Training in cryogenics and compressed gasses

Physics department security committee
Université de Sherbrooke

Physical properties:

Cryogen	Boiling point	Volume ratio (gas/liquid)	Density	Odour	Flammable?
N_2	77K (-196°C)	695	Slightly less than air	None	No
He	4.2K (-269°C)	757	Very low	None	No

- Gaseous nitrogen (N2) represents 78% of the air that we breathe. Oxygen represents 21% and argon 1% of the atmosphere. Water vapour is also mixed with the air.
- Liquid nitrogen costs roughly 50¢ per litre, whereas liquid helium (which is derived from sources of oil / natural gas) is sold at roughly $12/litre on the market.
- Our department's helium liquefier allows us to reduce the cost of production to $4/litre.

Transport classification

Cryogène	Classe	No UN	Appellation
N_2	2.2	UN1977	Refrigerated liquid nitrogen
He	2.2	UN1963	Refrigerated liquid helium

Class 2.2 – Non-Flammable and Non Toxic Gases

UN Number: Alphanumeric code (2 letters and 4 numbers) that designates a dangerous substance or a group of dangerous substances with the same characteristics. It allows for the rapid identification of a substance for transport within North America.

Pictogrammes for liquid nitrogen and helium

For vessels, in use and in storage, that contain cryogenic liquids (WHMIS 2015)

For transporters of cryogenic liquids

Pressurized liquid nitrogen vessels

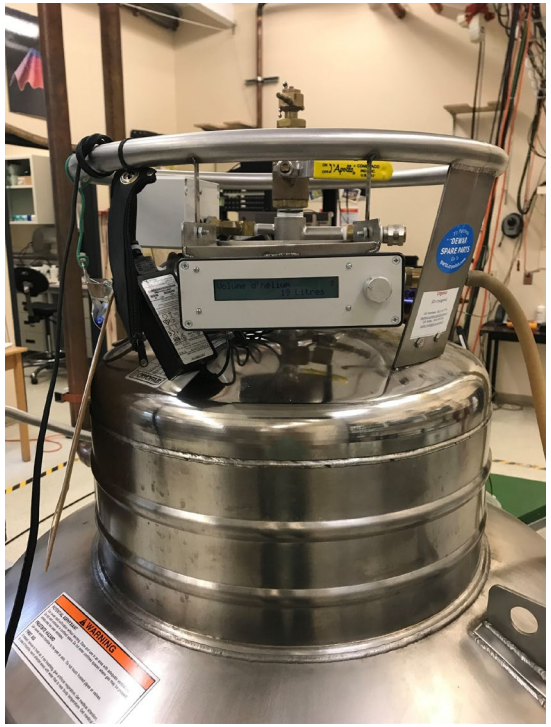
Manometer / pressure gauge (typically at 20 psi)
Pressure relief valve
Insulating vacuum space (do not modify)
Mechanical “pressure builder”
Do not force valves.
Watch your face. Caution when opening valves.
Presence of liquid oxygen during transfers (do not put near flames)

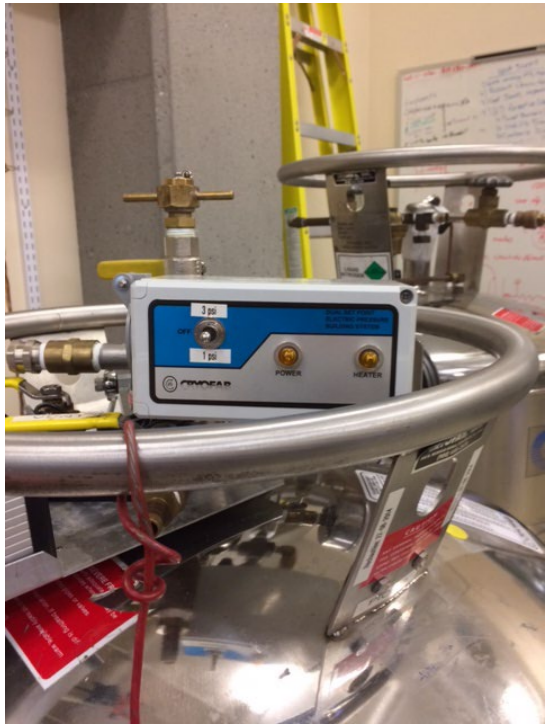
A technician will explain in detail the different components (valves, relief valves, etc.) of the vessels in the laboratory.

