

# CRYOGEN SAFETY MANUAL



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**I. Definition:** Cryogenic materials are very cold substances (gases, solids and liquids at or below -100° F) which are used in a wide variety of processes. Cryogenic liquids have boiling points below -130° F (-90° C).

Common cryogenic liquids : nitrogen (N), helium (He), argon (Ar), hydrogen (H), methane and solid carbon dioxide (dry ice, CO<sub>2</sub> ).

**II. Scope:** This document provides guidelines for the safe use of cryogenic fluids for personnel engaged in the transport, storage, or use of such fluids at IIT Hyderabad.

**III. Responsibility:**

It is responsibility of faculty, staff and students, for implementing the safety during handling of cryogenic materials.

Table 1: Lists the physical properties of common cryogens

	Chemical Symbol	Molecular Weight	Color of Gas	Color of Liquid	Odor of Gas	Boiling Point		Freezing Point	
						°F	(°C)	°F	(°C)
Argon	A	39	None	None	None	-303	(-186)	-308	(-189)
Ethane	C <sub>2</sub> H <sub>6</sub>	30	None	None	Slightly sweet	-126	(-88)	-297	(-183)
Ethylene	C <sub>2</sub> H <sub>4</sub>	28	None	None	Slightly sweet	-155	(-104)	-272	(-169)
Helium	He	4	None	None	None	-447	(-266)		
Hydrogen	H <sub>2</sub>	2	None	None	None	-423	(-253)	-434	(-259)
Krypton	Kr	84	None	None	None	-243	(-153)	-251	(-157)
Methane	CH <sub>4</sub>	16	None	None	None	-258	(-161)	-296	(-182)
Neon	Ne	20	None	None	None	-411	(-246)	-416	(-249)
Nitrogen	N <sub>2</sub>	28	None	None	None	-321	(-196)	-346	(-210)
Oxygen	O <sub>2</sub>	32	None	Lt. Blue	None	-297	(-183)	-362	(-219)
Xenon	Xe	131	None	None	None	-162	(-108)	-170	(-112)

#### **IV. PHYSICAL PROPERTIES ASSOCIATED WITH CRYOGENS:**

**Very low temperature:** cryogenes are gases liquefied at very low temperature, the extremely low temperature is a potential risk for cold burns, frostbites and hypothermia.

##### **Colourless and odourless:**

Cryogenes are colourless both in their liquid and gas state with the exception of liquid oxygen that is light blue. Most of them are also odourless. The fact that many cryogenes are colourless and odourless makes them impossible to detect and discriminate by eye or by the sense of smell.

##### **Very large expansion rate**

All cryogenes have a very large expansion rate, the volume of gas produced by the boiling liquid is several hundred times larger than the volume of liquid. For example the ratio volume of gas to volume of liquid at 1bar and 15° C is 738 for helium and 1417 for neon. The large volume of gas displaces the air and reduces the amount of available oxygen.

##### **Flammable**

Liquid hydrogen, methane, ethylene and ethane are flammable and special precautions must be adopted to manipulate, store and transport these fluids. Most of the commonly handled cryogenes are non flammable and non explosive, nevertheless liquid helium, nitrogen and neon although inert can encourage combustion.

##### **Toxic**

Some cryogenes are toxic. Ozone (O<sub>3</sub>), carbon monoxide (CO) and fluorine (F<sub>2</sub>) are highly toxic and must be treated with caution. Specific safety measures must be followed when working with these cryogenes.

#### **V. SAFETY HAZARDS ASSOCIATED WITH CRYOGENIC LIQUIDS:**

**1. Burns:** Liquid or low-temperature gas from any of the specified cryogenic substances will effects on the skin similar to a burn.

a. Contact of the skin with cryogenic liquids (or even cold gas) can cause severe cryogenic burns; the tissue damage that results is similar to that caused by frost bite or thermal burns. While the cold itself can reduce the feeling of pain, the subsequent thawing of tissue can cause intense pain.

b. Contact with non-insulated parts or equipment or vessels containing cryogenic liquids can produce similar damage. Unprotected parts of the skin may stick to low temperature surfaces and flesh may be torn upon removal.

c. Inhalation of cold vapor can cause damage to the lungs and may trigger an asthma attack in susceptible individuals.

d. Hypothermia is a condition associated to the decrease of body temperature below 35°C. The susceptibility of a person to hypothermia depends on the temperature, the exposure time and the person's physical conditions (older people are more likely to succumb).

