

# Cryogenic Materials

## STANDARD OPERATING PROCEDURE

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**Type of SOP:**       Process       Hazardous Chemical       Hazard Class

### 1. HAZARD OVERVIEW

This document describes the safety requirements that laboratory workers and supervisors must follow when using cryogenic gases in the laboratory. Its purpose is not to have any accident or risk. Liquid nitrogen is cryogenic material that exists at a very low temperature below  $-150^{\circ}\text{C}$ , and is used as coolants for various instruments and experiments.

### 2. HAZARDOUS CHEMICAL(S) OR CLASS OF HAZARDOUS CHEMICAL(S)

Nitrogen is not toxic, but simple asphyxiates. Cryogenic fluids are materials with extremely low boiling points (i.e., less than  $-150^{\circ}\text{F}$ ). At these temperatures, tissue burns may be sustained after contact with the fluids, surfaces cooled by the fluids, or by evolving gases. The hazard is comparable to that of handling boiling water. One special property of cryogenic liquids is that they undergo substantial volume expansion when converted to a gas phase, which can potentially lead to an oxygen deficient atmosphere where ventilation is limited.

1. Liquid Nitrogen

### 3. PERSONAL PROTECTIVE EQUIPMENT (PPE)

#### a. Eye Protection

ANSI compliant safety glasses with side shields should be worn. Chemical splash goggles should be worn when working with larger quantities. A face shield should be worn when splashing onto the face is a possibility.

#### b. Skin and Body Protection

Wear chemical resistant lab coat, long pants, and closed-toe shoes. These laboratory coats must be appropriately sized for the individual and be buttoned to their full length. Laboratory coat sleeves must be of a sufficient length to prevent skin exposure while wearing gloves.

#### c. Hand Protection

Wear the blue waterproof Cryo-gloves (Tempshield).

Additional PPE may be required if procedures or processes present additional risk. It is the responsibility of the PI to ensure that any additional PPE requirements are identified and communicated to research staff. Contact EH&S for consultation.

#### **4. ENGINEERING/VENTILATION CONTROLS**

Use liquid nitrogen only in well-ventilated areas. Even though nitrogen isn't toxic, but there is a danger of asphyxiation from unconsciousness, which occurs in a room if too much oxygen is displaced by nitrogen gas. Nitrogen expands its volume by a factor of ~700 when evaporated.

#### **5. SPECIAL HANDLING PROCEDURES AND STORAGE REQUIREMENTS**

Store and transport liquid nitrogen only in Dewars or cryogenic liquid cylinders designed for cryogen. Cryogenic liquid Dewars have to be stored in well-ventilated areas with the pressure relief valve in the opened position.

Use small quantities whenever possible. Monitor your inventory closely to assure that you have tight control over your material.

#### **6. SPILL AND INCIDENT PROCEDURES**

**Chemical Spill** - Dial 911 and EH&S 951-827-5528

**In the event of a large spill,**

- 1) Alert others immediately in the vicinity to evacuate.
- 2) Close the doors of the lab if opened.
- 3) Restrict access to the work area.
- 4) Do not attempt cleanup if you feel unsure of your ability to do so or if you perceive the risk to be greater than normal laboratory operations.
- 5) Call 911 from a campus phone or 951-827-5222 for EH&S from cell phone.

**Medical Emergency** - Dial 911 and EH&S 951-827-5528

Refer to "Injuries and Medical Treatment" Flipchart posted in the laboratory.

#### **7. DECONTAMINATION**

Wear proper PPE, decontaminate equipment and bench tops using soap and water. Dispose of all used contaminated disposables as hazardous waste following the Waste Disposal Section.

#### **8. WASTE DISPOSAL**

Return container and unused product to supplier. Do not attempt to dispose of unused product.

#### **9. PRIOR APPROVAL/REVIEW REQUIRED**

All work with cryogenic materials must be pre-approved by the Principal Investigator prior to use and all training must be well documented. In addition, the following shall be completed:

- Documented specific training and specific training on the techniques and processes to be used.
- Read and understand the relevant Safety Data Sheet.
- Demonstrate competence to perform work.

A review of this SOP and re-approval is required when there are any changes to procedures, personnel, equipment, or when an incident or near miss occurs.

## 10. DESIGNATED AREA

Work should be completed in a well-ventilated place or laboratory.

