Schlenk Line

Standard Operating Procedure

Lab: Engineering Sciences Building 155

Department: Materials Science and Engineering

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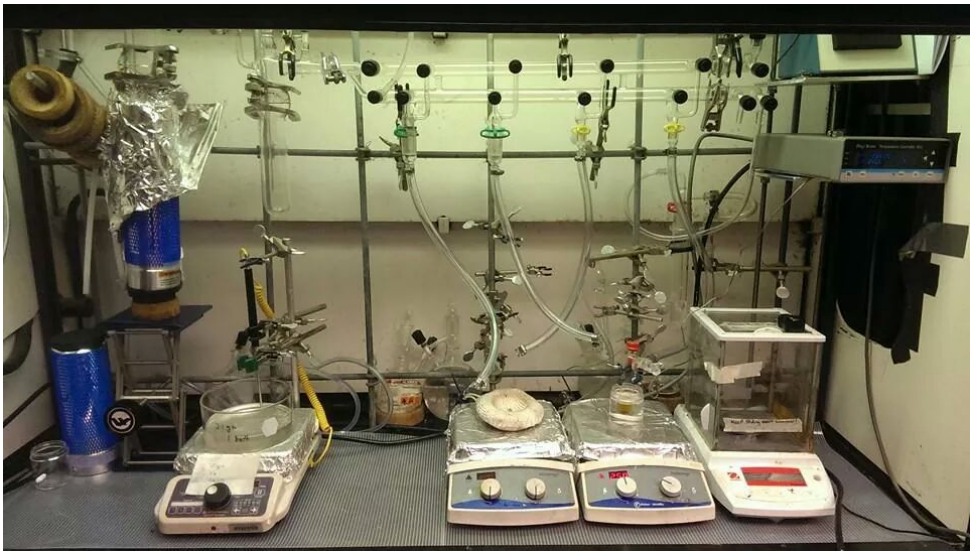
Section 1: Overview

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

Synopsis:

The dual vacuum manifold line, also known as a Schlenk line, is a standing piece of glassware used to surround the contents of a chemical flask in an inert atmosphere of Ar or N_2 gas. This instrument is critical for many important reactions including polymer chemistry, quantum dot synthesis, and any other variant of synthetic chemistry that requires the minimization of contact with air or other airborne contaminants. The Braun group has four different Schlenk lines—three located in the Beckman Institute, and one in the Engineering Sciences Building. In order to operate a Schlenk line for experimental chemistry, a user must be trained in methods to operate the valves and to introduce the proper gases as needed to ensure smooth operation and a desirable experiment. Proper use of a Schlenk line is quite complicated, hence the following procedures must be strictly observed after an initial series of trainings to avoid potential hazards and accidents that could compromise the expensive Schlenk glassware.

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



Materials and Equipment Hazards:

The Schlenk Line: The Braun group Schlenk lines were designed and blown by the chemistry department's glass shop in Noyes Laboratory. Any cracks or damages to the manifold, even if minor, will require the user to bring the manifold to the glass shop for repair. These manifolds were largely designed with O-ring fittings, and Teflon valves are used to seal ports to the reaction flasks, thus requiring no vacuum grease to achieve a proper seal. When using the Teflon valves, do not try to tighten them too much, as doing so may cause the glass to crack. Close the valve only enough to seal the reaction from either the vacuum or the nitrogen lines. Please do not use vacuum grease on any fitting other than those made PURELY of ground glass. These manifolds are separated into two main tubes—the top part accesses the vacuum line from a pump located beneath the fume hood, while the lower part passes nitrogen through the system.

The Vacuum Line:

The vacuum line begins in a cabinet below the fume hood, starting with a connection to a belt-driven pump, and connects to the Schlenk line via a glass hose on the left. Two thick glass cold traps precede the main manifold section, serving to condense any vapors into a solid to prevent any buildup inside the vacuum pumps. When the traps are submerged in liquid nitrogen-filled dewars, the vacuum pump removes gases like air or chemical by-products from the reaction flask, and deposits them in the cold traps.

