**Standard Operating Procedure**

**Cryogenic Liquid**

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| **Department:** |  |
| **Completion Date:** |  |
| **Approval (by PI / Lab Manger) Date:** |  |
| **Principal Investigator:** |  |
| **Principal Investigator Signature:** |  |
| **Internal Lab Safety Coordinator/Lab Manager:** |  |
| **Lab Phone:** |  |
| **Office Phone:** |  |
| **Emergency Contact:** | *(Name and Phone Number)* |
| **Location(s) covered by this SOP:** | *(Building/Room Number)* |

**Type of SOP:** ☐ Process ☐Hazardous Chemical ☐ Experiment ☐Equipment Use

**Contents**

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| --- | --- |
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***Read and review any applicable manufacturer/vendor safety information before developing standard operating procedure and performing work***

***\*\*\* NOTE: Each section needs to be complete with clear and detailed information based on the Red/italic font instruction. SOP must be approved and dated by the PI or lab supervisor.***

1. **Purpose and Scope of work/activity**

This document is intended to describe the best practice for cryogenic material and explain the hazards and risks associated with various type of cryogens and ways to have them in control. However, it is not designed to be substituted for hands-on training and supervision by experience laboratory personnel.

1. **Responsibility**
   1. The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP and a copies of any SDS provided by the manufacturer for any chemicals used.
   2. The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training annually.
   3. Any repair or maintenance must be done by a trained person

*Identify the personnel that have a primary roles in the SOP and describe how their responsibilities relates to this SOP. If necessary, include contact information.*

1. **Definition**

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| *Cryogen Liquid* | An extremely cold substance in liquid phase which produce large amount of gas at atmospheric temperature and pressure |
| *Asphyxiation* | Oxygen depletion can cause a condition of breathing difficulties (suffocation), loss of consciousness and ultimately death |
| *Dewar* | A vacuum-jacketed pressure vessel designed to hold liquids at below ambient temperature which is used for storing/transportation of cryogenic liquids |
| *SDS* | Safety Data Sheet |
| *Venting* | The discharge of gas vapor out of an over-pressurized container |

1. **Hazards Analysis**

The process of completing the hazard and risk assessment is needed for locations that storing quantities of cryogenics can present a significant risk of asphyxiation. For performing hazard & risk assessment follow the general rules listed below:

* Identify hazards
* Determine hazard effects and who might be harmed
* Determine the severity and probability of potential incident (Risk Assessment)
* Determine the potential likelihood of the potential incident (Hazard Control)

After Hazard Analysis, research group can develop a plan to minimize the risks of operating tube furnace in the process. This includes using **engineering control, administrative control and adequate PPE**. Safety needs to be continuously evaluated to identify additional controls and better work practices.

1. **Common Hazards**

Most of the cryogenic liquids are colorless, odorless liquid with a boiling point blow -150 °C (- 238°F). It means they stay in gaseous phase at normal temperature and pressures. They generate large volumes of gas when vaporize. The expansion ratio for the most common liquids are as below:

|  |  |
| --- | --- |
| Common Liquid | Expansion Ratio |
| Argon | 1 to 841 |
| Helium | 1 to 754 |
| Hydrogen | 1 to 848 |
| Nitrogen | 1 to 696 |
| Oxygen | 1 to 861 |

In general the main hazards associated with cryogenic liquids are as following:

|  |  |
| --- | --- |
| Hazard | How to control? |
| Cold burn/Frostbite   * Exposure to the cryogenic liquid itself or contacting with the substance that liquid is flowing through or stored | * Use compatible transfer hoses * Use appropriate PPE including **full face shield, and gloves** * Place a **Cold Burn Hazard** label   Image result for cryogenic burn label |
| Asphyxiation   * The main danger of using and storage of cryogenic liquid is asphyxiation. | * Use and store in the well-ventilated area * Use methods in *Appendix A* to calculate the potential for oxygen displacement * Use O2 monitor system if required * Contact EH&S for further assessment * Don’t get in the elevator with the container * Don’t fill more than 80% of the capacity of a secondary container |
| Over-pressurization   * Cryogens generate large volume of gas which can pressure build up inside the container | * Complete “*Compressed Gas Safety*” training * Use appropriate PPE * Review Safety Data Sheet * Ensure dewar is equipped with Pressure Relief Valve * If two or more containers connected to create higher product withdrawal rate, then vent valves should be connected as well. This practice is needed to equilibrate the containers pressure |
| Noise | * Use whisper valve if the vent noise is disturbing * Contact EH&S for noise evaluation |
| Physical Injury | * Always push the container with wheels instead of pulling * Use appropriate PPE |
| Thermal stress at material | * Fill slowly the secondary container to minimize thermal shock * Place materials slowly into cryogens |

