SAND2008-2582P

NOTE TO PROGRAMMER – THIS IS HOME PAGE

Introduction

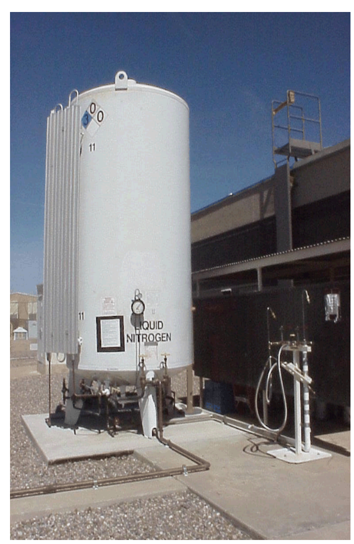
This is a web-based refresher course for Cryogen Safety (PRS115). Completion of the classroom taught Cryogen Safety class (PRS115) is a prerequisite for taking this web-based refresher course. For a more thorough alternative approach to the refresher requirement, you can repeat the classroom taught Cryogen Safety class (PRS115). Course material for the classroom taught Cryogen Safety (PRS115) is located on the SNL Pressure Safety Homepage at http://psi.sandia.gov/ . Cryogen Safety training should be considered awareness level only – and should compliment any applicable on-the-job or site-specific training.

At the end of this course you should be able to:

1. Identify pressure safety program requirements
2. Identify cryogenic fluid properties
3. Identify potential hazards and mitigations
4. Identify PPE (Personal Protective Equipment) requirements
5. Identify safety concerns and practices associated with laboratory storage Dewars

This course will consist of 4 modules. There will be an examination after each module. You must answer each of the questions correctly on each of these modules or you will be directed to review the module again and then retake the exam. The course modules are:

- Module 1 – Pressure Safety Program Overview of Requirements
- Module 2 – Cryogen Safety and Associated Hazards
- Module 3 – Laboratory Storage Dewars
- Module 4 – Additional Cryogen Safety Hazards



Module 1 – Pressure Safety Program Overview of Requirements

The goal of the SNL Pressure Safety Program is to provide a basis for a safe environment for pressure-related applications. Our program incorporates national consensus codes as well as DOE requirements. The major elements of the program are summarized:

- **Training and qualification of personnel**: required training includes Cryogen Safety (PRS115) for any cryogen applications and, where applicable, Pressure Safety Orientation (PRS150) and Advanced Pressure Safety (PRS250). Qualification may include site or organization specific training or equipment-specific training. Complete details on training and qualification requirements as well as recommended Qualification forms are in the Pressure Safety Manual Chapter 2.
- **Documentation:** in the form of a data package is required of all SNL pressure systems, including cryogen applications. The data package should reference literature from equipment manufacturers such as owner / equipment manuals, etc. and also document additional system designs such as piping / venting concerns, location and set pressure of pressure relief valves, etc. In addition, PPE (Personal Protective Equipment) requirements should also be documented in the data package. An alternative is to document the PPE requirements in an operating procedure.
- **Maintenance and reevaluation criteria**: should be established as applicable (such as the re-test or replacement intervals for pressure relief valves). Note these criteria and intervals in the data package.
- **The SNL Pressure Safety Homepage:** (http://psi.sandia.gov/) contains requirements and practices as described in the Pressure Safety Manual (CPR400.1.1.27) and the supplemental manual Safe Handling of Cryogenic Fluids (CPR400.1.1.36). The Homepage also contains design guidance, bulletins, etc. In order to receive updates and new information related to cryogenic or pressure applications, subscribe to "Pressure Safety Issues" via the SNL Subscription Notification Service.
- **Advice and Assistance**: can be provided by your Pressure Advisor (see the Homepage for a list of Pressure Advisors). Pressure safety SMEs are available in the Safety Engineering Organization to assist personnel with design, documentation, operational, or maintenance functions. Contact Roger Shrouf or Shane Page for further assistance.

Points to remember:

- Training requirement = PRS115 (add PRS150 and / or PRS250 as applicable for pressure applications)
    - may also include site specific or equipment specific training
- Systems documented (data package / manufacturers information)
- establish and document maintenance / reevaluation criteria (i.e., pressure relief valve replacement intervals)
- Homepage = psi.sandia.gov (Manual, Bulletins, assistance personnel / SMEs, other)

Module 1 – Pressure Safety Program Overview of Requirements

SUPPORTING INFORMATION

ES&H Manual and Supplements

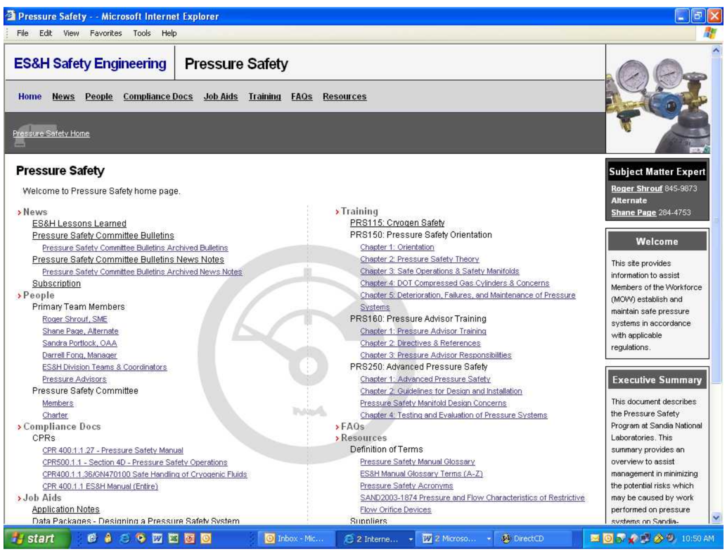
Pressure Safety Manual (cryo applications referenced)

Safe Handling of Cryogenic Liquids

SNL References

Facilities Administrative Procedures (defines cryogen system ownership and responsibilities)

Pressure Safety Home Page = http://psi.sandia.gov/



Point to Remember: In order to receive safety bulletins and other information related to pressure and cryogen safety, please subscribe to Pressure Safety Issues from the SNL Subscription Notification Service.