A valve located at the top of each trap can be opened to vent the system. This valve should be kept closed through the duration of an experiment, and opened only when the system is ready for dismantling. For most experiments, only one of the liquid nitrogen traps needs to be attached to a dewar; for experiments that generate a lot of liquid vapor, use a second trap to ensure as little backflow as possible into the vacuum pump.

The vacuum line terminates in a valve that connects to a pressure sensor. When operating a Schlenk line without any reaction flasks, the pressure reading that goes below 80 mTorr signifies a properly functioning system. This number should be monitored occasionally during the experiment to ensure proper function.

The Nitrogen Line:

The nitrogen line begins via a connection to an orange house nitrogen valve on the wall of the fume hood. The nitrogen is first connected via a hose into a drierite column, which dries and purifies the gases entering your reaction system. The drierite column should be replaced when the dessicant turns pink. Fresh drierite has a light blue color.

When the nitrogen is dry, the gas is fed into the Schlenk and directed into glassware via hoses connected to the nitrogen line valves. The valves serve can be opened and closed as need to control the pressure inside the reaction flask. The nitrogen line will terminate in a hose connected to bubblers containing mineral oil. The rate of bubbling gives the user a rough idea of the volume of nitrogen gas passing through the system. A bubble rate of approximate one to two bubbles passing through the mineral oil every second is ideal during most chemical reactions. When the Schlenk line is dismantled at the end of the day, you may lower the flow of nitrogen gas into the system. For reactions that require a high positive pressure of nitrogen increase the speed by incremental amounts as necessary. When your nitrogen flow is running, but you do not see any mineral oil bubbling, you are building pressure in the line and may encounter an explosion. Make sure that gas flow is always constant and flowing through the mineral oil unless dictated otherwise.

Hoses: Hoses can be purchased at any length from the chemistry department stockroom at the Roger Adams Laboratory. These hoses can be made of either a vacuum-grade rubber or thick Tygon. Thicker hoses should always be used with the vacuum line to avoid collapse of the hose walls when subjected to low pressure. The hoses should always fit tightly around the fittings and should have no surfaces gashes or tears. Use hose clamps to ensure a tight seal between glass fittings.

Vacuum Grease: Most of the experiments performed with Schlenk line glassware should be greased to place a barrier between the flask interior and the ambient environment. Your standard variety Corning vacuum grease is suitable for most experiment; for higher temperature reactions (greater than 200 degrees C), use a specialty high temperature vacuum grease (e.g. Apiezon H). No part of the Schlenk line itself should be greased. Only the joints below the Teflon valves can be greased in the Braun group manifolds.

Schlenk Glassware: Schlenk glassware has been specially made to withstand the
temperature and pressure fluctuations associated with airfree synthetic 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.

Dewars: Liquid nitrogen dewars are used to contain a cryogenic fluid like nitrogen during an experiment to lower the pressure and protect the vacuum pump from any vapors generated during the reaction. These dewars are manufactured with a special variety of vacuum glass to maintain an insulating environment. Be careful not to drop this, as it would shatter very loudly and cause shards to disperse over a large area.

Liquid Nitrogen: Liquid nitrogen is a cryogenic fluid that is used to lower the pressure of the system and to minimize the amount of vapors that flow back into the pump. At a temperature of 77K, liquid nitrogen is especially dangerous when it comes into prolonged contact with skin. Use specially designed cryogenic gloves when handling liquid nitrogen, and avoid wearing nitrile gloves when replacing the cryogen in the dewars.

Hazardous Conditions and Technique Hazards:

Maintaining a Pressure Equilibrium: The nitrogen line is designed to maintain equal pressure between each reaction flask and the manifold, and applies a slight positive pressure compared to the ambient environment to expel any oxygen. This is accomplished by adjusting the Teflon valves placed at various points throughout the nitrogen line. A nitrogen flow should always be running through line to maintain an inert environment; keep the flow low when the Schlenk line is not being actively used. You can monitor this flow through the gas bubblers. Without this exhaust line, nitrogen pressure builds up in the manifold and can result either in an explosion or an ejection of pieces of glassware. If you close or reduce the output to the mineral oil bubbler, ensure that your system has an alternative exhaust. 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 mechanical pump that is used to draw a high vaccum on the line. The main safety concern here is periodic maintenance. The pump oil should be changed regularly (every 4 months) to mitigate decomposition of organic products that can degrade equipment performance. If the pump exhaust emits a thick smoke or a foul odor, or if the pump is incapable of pulling a vacuum, 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. If using especially corrosive solvents (acids, thionylchloride, halogens, et.c) consult the vacuum pump manual for setting up the use of the pump gas ballast.

