**THIS IS A TEMPLATE/BASIC STARTING POINT. CUSTOMIZE THIS TEMPLATE WITH INFORMATION PERTINENT TO YOUR SETUP AND THE PROCEDURE YOU WILL BE USING/YOUR GROUP’S PERSONAL USE.**

STANDARD OPERATING PROCEDURE

Use this form to document the Health & Safety information associated with the procedure.

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Procedure Title:** | | Vacuum pump usage with Schlenk Line | | | | | | |
|  | |  | | | | | | |
| **Dept:** |  | |  | **Bldg/Rm:** |  |  | **Supervisor:** |  |

Always read and understand the safety data sheet (SDS) for a chemical or the equipment safety data sheet (EDS) before use or storage.

**Procedure Overview:** (brief description of the project)

1. The user will understand the procedure for operating vacuum pumps.

2. The user will understand the hazards involved with using vacuum pumps

3. The user will be able to prepare dry ice slurry for use in a cold trap.

4. The user will understand the rules governing vacuum pump use.

**Procedure:**

**WARNING:**

Evacuated glass vessels carry with them with risk of implosion, creating a shower of fragments which can injure the user. Also the Dewar flask used for the traps are also evacuated vessels and have the potential for implosion. Vessels should be taped or placed in a container to reduce scattering of fragments if an implosion should occur. A desiccator is a good example of a vessel that should be taped. While it is impractical to tape all your flasks, you should clamp your flask in place behind a shield or in a fume hood with the door lowered. Use as small a flask as possible so to minimize the stress placed on the vessel.

Vacuum pump must be properly ventilated – there should be two hoses connected – one ingoing and one outgoing. An oil-mist filter does NOT filter out volatiles and is not a substitute for proper ventilation. Ventilated pump cabinets in fume hoods contain fans – these are for cooling; they do not provide adequate ventilation. There are two ports/holes in the cabinet to allow for ventilation hoses.

**Cautions:**

1. Do not use the vacuum pump with dust, corrosive vapors, water vapor, or solvent are present without using a cold trap. The oil can be contaminated without a cold trap and then must be replaced

2. Do not allow the pump to suck air through the system for excessive periods as the oil will become oxidized and require replacement.

3. When not in use, keep the intake to keep out dirt and other contaminants

**Procedure:**

I. Evacuation of a system

1. Before beginning, check your apparatus for cracked glassware, loose joints, or strain points that can lead to leaks or implosion. Make sure the line is properly assembled and there is a trap and Dewar. Check the pump oil level. Correct any flaws you find.

2. Fill the Dewar around the traps with an appropriate cold bath. The two most appropriate choices are liquid nitrogen or dry ice used with either isopropanol or acetone.

Liquid nitrogen is obviously the more efficient choice, but presents a substantial hazard if significant volumes of air will be passed through or if the system is left open for long periods of time without the pump on. This is because it can condense air, particularly oxygen. Liquid oxygen is an extremely potent oxidizer that can react violently with organic compounds.

Dry ice should be thoroughly crushed before addition to a Dewar containing a vacuum trap. After the trap is filled, acetone or isopropanol should be slowly and carefully added just enough to cover the dry ice. This is conveniently done with a squirt bottle. Isopropanol tends to last longer and is a bit less hazardous than acetone and is thus recommended. As the dry ice is consumed, it can be replaced with more crushed dry ice. If the bath already contains acetone or isopropanol, but no dry ice, when you begin to use it, carefully add small amounts of dry ice to “pre-chill” the solvent before filling the bath. If you add the dry ice too fast, there will be terrible splattering.

4. Turn on the pump, and close the needle valve and/or turn the stopcock to evacuate the line, if applicable. You should hear a gurgling noise, which should disappear after a few seconds, as the air is pumped out of the vessel. Once you are satisfied that the line is functional, you can attach your sample.

5. If the sound does not fade, check the oil level on the pump and if the level is too low, turn off the pump, add oil and restart the pump as before. If the oil level is okay, there is probably a leak in the system or possibly a stopcock has been left open. Remember, that sucking large amounts of air through the pump can break down the oil, so correction of leaks is very important.