**2. Asphyxiation:** Asphyxia will occur when the oxygen content of the working environment is less than 20.9% by volume. Effects from oxygen deficiency become noticeable at levels below ~18% and sudden death may occur at ~6% oxygen content by volume.

**3. Explosion- pressure related:** Heat flux into the cryogen from the environment will vaporize the liquid and potentially cause pressure buildup in cryogenic containment vessels and transfer lines. Adequate pressure relief must be provided to all parts of a system to permit this routine outgassing and prevent explosion.

**Pressurization can occur due to the following:**

- a. Ice forming on the venting tube, plugging it and preventing gas release.
- b. Damaged equipment resulting in cryogenic fluids leaking into small areas, upon vaporization the cryogenic liquid vaporizes and causes pressure build up.
- c. Loss of vacuum inside a cryostat or Dewar(cryogenic vessel).
- d. If a liquid helium-cooled superconducting magnet "quenches" (changes spontaneously from a superconducting state to a normal state).
- e. Liquid nitrogen having permeated through sealed cryotubes containing samples which then return to room temperature.
- f. Direct contact of the cryogenic liquid with water in a tube results in rapid vaporization of the cryogenic liquid and can cause the tube to explode.

**Explosion-chemical related:** Cryogenic fluids with a boiling point below that of liquid oxygen are able to condense oxygen from the atmosphere. Violent reactions, e.g. rapid combustion or explosion, may occur if the materials which make contact with the oxygen are combustible.

**VI. CRYOGEN HANDLING:**

- Cryogenic liquids vaporize with a volume change ratio of 700-900 can cause violent changes in pressure, particularly if this occurs in a confined space. This in turn can result in an explosion. Vent systems must be in place to allow gas to escape from confined spaces.
- Explosion (excessive buildup of pressure in container of cryogenic fluid) Heat flux into the cryogen is unavoidable regardless of the quality of the insulation provided.
- Since cryogenic fluids have small latent heats and expand 700 to 800 times to room temperature, even a small heat input can create large pressure increases. Dewars must be moved carefully. Sloshing liquid into warmer regions of the container can cause sharp pressure rises.
- Pressure relief devices must be provided on each and every part of a cryogenic system. Satisfactory operation of these devices must be checked periodically and may not be defeated or modified at any time. Vents must be protected against icing and plugging. When all vents are closed, enough gas can boil off in a short time to cause an explosion.

- Vents must be maintained open at all times. Liquid helium is cold enough to solidify atmospheric air. Only helium should be introduced or allowed to enter the helium volume of a liquid helium dewar. Precautions should be taken to prevent air from back-diffusing into the helium volume.
- Some materials may become brittle at low temperature and fail in the case of overpressure or mechanical shock. Only suitable materials may be used to store or transfer liquid cryogenes.
- Fire/explosion (condensation of liquid oxygen): Liquid oxygen liquefies at a higher temperature than liquid helium or nitrogen. Consequently, liquid oxygen can condense on the exterior of cryogenic containers or transfer lines.
- An explosive situation may result if this oxygen-rich liquid is allowed to soak insulating or other materials which are not compatible with oxygen.
- Some oils can form an explosive mixture when combined with liquid oxygen. Surfaces where there exists a possibility of liquid oxygen condensation must be thoroughly cleaned and degreased.

## **VII. PREVENTIVE MEASURES:**

### **a) Training:**

All personnel working with cryogenic fluids must be thoroughly familiar with the hazards involved. They must also be familiar with all emergency measures that might be required in the event of an accident. Each personnel working with the cryogenes must receive training by the responsible scientist on the specific cryogenic equipment or system he is expected to use.

This will cover:

- Description of the equipment
- Operating procedures
- Maintenance schedule and procedures
- Specific hazards
- Reporting of incidents

### **b) Maintenance and Inspection:**

Cryogenic systems and equipment must be inspected and maintained on a regular basis by the user (laboratory head must insist on this to the lab personnel). The schedule and nature of the maintenance must be included in the operating procedures of the manual. The inspection and maintenance shall be documented. Every cryogenic system or equipment shall be inspected by qualified personnel before being put into operation for the first time or after modification. Inspection by qualified personnel shall also take place after an unusual incident which might affect the integrity and safety of a piece of cryogenic equipment. One should note that these requirements for inspection, maintenance, calibration and documentation extend to the monitoring systems for oxygen deficiency

### **c) Personal protective equipment**

Personal protective equipment should always be worn when handling cryogenics. The protective clothing is intended to protect from accidental contact with cryogenic liquids and from handling cold surfaces of instrumentations and tools that have been in contact with cryogenic liquids. When handling cryogenic liquids it is necessary to protect the whole body. Special precaution should be taken for those parts of the body most exposed to the cryogen, such as the hands and those most sensitive to cold, such as the eyes.

