oxygen concentration below 19.5% is considered an oxygen deficient environment.

- Asphyxia develops slowly as the oxygen content is gradually reduced from 21%
- Victims will not be aware of a problem and generally will not recognize the symptoms of gradual asphyxia from decreasing oxygen levels

Precautions to take when using cryogenic gasses include:

- Always wear safety equipment, including heavy loose-fitting leather or cryogenic gloves, and eye and face protection.
- All cryogenics should be used and stored in well-ventilated areas
    - High concentrations of escaping gas should not be allowed to collect in an enclosed area.
    - High concentrations of nitrogen and carbon dioxide reduce the breathable oxygen in the air
        - Filling between containers, leaking valves and liquid tank venting are some examples that could lead to an oxygen deficient atmosphere

2. *Contact Burns*

Source: https://documents.uow.edu.au/content/groups/public/@web/@ohs/documents/doc/uow158672.pdf

3. *Musculoskeletal injuries*

Two people should lift dewars greater than 20L capacity. Proper form and lifting technique should be used when lifting and moving any heavy objects. Visit https://hse.ok.ubc.ca/health/ergonomics/ for more information on Ergonomics.

SAFE HANDLING PROCEDURES

Liquid Nitrogen

- Always read the container label prior to use
- Cryogenic liquid containers must always be stored in the upright position
- Cryogenic liquids should **not** be handled in open pail-type containers or in unapproved dewars (use approved containers)
- Transfer of liquid into warm lines or containers must be done slowly to prevent thermal shock and possible buildup of pressure
- Avoid rough handling of liquid containers
- A cold outside jacket indicates a loss of vacuum (contact supervisor or vendor)
- Liquid cylinders should only be moved with proper handling equipment
- Prior to use, ensure the fittings on the regulator match the fittings on the liquid container
- Never use adaptors
- Never attempt to change or remove fittings
- Use containers specially designed to hold liquid nitrogen when dispensing
    - Check with the manufacturer of the container to ensure it is **approved** for holding liquid nitrogen

Handling Dry Ice

- Always store in a well-ventilated room
- Always wear insulated gloves when handling dry ice (do not use latex gloves)
- Wear safety glasses and lab coat
- Prevent contact with uncovered skin
- Dispose unwanted dry ice in a fume hood

LIQUID CYLINDERS

Liquid cylinders contain large volumes of liquid/gas. There are two general types (check the pressure gauge to determine the type of container):

1. Low Pressure for dispensing liquid **only** (operates at 22 psig)
2. High Pressure for dispensing liquid **and** gas (operates at 230 psig or above)

Always check the type of container that is being delivered, or before use (make sure you know the type of container that is used by your lab)

- One lab had ordered low pressure and received high pressure by mistake. The lab personnel assumed it was low pressure and began to use it. This could have resulted in an unsafe condition.

Low Pressure Liquid Cylinders

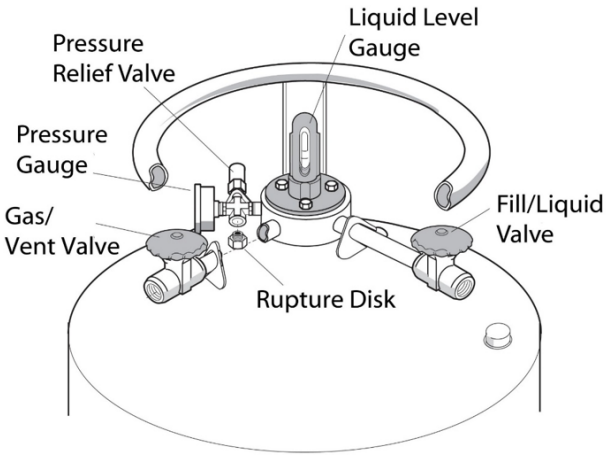

Image used with permission from Chart Industries

- *Liquid Valve* - Liquid is withdrawn through this valve
- *Pressure Gauge* - Displays internal pressure of the container
- *Liquid Level Gauge* - A float-type liquid level gauge-indicates approximate level of liquid
- *Vent Valve* -Primarily used in the fill process to vent the vapor space while filling. Can be used to vent unwanted pressure during storage and use

