STANDARD OPERATING PROCEDURE
USE OF SCHLENK LINE

Introduction

This SOP discusses the procedure and safety guidelines for using a Schlenk Line, also known as a Vacuum/Gas manifold.
The Schlenk Line is used for the handling of air or water sensitive chemicals. It consists of a gas manifold (for delivering either argon or nitrogen), a vacuum manifold (for evacuating glassware), and a vacuum pump (attached to the vacuum manifold). The inert gas is supplied either by a gas cylinder via a regulator or the house nitrogen, which then passes through the gas manifold, and finally bubbles through an oil bubbler.
This procedure applies to all staff, students and visitors that work in the laboratory and have the potential to use the line.

Safety

A Schlenk Line is often used for the handling of air or water sensitive chemicals, some of which could be very pyrophoric. Always read the MSDS of the chemicals that are to be used before using the line.
Operators must wear safety glasses, laboratory coat and gloves at all times when using the Schlenk line. New and infrequent users should also wear a face shield over their laboratory glasses.
The sash should be kept down at all times possible.

The major safety considerations are set out below:

EXPLOSION.
Explosions can occur in a number of ways, including:

- The use of pressurized gases. High vacuum manifolds are often connected to an inert or reactant gas supply line. One must ensure that the vacuum system is not closed when the gas supply is opened – there MUST be a source of pressure relief such as a bubbler. The pressure must be monitored with an electronic gauge, manometer or bubbler; make sure the valve to the pressure reading device is open to the manifold!
- Always TRIPLE CHECK that the manifold and supply line are connected to pressure relief (and your pressure sensor) before opening the gas supply. ALWAYS use an appropriate pressure regulator to avoid opening the line to more than 1 atm of pressure at any time.
- *Condensed gases.* Some gases, such as carbon monoxide and ethylene, are easily condensed into a liquid nitrogen-cooled trap. If the coolant level drops or you remove the nitrogen dewar without providing a means of pressure relief, the liquid may convert back to vapour. For example, just 10 mL of liquid CO (b.p. –191.5 °C) corresponds to 6.5 litres of gas. In a vacuum line with an internal volume of 500 mL the internal pressure would be 13 atm, more than enough to shatter the manifold with explosive force!
- *Runaway reactions.* Some reactions can occur violently and evolve large quantities of gas. Always provide a source of pressure relief!
- *Heating a closed system.* Never heat a vessel on a vacuum line without being open to a bubbler. Vacuum distillations must always have a pressure relief/regulator such as a manostat.
- Explosions of glass vacuum lines have lead to death and serious injuries. Always wear your safety glasses/goggles to protect your eyes. As the Schlenk is kept within the fumehood always keep the sash down when possible.

IMPLOSION.

- An unseen star crack or stress in a glass manifold can give rise to a catastrophic failure of the line while under pressure. Likewise, hitting the line with apparatus can cause a failure. While not usually as serious as an explosion, implosions generally involve sharp pieces of flying glass.

Liquid oxygen (when using a cold trap):

- If a constant stream of air is pulled through a vacuum trap cooled with liquid nitrogen, liquid oxygen may condense in the trap. Liquid oxygen is exceedingly dangerous and reacts violently with most organic substances, including Teflon tape, vacuum grease, and organic solvents. Even without this consideration, the pressure generated when a small quantity of liquid oxygen vaporises in a small space such as a vacuum manifold generates enough pressure to shatter the line.
- Should you lower the trap on your line and find a pale blue liquid, immediately replace the trap and back away. Consult your supervisor and researcher in charge of equipment IMMEDIATELY. Warn others of the danger, posting signs if necessary. To avoid such occurrences ensure that all experiments done while using a cold trap are only under nitrogen atmosphere only..

Risk Assessments

Refer to the following risk assessment forms:
Use of a schlenk line, assessed 02/02/10.
Use of glassware under vacuum, assessed 05/01/09.
Use of glassware, assessed 09/11/09.
Use of electrically operated equipment, assessed 20/06/07.
Waste solvent disposal, assessed 09/11/09.

Equipment and Maintenance / Handling and Storage / Labelling

The Schlenk line should be kept clean and free from any contaminants caused by experiments. Should any contaminant enter the line it must be cleaned ASAP. The joints of the line must be kept well greased and an appropriate level of oil in the bubbler maintained.

The pump should be serviced as described in its manufacturer's manual. If the maintenance is beyond the knowledge of the users the following technician should be contracted
Breakages and problems with the Schlenk line can be repaired by a glass blower. Before being repaired all silica grease must be removed from the joints by washing thoroughly with acetone.

Operating Procedures

When using the Nitrogen line:

1. Ensure all taps on the line are closed.
2. Turn on the nitrogen tap slowly, taking careful note of the bubbler’s flow rate. Turn nitrogen on until there is a constant stream from the bubbler.
3. Turn the appropriate tap connected to the experiment flask requiring nitrogen atmosphere.
4. To turn off nitrogen flow, close the tap connected to the experiment first, and then the nitrogen source.

When using the Vacuum line:

1. Ensure all taps on the line are closed.
2. Turn on Vacuum monitor.
3. Open the trap tap slowly to introduce the vacuum to the line.

4. Open the tap connected to the experiment concerned SLOWLY, paying close attention to bumping and degassing of the experiment. Close the tap if necessary to prevent contaminants entering the line.
5. Once tap is fully opened and stable continue to pay careful attention to the digital monitor to ensure no leaks are present.
6. To turn vacuum off, first turn the tap connected to the experiment then the tap connected to the trap. Once both are closed turn the pump off and finally release the vacuum by opening the trap tap slowly, and then the tap connected to the experiment extremely SLOWLY. Finish by closing both taps.

When switching between Nitrogen and Vacuum lines:

1. Follow the above procedures to introduce nitrogen to the experiment flask.
2. Follow the above procedure to introduce the vacuum to the line.
3. Turn the tap to the vacuum side extremely SLOWLY, paying close attention to both the bubbler and the experiment flask ensuring that the experiment doesn't bump or degas into the line.
4. When turning from vacuum to nitrogen be extra careful to ensure that the bubbler does not get sucked up into the line.
5. To turn off either follow the shutdown procedures outlined above.

   If the vacuum trap collects any waste it must be emptied after the experiment is completed. The user is responsible for researching if a liquid nitrogen trap is required for the experiment being performed.

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Waste Disposal

Solvent Disposal: Dispose of solvents in the appropriate waste container (e.g. water soluble waste solvent, water insoluble waste solvent, halogenated waste solvent). Refer to relevant chemical MSDS before disposal.

Oil Disposal: Dispose of waste oil in the water insoluble waste container.