Safe Handling & Use of Liquid Nitrogen and Other Cryogenic Gases

Laboratory Safety Training

Office of Engineering Safety
Texas Engineering Experiment Station (TEES)
& The Dwight Look College of Engineering

"A safe, healthful, and secure environment for scholarship and research."

Key Concepts

- Physical and Chemical Properties of Nitrogen
- Characteristics of Cryogenic Nitrogen and associated Hazards
- Handling Liquid Nitrogen
- Liquid Cylinder Construction
- Filling Cryogenic Dewar's
- Proper Personal Protective Equipment (PPE)
- Safely Moving Liquid Cylinders

Cryogenics

- The word *"Cryogenics"* is derived from two Greek words:
- *Kryos*, which means cold or freezing, and
- *Genes*, meaning born or generated

Cryogenic Gasses — Cryogenic Liquids

- When gasses are converted to liquid form they become cryogenic liquids
- Cryogenic liquids are liquefied gases that have a normal boiling point below -238°F (-150°C)
- The temperature difference between the product and the surrounding environment is substantial. Keeping this surrounding heat from the product requires special equipment to store and handle cryogenic liquids.
- A typical system consists of the following components: a cryogenic storage tank, one or more vaporizers, a pressure control system, and all of the piping required for fill, vaporization, and supply.
- The cryogenic tank is constructed like a vacuum bottle. It is designed to keep heat away from the liquid that is contained in the inner vessel. Vaporizers convert the liquid argon to its gaseous state. A pressure control manifold controls the pressure at which the gas is fed to the process.

Cryogenic Containers

- Cryogenic liquid cylinders are insulated, vacuum-jacketed pressure vessels. They come equipped with safety relief valves and rupture disks.
- The types of containers in use are the Dewar, cryogenic liquid cylinder, and cryogenic storage tank. Storage quantities vary from a few liters to many thousands of gallons.
- Since heat leak is always present, vaporization takes place continuously. Rates of vaporization vary depending on the design of the container and the volume of stored product.
- Containers are designed and manufactured according to the applicable codes and specifications for the temperatures and pressures involved.
- Liquid product is typically removed through insulated withdrawal lines to minimize the loss of liquid product to gas.

Cryogenic Containers, Cont'd.

- Insulated flexible or rigid lines are used to withdraw product from storage tanks.
- Connections on the lines and tanks vary by manufacturer.
- Liquid cylinders designed to dispense gaseous argon have valves equipped with standard Compressed Gas Association (CGA) outlets. Suitable pressure regulating equipment may be attached.
- Valves provided for the withdrawal of liquid product are also equipped with standard CGA outlets, but are different than the connections used for gaseous discs to protect the cylinders from pressure build-up.
- These containers operate at pressures up to 350 psig and have capacities between 80 and 450 liters of liquid.
- Product may be withdrawn as a gas by passing liquid through an internal vaporizer or as a liquid under its own vapor pressure.

Handling & Storage: Cryogenics

- Store and use cryogenics with adequate ventilation.
- Do not store in a confined space.
- Cryogenic containers are equipped with pressure relief devices to control internal pressure.
- Under normal conditions, these containers will periodically vent product. Do not plug, remove, or tamper with any pressure relief device.
- Never allow any unprotected part of the body to come in contact with un-insulated pipes or equipment that contains cryogenic product.
- The extremely cold metal will cause the flesh to stick fast and tear when one attempts to withdraw from it. Use a suitable hand truck for container movement.

Handling & Storage, Cont'd.

- Containers should be handled and stored in an upright position.
- Do not drop, tip, or roll containers on their sides.
- Do not remove or interchange connections.
- Contact the vendor if you experience any difficulty operating the container valve or with the container connections.
- Discontinue use. Use the proper connection. DO NOT USE ADAPTERS!
- Use piping and equipment designed to withstand the pressures to be encountered.
- On gas withdrawal systems, use a check valve or other protective apparatus in any line or piping from the container to prevent reverse flow.

Handling & Storage, Cont'd.

