

PPPL	PRINCETON PLASMA PHYSICS LABORATORY ES&H DIRECTIVES	
	ES&HD 5008 SECTION 9, CHAPTER 3 Cryogenic Safety	
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CHAPTER 3 CRYOGENIC SAFETY

3.1 INTRODUCTION

This section covers the use of cryogenic fluids (liquid helium, liquid nitrogen, liquid hydrogen) and their containment and transfer equipment. Hazards associated with cryogenic fluids include personnel exposure (cold burns, frostbite), material and construction compatibility, high pressure gases, explosions, implosions, toxicity, and asphyxiation.

3.2 SCOPE

This section provides guidelines concerning the safe use of cryogenic fluids. It applies to all PPPL personnel engaged in the storage, distribution, or use of such fluids.

3.3 DEFINITIONS

Cryogenics - The science and technology of producing and using low temperature matter, usually from 220° Kelvin to absolute zero (0°K = -273.16 °C = -459.69 °F).

3.4 HAZARDS

- 3.4.1 Physiological Hazards - Severe cold "burns" or frostbite may be inflicted if the human body comes in contact with cryogenic fluids, boiled-off vapor, or surfaces cooled by cryogenic fluids. The evolution of large volumes of gases associated with evaporation of cryogenic liquid spills can result in asphyxiation. For instance, nitrogen expands approximately 700 times in volume, going from liquid to gas at ambient temperature. Asphyxiation and chemical toxicity are hazards encountered when entering a cryogenic vessel that has been used to store cryogenic liquids if proper ventilation/purging techniques are not employed.
- 3.4.2 Material and Construction Hazards - The selection of materials calls for consideration of the effects of low temperatures on the properties of those materials. Some materials become brittle at low temperatures. Brittle fracture can occur very rapidly resulting in almost instantaneous failure. Low temperature equipment can also fail due to thermal stresses caused by differential thermal contraction of the materials. Over pressurization of cryogenic equipment can occur due to the phase change from liquid to gas if not vented properly. All cryogenic fluids produce large volumes of gas when they vaporize.
- 3.4.3 Flammability and Explosion Hazards - Fire or explosion may result from the escaping of flammable gases such as hydrogen, carbon monoxide, or methane. Escaping liquid oxygen, while not itself a flammable gas, can combine with combustible materials and cause spontaneous combustion. Oxygen clings to clothing and cloth items and presents an acute fire hazard for approximately one-half hour after exposure.
- 3.4.4 High Pressure Gas Hazards - Potential hazards exist in highly compressed gases because of the stored energy. In cryogenic systems high pressures are obtained by gas compression during refrigeration, by pumping of liquids to high pressures followed by rapid evaporation, and by confinement of cryogenic fluids with subsequent evaporation. If this confined fluid is suddenly released through a rupture or break in a line, a significant thrust may be experienced.

3.5 RESPONSIBILITIES

3.5.1 Personnel who are responsible for any cryogenic equipment must conduct a safety review prior to the commencement of operation of the equipment. Supplementary safety reviews must follow any system modification to ensure that no potentially hazardous condition is overlooked or created and that updated operational and safety procedures remain adequate.

3.6 REQUIREMENTS

3.6.1 OSHA Regulations - 29 CFR 1910.

3.7 GENERAL SAFETY PRACTICES

3.7.1 Personnel Safety

- A. Face shields and goggles shall be worn during the transfer and normal handling of cryogenic fluids.
- B. Loose fitting, heavy leather, or other insulating protective gloves shall be worn at all times when handling cryogenic fluids. Shirt sleeves will be rolled down and buttoned over glove cuffs, or an equivalent protection such as a lab coat will be worn in order to prevent liquid from spraying or spilling inside gloves. Trousers without cuffs will be worn.

3.7.2 Safety Practices

- A. Cryogenic fluids must be handled and stored only in containers and systems specifically designed for these products and in accordance with applicable standards, procedures, or proven safe practices.
- B. Transfer operations involving open cryogenic containers, such as dewars, must be conducted slowly to minimize boiling and splashing of the cryogenic fluid. Transfer of cryogenic fluids from open containers must occur below chest level of the person pouring liquid.
- C. Such operations shall be conducted only in well ventilated areas to prevent the possible gas or vapor accumulation, which may produce an oxygen-deficient atmosphere and lead to asphyxiation. The volumetric expansion ration between liquid and atmospheric nitrogen is approximately 700 to 1.
- D. Equipment and systems designed for the storage, transfer, and dispensing of cryogenic fluids shall be constructed of materials compatible with the products being handled and the temperatures encountered. There is no single source of information that will provide exact specifications and standards for cryogenic equipment. ASME Codes B31.1 through B31.7 apply. ASME Code B31.3 contains the majority of the relevant information. The American Society of Testing Materials (ASTM) handbook provides information concerning tensile strength of metals at various temperatures and other relevant information. The Code of Federal Regulations, 49 CFR, provides some useful guidelines, although it only references cryogenic vessels used in rail transportation. In each case, the design specifications are left to the discretion of the designing engineer.
- E. All cryogenic systems, including piping, must be equipped with pressure-relief devices to prevent excessive pressure build-up. Pressure-reliefs must be directed to a safe location. It should be noted that two closed valves in a line form a closed system. The vacuum insulation jacket should also be protected by an over-pressure device if the service is below 77° Kelvin. In the event a pressure-relief device fails, do not attempt to remove the blockage; instead call **Emergency Services** at Ext. 3333.
- F. If liquid nitrogen or helium traps are used to remove condensable gas impurities from a vacuum system that may be closed off by valves, the condensed gases will be released when the trap warms up. Adequate means for relieving the resultant build-up of pressure must be provided.