- *Pressure Relief Devices* (2) - Protect vessel from over-pressurization
  (1) Re-seating spring-loaded relief valve releases at 22 psig
  (2) Burst disk rated to protect the inner vessel

- *Outlet Restraints* - These are to prevent the dangerous practice of changing outlet connections at user sites. These restraints may be twist ties, wire, or other. Changing outlet connections is an extremely dangerous practice and can result in serious injury or death if an incompatible product is introduced into a user's system. Removal of these restraints will void all product warranties!!

More on Liquid Level Gauge…

The liquid level gauge is a float-type liquid level sensor that indicates the level of the liquid The gauge is an indication of *approximate* container content, and should not be used for judging the weight of the container. Containers are filled by Weight.

Signage

Caution signs should be posted in the area warning that liquid nitrogen is being stored and used.

Construction of Liquid Containers

Liquid containers are made *somewhat* like Thermos® bottles with a vacuum space and special insulation. But they are **not** Thermos® bottles.

- Even with the vacuum and insulation, heat leaks in to the cylinder causing the cryogenic liquid to vaporize and build pressure. The vaporization rate will depend on several factors including:
  - The product itself
  - Ambient temperature
  - Condition of the cylinder's vacuum
  - Etc…

Pressure relief devices

The liquid to gas conversion rate is about 2.3% per day under perfect conditions, so the actual vaporization rate experienced can vary. If the liquid is not used regularly, the vessel will be empty in a certain amount of time. It is important to estimate your use so the liquid will not be wasted.

- If gas product is not used, pressure will build until it is released by a control valve

- Note that this is a high-pressure container, with the gauge marked for 350 PSIG
- Source: https://users.stlcc.edu/departments/fvbio/Biosafety_Liquid_Nitrogen.pdf

- Hearing a slight hiss from a liquid cylinder is usually the normal operation of its pressure relief valve
- Liquid cylinders should always be stored and used in areas with appropriate natural or mechanical ventilation
- Never adjust, block, plug or attempt to repair anything on a liquid cylinder
- Pressure relief devices are prescribed based on the following formula for vacuum-insulated cylinders:
  - (Cylinder service pressure X 1.25) – 15 PSI = maximum pressure relief device rating (PSI)

Vendor checks / inspections

Before filling a liquid cylinder:

- Visual inspection
- Valve inspection
- Pressure relief valve inspection
- Safety burst / rupture disc inspection
- Vacuum casing burst / rupture disk inspection
- Tare weight

After filling a liquid cylinder:

- Vent valve
- Gross weight
- Net weight

Storage in cold rooms

Contrary to popular belief, storage of liquid containers in cold rooms will **not** slow down the liquid to gas conversion. Storage in cold rooms can actually create an oxygen deficient atmosphere if the room does not have adequate ventilation to remove the nitrogen gas generated. If there is a concern regarding oxygen deficiency in these areas, oxygen deficiency alarms should be installed.

Head Pressure

Head pressure results when heat leaks into the container. The safety relief valve will periodically release this pressure. If the safety relief valve malfunctions, a backup disk will rupture and relieve the pressure. The rupture of the backup disk will produce a loud sound and may release a large quantity of liquid and gas. Evacuation of the area is required to prevent asphyxiation

Warning!

Never plug, restrict, or remove any relief device. Never attempt to cap or seal a venting relief device in any way. Ice or frost buildup on a pressure relief valve can be removed with a damp cloth. Wear proper Personal Protective Equipment (PPE) when removing the frost.