- To prevent cryogenic liquids or cold gas from being trapped in piping between valves, the piping should be equipped with pressure relief devices.
- Only transfer lines designed for use with cryogenic liquids should be used.
- Some elastomers and metals such as carbon steel may become brittle at low temperatures and will easily fracture. These materials must be avoided in cryogenic service.
- All vents must be piped (routed) to the the building exterior, in compliance with university rules and applicable codes.

PPE and Prudent Work Practices

- One must be thoroughly familiar with the properties and safety considerations before handling a cryogenic liquid and its associated equipment.
- The eyes are the most sensitive body part to the extreme cold of the liquid and vapors of cryogenic liquids.
- The recommended personal protective equipment for handling cryogens includes a full face shield over safety glasses, loose-fitting thermal insulated or leather gloves, long sleeve shirts, and trousers without cuffs.
- In addition, safety shoes are recommended for people involved in the handling of containers.
- Depending on the application, special clothing suitable for that application may be advisable.

PPE, Cont'd.

- **A special note on insulated gloves:**
  - Gloves should be loose-fitting so they are able to be quickly removed if cryogenic liquid is spilled on them.
  - Insulated gloves are not made to permit the hands to be put into a cryogenic liquid. They will only provide short-term protection from accidental contact with the liquid.
- **In emergency situations, self-contained breathing apparatus (SCBA) may be required.**
- **Cryogenic liquids are to be handled by authorized persons only.**
- **Students should not handle cryogenics unless trained and supervised by their faculty or laboratory manager.**

How Liquid Gases are Manufactured

- **Normal air is cooled.**
- **As the temperature drops, the gases will drop out separately, according to their boiling points.**
  - Liquid nitrogen will drop out at –320° F (-196° C)
- **The liquefied gas is collected.**
- **The liquid boils violently in trying to get back to its normal gaseous state.**

Comparison of Liquefied (Cryogenic) Gases

Gas	N_2 (nitrogen)	O_2	Ar	H_2	He
Boiling Point °F	-320	-297	-303	-423	-452
Boiling Point °C	-196	-183	-186	-253	-268
Volume Expansion	696	860	696	850	745

Characteristics of Cryogenic Liquid Nitrogen

Liquid Nitrogen:

- **Boiling Point (BP) = -320° F (-196 °C)**
- **Volume Expansion Rate = 696**
- **Will not support combustion.**
- **Will not support life**
- **As a liquid it is non-magnetic**

Handling 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.
- Transfer of liquid into warm lines or containers must be done slowly to prevent thermal shock and possible buildup of pressure.
- Always wear safety equipment, including heavy loose fitting leather or cryogenic gloves, and eye and face protection.
- High concentrations of escaping gas should not be allowed to collect in an enclosed area.
- Avoid prolonged breathing of cryogenic liquid vapors.

Handling Liquid Nitrogen

- **Avoid rough handling of liquid containers**
- **A cold outside jacket indicates a loss of vacuum**
  - Immediately 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 any fittings**
- **Maintenance & repairs by authorized persons only (APO)**

Why should Liquid Nitrogen be Stored Properly?

- Liquid nitrogen, when returned to the gaseous state, can displace oxygen from the air and can create an oxygen-deficient atmosphere under the right conditions.
- High concentrations of nitrogen reduce the breathable oxygen in the air.
- 1 cubic foot of liquid nitrogen will expand to 696 cubic feet of 100% gaseous nitrogen at 70° F
- Oxygen deficiency can lead to asphyxiation:
  - Asphyxia develops slowly as the oxygen content of air is gradually reduced from 21%.
  - Oxygen concentration below 19.5% is considered an oxygen deficient environment
  - Safe Range: ≥19.5% and ≤23%
  - The victim will not be aware of a problem and generally will not recognize the symptoms of gradual asphyxia from decreasing oxygen levels.