3.7.3 First Aid

Workers will rarely, if ever, come into contact with cryogenic fluids if proper handling procedures are used. In the unlikely event of contact with a cryogenic liquid or gas, a cold-contact "burn" may occur. The skin or eye tissue will freeze. The recommended emergency treatment is as follows:

- A. **Emergency Services must** be contacted immediately by calling Ext. 3333.
- B. If the cryogenic fluid comes in contact with the skin or eyes, flush the affected area with generous quantities of cold water. Never use dry heat. Splashes on bare skin cause a stinging sensation but, in general, are not harmful.
- C. If clothing becomes soaked with liquid, it should be removed as quickly as possible and the affected area should be flooded with water as above. Where clothing has frozen to the underlying skin, cold water should be poured on the area, but no attempt should be made to remove the clothing until it is completely free.

3.8 CRYOGENIC FLUID PROPERTIES

Table 3.1 gives the properties of some cryogenic fluids.

3.8.1 Liquid Helium

- A. Liquid helium must be transferred via helium pressurization in properly designed transfer lines. A major safety hazard may occur if liquid helium comes in contact with air. Air solidifies in contact with liquid helium, and precautions must be taken when transferring liquid helium from one vessel to another or when venting. Over pressurization and rupture of the container may result. All liquid helium containers must be equipped with a pressure-relief device.
- B. The latent heat of vaporization of liquid helium is extremely low (20.5J/gm). Therefore, small heat leaks can cause rapid pressure rises.

3.8.2 Liquid Nitrogen

- A. Since the boiling point of liquid nitrogen is below that of liquid oxygen, it is possible for oxygen to condense on any surface cooled by liquid nitrogen. If the system is subsequently closed and the liquid nitrogen removed, the evaporation of the condensed oxygen may over-pressurize the equipment or cause a chemical explosion if exposed to combustible materials, e.g., the oil in a rotary vacuum pump. Additionally, if the mixture is exposed to ionizing radiation, ozone may be formed. The ozone will freeze out as ice and is very unstable. An explosion can result if this ice is disturbed. For this reason, air should not be admitted to enclosed equipment that is below the boiling point of oxygen, unless specifically required by a written procedure.
- B. Any transfer operations involving open containers such as wide-mouth dewars must be conducted slowly to minimize boiling and splashing of liquid nitrogen. The transfer of liquid nitrogen from open containers must occur below chest level of the person pouring the liquid.
- C. **Face shields and goggles shall be worn during the transfer and normal handling of liquid nitrogen.**
- D. **Loose fitting, heavy leather, or other insulating protective gloves shall be worn at all times when handling cryogenic fluids. Shirt sleeves will be rolled down and buttoned over glove cuffs, or an equivalent protection such as a lab coat will be worn in order to prevent liquid from spraying or spilling inside gloves. Elbow length cryogenic handling gloves are also acceptable. Full length trousers without cuffs will be worn.**

- E. Open-toed shoes, sandals, or other footwear which would allow liquid nitrogen to come in contact with the operator's feet, shall not be worn.
- F. Articles of clothing which may absorb liquid nitrogen readily and hold it next to the operator's skin will not be worn. This type of clothing includes sweaters, mufflers, or scarves, and bulky socks worn outside of boot tops.
- G. The fill station will be secured with a lock except when liquid nitrogen is actually being dispensed.
- H. The operator will remain at the fill station until the liquid transfer is complete. The equipment will then be secured and the key returned to the responsible person.
- I. Liquid nitrogen must be stored only in containers and systems specifically designed for cryogenic liquids and handled in accordance with applicable standards, procedures, or proven safe practices.
- J. Liquid nitrogen will be transported and handled in such a manner as to not endanger the operator or other persons in the area. Liquid nitrogen shall not be transported in elevators except under the following conditions:
 - 1. The elevator will be locked out or otherwise disabled so that persons not actually involved in the liquid nitrogen transfer are excluded from entering.
 - 2. An operator will load the cryogenic container into the elevator and start the car. The operator will **not** accompany the car to its destination.
 - 3. An operator will be stationed on the floor to where the liquid nitrogen is being transported to receive the cryogenic container.
 - 4. After the cryogenic container is removed from the elevator, the elevator can be returned to normal service.
 - 5. Should the elevator be incapable of the type of operation described in steps 1 through 4, an operator may accompany the container in the elevator provided that self-contained breathing apparatus is worn.
 - 6. Extreme caution should be exercised when transporting, transferring, or working with liquid nitrogen in areas where a spill could result in an oxygen deficient atmosphere. The volumetric expansion ratio between liquid and atmospheric nitrogen is approximately 700 to 1. Areas of particular concern are basements, corridors below ground level, small rooms, and the tunnel connecting C-Site and D-Site.

