Department of Environmental Health and Safety

Subject: Cryogenic Liquid Use (Research)		
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SUMMARY: Cryogenic liquids have boiling points below -130°F (- 90°C) and are used in research to provide extremely low temperatures for frozen storage and experimentation. Common cryogenic liquids of concern include nitrogen, helium, hydrogen, argon, methane, and carbon monoxide. Several hazards are associated with their use that must be properly controlled to avoid any contact with components or liquid, or exposure to its gases. This document describes administrative controls necessary to protect personnel from contact or exposure during handling of cryogenic liquids.

SCOPE: This Guideline applies to all University of Michigan-Dearborn personnel that use cryogenic liquids.

REFERENCE DOCUMENTS:

CGA P-12-2009, *Safe Handling of Cryogenic Liquids*

MIOSHA Part 75 Flammable & Combustible Liquids
http://www.michigan.gov/documents/CIS_WSH_part75_35492_7.pdf

NFPA 45, *Fire Protection for Laboratories Using Chemicals*

NFPA 55, *Compressed Gases and Cryogenic Fluids in Portable and Stationary Containers, Cylinders, and Tanks*

Air Products *Safetygrams*

EHS Personal Protective Equipment Guideline

DEFINITIONS:

Chemical Hygiene Plan (CHP) - a written policy developed and implemented by lab management which sets forth procedures, equipment, personal protective equipment, and work practices that protect employees from the health hazards associated with the use of hazardous chemicals. In essence, it is a lab safety manual.

Cryogenic Liquid (Cryogen) – a liquid with a boiling point below -130°F.

Cryogenic Liquid Cylinder – an insulated, vacuum-jacketed, pressure vessel equipped with safety relief valves and rupture disks to protect against excessive pressure build-up.

Dewar – open-mouthed, unpressurized, vacuum-jacketed vessels used to hold cryogenic liquids (usually argon, nitrogen, or oxygen).

Material Safety Data Sheet (MSDS) - informational tool developed by chemical manufacturers detailing safety and health aspects for a hazardous chemical. MSDS can be obtained from chemical suppliers, internet sites, and should be available to those working with the material.

Personal Protective Equipment (PPE) - items worn by workers to guard against hazards in the environment, primarily by creating a barrier between the hazard and the worker. Examples include: safety glasses, goggles, face shields, respirators, gloves, hard hats, safety-toed shoes, and hearing protection.

Standard Operating Procedure (SOP) - a concise, written, document that provides safety instructions specific to experimental materials and methods.

RESPONSIBILITY:

Deans, Directors and Department Heads

- Actively support this guideline within individual units.
- Ensure that adequate facilities, ventilation, and equipment are provided for the safe use of cryogens.
- Ensure an environment where principal investigators and other personnel are encouraged to follow this Guideline.
- Take disciplinary action against any person determined to be out of compliance with this Guideline.

Supervisors, PI, Lab Directors

- Implement procedures in accordance with this Guideline.
- Assure that staff is aware of this Guideline, instructed on the details of implementation, and provided with equipment and controls.
- Report all workplace accidents or injuries and complete the Illness or Injury Report Form.
- Implement contingency plan in the event a cylinder leaks; instruct student and staff on emergency procedures; and ensure procedures are documented.
- Contact EHS to request technical assistance.

Employees

- Comply with this Guideline and any further safety recommendations made by your supervisor or The Department of Environmental Health and Safety (EHS).
- Report any job related injuries or illnesses, questions on health and safety, or any unsafe or unhealthy working conditions to your supervisor.
- Contact EHS to evaluate potentially unsafe conditions.
- Conduct assigned tasks in a safe manner, wear appropriate personal protective equipment, and only use equipment for which you have been formally trained.
- Review chemical hazard information detailed on MSDS before beginning work with cryogens.

Building and Facility Managers

- Provide facilities and utilities that support the types of research performed and provide adequate engineering controls.
- Notify the dean, director, or department head about observed improper use of facilities or utilities.

EHS

- Review and revise this Guideline as needed.
- Provide training to the Principal Investigator and Laboratory Manager upon request and maintain records of training.
- Conduct safety audits and provide technical assistance when necessary.

PROCEDURES:

Injury

Personnel should be aware of the hazards associated with handling and usage of cryogens. If a lab worker is injured, the person should seek immediate medical attention. Their supervisor must complete a Work Connections Injury or Illness Report.

Routine Tasks and Maintenance Procedures

A. Use of Cryogenic Liquids

Cryogenic liquids have properties that make them more dangerous to use than other liquids: extremely cold temperatures, high liquid-to-vapor expansion ratios, and flammability for certain liquids.

Contact with cryogenic liquids, cryogenic equipment, or splashing liquid can cause severe tissue damage. Burns, frostbite, tearing of the flesh, and eye damage are all possible injuries. Vapors from boiling liquids can also cause eye damage, frostbite to the skin, and oxygen deficient environments.