ES&H Support Team (Safety Engineering) Pressure Safety Program Personnel

Safety Engineering / Pressure Safety SME support http://psi.sandia.gov/

- Roger Shrouf at 845-9873 or Shane Page at 284-4753

Industrial Hygiene Support

- assigned by organization I.H. Program Homepage @ https://oracleportalp.sandia.gov/portal/page/portal/Center_4100/4120_Home/4127_Home

Other Assistance Vendors / Consultants / Additional Information

Matheson TriGas - JIT supplier for gases and cryogenic liquids at http://www.mathesontrigas.com/

CRYOCO (training and consultation at http://www.cryoco.com/

Safety in the Handling of Cryogenic Fluids

by F. J. Edeskuty and W. F. Stewart

1996 Plenum Press N.Y. {ISBN # 0-306-45161-1}

Module 1 – Pressure Safety Program Overview of Requirements Test Bank

Question 1

What training is needed, at a minimum, to handle cryogenic liquids?

a. PRS115, Cryogen Safety

b. Applicable equipment or site specific training

c. **Both a and b**

d. None of the above

Which of the following training is required to handle cryogenic liquids?

a. PRS115 – Cryogen Safety

b. PRS150 – Pressure Safety Orientation

c. PRS250 – Advanced Pressure Safety

d. All of the above

Question 2

All pressure systems, including cryogenic systems, require documentation in the form of a data package.

a. True

b. False

What form of documentation is required for all SNL pressure systems?

a. Data Package

b. System operations manual

c. System maintenance manual

c. None of the above

Question 3

Where can you find requirements and practices as described in the Pressure Safety Manual and the supplemental manual Safe Handling of Cryogenic Fluids?

a. The SNL Pressure Safety Homepage (http://psi.sandia.gov/)

b. JIT Suppliers

c. Both of the above

The SNL Pressure Safety Homepage contains requirements and practices from what manual(s)?

a. Pressure Safety Manual

b. Safe Handling of Cryogenic Fluids Manual

c. None of the above

d. Both a and b

Module 2 - Cryogen Safety and Associated Hazards

After first describing cryogenic fluid properties, we will cover the following common hazards associated with cryogenic applications:

- Oxygen Enrichment
- Asphyxiation (oxygen deficiency)
- Pressure Build-up

Module 2 - Cryogen Safety and Associated Hazards

CRYOGEN PROPERTIES

Cryogenic liquids are gases that have been transformed into extremely cold liquids which are stored at low temperature and low pressures in specially constructed, multi-walled, vacuum-insulated containers. Cryogens commonly used in SNL applications include liquid nitrogen, liquid argon, and liquid helium.

The relationship of liquid nitrogen (LN_2) and liquid oxygen (LOx) should be noted on the chart below – with the normal boiling point of LN_2 being colder than LOx. The safety concern is that LN_2 is cold enough to condense the oxygen from the atmosphere – with the condensate being approximately 50% LOx. This creates a potential fire hazard. In addition, the large liquid to gas expansion ratio for the various cryogens should also be understood. A small amount of liquid accidentally released will boil-off rapidly (flash) to a much larger quantity of gas. This will be highlighted when we discuss asphyxiation hazards. Also note the potential pressure build-up if a cryogenic liquid is trapped and allowed to warm to room temperature.

Cryogen Properties

Temperature {Normal Boiling Point at 1 atm}

{ Kelvins }	{ ° F }	{ ° C }	Cryogen {Temp. @ 1 atm.}		Liquid to Gas Expansion Ratio	Pressure build-up of trapped liquid warmed to room temp.
273	32	0	{ice melts}			
194.6	-109	-78.3	carbon dioxide $CO_2 \approx$ 840 psig @ 70 ° F	{CO_2}	1 lb : ≈ 8.7ft^3	
111.6	-258	-161.4	liquid methane	{LCH_4}	1 : 578	
90.2	-297	-182.7	liquid oxygen	{LOx}	1 : 860	
87.3	-302	-185.5	liquid argon	{LAr}	1 : 847	
77.4	-320	-195.6	liquid nitrogen	{LN_2}	1 : 696	43,000 psig
20.3	-423	-252.7	liquid hydrogen	{LH_2}	1 : 851	28,000 psig
4.2	-452	-268.8	liquid helium	{LHe}	1 : 757	18,000 psig

Points to remember:

- Air can be condensed to an oxygen enriched liquid at LN_2 temperatures
- Argon, nitrogen, and oxygen can be condensed to solids at LH_2 or LHe temperatures
- All of the above substances are condensed to solid at LHe temperatures (except helium itself)