It is normal for ice to form near the neck of the dewar, especially if the ambient humidity is high. However, if a vessel is iced up over a large surface area, it is important to report it to a technician.

Pressurized helium dewars

Manometer / pressure gauge (low pressure, since should be connected to helium recovery system)
Pressure relief valve / check valve
Insulating vacuum space (not to be modified)
Do not force valves.
Protect your face, do not open valves with your face too close.
There should not be any build up of ice anywhere on the cryostat.
Electric “pressure builder” (heats a resistance to evaporate helium)





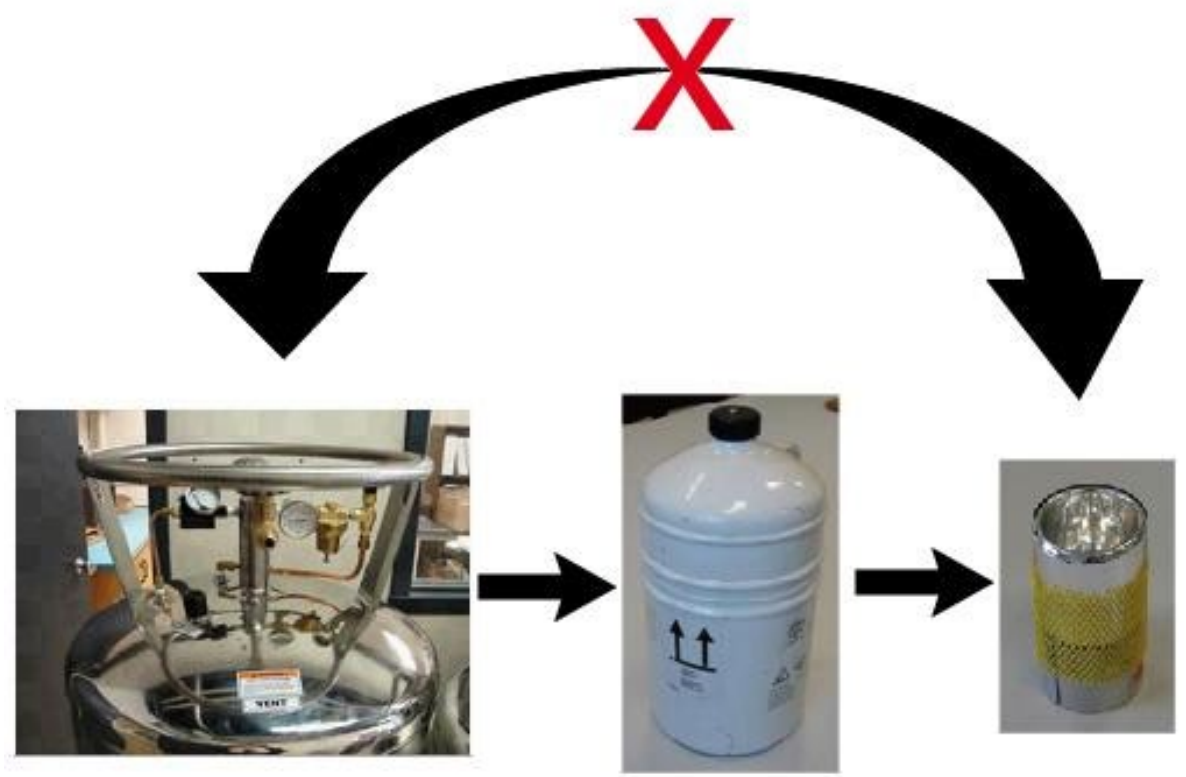
Unpressurized containers

Never seal them completely!