**Note:**

* *Never remove or exchange the recommended Compressed Gas Association (CGA) connection, or use adapting fittings*.
* *To avoid excessive splashing of liquid at pressures more than 22 psig into open vessel, extra precaution is needed. Please read product labels before using*
* *To avoid any physical injuries, it’s recommended to push the container instead of pulling it as it shown below*

|  |  |
| --- | --- |
|  |  |

* Be aware of the formation of liquid oxygen in cold-traps. Mixture of liquid oxygen and organic solvent can cause explosion

1. **Hazard Control**
   1. **Engineering Control**

In addition to the information below, follow procedures as specified in the lab-specific section of this SOP.

|  |  |
| --- | --- |
| Room Ventilation, | * Liquid containers must be stored in well-ventilated areas or areas with forced ventilation * Containers should be stored from the air intakes, high traffic areas, floor drains and other underground openings |
| Oxygen Deficiency Monitor | * Oxygen monitor is required in rooms with poor ventilation system to monitor the oxygen level or concentration |
| Pressure relief Valve | * Containers must be equipped with pressure relief valve which protects the container from over-pressurization |

*List any gas monitoring system available for the process with the calibration/service dates in this section.*

* 1. **Administrative Control**

In addition to the practices described below follow procedures as specified in the lab-specific and special handling/use sections of this SOP.

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| Documentation | * Safety Data Sheet (SDS) for each material should be reviewed. * Standard Operating Procedure (SOP) must be completed to verify your experimental set-up and procedures prior to use. * Manufacturer Operating Manual should be reviewed prior to use the equipment. |
| Storage & Location | * Designated areas must be identified for. * Gas cylinders/dewars and associated piping must be well secured. * Incompatible chemical/gases must be segregated. Examples include flammable and oxidizers that must be segregated by minimum of 20 ft distance * Cryogenic liquids must be stored in well ventilated locations |
| Training | * Training must be provided prior to conducting any work with the equipment * Basic trainings are: ***lab safety fundamental, compressed gas safety, waste management***   Note: Training should be renewed under the following conditions:   * Changes in the workplace render previous training obsolete * Changes in the type of cylinders systems or equipment used render previous training obsolete * Inadequacies in an employee’s knowledge of compressed gas cylinders or equipment or observed behavior indicate that the employee has not grasped the required training * Any deviation from this SOP requires approval from PI. |

* 1. **Personal Protective Equipment (PPE):** 
     1. Full face shield
     2. Apron
     3. Thermal resistant gloves
     4. Lab coat
     5. Long pants
     6. Closed toed shoes

**Note:** Additional PPE could be required if the process present additional risk. It would be PI responsibly to identify and communicate any additional PPE requirements.

*List all additional PPE required for the specific process*

1. **Anatomy of the Liquid Cryogenic Container**

It is important to be familiar with the container design for handling gas or cryogenic liquid in a safe manner. Two different containers are compared in the following table:

|  |  |
| --- | --- |
| Low-Pressure Liquid Container | High-Pressure Liquid Container |
|  |  |
| Liquid Valve: Add or withdraw the liquid product through the connection controlled by the valve.  Specific CGA connection is required for the appropriate cryogenic liquid | |
| Relief Valve: To protect the container from over-pressurization. It consists of two relief devices; first is a reseating spring-loaded relief valve which depending on the setting will relieve pressure at 22 psig, 230 psig or 350 psig. The second is a burst disk rated to protect the inert vessel | |
| Vent Valve: To release un-wanted pressure during storage and use. | |
| Contents Gauge/Liquid Level Gauge: A float type level gauge to indicate the approximate amount of container contents | |
| Pressure Building Valve: to create enough operating pressure. For more information visit <https://www.airproducts.com/~/media/Files/PDF/company/safetygram-27.pdf> | |
| Gas Use Valve: To withdraw gaseous product through the vaporizer and/or the economizer | |