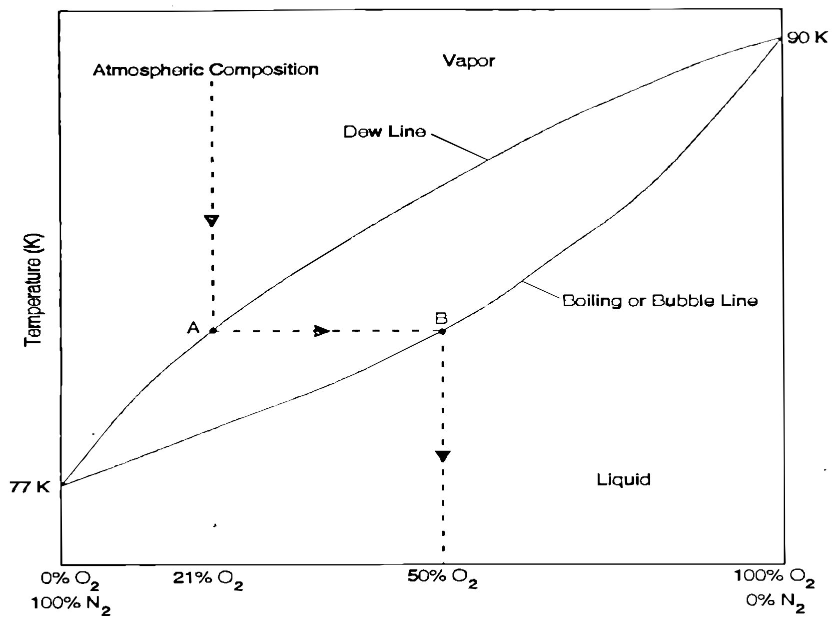
Module 2 - Cryogen Safety and Associated Hazards

Oxygen Enrichment Hazards

Oxygen (liquid or gas phase) can be very reactive and enhance combustion. Concentrations of 23 % or greater should be considered oxygen enriched. Condensation of air at liquid nitrogen temperature (or colder temperatures such as liquid helium or liquid hydrogen) produces an oxygen enriched condensate - at liquid nitrogen temperatures, the condensate is ≈ 50 % liquid oxygen. This condensate would be slightly blue in color. Lessons Learned Info – liquid nitrogen cold traps, accidentally left open to the atmosphere, can condense oxygen enriched air into the trap.

This oxygen enriched liquid can cause a fire / explosion hazard if it condenses into incompatible cryogens such as liquid hydrogen – or drips onto an incompatible surface such as asphalt - or contacts materials contaminated with incompatible oils or greases. Good practices include the use of insulation to prevent the exposure of low temperature surfaces to the atmosphere and keeping equipment used for cryogenic liquids clean and free of hydrocarbons such as oily contamination - as if it were to be used in an oxygen application. Vacuum pumps used to pump oxygen enriched gases must be rated for oxygen service.

Air – condensed at liquid nitrogen temperatures – results in an oxygen rich condensate.



Points to remember:

- The presence of oxygen in concentrations greater than 23 % can greatly enhance combustion.
- Liquid nitrogen (or cryogenic liquids colder than liquid nitrogen such liquid helium or liquid hydrogen) will condense air into an oxygen enriched condensate.

Module 2 - Cryogen Safety and Associated Hazards

Asphyxiation (O_2 Deficiency) Hazards

The use of cryogenic fluids can present a very serious asphyxiation hazard. Unfortunate accidents, including fatalities, occur every year from asphyxiation – this may be the single greatest hazard associated with cryogen applications. Cryogens have large liquid to gas expansion ratios – the gas that evaporates from even a small liquid release can displace air and cause an oxygen deficient environment. Cryogens should be used in large areas with good ventilation. Some gases, such as argon or carbon dioxide, are heavier than air and can create asphyxiation hazards in low lying areas or recessed pits. Nitrogen gas (molecular weight = 28) is slightly lighter than air (molecular weight = 28.96) – but nitrogen gas coming directly from liquid nitrogen releases is cold and dense and behaves as if it were a heavier gas until warming occurs.

% Oxygen	**"At-rest" Symptoms**
20.9 % ……………	none – standard atmospheric conditions
19.5% …………….	OSHA "oxygen deficient" level – no noticeable symptoms (some minor physiological effects)
16% ………………	Impaired thinking, reduced coordination, …
14% ………………	abnormal fatigue, emotional upset, impaired coordination / judgment, ...
10 - 12.5% ……...	impaired respiration (potential permanent heart damage), nausea, …
< 4% ……………..	Unconscious within seconds (death within minutes)

Be aware – an uncontrolled release of house nitrogen (gas or liquid phase) may create a < 4% oxygen environment in the typical R&D laboratory!

The asphyxiation hazard is directly related to the volume of the cryogenic liquid source – with lab Dewars presenting significantly less hazard than "house" systems. ("House" refers to systems with a very large tank outside – and distribution piping within the building.) Select Dewars as the source for cryogenic liquids whenever possible. The asphyxiation hazard is also related to the volume of the room where they are used and the ventilation rate within that room. Always use cryogens in large, well ventilated areas.

Manual valves left open and unattended (and forgotten) represent a common accident scenario for developing asphyxiation hazards. Tragically, fatalities have occurred when a user opens a valve to fill a Dewar, leaves the area and subsequently forgets about the Dewar. Eventually, the user remembers the Dewar was filling (now overfilling). The user runs in to close the valve – and potentially in to a severely oxygen deficient environment. Manual valves on cryogenic liquid systems should be attended when open and in use. Automated systems can be purchased for automatic shut-down of the source when the Dewar is filled.

Good engineering practices and procedures can help to reduce the asphyxiation hazard – but oxygen monitoring may still be required in many instances. For example, any use of a "house" source of liquid cryogen, such as liquid nitrogen, will also require oxygen monitoring as per the requirements of the ES&H Manual Chapter 6-T. Your ES&H Support team can help to evaluate, mitigate and document potential asphyxiation hazards.