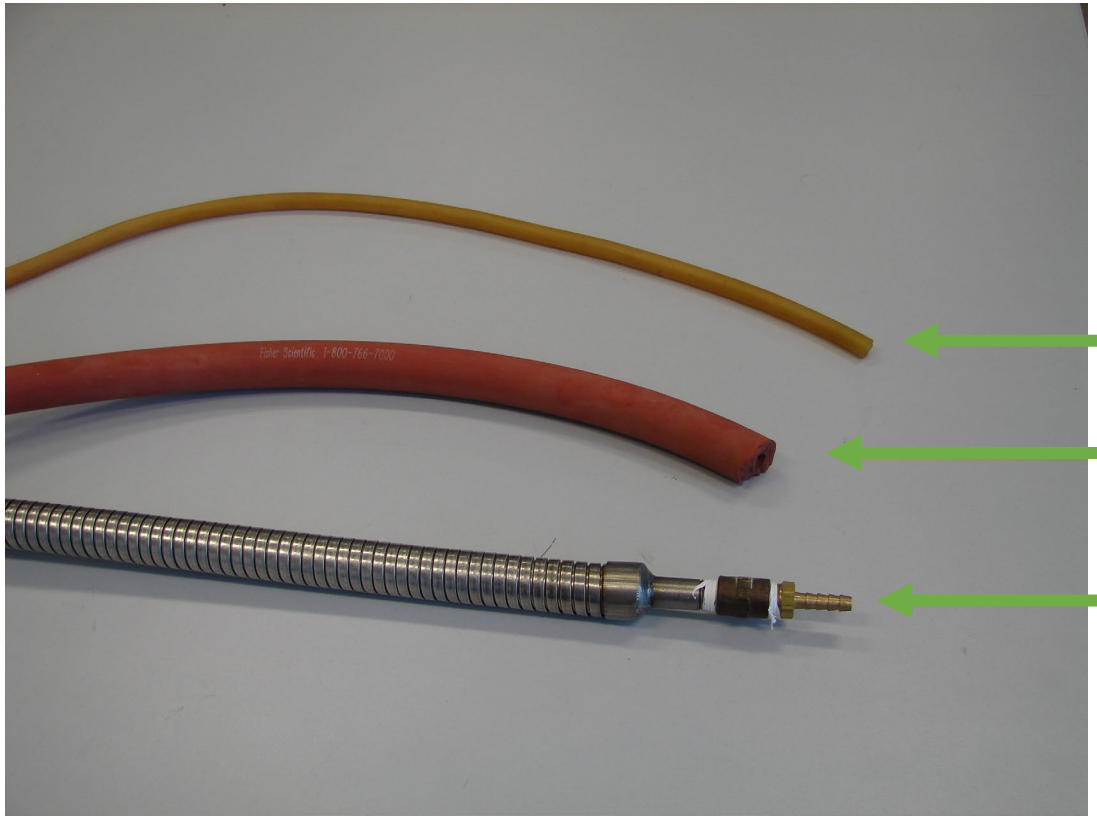
Avoid glass thermoses

Use a loose stopper or cap

Can overflow when filling



Liquid nitrogen transfer tube



Avoid (breaks easily)

Not as bad (but still breakable)

Best choice

A transfer tube should never be closed at both ends when it contains a cryogenic liquid or cold gas, as later expansion can lead to an explosion.

Transport of LN_2 and LHe

- Vessels must always be kept upright.
- It is forbidden to accompany a dewar inside of an elevator, as there is a risk of asphyxiation.
- Transport of pressurized dewars containing liquid cryogens should be left to the technicians of the “cryoteam”.

Transport of LN_2 and LHe

Use the support base that is available in room D2-2057 when transporting a 4 litre dewar of liquid nitrogen in the trunk of a vehicle.

Note: Liquide nitrogen should only be transported in a vehicle by a user that has taken appropriate training and holds a valid certificate. The training is provided by the SSMTE here at the U. de Sherbrooke and remains valid for a period of 3 years.

Manipulating liquid cryogens

- Request help from a knowledgeable technician, research professional or professor for your first use of a dewar of liquid nitrogen or helium.
- Training is required in order to carry out liquid helium transfers and use liquid helium cryostats.
- Every cryostat has its unique features. Make sure you are informed of possible dangers before you use them.
- Only use approved cryostats for liquid cryogens.
- Verify that valves and connections are in the correct configuration before and after the use of a cryostat or dewar containing a liquid cryogen.
- Ensure that the exhaust ports of helium dewars and helium cryostats are connected and open to helium recovery lines following a transfer.
- Do not leave cold cryostats or dewars open to the air. In addition to loss of helium, water ice or frozen air can accumulate within the vessel, potentially leading to a blockage and a risk of explosion.

