General Schlenk Line Use

Standard Operating Procedure

Lab: 162 Davenport

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Section 1: Overview

Type of SOP: ☒Process ☐Hazardous Material ☐Hazardous Class of Materials ☒Equipment

Synopsis:

The purpose of this SOP is to allow the easy and safe operation of the Schlenk line (i.e., vacuum and inertgas manifold).

Section 2: Risk Assessment Summary (Hazards and control measures)

Information obtained from performing a risk assessment should be entered into this section.

Materials:

Material (name, CAS #, other ID)	**Hazards**
Argon	Asphyxiant.

Relevant References for Material Hazards:

Argon: https://www.airgas.com/msds/001004.pdf

Equipment Hazards:

Glass may shatter/break if used improperly.

Hazardous Conditions:

Cold trap (L-N_2, Acetone + Dry ice) with heating elements nearby pose a glass-shatter hazard.

Technique Hazards:

None.

Personal Protective Equipment

Safety glasses, nitrile gloves.

Engineering Controls

Oil bubbler to observe inert-gas flow; Cold trap to condense liquids and prevent solvents from reaching the vacuum pump.

Section 3: Procedures I. General Operation

Many uses of the Schlenk line will be customized to whatever chemistry is being done, but the general principles behind its use will remain *roughly* constant.

Before turning on the pump:

1. Before you turn on the vacuum pump, make sure that the exhaust of the pump is being directed within the fume hood (Fig. 1). If this is not the case, then any solvent fumes that reach the pump will be vented into the open atmosphere and users will be exposed to any *hazardous* vapors that may be given off.
2. Before turning on the pump, ensure that the solvent trap is put together (Fig. 2), otherwise the pump will be pumping continuously against the atmosphere, which is not good for the pump's lifetime.
3. Lastly, before turning on the pump, ensure that all stopcocks are in the "closed" position (Fig. 3, pos. "B") to ensure that the vacuum will be only applied to the manifold rather than pull what is ever attached to the end of a given line.
4. Turn on the pump and *use your ears* to listen for any odd sounds or as if it were continuously pumping and not pulling a vacuum.

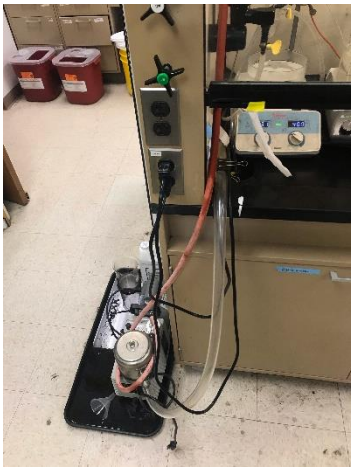

Figure 1. Vacuum pump with exhaust being vented into fume hood (clear tube)

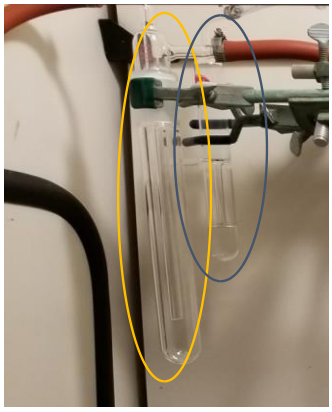

Figure 2. Solvent trap (orange oval), oil bubbler (blue oval)

Before turning on the gas:

1. Before you turn on the gas, make sure that all stopcocks are in the closed position (Fig. 3, pos. "B"). *Note: the copper wire is present to avoid the stopcocks from popping off due to any pressure buildup from Ar gas. This way, if they do pop out they will not fall to the bench and break.*

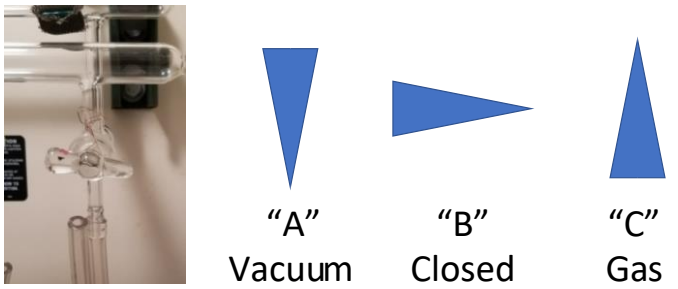

Figure 3. Settings on the stop cock. The head of the stopcock is roughly a triangle, which corresponds to a vacuum ("A", pointing down), closed ("B", pointing to the side), and gas ("C", pointing up).

2. To turn on the gas, first check that the Argon cylinder is open, then open the ball valve connected to the Argon rotameter. (The delivery pressure on the Argon cylinder should be set to 40 psi)
3. Initially set the flow rate to ~300 cm^3 min^{-1} for 2-3 minutes to flush out/purge the stainless steel lines and the Schlenk line.
4. You must confirm that gas is flowing through the system by visualizing a flow of bubbles in the oil bubbler (Fig. 2). Note: the bubbler should be filled with silicone oil (100 cSt) ~ 1 cm above the bottom of the glass rod that protrudes into the bottom.
5. Once lines are purged, turn flow rate to ~50-100 cm^3 min^{-1} to conserve Ar while maintaining the air-free conditions.