Proper Storage of Liquid Nitrogen

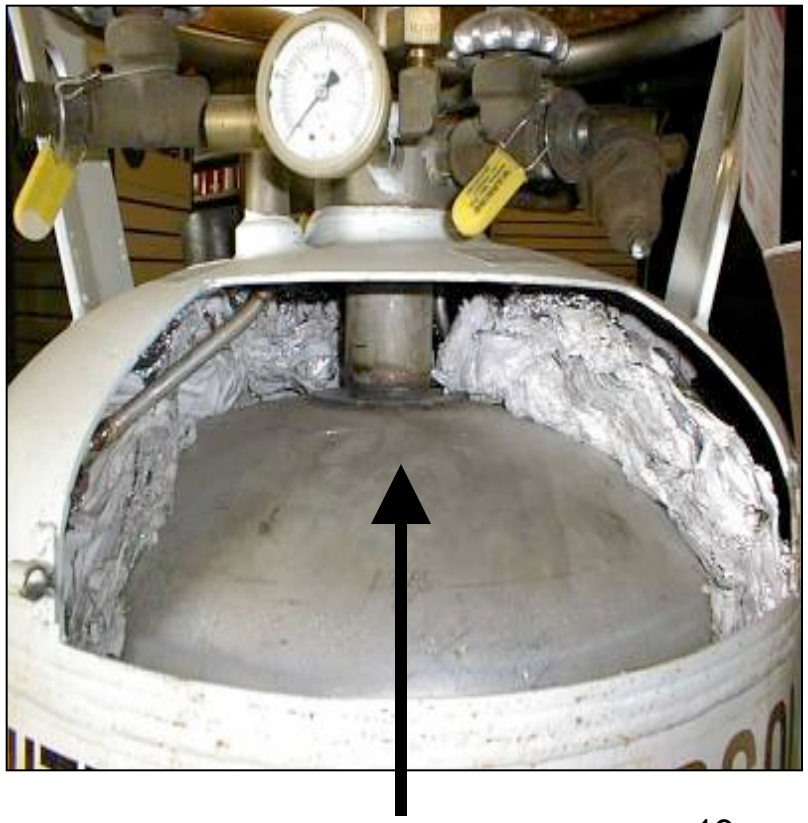
- All cryogenic liquids should be used & stored in well-ventilated areas.
- Caution signs should be posted in the area warning that liquid nitrogen is being stored and used.
- Oxygen monitors and alarms can be installed in areas where liquid nitrogen is stored and ventilation is minimal.
- Use only containers specially designed to hold liquid nitrogen. Check with the manufacturer of the container to ensure it is approved for holding liquid nitrogen
- 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.
- Filling between containers, leaking valves, & liquid tank venting are some examples that could lead to an oxygen deficient atmosphere.

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 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 valve marked for 350 psig.

Pressure in Pounds Per Square Inch (psi)

- The pound per square inch or, more accurately, pound-force per square inch (symbol: psi or lb-f/in^2 or lb_f/in^2) is a unit of pressure or of stress based on avoirdupois units. It is the pressure resulting from a force of one pound-force applied to an area of one square inch:
  - 1 psi = 6.894757 kPa {Pascal or Pa is the SI unit of pressure.}
- Other abbreviations are used that append a modifier to "psi".
- psia (pounds-force per square inch absolute) gauge pressure plus local atmospheric pressure.
- psid (psi difference) difference between two pressures.
- psig (pounds-force per square inch gauge).
- psivg (psi vented gauge) difference between the measuring point and the local pressure.
- psisg (psi sealed gauge) difference between a chamber of air sealed at atmospheric pressure and the pressure at the measuring point.