Evening and weekend helium transfers

A one-time written authorization from your supervisor is required in order to carry out helium transfers alone in the evenings or on weekends.

The department requests that users limit their manipulations of cryogenic liquids to normal working hours during the week when possible. Make efforts to avoid carrying out transfers at night and on weekends and plan your experiments appropriately.

Risk of asphyxiation caused by the displacement of oxygen

Avoid using cryogenic liquids in closed spaces. Leave the doors to the laboratory open during your transfers of liquid nitrogen and helium.

Oxygen concentration	Effect on the human body
23.0%	Oxygen enriched atmosphere. Threshold beyond which it is forbidden to enter.
21.0%	Normal concentration of oxygen.
19.5%	Minimum concentration in order to enter into a closed space without an independent breathing apparatus.
12 – 16%	Irregular breathing, anxiety, abnormal fatigue when moving, insufficient concentration to keep a flame lit.
10 – 11%	Accelerated breathing and heartbeat, euphoria, headaches.
6 – 10%	Nausea, vomiting, inability to move freely, possible loss of consciousness or collapsing while remaining conscious.
< 6%	Respiratory arrest followed by cardiac arrest, death within several minutes.

Risk of asphyxiation caused by the displacement of oxygen

Table: Concentration of oxygen after a release of liquid nitrogen.

Room volume m3	Volume of liquid nitrogen spilled, litres						
	1	2	3	4	5	10	25
10	19.6	18.1	16.7	15.3	13.8	6.7	
25	20.4	19.9	19.3	18.7	18.1	15.3	
50	20.7	20.4	20.1	19.9	19.6	18.1	
75	20.8	20.6	20.4	20.2	20.0	19.1	16.2
100	20.9	20.7	20.6	20.4	20.3	19.6	17.4

Elevator →

Office →

A typical laboratory has a volume of 250 to 500 m^3.

Risk of asphyxiation caused by the displacement of oxygen

Oxygen level detectors are present in several laboratories as well as in rooms containing clusters of cylinders for recovering helium gas. If an alarm sounds, leave the room immediately and call security. **(Emergencies: 811)**

The most recently renovated labs have an alarm that is directly linked to campus security.

Do not go into a room in which the alarm is sounding.

WARNING: Some research labs (In the older part of the D2 building) are not equipped with oxygen detectors and are not adequately ventilated. Use increased caution when manipulating cryogens in these rooms.

Risk of frostbite

Prolonged exposure of skin with cold gases or contact with cold surfaces can cause frostbite. Skin can take on a yellow colour and waxy texture. An initial absence of discomfort is followed by intense pain when human tissues taws. Be particularly careful with your eyes. Personal protection equipment is obligatory for any kind of manipulation of cryogenic liquids.

Avoid wearing jewelry (rings, bracelets, watches).
Wear long pants and sleeves and closed shoes.
Safety glasses are absolutely essential.

Liquid nitrogen is sometimes used for culinary experiments (eg. Ice cream). Never ingest liquid nitrogen as it can cause serious injury.

Risk of frostbite: first aid

- Remove any clothes that could slow blood circulation in the frostbitten area.
- Apply warm water. Never apply dry heat.
- Do not try to thaw skin too quickly.
- Do not rub skin as this can further damage tissues.

- If eyes are affected, rinse them liberally with warm water (for 15 minutes).

- Frozen tissues are painless and have a waxy, yellow aspect. Thawing can take 15 to 60 minutes and should continue until blueish skin turns pink or red. Doctors may administer morphine or tranquilizers to help reduce pain.

- If the affected region thaws before arriving at the doctor's office / hospital, cover it with dry and sterile bandages and with a large and thick blanket.