Points to remember:

- Cryogenic liquids have large liquid to gas expansion ratios
- Spills and accidental releases can easily create an oxygen deficient environment within a lab
- The asphyxiation hazard is proportional to the volume of the source – use small volume dewars whenever possible
- Manual valves should be attended when open and in use in order to prevent accidental releases of cryogen
- See ES&H Manual Section 6T for complete details on potential Asphyxiating Environments

Module 2 - Cryogen Safety and Associated Hazards

Pressure Build-up Hazards

Trapped cryogens, upon warming, can generate very high pressures. As an example, liquid nitrogen trapped between two closed valves in a piping system, will generate a pressure of approximately 43,000 psig upon warming to room temperature. Pressure relief must be considered for the following areas:

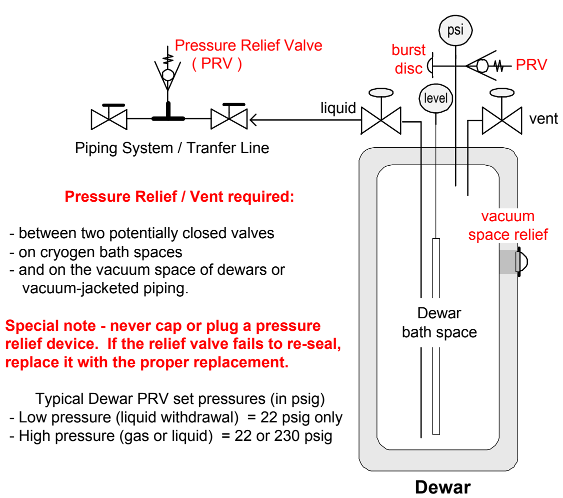
Piping: Pressure relief valves (PRVs) or other vents must be rated for the application (LN_2 , CO_2 , etc.), properly installed to avoid cold shock and ice build-up, properly vented to a safe location, and maintained / replaced periodically.

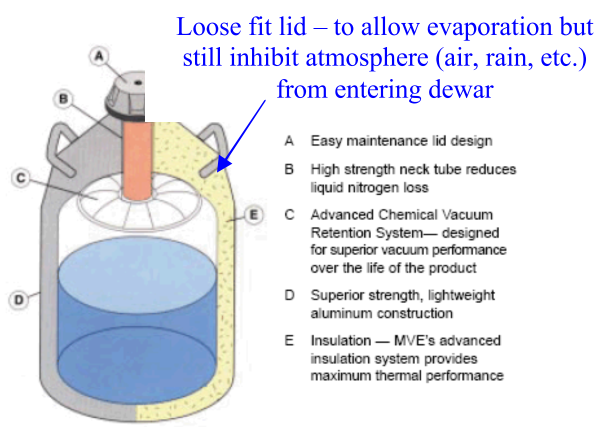
Special attention should be given to pressure relief devices (relief valves or rupture discs) connected to house systems because of their potential to fail to re-seal and release large quantities of cryogen. These applications call for a Facilities design where the relief device must vent to a safe location.

Vacuum insulation: Cryogenic liquids are frequently handled in piping or containers that are insulated by vacuum. This vacuum insulation space could become pressurized by cryogenic liquid leaking into the space. Upon warming, the release of condensed liquids or solids within the space (such as condensed air from an air leak in the vacuum space of a liquid helium Dewar) could also create a pressure hazard. Pressure relief for vacuum sections is frequently provided by relief valves or burst disks, vacuum valves with a relief feature incorporated into the design of the valve, or by an o-ring sealed flange held in place by the force of vacuum itself. Another good practice is to actively pump on the vacuum space during warm up.

Cryogen Baths: Even when the bath space is well insulated, the cryogen bath must allow for venting gases from the normal evaporation of cryogens. This is frequently accomplished by using open vent tubes, pressure relief valves and / or burst discs, or intentionally loose fitting caps or covers. An experimental volume is sometimes designed by a researcher and must also address concerns such as pressure relief, condensed air, and ice build-up.

Cold Trap Applications: Cold traps will, upon warming, release (or re-generate) the trapped material. The potential regenerated pressure should be pumped off or otherwise properly vented to prevent an overpressure upon warming.





These 1960s vintage Linde Dewars (25 liters in size) lack the required pressure relief on the vacuum space and can lead to accidents. If you have this type of Dewar, please contact your Safety Engineering representative for guidance.

LN2 Dewar Rupture

Points to remember:

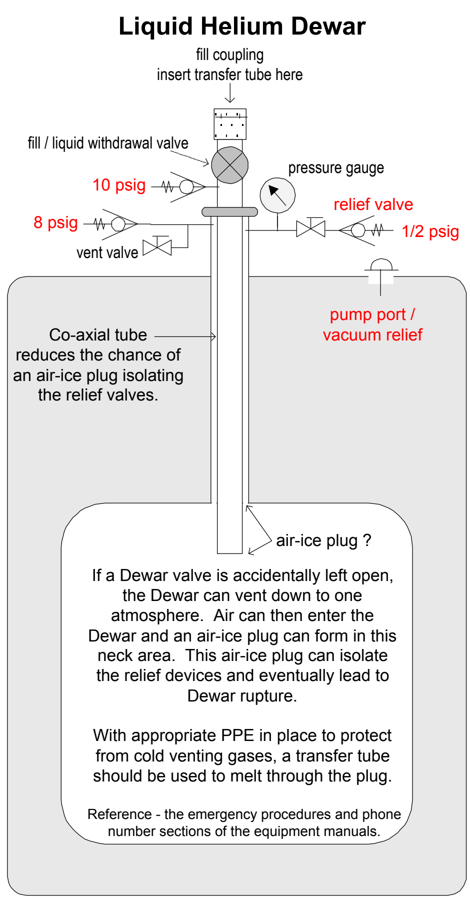
- Trapped cryogens, upon warming, generate very high pressures
- Pressure relief is required for all cryogen baths and potentially trapped spaces – including vacuum spaces
    - Use pressure relief valves, burst discs, open vent tubes, loose fitting caps / lids to allow venting
    - Pressure reliefs must vent to a safe location