Pressure Relief Devices

- Hearing a slight hiss from a liquid cylinder is usually the normal operation of its pressure relief device.
- 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.
- Maintenance & repairs by Authorized Persons Only (APO)

Pressure Relief Device Formula

- **4L 292 = 350 safety**
- **4L 200 = 235 safety**
- **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.
    - Example (200 x 1.25) – 15 psi = 235 psi

Liquid-to-Gas Conversion

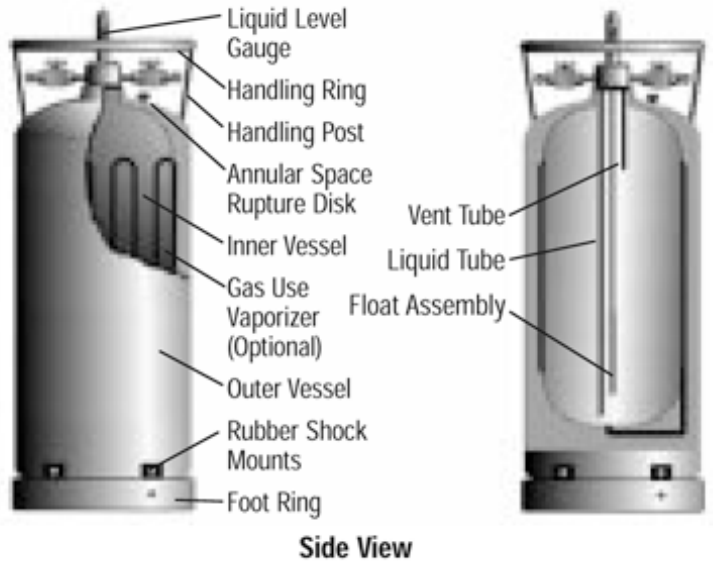
- **Since liquid is converted to gas at about 2.3% per day even under ideal container conditions, if the liquid is not used regularly, the vessel will be empty in a certain amount of time.**
- **It is important to estimate your use so the liquid will not be wasted.**
- **Always purchase "*Just the right amount, at just the right time*."**