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.

Liquid Nitrogen: Liquid nitrogen is a cryogenic cooling agent that is used to lower the pressure of the system and to minimize the amount of vapors that flow back into the pump. At a temperature of 77K, liquid nitrogen is especially dangerous when it comes into prolonged contact with skin. Use specially designed cryogenic gloves when handling liquid nitrogen, and avoid wearing nitrile gloves, long-sleeve shirts, and wrist accessories when replacing the cryogen in the dewars.

Condensing Oxygen: This is the most dangerous hazard associated with Schlenk line operation. Liquid oxygen is deposited into your solvent traps when air from the ambient environment is pulled through the vacuum line and 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 environment for a period of time, oxygen can condense. 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 have been deposited into 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.

Fortunately, liquid oxygen has a very distinct pale blue color. However, if you are removing your liquid N_2 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 N_2 in the dewars dissipates. Close the doors on your hood and warn any other lab personnel present of the explosion hazard. Evacuate the lab and notify Prof. Braun. After the traps have come to room temperature, consider them still dangerous as peroxides may have formed. Rinse the traps with water into a clean beaker and test the solution with the peroxide strips if peroxides are present, neutralize them with sodium thiosulfate or sodium sulfite before disposing of the waste.

Personal Protective Equipment

1. Use specially designed cryogenic gloves to protect your extremities when transferring liquid nitrogen into a dewar.
2. Wear lab glasses to shield your eyes from potential cracks in the glassware.

Section 3: Procedures

1. Before starting the pump, make sure that all the valves are closed to the ambient environment.
2. Turn on the vacuum pump. Allow the pump to run for 10 minutes. The system pressure should go below 100 mTorr.
3. Fill a liquid nitrogen dewar with cryogenic liquid. Place the liquid underneath the traps, and allow the pump to run for another 5 minutes.
4. When introducing a reaction vessel, perform a pump/purge cycle three times. This is done by attaching a flask with hoses to the manifold valves, ensuring that all ports are well sealed, and vacuuming the flask down to about 60 mTorr. When this vacuum is achieved, close the vacuum valve, close the valve to the nitrogen bubbler, and open the nitrogen valve to the flask. Close the nitrogen valve to the flask when the pressure equilibrates, and open the nitrogen valve to the bubbler. Repeat this process three times. When three pump/purge cycles are completed, you may begin your reaction.
5. Make sure that the nitrogen dewar is periodically filled with cryogen through the reaction.

Section 4: Waste Disposal/Cleanup

1. When you are ready to dismantle the Schlenk line, make sure that all ports are closed to ambient environment.
2. Disconnect the reactions from the line. Clean your workstation.
3. Turn off the pump. Immediately remove the nitrogen dewar and set down in a safe place within the fume hood.

4. Allow the contents of the cold trap to come to room temperature. When thawed, remove the cold trap and rinse with acetone and isopropanol. Use a stream of nitrogen to dry the cold trap, and replace the trap onto the Schlenk line.

Section 5: Emergency Response

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Section 6: Additional Information

Vacuum pump maintenance:

1. Make sure to change the vacuum oil every 4 months.
2. Attach a Tygon hose onto the vacuum pump's oil port. Open the valve and allow the oil to drain completely.
3. Fill the pump to the prescribed level in the glass window on the side of the pump.

Checklist:

☐*Make sure that all ports are closed before operating Schlenk line.*
☐*Without cryogen, vacuum pump should be able to pull pressure down below 100 mTorr.*
☐*Make sure that all valves are sealed to avoid drawing excess air into the system and causing an oxygen explosion.*
☐*Wear the proper PPE when handling liquid nitrogen.*
☐*Always maintain a steady level of liquid nitrogen in the dewars to maintain an ideal pressure.*
☐*Clean the traps well after using the Schlenk lines.*