Special Considerations for Liquid Helium Dewars

Dewar explosions are rare – with most occurring in liquid nitrogen, argon, or oxygen applications where the relief devices were intentionally plugged. Leaking relief devices should be replaced – never cap / plug a relief device in an attempt to stop the device from leaking. Liquid helium Dewars can overpressurize to the point of failure even with the relief valves still in place. An overpressure can be caused by an ice plug formed in the neck of the Dewar which can isolate the helium reservoir from the relief devices located on the top of the Dewar.

Liquid helium Dewars are normally stored at less than 1 psig - slightly higher pressures are used when transferring liquid from the Dewar. If the Dewar valve(s) is accidentally left open, the Dewar pressure will vent completely down to atmospheric pressure. It is then possible for air to enter the neck of the Dewar. Under these conditions, air (primarily nitrogen and oxygen) can freeze into a solid plug at the bottom of the tube leading to the helium reservoir. This air-ice plug can isolate the relief devices on the top of the Dewar – eventually leading to a catastrophic rupture of the Dewar. The co-axial tube can help to limit the formation of the air-ice plug to only the inner tube and thereby not isolate the relief devices. This tube is not always in place on all Dewars. Note – nitrogen / argon / oxygen Dewars are configured differently – accidentally leaving valves open will not isolate the relief devices.

If an air-ice plug is suspected, the user should break through / or melt through the plug (with appropriate PPE in place) in order to prevent pressure build-up. The user's helium transfer probe can typically be used for this purpose – or a probe incorporating a helium gas purge could be used. Consult the Dewar Operator / Owner Manual or contact the Dewar manufacturer for more information on these topics of concern.



Module 2 - Cryogen Safety and Associated Hazards Test Bank

<u>**Question 1**</u>
Air, condensed at liquid nitrogen temperatures, produces a potentially hazardous oxygen enriched condensate.

a. **True**
b. False

Pressure relief for vacuum insulation space can be provided by

a. pressure relief valves
b. actively pumping on the vacuum space during warm up
c. o-ring sealed flange held in place by the force of vacuum
d. **any or all of the above**

<u>**Question 2**</u>
Pressure relief for cryogen piping systems

a. is required between any two potentially closed valves
b. is only required for "house" cryogen piping systems
c. must vent to a safe location
d. both a and c

A cryogen bath space

a. requires venting for the normal evaporation of the cryogenic liquid
b. does not require venting if the bath space is well insulated

<u>**Question 3**</u>
The asphyxiation hazard may represent the single greatest hazard associated with the use of cryogenic liquids.

a. True
b. False

The asphyxiation hazard associated with cryogenic applications

a. is of greater concern with large volume cryogen sources such as "house" liquid sources
b. is of greater concern when using small hand-held laboratory dewars
c. is of greater concern when the lab space is quite small and / or poorly ventilated
d. **both a and c**

<u>**Question 4**</u>
Which of the following presents the greatest asphyxiation hazard?

a. "House" or large volume liquid nitrogen
b. an 8 liter Dewar of liquid oxygen
c. a small 4 liter Dewar of liquid nitrogen
d. None of the above

Nitrogen is an asphyxiant gas which provides adequate warning properties – and oxygen monitoring is not applicable for nitrogen hazards.

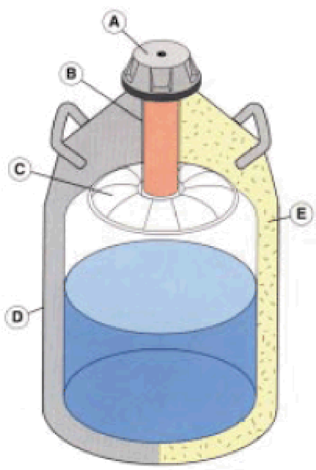
a. True
b. False

Module 3 - Laboratory Storage Dewars

Open / non-pressurized storage Dewars are used for hand pouring or other common laboratory applications. These Dewars are typically vacuum insulated with overpressure protection provided on the vacuum space - and supplied with intentionally loose fitting caps or covers to allow the venting of normal boil-off gases without pressurization of the bath space. Use special caution when working with glass dewars. As in all pressure (or in this case, vacuum) applications, the glass can implode or otherwise shatter and throw pieces some distance – eye protection shall be worn when working with glass Dewars. Due to their construction, glass dewars typically do not have overpressure protection on the vacuum space. In addition, it is common to shield the outer jacket of glass Dewars by using tape or a plastic mesh to contain fragments if the Dewar implodes.

Open / non-pressurized Dewars should be filled by inserting a transfer tube into the Dewar. With the operator standing back a safe distance (i.e., hands and face away from the point of discharge) slowly open the valve to fill the Dewar. Smaller Dewars are typically used in hand-pouring type of operations. For larger Dewar sizes, liquid can be dispensed through transfer tubes (rather than lift and pour) to reduce the potential for back injury. Use only OEM (Original Equipment Manufacturer) transfer tubes incorporating proper overpressure protection. When filling Dewars, do NOT leave manual valves open and un-attended. Auto-fill systems are available from various suppliers to fill Dewars and automatically shut-off when full.