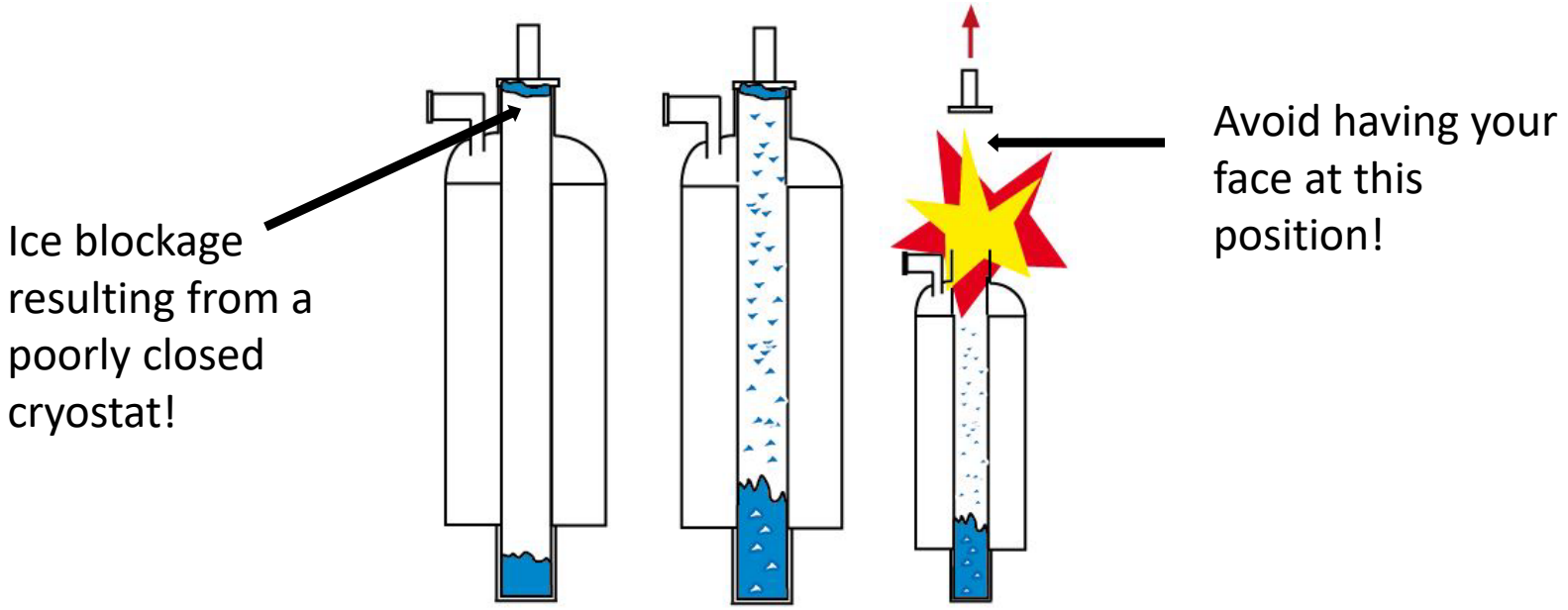
- Alcoholic beverages and cigarettes are proscribed as they can reduce blood circulation to the frozen tissues. Offer warm beverages and food.

Risk of forming liquid oxygen

- During filling of a vessel of liquid nitrogen from a second, pressurized vessel, there is generally condensation of liquid oxygen on very cold metallic surfaces.
- Avoid flames.
- Pure oxygen reacts very strongly with oil or grease, leading to a risk of explosion. Avoid grease for connections on cryogenic liquid vessels.

Risk of explosion

- Failure of the pressure relief valve.
- Outside fire.
- Sudden loss of vacuum isolation (the burst disc should function, but best not to test it…).



If the neck of a dewar or cryostat is blocked with ice, unblock it by following the manufacturer's instructions : contact a member of the cryoteam or another technician.

Risk of explosion

Explosion of a pressurized dewar containing liquid nitrogen in the chemistry department in the 1990s.

An exploding dewar can destroy a wall made from cement blocks!

Risk of explosion

- A soft hissing from a pressurized nitrogen vessel is normal.
- If the dewar is iced up over a large surface, there is a problem: the vacuum insulation is no longer adequate.
- If the pressure relief valve hisses noisily and continually, is is likely damaged.
- If the pressure in a liquid nitrogen reservoir is above 20 psi, there is likely a problem with the pressure relief valve.
- Liquid helium vessels and cryostats should all be connected to the helium recovery system and the pressure should be slightly above atmospheric pressure.

If in doubt, contact a member of the cryoteam, or a technician, immediately.

- Do not leave liquid helium or nitrogen dewars in the hallways.
- **In labs, place them far away from where people are working.**

Personal protection equipment

- For pressurized vessels :




- For unpressurized vessels :



- Wear long pants.
- Wear closed shoes (closed at both ends).

