V. General Procedures

E. Using the Schlenk Line

Overview

The Schlenk line is an extremely useful system for many chemists, allowing the conduction of air- and/or water-sensitive reactions without the expense and restriction of a glove box. The Schlenk line can be configured to pipe in any gas, but argon and nitrogen are most commonly used to provide an inert atmosphere for sensitive reactions. When used correctly, the Schlenk line is an invaluable asset; however, correct use is complicated and involves many extra steps. Additionally, there are several serious hazards that can present themselves with inappropriate use. The new user must be acquainted with the following procedures if he is to successfully utilize the line without damage to himself or others.

Common Materials

The Schlenk Line: The lines in the Joy group laboratory consist of two separate glass tubes. The top tube is the vacuum line, and the bottom tube is the nitrogen line.

The Nitrogen Line: The nitrogen line begins in the wall of the hood with the common nitrogen source. The nitrogen is then fed into the Schlenk line, where it can be directed into glassware by opening the ports on the line (partially unscrewing the bottom stopper). The nitrogen line continues to a mineral oil bubbler, which serves as a gas barrier between the nitrogen line and the ambient oxygen-containing atmosphere of the hood. The bubbler is also an indicator to the user that a pressure equilibrium is established in the line. When your nitrogen flow is running, if you do not see bubbles coming out of the mineral oil bubbler, you are building up pressure in the line and headed for an explosion. Gas flow into the bubbler can be adjusted by the valves at various points throughout the line.

The Vacuum Line: The vacuum line begins beneath the hood at the vacuum pump. The pump removes gases from the vacuum line, pulling them down into the pump and then exhausting them. The pump can be isolated from the vacuum line by means of the valve where the vacuum pump hose meets the line. The vacuum line can be vented (returned to ambient pressure) by the slow opening of this valve. Beneath this valve is a glass cylinder called a solvent trap.

Similar to the nitrogen line, the vacuum line continues past several ports that can be opened to expose glassware to vacuum.

Hoses: Made of tough, stretchy materials such as rubber or Nalgene, hoses form an airtight seal between the Schlenk line and any connected reaction flasks. These should always be checked for appropriate fit and for gashes or holes.

Vacuum Grease: The vacuum grease in use in the Joy lab contains siloxanes and silica. It is used to seal any joints in the glassware setup to improve the gas barrier between the flask and the ambient environment. Do not grease any portion of the Schlenk line itself, it has been designed

to keep an air tight seal without the use of grease and the application of grease can reduce its effectiveness.

Schlenk Glassware: Schlenk glassware has been specially made to withstand the temperature and pressure fluctuations associated with Schlenk procedures, specifically to withstand high vacuum without implosion. Glassware should always be inspected for cracks and defects before use. There are many various pieces, such as gas adapters, multi-necked flasks, distillation heads, etc., and the exhaustive list will not be covered here.

Dewars: These dewars are used for holding the liquid nitrogen coolant that will be used to cool the traps during vacuum procedures. Though they may look metal, they are made of a special type of glass and also contain a vacuum chamber, so they will break loudly and violently if dropped.

Safety Concerns

Keeping Pressure Equilibrium: The nitrogen line is designed, when used properly, to maintain equal pressure between each reaction flask and the inside of the line, and a slight positive pressure compared to the ambient environment (so that oxygen stays out). This is accomplished through the flow adjustment valves at various points throughout the nitrogen line. When you activate the nitrogen flow using the faucet on the hood, check first to make sure that there is gas exiting the mineral oil bubbler. If not, open the flow adjustment valves throughout the line until gas is seen. Without this exhaust, nitrogen pressure builds up in the line and can cause an explosion or ejection of stoppers, septa, etc. from glassware setups. If you close or reduce the output to the mineral oil bubbler, be sure that your system has an alternative exhaust to avoid a dangerous situation! Keep in mind that if you have multiple reactions running on the same Schlenk line the headspace of the flasks will mix. Do not run reactions that are not compatible (i.e. will react) on the same line at the same time.

The Vacuum Pump: The vacuum pump is a powerful machine that is used to create high vacuum within the line. The main safety concern here is periodic maintenance. The pump oil should be changed regularly to mitigate decomposition by organic solvent vapors that may be pulled into the pump. If the pump ever exhausts thick smoke or a foul odor, or if the pump is performing worse than usual, the pump oil should be changed immediately. The vacuum pump should be capable of pulling a vacuum of about 70-80 mtorr with no cooling traps installed, and at most 30-40 mtorr with traps installed. If you are commonly changing your pump oil due to solvent intake, review the proper procedure for cooling the traps on your line.