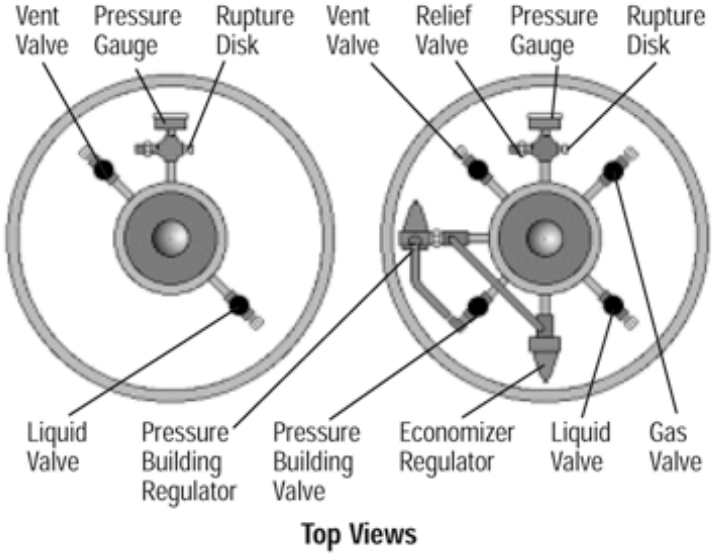
EXAMPLES: Liquid Cryogenic Cylinders

Examples: Liquid Cryogenic Cylinders

Examples: Liquid Cryogenic Cylinders

Examples: Liquid Cryogenic Cylinders





Typical Cryogenic Cylinder & Apparatus

Examples: Liquid Cryogenic Cylinders

Cryogenic Dewars

Examples: Liquid Cryogenic Cylinders

Cryogenic Dewars

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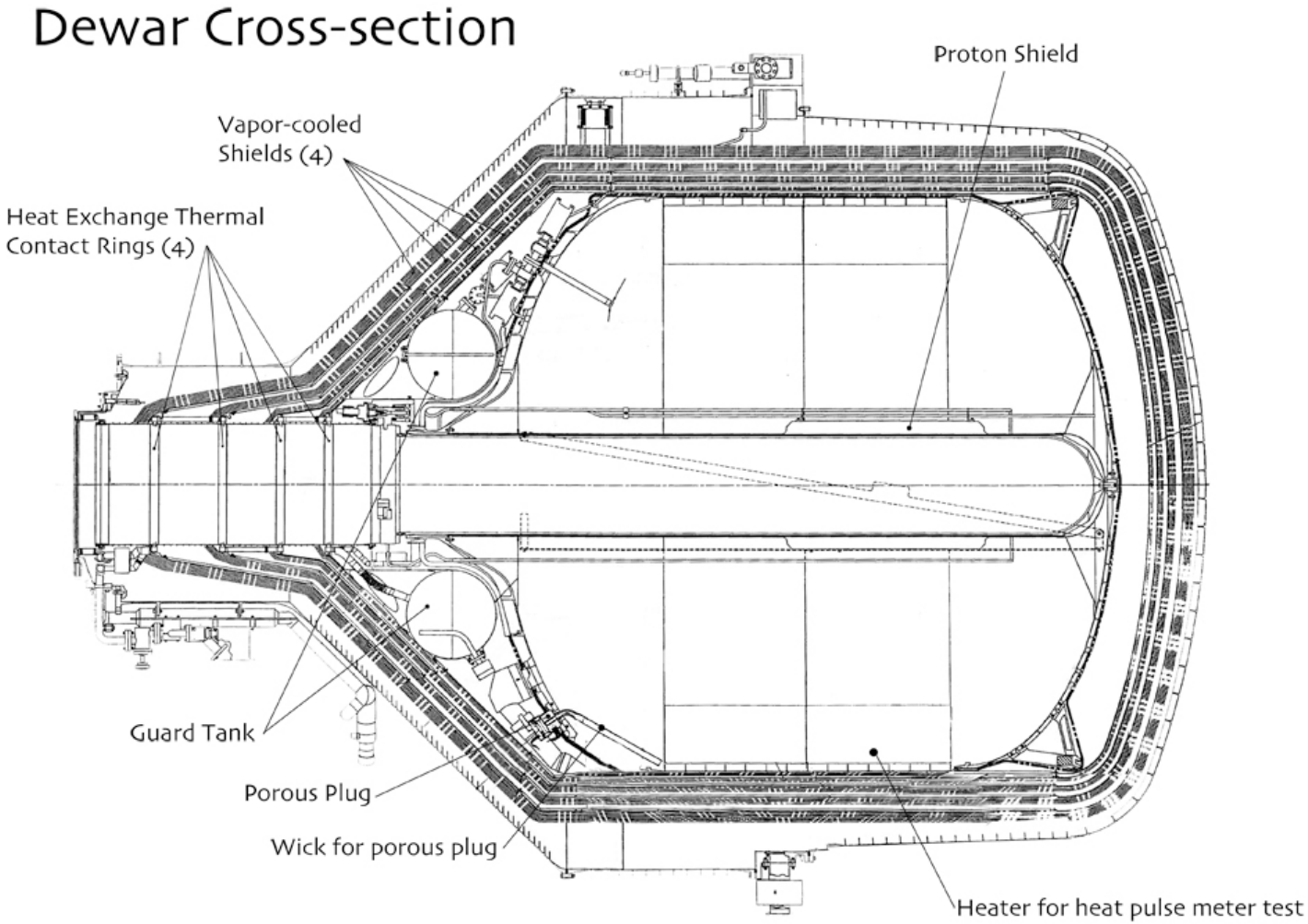
Cryogenic Dewars

Examples: Liquid Cryogenic Cylinders



Cryogenic Liquid Containers
(aka, Liquid Cylinders)

- **Advantages:**
  - Contain large volumes of gas
  - Provides a source of Cryogenic Liquids which can be easily handled

- **Two general types:**
  - Low Pressure - For dispensing of liquid only.
  - High Pressure - For dispensing of liquid and gas

Cryogenic Liquid Containers (Liquid Cylinders)

- **Always know the type of container(s) used by your lab!!**
- **Low pressure is used only for the delivery of LIQUID, not gas**
- **It's operating pressure is 22 psig**
- **Always check the type of container that is being delivered or before use.**
- **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 has an operating pressure of 22 psig!!!
  - High pressure operates at 230 psig or above.
- **Always check the pressure gauge to determine the type of container.**

Liquid Containers should always be inspected by the Vendor before and after filling.