The lid (A – in the drawing below) is intentionally loose fitting to allow for the venting or normal boil-off gases, but it will still inhibit atmosphere (air, rain water, etc.) from entering the Dewar. If rain water enters the dewar, it is possible to form an ice plug across the neck tube with resultant pressurization of the dewar. The lid can then be ejected with significant force in a directional fashion from the dewar neck. Another hazard that can occur is sudden boil-off of the liquid resulting in a splashing from the dewar neck. Pay attention to these potential directional exposures and do not place your head / face directly above the Dewar opening. These hazards validate the need for the appropriate level of PPE when handling liquid cryogens.



A Easy maintenance lid design

B High strength neck tube reduces liquid nitrogen loss

C Advanced Chemical Vacuum Retention System— designed for superior vacuum performance over the life of the product

D Superior strength, lightweight aluminum construction

E Insulation — MVE's advanced insulation system provides maximum thermal performance

Containers for cryogen applications must be properly rated for the application. Do NOT use consumer containers (such a Thermos bottle) to contain cryogens. Although the Thermos bottle is capable of holding a cryogenic liquid, it would be unsafe to use it in a cryogenic application. The lid design does not allow for proper venting of the boil-off gases and the vacuum space does not incorporate overpressure protection.

Points to remember:

- Open / non-pressurized storage dewars must have lids / covers that allow for the venting of boil-off gases – and should have overpressure protection on the vacuum space.

- Glass Dewars can shatter / implode – PPE should be used when working with any glassware under vacuum (or pressure)
- Keep hands and face away from potential directional splashes.
- When filling Dewars, do NOT leave manual valves open and un-attended.

Module 3 - Laboratory Storage Dewars

Pressurized Laboratory Storage Dewars for Liquid Nitrogen or Argon Applications

Pressurized storage Dewars can be used to dispense liquid or, in some cases, low pressure gas can also be supplied. The pressure in the Dewar is determined by the set pressure of the relief valve. There is typically a burst disk in parallel with the relief valve. Dewars configured for liquid withdrawal service only must limit the Dewar pressure to 22 psig. Dewars configured with pressure building and vaporizer coils can be configured to supply liquid at 22 psig or gas at pressures up to 230 psig. It's important to remember that Dewar relief valves will vent gas under normal operating conditions. Especially at higher pressures (230 psig) this venting can be rather startling to nearby workers. When used as a gas source, frost can develop on the outside of the Dewar in a geometric pattern indicating the position of the internal "vaporizer coil". Frost on the outside of a Dewar configured for liquid withdrawal would probably indicate a loss of vacuum insulation. OEM (original equipment manufacturer) equipment manuals can give detailed information on the valve configuration and operation of these Dewars.

Very severe accidents have occurred when users cap or plug the Dewar's pressure relief devices in a misguided attempt to address what they think is "leakage" of the relief device. Never cap or plug a relief device – if you suspect the relief device itself has failed to re-seal, replace the relief device with the exact replacement. Upon request, the cryogen supplier (Matheson Trigas at SNL/NM and SNL/CA) can perform this type of Dewar maintenance.

Points to remember:

- Dewars will vent pressure through their relief valve under normal operation.

- Dewars can be configured to dispense liquid only (at 22 psig) or dual purpose Dewars can be configured to dispense liquid (at 22 psig) or gas (typically at 230 psig)

- Never cap or plug relief devices – replace relief devices when needed.

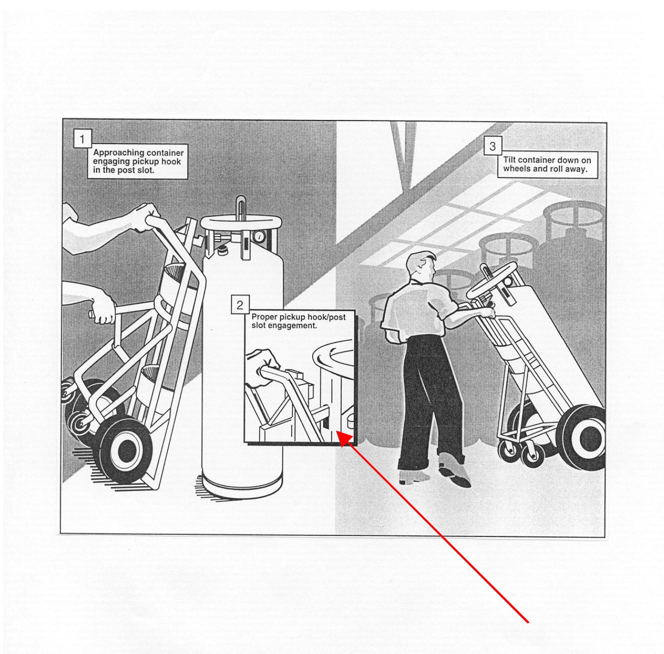
Module 3 - Laboratory Storage Dewars

Lifting Hazards and Dewar Handling

Injuries such as back strain frequently occur during the lifting and handling of Dewars. Equipment for the low pressure transfer of cryo liquids is available and will eliminate the need to lift and pour from Dewars. Use only OEM (Original Equipment Manufacturer's) equipment for these transfers. The OEM equipment will be equipped with the appropriate overpressure protection and limit the pressure in the Dewar to a safe level.