MOVING LIQUID CONTAINERS

Liquid cylinders range in different weights and sizes. They are heavy and cumbersome, especially when filled with liquid nitrogen. They may require two people to handle in order to do so safely. Containers can cause crushing injury to the feet. Wear proper shoes. (Tennis shoes and open toed shoes are not proper foot protection!).

- **Never** try to roll liquid cylinders by using the Liquid Level Gauge Tube
- **Use** that cylinder's halo ring for moving

- Always use the specially designed **cylinder cart** when moving liquid cylinders
    - If the cart has a height adjustment for different cylinders, be sure it's adjusted to the **proper** height
- Always use the specially designed and designated hand cart
- Do **not** roll, either vertically or horizontally
- Push, **don't** pull
- If the container tips over, **let it go**

Elevator transport

The immediate hazard is oxygen depletion if spilled. Use the freight elevator if possible. If a passenger elevator is used, it should be locked out to all other users. **Do not** transport cryogenics at any time in an elevator with any other personnel in the car unless they have a supplied air respirator.

- When a container has been placed on an elevator, the elevator must travel between floors unoccupied
- All elevator doors must be manned to prevent entry
- Person/s must be stationed at all "in-between" floors to prevent riders from entering elevator

- The sender should remain outside the elevator and activate it to the desired floor. Another person should be available on the receiving floor to take the liquid container off the elevator at its destination
- Calculation of oxygen displacement:
    - 1 litre of DI @ 14% = 140 cc/hr
    - Expands to 540 x 140 = 75600 cc/hr
    - Elevator = 2m x 2.5m x 3m = 15 x106 cc
    - 75600/15,000,000 = 0.5% concentration of CO2 after one hour in elevator
- Not an immediate risk of oxygen depletion…personnel permitted in elevator. (*The TLV (and PEL) assigned to carbon dioxide is 5000 ppm (NIOSH has recommended a Standard of 1.0% or 10,000 ppm for a 10-hr work shift with a ceiling of 3.0% or 30,000 ppm for any 10-min period). These concentrations are an expression of good practice rather than a line between "safe" and "deadly."*)

EMERGENCY RESPONSE

If there is a large spill or rupture of a container

1) Call 911 and warn others in building
2) Evacuate! There may be oxygen deficiency in the are of the spill
3) If there is injury to the body from liquid nitrogen, seek immediate medical assistance
4) If liquid is splashed in the eyes, flush with water for at least 15 minutes.
    a. Seek immediate medical attention. Call Campus Security for first-aid at 250-807-8111.
5) Skin contact may cause frostbite and burns.
    a. Soak affected part in tepid water and seek immediate medical attention. Call first aid. Skin contact is a medical emergency. Lack of prompt medical attention may result in amputation.

PERSONAL PROTECTIVE EQUIPMENT (PPE)

PPE is the **last line** of defense, and should never be your first means of controlling a risk.

Hazards can be controlled in the following ways and in the following order:

1) Minimize/Eliminate/Substitute
2) Engineering/Task Design
3) Administrative/Work Procedures
4) Personal Protective Equipment (PPE)

If there is any chance of **contact** with cryogenics, or if **handling / transporting**, wear the following PPE

- Full Face Shield with safety glasses
    - Due to the potential for splashing & shattering
- Heavy, Loose Fitting leather or Cryogenic Gloves
    - Protection for skin
- Lab Coat, Long Sleeve Shirt, or Arm Protection
    - Protection for skin

- Pants should be cuff-less
  - Cuffs can hold liquids / vapours
  - Do not tuck pants into shoes, boots
- Proper laboratory footwear
  - Footwear that does not completely cover the surface of your foot does not provide adequate protection
  - The mesh on running shoes does not provide enough protection for your feet
  - Consider steel toed footwear if there is a chance of heavy items landing on feet