Design of cryogenic systems

A risk evaluation form must be filled out and signed by the supervisor of the lab when designing a new cryogenic set-up.
(A model document is available at https : //www.physique.usherbrooke.ca/securite/)

- Any system that is under vacuum (for example for thermal insulation) or under pressure for cryogenic use must include pressure relief valves, both on the vacuum space and on the pressurized space.
- During design, consider in which direction the most mobile pieces of the set-up could be projected in case of an explosion.
- Carefully consider which materials to use and ensure that they are compatible with extreme cold.
- Make sure to have your design approved by the team of research technicians and professionals.

Public demonstrations

- Wear safety glasses.
- It is difficult to manipulate the levitating train with cryogenic gloves, but make sure to remove jewelry and watches.
- Wear closed shoes and long pants.
- Ask that spectators keep their distance so as to avoid splashing them with liquid nitrogen – especially children!
- Do not allow the public to manipulate cryogenic components (liquid nitrogen, levitating train, superconducting pucks, etc.).
- Do not allow spectators to get too close to neodymium magnets.
- Do not break frozen objects, as this presents a risk to spectators' eyes.

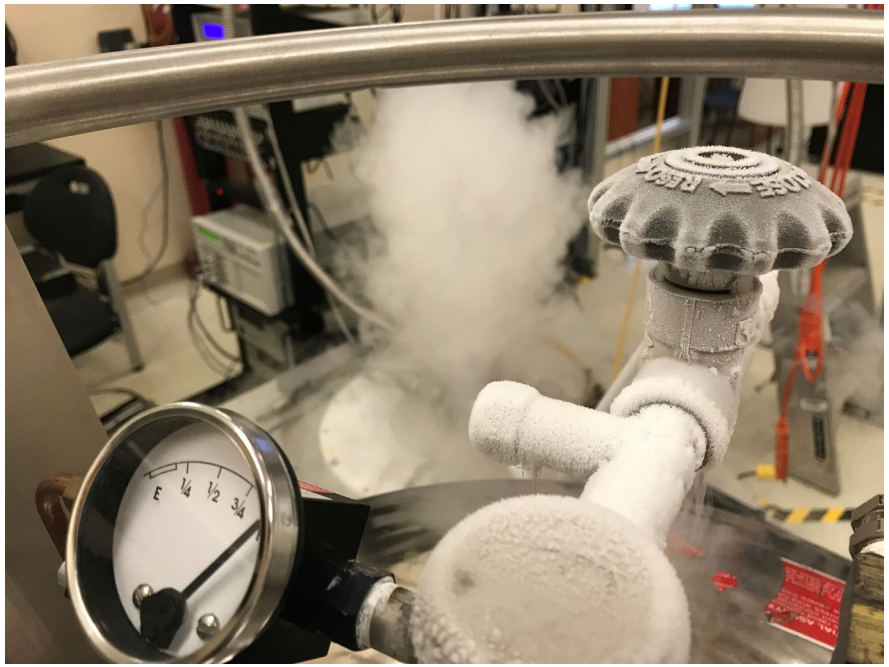
Good procedures to adopt in the lab

- Place cryogenic vessels far away from people and desks in the labs.
- Know the location of emergency showers and eye-rinse stations.
- Avoid working alone (especially nights and on weekends).
- Wear appropriate personnel protection equipment.
- Do not hang out in the mist produced during nitrogen transfers.
- Do not put your face in front of a pressure relief valve of a cryostat, especially when there is risk of a superconducting magnet “quenching”.
- Keep your face away from any pieces of the apparatus that could be ejected because of high pressure or explosion.
- Use pliers or cryogenic gloves to manipulate objects that were in contact with cryogenic liquids.
- Do not pour cryogenic liquids down the sink.
- Avoid using materials that become fragile when cold.
- Avoid excessive humidity as this can lead to excessive ice formation and may damage vacuum couplings.
- Limit access to storage spaces.

In conclusion

- Be aware of what is happening in your work space so as to detect problems before they become dangerous.
- Use adequate personal protection equipment.
- In case of emergency dial 811.
- When in doubt, ask for help from a professional or technician or else remove yourself from the situation.

Good luck with your experiments!



Use of compressed gas

- You should carry out a risk analysis with the lab's technical staff before using any gasses that are not inert.