Use properly fitting carts or Dewars with built-in casters for Dewar transport. Be especially careful to avoid tipping the Dewar over when transporting Dewars across thresholds and level changes. For outside or loading dock applications, secure Dewars from rolling away on non-level surfaces or being blown around by high winds. Seismic activity may also be a concern at some locations. For transport in elevators, only persons associated with the transport should be in the elevator – politely ask people unassociated with the Dewar transport to wait for the next elevator.

Note the magnified section of the insert, the lifting/hand truck connection is in the vertical slot in the post and not on the valve protection ring. The ring is not rated for lifting and the hook can easily slip off the ring.



Points to remember:

- Use properly fitting carts or Dewars with built-in casters
- To avoid lift and pour injuries, use low-pressure transfer equipment safely designed by the equipment supplier
- Secure Dewars from rolling away on sloped surfaces or being moved around by the force of the wind.

Module 3 - Laboratory Storage Dewars

Dewar Filling Procedures and Concerns

Dewar fill procedures are equipment and site-specific, however there are some general issues to remember. Always wear the appropriate level of PPE (Personal Protective Equipment) as determined for your fill station. Whenever possible, consider filling Dewars outside rather than inside. This reduces the potential hazards associated with the normal venting that occurs when filling and the hazards associated with potential over filling. The use of a "house" liquid nitrogen supply within a building will require oxygen monitoring as per the requirements of ES&H Manual Chapter 6-T.

When filling open Dewars, the fill station should be designed so that a transfer tube can be inserted into the Dewar that allows the operator to stand back with hands and face away from the point of discharge. A phase separator should be used to minimize splashing. Slowly open the valve for transfer of liquid. Slowly pre-cooling the transfer line and the Dewar is needed in order to minimize the potential flex line whipping hazard as well as minimize boiling and splashing.

Do NOT leave manual valves open and unattended when filling Dewars. Large volume accidental releases have occurred when users left the area – intending to come back in a few minutes. Inevitably, some distraction will cause the user to forget about the open valve, leading to overfilling and the resultant potential hazards of asphyxiation, freezing of floor materials and nearby equipment, etc. This concern is greatest for filling operations inside a building and from a large volume source.

When filling pressurized Dewars, fill stations located within buildings should have a connection for the Dewar vent line – to direct the vent gases to an exhaust system or otherwise to an outside location. Automated systems provide automatic shut-off of the liquid source when the Dewar is full – thereby eliminating the human error of accidentally leaving the valve open. The automated system design should be considered for pressurized Dewar fill stations.

When filling pressurized Dewars, be sure to open the Dewar's vent valve to vent off excess pressure. Dewars should have at least a 10 % air space above the liquid level when full. Do NOT overfill Dewars beyond the point of liquid droplets being emitted from the vent valve. Never fill a Dewar to the point of liquid discharge from the relief device. This would represent a dangerous overfilled condition. Most manufacturers recommend filling Dewars by weight.

Points to remember:

- Cryogenic liquids used from a "House" source will require oxygen monitors as per the requirements of ES&H Manual Chapter 6 T.

- Fill stations should be designed to allow the operator to stand back with hands / face away from the point of discharge while filling.

- Never leave manual valves open and un-attended.

Module 3 - Laboratory Storage Dewars Test Bank

Question 1

Pouring from a small (4 liter or less) open / non-pressurized Dewar presents no hazard and requires no safety precautions.

a. True

b. False

Fill stations should be designed to allow operators to stand back with hands and face away from the point of discharge.

a. True

b. False

Question 2

The relief valve on a pressurized liquid nitrogen storage Dewar

a. will vent gas under normal working conditions

b. can be capped or plugged if the relief valve is found to be "leaking" gas

c. can be replaced by the cryogen supplier

d. Both a and c

The relief valve on a pressurized liquid nitrogen Dewar could be

a. set at 22 psig for liquid withdrawal from the dewar

b. venting gas under normal working conditions

c. set at 230 psig for gas withdrawal from the dewar

d. All of the above

Question 3

Consumer containers, such as Thermos bottles, should not be used for cryogenic liquids because

a. cryogenic liquids are too cold

b. the lid does not allow for venting of the boil-off gases and the vacuum space does not have pressure relief

c. they do not provide enough insulation for cryogenic liquids

d. all of the above

Consumer containers, such as a Thermos bottle, can be safely used for cryogen applications

a. True

b. False

Module 4 - Additional Cryogen Safety Hazards

After first describing cryogenic fluid properties, we will cover the following additional hazards associated with cryogenic applications including the following:

- Thermal (Freezing) / Ice build-up
- PPE concerns
- Brittle Materials
- Miscellaneous Cryo-related Hazards

Module 4 - Additional Cryogen Safety Hazards

Thermal (Freezing) Hazards and Ice Build-up

Thermal: the temperatures associated with cryo applications are low enough to freeze tissue and condense atmospheric moisture. Thermal hazards include exposure to splashes or pressurized sprays, or contact with cold metal parts such as valve handles or un-insulated flex lines.

Use special caution when inserting samples, probes, etc. into a cryogen bath – the boil-off rate will increase suddenly and splashing can occur. In some instances, the liquid in a bath may become super-heated above the normal boiling point (unforeseen by the user). Boil-off and splashing from this situation will be especially hazardous.