To minimize exposure during use implement the following procedures:

- Perform a Personal Protective Equipment (PPE) assessment in accordance with the EHS Personal Protective Equipment Guideline to determine the level of protection needed for the task. Typical PPE used to work with cryogenic liquids includes safety goggles, insulated gloves, lab coat or apron, and a face shield. Gloves should be loose enough to easily toss off in the event of a spill.
- Long sleeve shirt and pants should be worn. Shoes must be closed- toed leather or safety shoes. Pants, without cuffs, should be worn over the shoes. Never wear sandals. Do not wear jewelry or other materials that could trap liquid to the skin if spilled.
- Stay out of the vapor pathway.
- Use fume hoods when working with cryogens if possible.
- Always use tongs when handling objects in liquid.
- Only use approved materials with cryogens. Unapproved materials (such as plastic, rubber, wrought iron, hollow tubes, and carbon steel) will become brittle and shatter or, in the case of hollow tubes, become over pressurized.
- Periodically inspect equipment and remove ice and frost blockages from openings to prevent over pressurization. *Do not tamper with pressure relief valves. Report any leaks or improperly set relief valves to the manufacturer.*
- Equipment should be kept clean without the use of corrosive cleaning materials that could damage the metal jacket.

B. Dispensing and Transport of Cryogenic Liquids

Special precautions must be taken to prevent a spill while dispensing or transporting cryogens, in addition to minimizing exposures from

liquids and vapors. The high liquid to vapor expansion ratio could rapidly displace all oxygen in a room and result in asphyxiation.

Implement the following procedures to minimize exposure:

- Always wear proper PPE when dispensing or transferring cryogenic liquids.
- When obtaining liquid from a large dispensing dewar or cryogenic liquid cylinder, cool the secondary container by adding a little cryogenic liquid first. Then:
  - Dispense slowly to mitigate thermal stress
  - Stay in constant attendance of the filling operation
  - Do not overfill
  - Do not allow the cryogenic liquid to fall through a distance to reach the receiving vessel
- When manually pouring liquid into a smaller dewar, ensure: o Secondary container is secured
  - Pour slowly to prevent excess splashing
  - Do not overfill
  - Use a phase separator, if available, to control the vapor path while pouring
- Use no fewer than two personnel to transport cryogenic liquids and use handcarts equipped with brakes for large dewars and cryogenic liquid cylinders. BEST PRACTICE IS TO AVOID TRAVELING IN AN ELEVATOR WITH A DEWAR. Spills or elevator failures may be dangerous in this restricted space by displacing oxygen if the cylinder failed or leaked. If this is not avoidable, make sure to use the buddy system and have another employee remain outside the elevator during transport.
- Always use care when handling equipment. Damage to dewars could result in the loss of vacuum and increased evaporation.
- When carrying a dewar, wear PPE and hold it as far away from the face as possible. Containers that cannot be easily and safely carried should be placed on a stable wheeled base designed for the dewar.

C. Storage of Cryogenic Liquids

A cryogenic liquid storage unit left open to the atmosphere, or catastrophic failure of a storage unit, could create an oxygen deficient atmosphere.

Follow these procedures to reduce the likelihood of this occurrence:

- Glass dewars must have an exterior coating/cover to minimize projectiles in the event of an explosion. Newer dewars may have a plastic mesh over the exterior for this purpose. Older dewars must be thoroughly taped or replaced.
- Store dewars in well-ventilated rooms with a minimum of six air changes per hour. If the ventilation rate is unknown, contact EHS to evaluate the storage area.
- EHS may recommend the installation of oxygen detection systems and alarms for cryogenic liquid storage areas depending on location, ventilation, and quantity of material stored.
- Bulk cryogenic liquid dispensing areas within buildings must also be well ventilated. EHS recommends continuous oxygen monitoring equipment in these areas.
- Dewars and cryogenic liquid cylinders should be placed so that vents and openings are oriented away from personnel and lab equipment.
- Do not store cryogenic liquids with corrosive or flammable chemicals.
- Storage of cryogenic liquid cylinders or dewars in hallways, unventilated closets, environmental rooms, and stairwells is prohibited.
- No more than one backup dewar or cryogenic liquid cylinder is allowed per piece of equipment. Additional dewars or cryogenic liquid cylinders must be stored in areas designed for such storage. Contact Facility Operations Call Center 3-5270 to evaluate potential storage locations.

D. Special Precautions for Flammable Liquids and Oxygen

Flammable cryogenic liquids like methane, hydrogen, and carbon monoxide introduce an additional hazard. Oxygen does not burn, but accelerates and supports combustion. High concentration oxygen atmospheres substantially increase combustion rates of other materials and may form explosive mixtures with other combustibles.

It is important to implement the following procedures when using flammable cryogenics and oxygen.

- Contact Facility Operations Call Center 3-5270 to assess engineering and work practice controls if you plan to work with these materials.
- There are several industry guidelines that refer to flammable liquids that should be considered in addition to these recommendations (see Reference Documents).
- All combustible materials should be kept away from flammable liquids and oxygen. There should be "No Smoking" signs posted, and no sources of ignition should be present in this area.
- Oxygen dewars and equipment should be kept very clean. Surface contamination could become ignited if oxygen leaks from the dewar and provides a local oxygen enriched area.
- Stationary equipment should be properly grounded and mobile equipment should be properly bonded when dispensing (See Reference Documents for more detailed standards for electrical requirements).
- Valve operation should occur very slowly to prevent ignition of contaminants in the system.
- Hydrogen venting should be independent from other ventilation systems and may require a nitrogen purge capability.