6. Use a manometer or thermocouple gauge to measure the pressure of the system.

II. Restoring the system to atmospheric pressure

1. Remember that it is not always the case that you need to bring the entire system up to atmosphere after using it. Often, it is wiser to leave parts of the system (only between the pump and trap if the trap is to be allowed to warm) under vacuum. If you decide not to bring the whole system up, isolate the pump and line by closing the appropriate valves and isolate your evacuated vessel if you want to keep it under vacuum. Then you can open a valve that will expose the intermediate part of the line to atmosphere and remove your vessel.
2. If you are going to bring the whole system up, first seal off your sample if that is appropriate.
3. Disconnect a hose, or open a valve to allow air into the system and bring the vacuum pump back to atmospheric pressure.
4. Turn off the pump and then remove your sample.
5. If the evacuated vessel has been 'isolated' by means of a valve, the hose to the pump can be disconnected and if the vessel is to be filled with an inert gas, the hose can be replaced with a nitrogen or argon line connected to a bubbler. Do not use an oil bubbler for this purpose. After establishing that gas is flowing with such a bubbler, seal it off with an appropriate valve. Otherwise you will simply suck oil into your sample. Use of a mercury bubbler (>760 mm column) is appropriate for this task.
6. Remove the contents of the trap, if any, and if it is waste, dispose of it appropriately. Clean the trap for the next user.

**Hazard Control Measures:**

(Lab coat, eye and hand protection, and closed toe/heel shoes must be selected as required by Section D of the ISU Laboratory Safety Manual.)

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
|  | **Latex gloves** |  | **Insulated gloves** |  | **Face Shield** |  | **Respirator** |
|  | **Nitrile gloves** |  | **Safety glasses** |  | **Lab Coat** |  | **Fume hood** |
|  | **Neoprene gloves** |  | **Vented goggles** |  | **Apron** |  | **Biosafety cabinet** |
|  | **Vinyl gloves** |  | **Splash goggles** |  | **Dust mask** |  | **Glove box** |
|  | **Closed Toe/Closed Heel Shoes** | | |  | **Flame Resistant Lab coat** | | |

**Maintenance:**

1. The oil in the pump should be changed every 4 months or immediately upon suspicion that the pump oil has become contaminated. A clue to contamination is a chemical odor emanating from the pump.
2. Once a year, or more often after heavy use, have the pump examined by machine shop personnel to examine the baffles
3. Check the drive belt once a year. BE SURE THE PUMP IS UNPLUGGED BEFORE REMOVING THE BELT COVER.
4. Consult the Owner's Manual for more information.

**Emergency Procedures:**

1. The most likely emergency situation to occur is a power failure. If this occurs, turn off the main power switch to the vacuum pump. Close the valves, if any, to the vacuum pump. Once power has been restored, vacuum pump may be restarted as before.
2. If you encounter a trap that has condensed air or oxygen, MAKE SURE THE SASH OF THE HOOD IS CLOSED OR THAT BLAST SHIELDS ARE IN USE. Once that is done, turn off the pump. Wear a face shield. Next open the system to atmosphere if it has not been already and preferably remove all organics. Lower the cold bath from the trap to allow it to warm to room temperature. Then you should allow the liquid air and nitrogen to evaporate. Tag the area out until the liquid air is completely gone.

**General Considerations:**

1. The pumps are shared devices. Although there will be someone “in charge” of them, it is your duty to make sure that you leave them in proper condition. This includes cleaning the traps, changing the oil, etc.
2. If the pump is permanently stationed at your lab area, you are responsible for the maintenance of the pump. So to minimize down time, service your pump regularly and keep the oil clean.

**Training Requirements For Operating the Vacuum Pump:**

Each person using the vacuum pump must:

1. Read and understand this document.

2. Be able to properly;

a) Operate a vacuum pump, both evacuating a system and returning it to atmospheric pressure.

b) Understand the rules regarding the use of the pumps

c) Know what to do if the power goes out

**Using Substances Requiring Special Procedures?** No  Yes

(If Yes; identify authorized personnel, designate a use area and specify specialized safety precautions here. Refer to Section B in the ISU Laboratory Safety Manual for details.)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Written By:** |  |  | **Date:** |  |
|  |  |  |  |  |
| **Approved By:** |  |  | **Date:** |  |

(PI or Lab Supervisor)

1. **HAZARD ASSESSMENT**

Use the hierarchy of controls to document the hazards and the

corresponding control measure(s) involved in each step of the procedure.

Consider *elimination or substitution* of hazards, if possible.

*Engineering Control(s):* items used to isolate the hazard from the user (i.e. fume hood, biosafety cabinet).

*Administrative Control(s):* policies/programs to limit the exposure to the hazard (i.e. authorizations, designated areas, time restrictions, training).

*Required PPE*: indicate PPE including specific material requirements if applicable (i.e. flame resistant lab coat, type of respirator or cartridge).

|  |  |  |  |
| --- | --- | --- | --- |
| **Hazard** | **Engineering Control(s)** | **Administrative Control(s)** | **Required PPE** |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |
|  |  |  |  |

1. **Training Record**

Use the following table to record the training associated with this Standard Operating Procedure.

|  |  |  |
| --- | --- | --- |
| **Print Name** | **Signature** | **Date** |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |

**Note: Attach to or file with written materials and methods**