## 11. SAFETY DATA SHEETS

Online SDS can be found at <https://ehs.ucop.edu/sds/#/>

## 12. DETAILED PROTOCOL

All lab workers who will be using cryogenic materials must review this SOP and sign the associated training sheet. Lab workers must have specific training on the proper handling of cryogenic materials and understand the hazards.

Lab workers using cryogenic materials must demonstrate competence to the Principal Investigator or designee by being able to 1) identify the hazards and list any particularly hazardous handling techniques (use of a schlenck line, rotary evaporation, canula transfer, extremes of pressure or temperature, etc.), 2) list the foreseeable emergency situations, 3) describe the proper response to the emergency situations, and 4) know the control measures to minimize the risks.

When working in the lab, a laboratory worker must:

- 1) not work alone;
- 2) be cognizant of all of the SDS and safety information presented in this document;
- 3) follow all related SOPs in the laboratory SOP bank (PPE, syringe techniques, waste disposal, etc. as appropriately modified by any specific information in the SDS information presented in this document);
- 4) employ a minimum amount of cryogenic materials in any given reaction (larger quantities REQUIRE the approval of PI or designee), and
- 5) discuss ALL issues or concerns regarding cryogenic materials with the PI prior to its use.

### Transporting Liquid Nitrogen from Cylinder to Dewars

1. Cylinder for cryogenic liquids must not be closed completely. Even though liquid nitrogen is stored in vacuum-jacketed vessels (dewars), there is always some heat

leak into the dewar such that there is boil-off of the liquid at all times. Pressure will build up if no exit is available to the gas, and then the container would ultimately explode. A pressure relief valve is attached to the cylinder to keep the internal pressure very low. Make sure you are aware of the presence of a protective pressure relief valve on any cylinder that you handle.

2. If there is a crack in the metal between the liquid and the vacuum space of a cylinder, a rapid pressure build-up in this relatively confined space will occur since it would no longer be insulated by vacuum. For this reason, all metal dewars must be constructed such that a pressure relief valve or rupture disk connected to the vacuum space will relieve excess pressure prior to sufficient build-up to cause explosion. For the same reason, all glass dewars must be wrapped with tape or netting to prevent flying glass in the event of an explosion.

3. Do not leave openings to cold dewars wide open to the atmosphere for any longer than is absolutely necessary for the manipulations required for transferring liquids. The temperature of liquid nitrogen at atmospheric pressure is  $-196^{\circ}\text{C}$ . Air (and its contents) will condense into the dewar and can cause blockages that are potentially dangerous and that will almost certainly interfere with some aspect of the liquid transfer or with the operation of the instrument in the long run. The freezing point of liquid oxygen is  $-183^{\circ}\text{C}$ , above that of liquid nitrogen. Having liquid oxygen in liquid nitrogen is an explosion hazard!

4. The very cold temperatures of cryogenic fluids necessitate complete avoidance of contact with the skin. Frostbite, i.e. “burns” from extreme cold, will occur very quickly upon contact, especially if clothing, shoes, etc., hold the liquid tightly to the skin. Take care about spills and use appropriate hand protection while transferring these liquids. Use thermally insulated gloves; thin gloves will not help. Take care to avoid contact with the portions of the transfer line that have been inserted into the dewars during the transfer as they will be cold enough to cause cold “burns” for quite a while after removal from the dewars.

5. Liquid helium transfer lines are vacuum jacketed and should not be very cold to touch during the initial cool down of the transfer line. If a transfer line appears to be too cold, it is time to re-pump the vacuum space. The heat of vaporization of liquid helium is very small, i.e.,  $\sim 10\%$  of that of liquid nitrogen. Thus, helium is never seen as a liquid during transfer operations. A “plume” or “flame” is the indicator that liquid helium is coming through the transfer line during the cool down and is ready to be inserted.

If there is an unusual or unexpected occurrence when using liquid nitrogen, the occurrence must be documented and discussed with the Principal Investigator or Lab Supervisor and others who might be using cryogenic materials. Unusual or unexpected occurrences might include a fire, explosion, sudden rise or drop in temperature, increased rate of gas evolution, color change, phase change, or separation into layers.

SOP Reviewed and Approved by:

Print name: \_\_\_\_\_

Signature: \_\_\_\_\_

Approval Date: \_\_\_\_\_