Before using a compressed gas cylinder

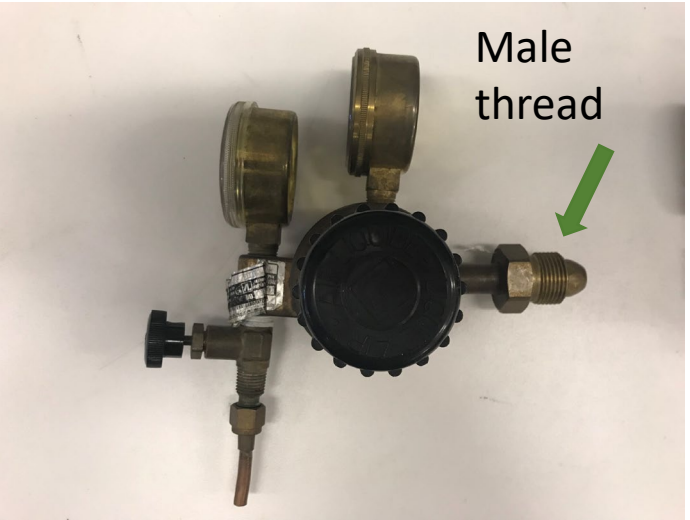
- The cylinder must be fixed to the wall or something else solid.
- The protective cylinder cap over the valve can only be removed when the cylinder is fixed in place.
- Never insert a tool into the cap to unscrew it.
- Request technical assistance in the case of a blocked cap.
- Never open the valve of a gas cylinder without a regulator in place.
- A compatible regulator must be adequately connected to a gas cylinder (without grease or teflon tape)
- Never modify or attempt to adapt a regulator.

Cylinders and connectors

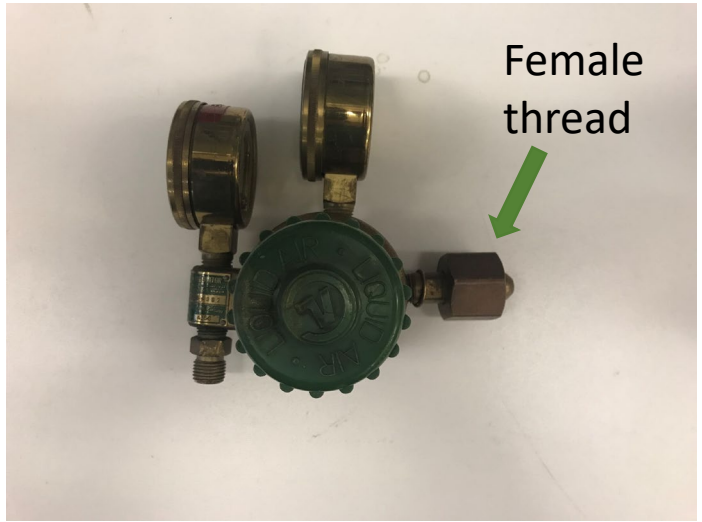
Compressed gas cylinders should only be connected to pressure regulators and equipment designed for the specific gas contained in those cylinders

- Regulators for inert gasses (N_2, Ar, He) are all identical.
- Oxygen and hydrogen, for instance, require specific regulators.

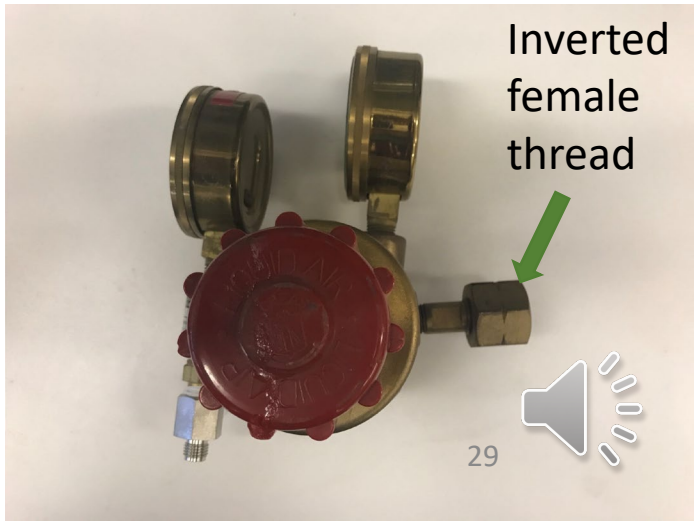
Regulator for inert gasses (N_2, Ar, He).



