Safe Use of Schlenk and Vacuum Lines

Adapted from UCLA Department of Chemistry

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Objectives

- Introduction
- Start Up
- Shutdown
- Cleaning
- Changing Pump Oil
- Dealing with Liquid O_2

What are Schlenk and Vacuum Lines?

- Schlenk lines and vacuum lines provide a means of manipulating pyrophoric and water reactive compounds without the use of a glove box.

- The apparatuses consist of a glass manifold with stopcocks and a connection to a vacuum source.

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Schlenk and Vacuum Hazards

- The use of Schlenk and vacuum lines pose serious hazards:
  - Explosions
    - Pressurized gases – high vacuum manifolds are connected to an inert or reactant gas line. This system must not be closed when the gas supply is opened – there must be a source of pressure relief (bubbler). This pressure must be monitored with an electronic gauge, manometer, or bubbler.
    - Condensed gases – some gases are easily condensed into a liquid nitrogen cooled trap (CO and Ethylene, for example). If the nitrogen dewar is removed without providing pressure relief, the liquid may convert to vapor quickly and cause an explosion.
    - Runaway reactions – could release large quantities of gas.
    - Heating – lines should never be heated without pressure relief.

Schlenk and Vacuum Hazards Cont.

- Implosions
  - Cracks or stars in the glass can cause failure of the line.

- Liquid Oxygen
  - Air in the system could cause oxygen to condense. Liquid oxygen is extremely dangerous and reacts violently with organic substances (Teflon, vacuum grease, solvents…).

PPE Required

- Laboratory Coat (Flame Resistant)
- Gloves (ensure compatibility with solvents)
- Safety Goggles
- Face shield (if using outside of a fume hood)
- Cryogen/Leather gloves for liquid nitrogen

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Start-Up Procedures

1. Make sure all of the stopcocks and seals are properly greased.

2. Ensure that the system is free of cracks, stars, and chips.

3. Make sure the vacuum trap is completely dry and clean.

4. Check to see if there is enough liquid nitrogen to fill the dewar, plus a little extra. (The dewar will have to be refilled every 6-8 hours).

Start-Up Procedures Cont.

5. Insert traps into empty dewars.

6. Ensure that all valves are closed, and start the line to the vacuum pump.

7. Fill the dewar with liquid nitrogen. Wrap the top with aluminum foil. *Extremely important that the vacuum pump is activated before any component of the system is under liquid nitrogen. Otherwise, oxygen could condense.

8. Watch the gauge to ensure that the pressure is dropping.

Shut Down Procedures

1. Remove the liquid nitrogen dewar.

2. Shut off the vacuum pump and vent the system.

3. Allow the system to come to room temperature before disposing of any remaining solvents.

Never vent the system to air or turn off vacuum when any portion of the system is still at liquid nitrogen temperatures. This could cause oxygen to condense.

Cleaning the System

1. Clean the system with hexanes and dispose of it in organic “A” waste.

2. If necessary, soak in a base bath composed of isopropyl alcohol and potassium hydroxide.

3. Use Apiezon M grease to grease the joints. Applying heat to the joints via heat gun before applying the grease is helpful.

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Changing Vacuum Pump Oil

1. Ensure that the pump is off.
2. Use screwdriver to remove the hose from the pump to the manifold.
3. Place pump on bench top.
4. Find an empty container for pump oil waste.
5. Attach a latex or tygon tube to the drainage nozzle and open to allow the oil to flow out. Tilt the pump to drain the remaining oil.

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Changing Vacuum Pump Oil

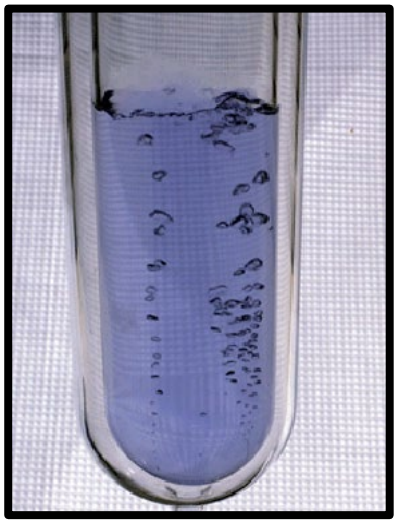
6. Fill with fresh pump oil. Seal the top with a vulcanized rubber stopper.

7. Plug in and run the pump for 1 minute. Drain the oil.

8. Repeat the above steps until the oil is free of particulates.

9. Place the pump on the floor and reassemble.

Liquid Oxygen Procedure



Liquid oxygen is highly explosive in the presence of organic compounds. The presence of liquid oxygen is a very dangerous situation and should be handled by training personnel.

1. If the liquid nitrogen dewar is removed and the remaining liquid has a blue color (or if any liquid is observed), assume liquid oxygen is present.

2. Immediately replace the liquid nitrogen.

3. Inform others of the situation and evacuate the lab area except for a buddy at a safe distance.

4. Place a blast shield around the traps and remove any surrounding organic materials.

Liquid Oxygen Procedure

5. Remove the liquid nitrogen dewar and quickly vent the system. Lower the fume hood sash completely.

6. Immediately leave the laboratory and warn others not to enter.

7. After the system has warmed to room temperature, consider the traps still dangerous. Organic peroxides may have formed. Pour remaining liquid into a clean beaker and flush the trap 5 times with water. Do this behind the blast shield with the sash lowered.

8. Check the solvent for peroxides using a potassium iodide test strip. If a purple color forms on the test paper, peroxides are present. In that case, reduce the solution by adding sodium thiosulfate or sodium sulfite before disposing of waste.

As soon as possible, make sure you research director is informed of the situation.

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