E. Special Precautions for the Use of Cold Traps

Cold traps are used when using instrumentation, a building vacuum system, water aspirator, or a vacuum pump. Cold traps prevent the introduction of liquids and vapors into and out of the system by providing a low temperature surface for molecules to condense. When using liquid nitrogen (LN2) in cold traps, the following procedures must be implemented to prevent over pressurization and explosion:

- Do not open the system to the atmosphere until the trap is removed.
- In the event that the system is opened with the trap still in place, there is a possibility that oxygen will condense out of the air and combine with the organic material inside the trap.

There are two possible scenarios that could result: it could immediately create an explosive mixture and explode, or the oxygen could stay condensed in the liquid. DO NOT RECLOSE THE SYSTEM. The condensed oxygen will vaporize after the trap is removed or the bath has evaporated resulting in an over pressurization and possible explosion.

F. Special Precautions for the Use of Cryotubes

- Cryotubes containing samples stored under liquid nitrogen may explode without warning. Tube explosions are caused by liquid nitrogen entering the tube through minute cracks and then expanding rapidly as the tube thaws.
- Cryogenic storage vials are designed for vapor phase storage in the extremely cold nitrogen gas that sits just above the reservoir of liquid nitrogen in the bottom of the freezer or dewar. If the freezer/dewar is overfilled with liquid nitrogen and the vials are immersed, leakage of liquid nitrogen into the vial occurs. To avoid this problem do not overfill the freezer/dewar with liquid nitrogen and visually check each cryotube prior to filling to ensure there are no defects around the rim.
- Cryotubes should never be re-used.
- As a precaution, slowly remove vials from the dewar, holding the vial in the neck of the dewar for a moment before bringing them into room atmosphere. A tube that is going to explode will usually do so early in the warm-up process.
- PPE for thawing cryotubes should include safety glasses, face shield, insulated heavy gloves, a buttoned lab coat, closed toe shoes and pants.
- Cryotubes should be kept in a heavy, walled container or behind a safety shield while warming.

G. Emergency Procedures

Liquid Nitrogen (LN2) is the most commonly used cryogenic liquid. Oxygen depletion resulting from nitrogen gas may occur rapidly with no warning properties. A person entering an oxygen deficient environment may become disoriented and unable to respond properly.

Nitrogen gas is odorless, colorless, tasteless, and inert. The failure of a large cryogenic liquid cylinder could spill 180 L of LN2 - in gas form this will completely displace all oxygen in a 21x21x10 ft. room. A much smaller spill in the same room could still create a safety hazard.

Simply reducing the oxygen content in a room below 19.5 % is considered an oxygen deficient environment.

Implement the following procedures to minimize the risk of asphyxiation:

- Periodic equipment inspections, removal of ice blockages, and replacement of damaged or old storage units will reduce the probability of the catastrophic failure of a storage unit. Ice blockages that prevent the container from venting properly can cause an explosion hazard. Contact the University Police Department immediately at 593-5333 if ice blockages are observed.
- If a spill occurs ***immediately exit*** the area. With adequate ventilation it may be appropriate to return to the area after thirty minutes. For large spills contact the University Police Department immediately at 593-5333 to monitor oxygen levels in the area and determine when it is safe to re-enter.
- If experiencing symptoms such as lightheadedness, dizziness, or confusion, immediately seek fresh air and receive medical attention.
- If an employee becomes unconscious in a cryogenic liquid storage area they should only be retrieved by personnel using proper PPE (such as a Self Contained Breathing Apparatus). The University Police Department should be immediately notified at 911 from a campus phone or (313)593-5333 from a cell phone to coordinate emergency rescue services. Over fifty percent of deaths associated with asphyxiation in confined spaces occur to would-be rescuers.
- Once personnel have been removed to fresh air, provide rescue breathing or CPR until paramedics arrive.

In the event of contact with cryogenic gases or liquid:

- Immediately remove any clothing that has been contaminated. *In the event of clothing contamination with oxygen, hydrogen,*

or carbon monoxide, it is important to remove clothing, evacuate personnel from the facility, and keep away from ignition sources.

- Flush or soak the area with warm water (no greater than 105^{o}F).
- Do not apply dry heat or rub damaged flesh or eyes.
- Employees should notify their supervisor of injuries and report to Henry Ford Hospital or Oakwood Hospital for a medical evaluation and follow-up. The supervisor must complete the Work Connections Injury and Illness form to report the incident.

TECHNICAL SUPPORT:

Facility Operations Call Center 3-5270 will provide technical support for the proper use and storage of cryogenic liquids.

Additional Requirements for Toxic, Pyrophoric and Flammable Gases

Evaluating Flammable and Toxic Compressed Gases in Laboratories

Hazards of Common Compressed Gases

Maximum Allowable Quantities

Generic Compressed Gas Cylinder SOP Template