Vacuum Gas Lines and Use of Desiccators Under Vacuum

Standard Operating Procedure (SOP)
Ragauskas Group ● June 2010
• Also called vacuum gas manifold or Schlenk Line
• Developed by Wilhelm Schlenk
• Dual manifold with multiple ports
• One manifold connected with purified inert gas or air
• The other one connected with high-vacuum pump

http://www.ilpi.com/inorganic/glassware/vacline.html
Major components

- Vent
- Cold trap to condense solvent vapors or gaseous reaction products
- Safely and successfully manipulate air sensitive compounds
- High vacuum to remove the last traces of solvent from a sample
- Inert gas is used to remove oxygen for the prevention of samples to be oxidized
Main techniques involved

- Counter flow additions, where air-stable reagents are added to the reaction vessel against a flow of inert gas
- The use of syringe and rubber septa to transfer liquids and solutions
- Cannula transfer, where liquids or solutions of air-sensitive reagents are transferred between different vessels stoppered with septa using a long thin tube known as a cannula. Liquid flow is achieved via vacuum or inert gas pressure
Potential Dangers

- Implosion or explosion
  - Implosion can occur due to the use of a high vacuum and flaws in the glass apparatus
  - Explosion may happen due to the common use of liquid nitrogen in the cold trap.
  - The form of liquid oxygen in the cold trap (a pale blue liquid) may cause an explosion due to the reaction of the liquid oxygen with any organic compounds trapped in the cold trap.
VWR Scientific Immersion Chiller Model 1109

Cooling Capacity: 265W at –30°C
1050W at 20°C

Inlet and Outlet: —

Temperature Range: –45 to 40°C

Probe Diameter: 7.6 cm

When the chiller is running, make sure no external force on the hoses to cause any cracks.

Technical data source:
https://www.vwrsp.com/catalog/product/index.cgi?object_id=0006626&class_id=5002752
PPE Needed

- Goggle or safety glass
- Lab coat
- Gloves

Make sure no cracked glassware or desiccators are used!
Operate the vacuum line

1. Ensure all seals and the manifold stopcocks are properly greased
2. Make sure the vacuum trap is dry and clean
3. Enter the trap into the cooling dewars
4. Close all valves and open line to the vacuum pump
5. Be sure the dewar contains enough coolant to fully submerge the trap(s)
6. Start the chill and let it run for 1 h, and then start the vacuum pump
7. Ensure the pressure drops properly by watching the gauge
8. Make sure the system is under vacuum and condensate is below the level of coolant to prevent explosion
Shut down

• Turn off the vacuum pump
• Vent the manifold
• Empty the cold trap by allowing the trap to thaw before safe disposal of any condensed solvents (done periodically for large double cold trap).
• Special caution should be taken if liquid nitrogen has to be used. **Never** vent the system or turn off the vacuum pump before the liquid nitrogen is removed.
Clean the system

- **PPE:** goggle/safety glasses, gloves, lab coat
- **Materials:** Hexane, Vacuum grease
- **Procedure**
  - Dissemble the system with caution
  - Clean the different parts with hexane
  - Dispose hexane as normal organic waste
  - If needed, soak any glass parts of the manifold in base bath. Clean isopropanol with KOH added
Re-assemble

• Grease the joints with vacuum grease
• Slightly heat the joints with a heating gun before grease application
Vacuum pump oil change

- **PPE:** Goggle/safety glass, gloves, lab coat
- **Tools and supplies:**
  - screwdriver, tygon or latex tubing (18~36 in), vacuum pump oil (4 L), container for waste oil, squirt bottle of hexanes
Vacuum pump oil change

• Procedure
  1. Ensure the pump is off (power cord unplugged)
  2. Remove the top sealing lid
  3. Drain the waste oil to an empty container (big enough) through a hose
  4. Tilt the pump as necessary
  5. Partially fill the pump with fresh oil
Vacuum pump oil change

- Procedure (continued)

6. Seal the top and run the pump for 1 min. Drain again

7. Repeat steps 5 and 6 until no particulate materials can be observed from the drained oil

8. Fill the pump with fresh oil to a level marked by the level marks

9. Replace the sealing lid and tighten
The pump exhaust is vented to a fume hood
liquid $N_2$ as coolant

Only use liquid $N_2$ when you have to

Use danger sign and a blast shield
Use of vacuum desiccator

A safety cage is required to be around a desiccator under vacuum!!

Make sure the safety cage is positioned safely

Use a plastic vacuum desiccator if you can find such one and it satisfies your needs
The following three slides are just for your information. You should follow the SOP stated in the previous slides!
Dealing with Condensed O₂

- Liquid oxygen has a light blue color
- Explosive in the presence of organic compounds, including the grease
- Oxygen condenses when liquid nitrogen is used to cool the trap. Dangerous!!!!!!!
- If a blue color is observed and you suspect that liquid oxygen is formed once the liquid nitrogen dewar is removed, call EH&S @ 404-385-2964 for special care!
Deal with Condensed O$_2$

- Once a blue color is observed in the cold trap, keep the vacuum system running
- Evacuate all unnecessary personnel
- Place a blast shield around the traps and remove any organic materials nearby
- Remove the liquid nitrogen dewar, quickly vent the system and lower the hood slash as low as possible
- Leave the lab area and warn others not to enter
Deal with Condensed $O_2$

- Wait enough time for the system to warm up to RT (Still dangerous due to the possible formation of organic peroxide)
- Pour the liquid into a clean beaker and flush the trap several times with water. Remember to do this behind the blast shield and with the hood sash lowered
- Use a KI test strip to test peroxide existence. If a purple color is formed, peroxides are present. Use sodium thiosulfate or sodium sulfite to reduce the solution before the waste disposal
• Always check your research director for the most authoritative information if you are not completely sure about the safety issue.

• Questions?