Oxygen regulator (O_2).



Hydrogen regulator (H_2).



The regulator

- A regulator is equiped wih two pressure gauges : one indicating the pressure within the cylinder and the other indicating the output pressure.
- It also has a pressure adjusting knob that allows you to set the desired output pressure.
- Usually there is also a small valve that is used to close the gas output.

Safety glasses are obligatory when manipulating compressed gasses.

Mode of operation

- Close the regulator by unscrewing the pressure adjusting knob (by turning counter-clockwise).
- Open the cylinder valve/tap (3/4 of a turn for rare gasses, completely for oxygen, verify the procedure for other gasses).
- Open the regulator to obtain the desired pressure (by turning the pressure adjusting knob clockwise).
- Open the small output valve that connects the regulator to the system you are seeking to pressurize.

When finished with the gas cylinder:

- First close the cylinder valve, allowing you to evacuate the pressure built up in the regulator.
- Then close the regulator (turn the pressure adjusting knob counter-clockwise) and finally close the small valve leading to your system.

Safety devices

- Compressed gas cylinders must be tested or inspected every 5 or 10 years. The date of each test should be engraved on the cylinder.

Safety / pressure relief valves: release valves are usually built into the cylinder. They are normally kept closed with a spring.

Burst disc: usually these discs are metal. They will burst or break open at a certain pressure releasing the gas contained within before a pressures reach a value that can cause a dangerous explosion.

Fusible plug: in this case it is high temperatures rather than high pressures that lead to the opening of this safety device.

Appropriate ventilation for poisonous gasses

- Certain poisonous gasses, like fluorine, require permanent air extraction.
- Cylinders containing these gasses are usually enclosed in a ventilated cabinet from which air is continuously extracted via the building roof.
- Any manipulations (regulator installation, connection to the experimental apparatus) must be carried out by a qualified technician.
- A gas detector and alarm must also be present in the lab where these gasses are to be used.

- In the case of inert gasses, simply avoid enclosed spaces.

Storage (for technical staff only)

- Always close the cylinder valve before removing the regulator and reinstalling the cap.

- Do not mix different gas types.
- Oxidants must be kept at least 6 metres away from combustible gasses or other combustible materials.
- The cap should immediately be put back onto the cylinder when it is not in use.
- Cylinders should not be stored near electrical panels.
- A chain needs to be used to prevent cylinders from falling over.
- Separate empty and full cylinders.
- Avoid competely emptying a cylinder (leave a pressure of about 20 psi).

- When not in use, oxygen cylinders must be stored outside (request assistance from technical staff).

Transporting compressed gas cylinders

- A specific type of cart should be used to transport cylinders within the building. It is stored in the helium liquefier room.
- A gas cylinder must be capped before being detached from the wall and moved. The cylinder must be solidly attached to the cart when moving.
- Users must not accompany compressed cylinders inside of a closed elevator.

Best practices

- Wear safety glasses.
- Never place anything on or hang anything from the valve of a gas cylinder (for example clothing).
- Remove the cap from a cylinder only once it is well secured to the wall.
- Never force a valve (contact the technical staff instead).
- Allow a frozen cylinder or very cold cylinder to warm up gradually before using.
- Only open the cylinder valve to 3/4 of a turn, which will allow you to quickly close it if there is a problem.
- Close the cylinder valve if it is not being used.
- If you are using compressed gas for a long duration, post a sign / note so that your colleagues can see that it is in use and has not just been left open by mistake.

Enclosed spaces should be avoided

Assistance and first aid

In case of emergency, dial 811 on campus from a land line or 819-780-0811 on a cell phone.
An application “Sécurité UdS” is also available.

For any questions concerning procedures, safety or first aid related to cryogenic experiments apparatus and compressed gasses, contact :

- The cryoteam, cryoteam@usherbrooke.ca
- SSMTE, info.sst@USherbrooke.ca
- UdeS security: 811

For additional information, please visit :
https://www.physique.usherbrooke.ca/securite/

Complete the exam on Moodle to obtain your authorization to work with cryogenic liquids and compressed gases.