Call the vendor immediately if you suspect a problem or concern.

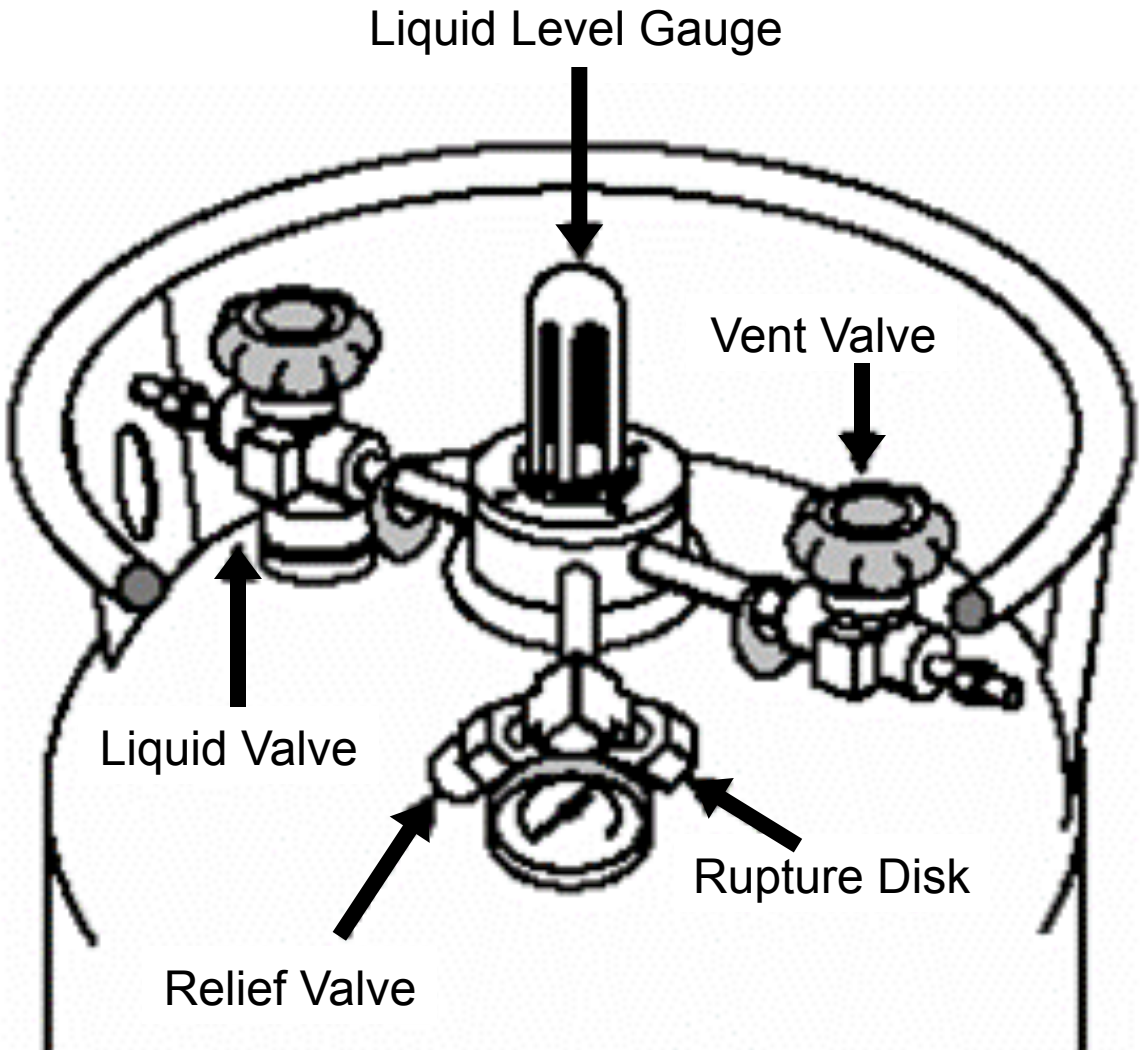
Items that are usually checked by the vendor before filling a liquid cylinder