#### Eye protection

Safety glasses must be worn at all times while handling cryogenic liquids, because the liquid is almost always boiling and can splash into the eyes. Safety goggles provide the best protection for the eyes. When filling dewars or transferring cryogenic liquids from one container to another, face shields must also be worn.

#### Hand protection

When cryogenic liquids are spilled on the skin, a thin gaseous layer apparently forms next to the skin. This thin layer protects the skin from freezing, provided the contact with the cryogen is brief and in small quantities. The most likely cause of frostbite to the hands and body is by contact with cold metal surfaces. Because there is no protective layer of gas formed, frostbite will occur almost instantaneously when touching surfaces that have been cooled by cryogenic liquids.

Cryogenic gloves should be used when working with cryogenics. A wide selection can be found on the market. Non absorbent leather gloves are recommended. Gloves should be a loose fit so that they can easily be removed if liquid should splash onto or into them. Gloves with a wide cuff are not recommended because liquid can easily splash into them. Never use rags to handle cold surfaces. Use tongs to add or remove materials from cryogenic liquids.

#### Protective clothing

Closed toed shoes are required when handling cryogenic liquids. Long trousers and long sleeve shirts are recommended for leg and arm protection. An apron made of leather or other material recommended for use with cryogenics is indicated when large quantities of cryogen are handled.

#### **d) Safe Handling Procedures :**

##### Preparation

1. Always be familiar with the hazards of the liquid in use.
2. Work in an open, well-ventilated location.
3. Ensure that safety glasses and if necessary face shields are worn.
4. Have pot holders or appropriate gloves on hand.
5. Examine containers and pressure relief valves for signs of defect. Never use a container which has defects.
6. Any exposed glass areas of dewars should be taped to prevent the generation of broken glass projectiles in the event the container implodes.

7. Ensure that all equipment and containers are free of oil, grease, dirt or other materials which may create a hazardous condition upon contact with the cryogenic liquid. Researchers should wash their hands and arms with soap and water, rinsing and drying thoroughly prior to handling the liquid. Clothing should be relatively clean.

8. Select working materials carefully. Cryogenic temperatures may alter the physical characteristics of many materials.

### **Transfer and use**

1. Use only fitted transfer tubes designed for use with the dewar container. Damaged transfer tubes should be replaced. Do not handle transfer tubes with bare hands as the fitting is not insulated.

2. When transferring into a secondary container, do not fill the secondary container to more than 80% of capacity (60% if the temperature is likely to be above 30 C).

3. Do not lower experiments into storage dewars unless provisions have been made to vent the dewar and prevent freezing in the narrow neck.

4. Immediately re-cap any container to prevent atmospheric moisture from entering and forming an ice plug.

5. Provide proper venting for the dewars used in experiments

6. Use care in transporting fragile cryogenic containers. Use a hand truck for transport. Always transport cryogenic liquids in service elevators when available.

### **Storage**

1. Store in a well ventilated area to prevent buildup of flammable gases or air displacement.

2. Use only approved storage vessels having pressure relief valves.

3. Avoid contact of moisture with storage containers to prevent ice plugging of relief devices.

4. Periodically check container necks for ice plugging; core out plugs if present.

5. Keep all sources of ignition away from cryogenic liquids.

### **VIII. FIRST AID:**

All personnel using cryogenic liquids must also be familiar with first aid procedures for treating frostbite:

1. Warm the affected area of the body rapidly by immersion in water not to exceed 105° F, with body heat, or by exposure to warm air. In the event of massive exposure, the emergency shower should be used to warm the body. All clothing must be removed prior to showering. Maintain the affected area of the victim at normal body temperature until medical help arrives.

2. Calm the victim and avoid aggravating injury. People with frostbitten feet should not walk on them. Do not rub or massage the affected parts of the body.

3. Prevent infection--use a mild soap to clean the affected area. Dressings need not be applied if the skin is intact.

4. If affected, flush eyes with warm water for 15 minutes.
5. For any serious injuries contact Medical Officer at xxxx6144

References:

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3. <http://www2.lbl.gov/ehs/pub3000/CH29/CH29.html#29e>
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