3.8.3 Liquid Hydrogen

- A. Liquid hydrogen is not currently used at PPPL. Anyone proposing the use of liquid hydrogen must first obtain the approval of **Industrial Hygiene and the Safety Review Committee**.
- B. Because of its wide flammability range and ease of ignition, special safety measures must be invoked when using liquid hydrogen.
- C. Liquid hydrogen must be transferred by helium pressurization in properly designed transfer lines in order to avoid contact with air. Properly constructed and certified vacuum-insulated transfer lines should be used.
- D. Only trained personnel familiar with liquid hydrogen properties, equipment, and operating procedures are permitted to perform transfer operations.

- E. Transfer lines in liquid hydrogen service must be purged with helium or gaseous hydrogen, with proper precautions, before using.
- F. The safety philosophy in the use of liquid hydrogen can be summarized as the following:
 - 1. Isolation of the experiment.
 - 2. Provision of adequate ventilation.
 - 3. Exclusion of ignition sources plus system grounding/bonding to prevent static charge build-up.
 - 4. Containment in helium purged vessels.
 - 5. Efficient monitoring for hydrogen leakage.
 - 6. Limiting the amount of hydrogen cryopumped in the vacuum system.

3.9 REFERENCES

Compressed Gas Association Inc., Safe Handling of Cryogenic Liquids, Compressed Gas Association, Arlington, Virginia, current edition.

Russell B. Scott, Cryogenic Engineering, D. Van Nostrand Company, Princeton, New Jersey, current edition.

Norman Steere, Handbook of Laboratory Safety, Chemical Rubber Company, Cleveland, Ohio, current edition.

ASME Publication, American Society of Mechanical Engineers, 345 East 47th St., New York, NY 10017.

American Society of Testing Materials (ASTM) Handbook.

Code of Federal Regulations 49 CFR.

NFPA Publications, National Fire Protection Association, 1 Batterymarch Park, P.O. Box 9101, Quincy, MA 02269-9101.

TABLE 3.1 PROPERTIES OF CRYOGENIC FLUIDS

Gas	Normal B.P.		Volume Expansion to Gas	Flammable	Toxic	Odor
	°C	K ^a				
Helium-3	-269.9	3.2	757 to 1	No	No ^c	No
Helium-4	-268.9	4.2	757 to 1	No	No ^c	No
Hydrogen	-252.7	20.4	851 to 1	Yes	No ^c	No
Deuterium	-249.5	23.6	--	Yes	No ^c	No
Tritium	-248.0	25.1	--	Yes	Radioactive	No
Neon	-245.9	27.2	1438 to 1	No	No ^c	No
Nitrogen	-195.8	77.3	696 to 1	No	No ^c	No
Carbon monoxide	-192.0	81.1	--	Yes	Yes	No
Fluorine	-187.0	86.0	888 to 1	No	Yes	Sharp
Argon	-185.7	87.4	847 to 1	No	No ^c	No
Oxygen	-183.0	90.1	860 to 1	No	No ^d	No
Methane	-161.4	111.7	578 to 1	Yes	No ^c	No
Krypton	-151.8	121.3	700 to 1	No	No ^c	No
Tetrafluoromethane	-128.0	145.0	--	No	No ^c	No
Ozone	-111.9	161.3	--	No	Yes	Yes
Xenon	-109.1	164.0	573 to 1	No	No ^c	No
Ethylene	-103.8	169.3	--	Yes	No ^c	Sweet
Boron trifluoride	-100.3	172.7	--	No	Yes	Pungent
Nitrous oxide	-89.5	183.6	666 to 1	No	Yes	Sweet
Ethane	-88.3	184.8	--	Yes	No ^c	No
Hydrogen chloride	-85.0	188.0	--	No	Yes	Pungent
Acetylene	-84.0	189.1	--	Yes	Yes	Garlic
Fluoroform	-84.0	189.1	--	No	No ^c	No
1, 1-Difluoroethylene	-83.0	190.0	--	Yes	No ^c	Faint ether
Chlorotrifluoromethane	-81.4	191.6	--	No	Yes	Mild
Carbon dioxide	-78.5 ^b	194.6	553 to 1	No	No ^c	No

^a K = -273.16 °C = -459.69 °F

^b Sublimes.

^c Nontoxic, but can act as an asphyxiant by displacing air needed to support life. As with most chemicals, even harmless materials can be toxic or poisonous if taken in sufficient quantities under the right circumstances.

^d Pure oxygen is toxic at pressures greater than two atmospheres absolute.