- Visual Inspection
- Valve Inspection
- Pressure relief valve inspection
- Safety burst / rupture disc inspection
- Vacuum casing burst / rupture disk inspection
- Tare weight
- DOT-4L markings
- Vent valve
- Gross weight
- Net weight

Content Gauge on Liquid Cylinders

- The container contents 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 always filled by weight!

Illustration of Typical Gauge & Valve Configuration



Low Pressure Liquid Container Components

Liquid Withdrawal Valve

- Liquid is withdrawn through this valve

Pressure Gauge

- Displays internal pressure of the container

Contents 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

Low Pressure Liquid Container Components

Pressure Relief Devices (2)

- Protect vessel from over-pressurization
  - Re-seating spring-loaded relief valve releases at 22 psig
  - Burst disk rated to protect the inner vessel

Outlet Restraints

- These are to prevent the dangerous practice of changing outlet connections at user sites.
- Restraints may be twist ties, wire, or other.
- Removal of these restraints will void all product warranties!!
- 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

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 appropriate Personal Protective Equipment (PPE) when removing the frost.

Moving Liquid Cylinders

- **Liquid cylinders are heavy and cumbersome, especially when filled with liquid nitrogen.**
- **Cylinders may require two people to handle, in order to do so safely.**
- **Containers can cause crushing injury to the feet. Wear proper safety shoes.**
  - Tennis shoes and open toed shoes are not proper foot protection!!
- **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.**
- **Do not roll, either vertically or horizontally !**
- **Push, Don't Pull !**
- **If the container tips over, let it go !**

Moving Liquid Cylinders

- **Never try to roll liquid cylinders by using the Liquid Level Gauge tube.**
- **Note the proper stance and use of the cylinder's halo ring for moving.**

Elevator Transport

- **Use a freight elevator if possible.**
- **If a passenger elevator is used, it should be locked out to ALL other users.**
- **Do not transport a liquid container at any time in an elevator with any other personnel in the car, unless they have a supplied air respirator.**

Emergencies

- **If there is a large spill or rupture of a container,**
  - immediately call 9-911 or 911!
- **Warn others in building**
- **Evacuate!!**
  - There may be oxygen deficiency in the area of the spill!!
- **If there is injury to the body from liquid nitrogen, seek immediate medical assistance**

Emergencies

- **If liquid is splashed in the eyes, flush with water for at least 15 minutes. Seek immediate medical attention. Call 9-911 !**
- **Skin contact may cause frostbite and burns. Soak affected part in tepid water and seek immediate medical attention. Call 9-911 !!**
- **Skin contact is a medical emergency. Lack of prompt medical attention may result in amputation!!!**
- **CALL 9-911 or 911 !!!**

Potential Problems

- Head pressure
- Results when heat leaks into the container
- The safety valve will periodically release this pressure
- If the safety 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

Personal Protective Equipment (PPE) for Cryogenics

- **If you fill cryogenic liquids the possibility of cryogenic liquid coming in contact with the skin is reduced with the use of proper Personal Protective Equipment (PPE).**
  - Full Face Shield with safety glasses
  - Heavy, Loose Fitting leather or Cryogenic Gloves
  - Long Sleeve Shirt, or Arm Protection
  - Pants should be cuff-less
  - Do not tuck pants into shoes, boots.

Removing Liquid from Cylinders

- **Caution!!**
- **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**
- **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!**

Compressed Gas Association (CGA)

Caution!!!