1. **Special Handling Procedures and Storage Requirements**

*Describe special handling and storage requirements for hazardous chemicals/gases in the laboratory, especially for highly reactive, unstable and highly flammable materials and corrosives. Describe transport and secondary containment requirement, between the laboratories or between facilities.*

1. **Location of Nearest Emergency Safety Equipment**

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| Items | Location |
| Eyewash/Safety Shower |  |
| Chemical Spill kit |  |
| First aid kit |  |
| Fire extinguisher |  |
| Telephone |  |
| Fire alarm manual pull station |  |
| Others/Details: |  |

1. **Shipping and Receiving Requirements**

*Describe shipping or receiving requirement for cryogenic liquids.*

1. **Step-by-step Operation Procedure**

*Describe the step-by-step procedure for using the cryogenic liquid containers properly. Include details for potential tank failure if something is done improperly in the procedure and the procedure of liquid/gas withdrawal.*

1. **Emergency Response Plan**

A laboratory emergency response plan should be developed that address accidental releases and emergency response to this process. This plan should be part of the training procedure and be documented.

**Only trained emergency response personnel with proper personal protective equipment (i.e., SCBAs, Level A suits for gases with a Cal/OSHA Skin notation) and appropriate gas monitoring instruments should be allowed to enter the toxic gas release area.**

The following steps should be taken following below incidents:

*In this section describe additional procedure for any other emergency events that may happen. Indicate how accidental events should be handled and by whom. List emergency contact number.*

1. **Reference**

The following links are referenced to support safe operation of cryogenic liquid containers:

1. Cryogenic liquid containers from Air Products manufacturer:

<https://www.airproducts.com/~/media/Files/PDF/company/safetygram-27.pdf>

1. General use SOP for cryogenic liquid

<https://ehs.stanford.edu/wp-content/uploads/sops/Cryogenic-Liquids.pdf>

1. Cryogenic substances SOP from Marywood University

<http://www.marywood.edu/science/pdf-files/Cryogenics%20SOP.pdf>

1. Oxygen depletion calculations

<https://intranet.birmingham.ac.uk/hr/documents/public/hsu/information/hazardoussubstances/Appendix3Oxygendepletioncalculations.pdf>

**Appendix A: Assessment of Ventilation Requirements**

The assessment of ventilation requirements is important to mitigate the consequences of a gas leakage that can cause asphyxiation. Gas leakage could change the concentration of the air components. Air contains 78% Nitrogen, 21% Oxygen and 1% Argon. An atmospheres containing less than 18% oxygen is dangerous and entry into atmosphere containing less than 20% of oxygen must be avoided.

How to calculate the air concentration?

Case 1:

This case is about a situation that gas releases by certain evaporation rate from vessels or pipework.

Determination of the gas concentration depends on the amount of gas release, room volume and air change per hour which shown in the following equation:

Where  
L = gas release (ft3/hr) = Cryogen volume X Expansion factor / 1000  
V = room volume (ft3)  
n = air change per hour

Case 2:

Oxygen depletion resulting from a large spillage of a cryogenic liquid or sudden release from a pressurized vessel. Always “worst Case Scenario” must be considered

Determination of the oxygen concentration (%) is as following:

Where: for nitrogen:

Vo = 0.2095 (Vr – Vg)   
Vr = Room Volume (ft3)  
Vg = maximum gas release = liquid volume capacity of the vessel V \* gas expansion factor

**I have read and understand the content of this SOP:**

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| --- | --- | --- | --- |
| **Name** | **Signature** | **Identification** | **Date** |
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