LIQUID DISPENSING

- Remind yourself of the hazards
- Always have a co-worker present
- Two-person lift of Dewars greater than 20 litre capacity
- Ensure area/room is well ventilated
- Avoid using a funnel
- Always wear a full-face shield, goggles, leather or cryogenic gloves, safety shoes, and aprons when transferring liquid.
- Ensure that the liquid cannot collect in pants cuffs or travel down into shoes. Do not tuck cuffs into pants! Cuff gloves over sleeves.
- Transfer of liquids at pressures higher than 22 psig into open vessels such as small dewars can lead to excessive splashing. This could result in injury from freezing of the body part
- Caution!!!
  - Before use, always confirm that the fittings are appropriate for the product identified on the cylinder label.
  - If a mismatch appears, do not attempt to use the container.
  - If help is needed, ask your supervisor or supplier
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Liquid Withdrawal

- Ensure that withdrawal hose is equipped with a phase separator to prevent splashing.

Check with supervisor or supplier

- Never dispense liquid into an unapproved container, such as a Thermos® bottle. It will shatter!

OTHER CRYOGENIC FLUIDS

Liquid Helium	Liquid Oxygen	Liquid Hydrogen	Liquid Argon
• Super cold at -452F (-268°C) • Special container needed for more insulation • Personal Protective Equipment (PPE) extremely important • Like Liquid Nitrogen, an inert gas • Will not support life or combustion • Asphyxiation potential same as Liquid Nitrogen	• Not as cold as liquid nitrogen • Incompatible with flammable, organic, and combustible materials • Avoid contact with heat, sparks, and flame. • NO SMOKING OR OPEN FLAMES signs should be posted in areas that use liquid oxygen • Highest volume expansion rate of the Liquid gasses (860)	• Super cold at -423F (-253°C) • Hydrogen gas is explosive • Do not use near open flames or other sources of ignition. • Asphyxiation potential same as Liquid Nitrogen • Second Highest Expansion Rate of the Liquid gasses (850)	• Like Liquid Oxygen, an inert gas • Will not support life or combustion • Asphyxiation potential same as Liquid Nitrogen

TROUBLESHOOTING

Issue:	Possible Cause:	Recommended Response:
Container top covered with frost	High product use	Normal Operation
Gas vents intermittently through safety relief valve	Probably normal operation. Gas generated due to heat leak into cylinder causes head pressure to build	Ensure inactive containers are stored in well ventilated area. Rotate inventory.
Gas vents continuously through safety relief valve	Possible relief valve failure or excessive heat leak	Removed container or vent the exhaust to a well-ventilated area. Relieve product through vent valve. Check to see if safety relief valve is frozen open. Contact supervisor or supplier for assistance.
Gas vents during use through safety relief valve	Set point on regulators exceeds safety relief valve setting	Reduce set point on pressure building regulator. Contact supervisor or supplier for assistance.

Issue:	Possible Cause:	Recommended Response:
Pressure in the container is low	Leak from container	Use appropriate leak detection fluid to check for leaks in connections. Examine container for signs of frost. If leaks are on the container itself, contact supervisor or supplier.
	Pressure building valve is not fully opened	Open valve fully
	Pressure building regulator is not set high enough	Adjust to increase pressure. Contact supervisor or supplier
	Pressure building valve is open	Close the valve if frost is visible on the pressure building vaporizer near the bottom of the tank. Contact supervisor or supplier.
Pressure in the container is too high	Leaking or improper setting of pressure building regulator	Reduce regulator settings to achieve desired pressure level. Contact supervisor or supplier.
	Vacuum integrity failing	If container walls are covered with frost, contact supervisor or supplier.
Container has isolated spots of frost	Container may have been damaged, compromising the integrity of insulation	Contact supervisor or call supplier for replacement
Container surface is uniformly covered with frost	Vacuum integrity compromised	If accompanied by a high rate of product venting through the safety relief valve, or high rate of pressure increase, call supplier.

QUIZ

Please complete the short quiz provided by your supervisor. Contact Health, Safety & Environment if you need the quiz or the answers.

Questions? Contact Health, Safety & Environment at hse.ok@ubc.ca.