- Before use, always confirm that the Compressed Gas Association (CGA) 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, notify your supervisor or supplier/vendor

- **Compressed Gas Association (CGA)**
  - http://www.cganet.com/

Liquid Helium

- Super cold at - 452° F (- 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

Liquid Oxygen

- Not as cold as liquid nitrogen
- Incompatible with flammable, organic, and combustible materials.
- Avoid contact with heat, sparks, and flame.
- Post “NO SMOKING OR OPEN FLAMES” signs in areas that use liquid oxygen
- Highest volume expansion rate of the Liquid Gases (860)

Liquid Hydrogen

- Super cold at - 423° F (- 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 Gases (850)

Liquid Argon

- Argon is the most plentiful of the rare gases, making up approximately 1% of the earth's atmosphere. It is nontoxic and largely inert
- It is monatomic and extremely inert, forming no known chemical compounds
- Will not support life or combustion, and has no warning properties
- Liquid argon has a boiling point of -302.6°F (-185.9°C)
- Extensive tissue damage or burns can result from exposure to liquid argon or cold argon vapors
- It can act as a simple asphyxiant by displacing the oxygen in air to levels below that required to support life
- Inhalation of argon in excessive amounts can cause dizziness, nausea, vomiting, loss of consciousness, and death. Death may result from errors in judgment, confusion, or loss of consciousness that prevents self-rescue. At low oxygen concentrations, unconsciousness and death may occur in seconds and without warning.

Resolving Common Problems

Issue: Gas vents intermittently through safety relief valve

Possible cause:

- Probably normal operation.
- Gas generated due to heat leak into cylinder causes head pressure to build

Recommended Activity:

- Ensure inactive containers are stored in well ventilated area.
- Rotate inventory.
- Purchase “Just the right amount, at just the right time.”

Resolving Common Problems

Issue:

- Gas vents continuously through safety valve

Possible Cause:

- Possible relief valve failure or excessive heat leak

Recommended Activity:

- Remove 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.
- Call vendor for inspection and/or replacement.

Resolving Common Problems

Issue:

- Gas vents during use through safety relief valve

Possible Cause:

- Set point on regulators exceeds safety relief valve setting

Recommended Activity:

- Reduce set point on pressure building regulator.
- Contact supervisor or supplier for assistance, maintenance, and/or repair.

Resolving Common Problems

Issue:

- Pressure in the container is low

Possible Cause:

- Leak from container

Recommended Activity:

- Use appropriate leak detection fluid to check for leaks in connections.
- Examine container for signs of frost.
- If leaks are on container itself, contact supervisor or supplier

Resolving Common Problems

Issue:

- Pressure in the container is low

Possible Cause:

- Pressure building valve is not fully opened

Recommended Activity:

- Open valve fully

Resolving Common Problems

Issue:

- Pressure in the container is low

Possible Cause:

- Pressure building regulator not set high enough

Recommended Activity:

- Adjust to increase pressure-contact supervisor or supplier

Resolving Common Problems

Issue:

- Pressure in the container is low

Possible Cause:

- Pressure building valve is open

Recommended Activity:

- Close the valve if frost is visible on the pressure building vaporizer near the bottom of the tank
- Contact supervisor or supplier

Resolving Common Problems

Issue:

- Pressure in the container is too high

Possible Cause:

- Leaking or improper setting of pressure building regulator

Recommended Activity:

- Reduce regulator setting to achieve desired pressure level
- Contact supervisor or supplier

Resolving Common Problems

Issue:

- Pressure in the container is too high

Possible Cause:

- Vacuum integrity failing

Recommended Activity:

- If container walls are covered with frost, contact supervisor or supplier

Resolving Common Problems

Issue:

- Container top covered with frost

Possible Cause:

- High product use

Recommended Activity:

- Normal operation

Resolving Common Problems

Issue:

- Container top covered with frost

Possible Cause:

- High product use

Recommended Activity:

- Normal operation

Resolving Common Problems

Issue:

- Container surface is uniformly covered with frost

Possible Cause:

- Vacuum integrity compromised

Recommended Activity:

- If accompanied by a high rate of product venting through the safety relief valve, or high rate of pressure increase, call supplier

Prepared by:

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SAFETY
FIRST
THE SAFE WAY IS
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