Cracked Glassware: Star cracks and hairline fractures result in glassware from normal handling, especially during cleaning. These easily-overlooked defects weaken the structure of the glass, making it more susceptible to explosions in increased-pressure situations (cannula transfers, refluxes, etc.) and implosions in low-pressure situations (vacuum). Glassware should always be carefully inspected for these defects before use on the line. Broken glassware can be repaired for use later or discarded.

Heating/Freezing Flasks on the Line: All of the same rules apply here as in the Pressure Equilibrium and Cracked Glassware sections. Especially when heating, an exhaust is necessary

to ensure that pressure in the flask does not build to intensely high or drop to intensely low levels. Breakage in glassware can be avoided by ensuring that any flask undergoing temperature change is open, either to vacuum or to the nitrogen line as the procedure requires. Heating/freezing a closed system is very rarely necessary and should be done extremely carefully, if at all!

Liquid Nitrogen: Liquid nitrogen is a cryogenic cooling agent that is used to prevent organic solvents from entering the pump oil. It's temperature is -196 °C, so skin contact should be avoided. Burns and blisters may result from contact, though the pain is not more than a slight stinging. Insulated gloves should be used while handling and pouring liquid N2. NITRILE GLOVES SHOULD **NOT** BE WORN, AND ANY HAIR TIES ETC. SHOULD BE REMOVED FROM WRISTS BEFORE USE. These items will freeze to the skin and prolong the exposure, leading to more serious burns!

Condensing Oxygen: This is the most dangerous hazard associated with Schlenk line use. Liquid oxygen is deposited into your solvent traps when air from the ambient environment is pulled through the vacuum line and then cooled by the liquid nitrogen in the traps. This does not occur during normal use, such as when evacuating a flask containing air, for example. When there is a leak in your vacuum line, or when an active vacuum port has been mistakenly left open to ambient for a period of time, oxygen can be condensed. This is why it is important to watch the pressure meter. If your pressure is suddenly and inexplicably rising, seal off your line ASAP.

Liquid oxygen is extremely reactive and very, very dangerous. The hazard presents itself when the traps are removed and the system begins to heat to room temperature. The oxygen may react explosively with organic solvents that are also in the trap. If that does not happen, then the expansion of the gas due to normal heating can build up pressure within the Schlenk line and cause an explosion in that manner. (Gases take up far more space than liquids do – remember your general chemistry!)

Fortunately, liquid oxygen has a very distinct pale blue color. However, if you are removing your liquid N2 dewars and you suspect that you have condensed oxygen, replace the dewars immediately, turn off the vacuum, open the system to the atmosphere (with the closest and largest ports available), close the sash, and allow the system to warm slowly as the N2 in the dewars dissipates. Close the doors on your hood and warn any other labmates present of the explosion hazard. Evacuate the lab and notify Dr. Joy. After the traps have come to room temp, consider them still dangerous as peroxides may have formed. Rinse the traps with water into a clean beaker. O_2 CONDENSATION IS ONLY ACHIEVED THROUGH IMPROPER SCHLENK LINE USE. THINK, AND DOUBLE CHECK, BEFORE DOING ANYTHING ON YOUR LINE.

Procedure for Starting Vacuum

Pump remains "off" when not in use. To turn pump on, turn on/off switch to "on". To evacuate the line:

1. Close vent and inlet valves on vacuum line.
2. Trap should be at room temperature (not immersed in dewar containing liquid N_2).

3. Then immerse trap in dewar containing liquid N_2.

When finished, Steps 4, 5 and 6 need to be done in rapid succession:

1. Remove dewar from under trap.
2. Shut off vacuum pump (turn switch on pump to “off”).
3. Open inlet valve on vacuum line.
4. Clean trap after it reaches room temperature.

Important Issues

- Always have the trap immersed in liquid N2 when drying (dewar lasts approximately 20 hours when filled with liquid N2)
- Never condense air in the trap (e.g. leave inlet valve on oven open to the trap while it is immersed in liquid N2 and closed to the manifold).
- If the pump seizes for any reason, turn it off and inform the person(s) in charge.