Points to remember:

- Cold metal parts will freeze tissue (skin moisture freezes and sticks to the surface)
- Cryogenic liquids and cold boil-off gases can freeze tissue
- Special hazards from sudden / rapid liquid boil-off

Ice build-up: it is important to use hardware (such as relief valves, regulators, etc.) rated for cryogenic applications and properly installed to minimize the accumulation of ice. Specifically, vent tubes or pressure relief devices can freeze over from condensed atmospheric moisture and inhibit the intended venting / pressure relief. An extended length of poor conducting material (i.e. stainless steel) can be used to stand-off critical elements. Surgical tubing is sometimes used to prevent ice formation at the end of open vent tubes.

In addition, ice formation on piping or components can also cause mechanical stresses which can damage the piping or equipment. Upon melting, ice can present water-related hazards to nearby electrical equipment or present a slip hazard. Ice formation on certain cryogen components (such as vaporizers or un-insulated transfer lines) is normal and expected.

Points to remember:

- Maintain relief devices (relief valves, vent tubes, etc.) free of ice to allow proper operation
- Ice formation will be "normal operation" for vaporizers
- Ice eventually melts and creates water related hazards

Module 4 - Additional Cryogen Safety Hazards

Personal Protective Equipment (PPE) Concerns

Engineering controls and Personal Protective Equipment (PPE) are used to address the thermal hazards associated with cryogenic liquid applications. Examples of preferred engineering controls include safe designs for transfer equipment where the operator's hands / face are away from the point of discharge, insulated transfer lines to protect the operator, and the use of phase separators to minimize splashing for liquid withdrawal applications.

Procedures can also enhance safety, such as opening valves slowly to reduce the potential of flex line whipping and to minimize splashing and liquid boil-off, or properly venting pressure from transfer lines prior to cracking fittings.

Personal Protective Equipment (PPE) - in addition to engineering and procedural controls, the applicable level of PPE should be selected based on the criteria of the specific process (such as the pressure or volume of the cryogen source, the potential for splashing, proximity of hands / face to the point of discharge, etc.). Document PPE selections in the system data package or an operating procedure. At a minimum, the eyes must always be protected when handling cryogens. Eye protection is considered primary and can be provided by the use of safety glasses with side-shields or goggles. Face shields are considered secondary and should be worn over eye protection for more severe applications.

Additional PPE options include:

- Gloves (not rated for submersion)
- Long sleeves
- Apron
- Safety shoes (for cylinder handling personnel)
- Hearing protection

Avoid entrapment when selecting PPE. If goggles are used, they should be of the indirect venting style in order to minimize the potential for liquid entrapment. Gloves should be loose fitting to allow for rapid removal in the event of a splash entering the glove and being trapped against the skin. An alternative approach is to use gloves with a tight-fitting cuff that would not allow a liquid splash to enter the glove. Trousers should be worn over boots in order to avoid splashes from entering the boot. Open-toed shoes, such as Sandals or flip-flops, increase the risk of exposure and are not allowed in cryogen applications.

Points to remember:

- Consider engineering controls first (phase separators, insulated lines, a design that allows hands and face away from point of discharge, etc.)

- Also consider procedural controls (slow transfers / slow cool-downs to minimize splashing)

- PPE = eye, face, skin, or other, as applicable to the process – and documented in the data package or operating procedure

Module 4 - Additional Cryogen Safety Hazards

Low Temperature Materials Concerns

Some materials may become brittle at low temperatures and are unacceptable for pressure applications at cryogenic temperatures. Materials such as the 300 series stainless steels, copper, or brass maintain their ductility and are fine for use at cryogenic temperatures.

"Tygon" (or vinyl) tubing embrittles and easily breaks at cryo temperatures. Silicone (or surgical) tubing maintains some ductility but will develop small cracks with time and need frequent monitoring and replacement. This type of tubing should only be used in non-critical, low pressure, small volume applications. Stainless Steel (SS) flex lines are the recommended component for applications requiring flex lines and can be of the insulated, un-insulated, or vacuum-jacketed style. Swagelok and VCR connections made of 300 series SS are acceptable for use in cryogen applications.

Points to remember:

- Many materials embrittle at low temperatures
- Components used in cryogen systems must be rated for both the pressure and temperature of the application

Module 4 - Additional Cryogen Safety Hazards

Miscellaneous Cryo-related Hazards

Some additional hazards that may be associated with cryogen applications may include:
- Flammability / as in the use of liquid hydrogen
- Toxic / air quality issues such as the venting carbon dioxide into lab spaces
- Ozone production from liquid nitrogen with condensed air and the conversion of oxygen to ozone in high ionizing radiation fields

Points to remember:

- Consult your ES&H Support team for guidance in addressing these hazards

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Module 4 - Additional Cryogen Safety Hazards Test Bank

Question 1

A direct splash from a cryogenic liquid is the only thermal concern associated with cryogenic liquid applications.

a. True
b. False

Thermal hazards associated with cryogenic liquid applications include

a. ice build-up on relief valves or vent tubes
b. frozen tissue from contact with cold parts
c. frozen tissue from splashes, sprays or cold boil-off gases
d. all of the above

Question 2

Engineering controls to prevent exposure to cryogenic liquids may include

a. use of a phase separator to reduce splashing during open dewar filling
b. a system design that allows the operator to stand back – with hands and face away from the point of discharge
c. insulated lines
d. any or all of the above

Metals do not embrittle at low temperatures – any metal can be used in cryogenic applications.

a. True
b. False

Question 3

As a minimum, eye protection is required when handling cryogens.

a. True
b. False

The following factor should be considered when selecting PPE for cryogen applications:

a. the color of the gloves to be worn
b. the potential for entrapment of liquid against the skin
c. the criteria of the specific process (such as the pressure or volume of the cryogen source)
d. both b and c