Figure 4. Rotameter to control Argon flow rate into the Schlenk line.

Basics of using the Schlenk line:

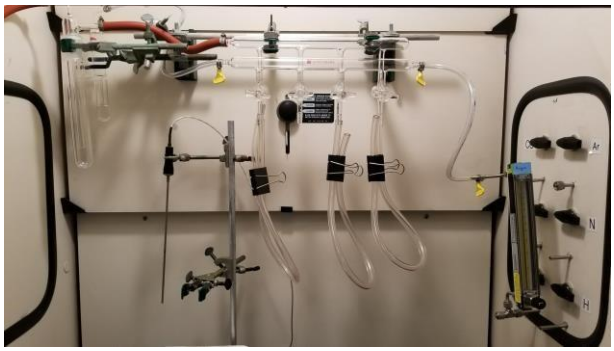

Figure 5. Complete Schlenk line

1. When first putting a flask on the Schlenk line, make sure you have the proper barbed vacuum adapter (e.g., 14/40, 24/40, etc) to match your flask.
2. Carefully attach the vacuum adapter to the end of one of the Tygon tubes that is connected to the manifold (*do not use too much force, it is easy to snap the glass*).
   Use a ring stand with standard clamps to support the reaction vessels.
3. Note: I recommend using 2- or 3-neck flasks with a rubber septa on two of the ends so that solvents and reagents can be added.
4. If you are planning on evaporating a significant amount of solvent (> 2-3 cm^3), set up a cold trap using a dewar filled with ice/H_2O, Acetone/dry ice, or frozen EtOH/H_2O slurry with the vacuum trap to keep this solvent from reaching the pump.
5. Start by turning the stopcocks to the vacuum (Fig. 3, pos. "A") to remove air from your flask. *Be patient, it takes minutes to reach a decent vacuum on your flask (~3-5 minutes).*
6. Use a heat gun to heat your flask under vacuum to desorb H_2O if your chemistry is sensitive to H_2O. Be patient, using a heat gun can take ~10-15 minutes to desorb most water. *Note: be careful when using the heat gun as the glass can reach >200 ^oC and that hot glass looks the same as room temperature glass. The heat gun can also melt the tygon tubing, so be careful where the heat gun is pointed. This should* ***only*** *be done with empty flasks – NO SOLVENT IN THE FLASK WHEN DRYING WITH A HEAT GUN.*
7. Now turn the stopcock to the gas (Fig. 3, pos. "C") to purge Ar into your flask.
8. Repeat steps 5 and 7 (step 6 only needs to occur once) two-to-three times to ensure all air is removed.

9. Make sure the stopcock is turned to the gas position, so that your flask is under a small partial pressure of Ar, and add your reagents using a needle/syringe through the rubber septa.
10. DO NOT pull vacuum while a significant amount of solvent (> 5 cm^3) is in the flask, the Schlenk line is not meant to remove solvent from reactions – use a rotary evaporator for this.

Finishing up:

1. Turn off the vacuum pump and re-pressurize the manifold by turning a stopcock connected to a line with no flask attached to the vacuum position and then turn the stopcock back to the closed position when re-pressurized.
2. Remove the solvent trap by carefully twisting and pulling down the trap and clean it if a significant amount of solvent/material made its way there.
3. When done with using the manifold, turn the stopcocks to the closed position (Fig. 3, pos. "B").
4. Turn off the Ar flow (i.e., close the ball valve on the side of the fume hood) to preserve the gas
5. Clean up everything. Make sure that all equipment (e.g., heat gun, reaction flasks, condensors, vacuum adapters) are put away and all messes that may have formed during your reaction/synthesis are cleaned up.

IV. Troubleshooting guide

Problem: Pump is not pulling vacuum

The pump will occasionally need to have its oil changed. This may be part of the cause for a poor vacuum. Also, do not leave the vacuum on for extended periods if it will not be in use. This will deteriorate its lifetime. The pump should only be left on when you are actively using the schlenk line.

Problem: No gas is coming out after I open the ball valve

Check to make sure that the Ar cylinder is not depleted (> 200 psi). If it is, close the valve in the gas cabinet, change the cylinder, then purge the line so that no air makes it way to the manifold.

Problem: The manifold was broken during use

For example, if there is a star-crack or one of the lines was snapped off during use, carefully un-do the tubing and bring the manifold to the glass shop for repair.

Section 4: Waste Disposal/Cleanup

The solvent trap should be cleaned out after each use when solvent has accumulated in it. This involves repressurizing the manifold, removing the trap, and cleaning with water/soap and acetone.

Section 5: Emergency Response

Section 6: Pump Maintenance

- The vacuum oil should be changed every few months. Steps:

1. Release old oil from the pump
2. Fill with flushing oil and let pump run for 30 minutes.
3. Release flushing oil
4. Fill with new oil

Emergency Response should be a component of the Laboratory Safety Plan. If there are particular response measures that are required by this procedure, include them here.

Training Documentation

Signing this document means that you have read and understand all aspects of this Standard Operating Procedure. The supervisor is the person that acknowledges you took the training and understand the procedure. They can be a lab manager or researcher assigned by the PI to oversee this particular SOP.

Name (Printed)	Name (Signed)	Supervisor	Date
