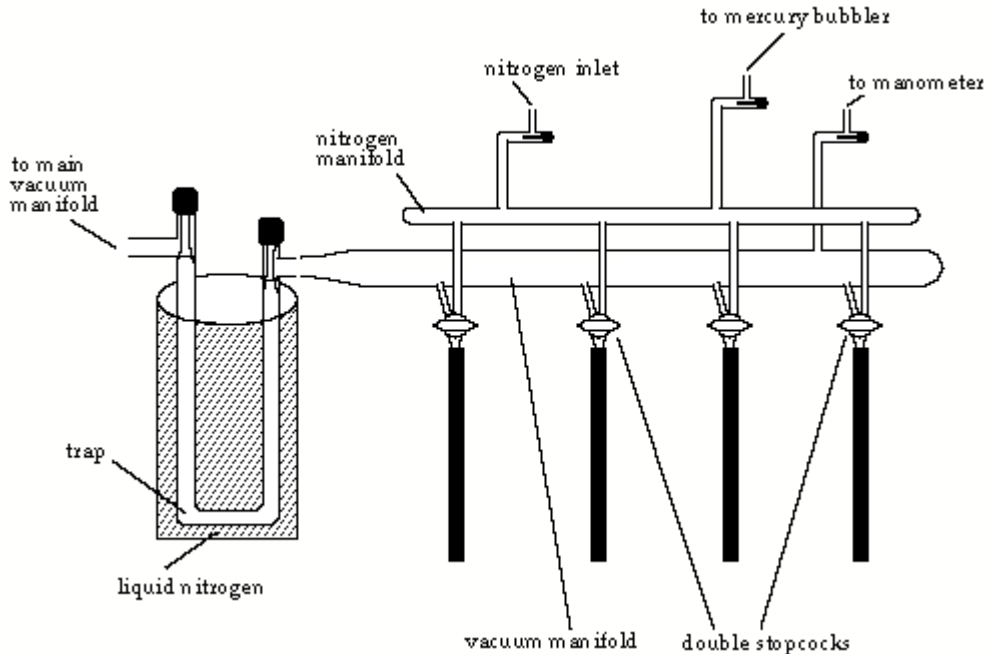
Trap, Vacuum line, Schlenk techniques

Make sure that you never pump air in a cold trap. Oxygen will condense and explode!

The schlenk techniques are named for the Schlenk who investigated the complex structures and equilibria for common Grignard reagents in solution. These techniques have been modified and refined to deal with transition metal compounds that are sensitive to the components of normal air (dioxygen and water are typically the most problematic) and are thus also referred to as "Inert Atmosphere Techniques". Moderate-to-quite-air-sensitive compounds are routinely manipulated by these procedures in modern laboratories. Extremely air sensitive or very volatile compounds, however, are not amenable to Schlenk techniques and are handled using "**High Vacuum Line Techniques**". Consult HU if you need to use these more specialized techniques.

I. Schlenk Techniques

The basic piece of equipment used in this project is the double manifold, or **Schlenk line**:



The designs of Schlenk lines vary in individual labs. For example, at EPFL, we are not allowed to use mercury bubbler, so we use a silicon oil bubbler. We use a digital vacuum manometer to monitor the vacuum, whereas in most places in the US Hg manometers are common.

Inert gas (argon or nitrogen) is provided through the top manifold. The inert gas enters from a tank via the indicated stopcock and any over pressure exits through a oil bubbler (not shown). The vacuum manifolds in Mead are all connected to one main manifold, which is isolated from the oil pump by a liquid nitrogen trap (solvents degrade pump oil). The vacuum in the individual manifolds is roughly indicated by the manometer. The main vacuum line is equipped with an electronic vacuum gauge.

There are certain hazards associated with this apparatus. First of all, any time there is pressure or vacuum in use there is a possibility of glassware failing due to fatigue. Using the oil bubbler as an outlet for the over pressure of nitrogen greatly reduces the chance of explosion, but the risk of implosion is not as readily controllable. Even glassware that is in apparently good condition can fail under vacuum as small as that provided by a water aspirator – the probabilities increase somewhat on a vacuum manifold, especially when the apparatus is subjected to thermal shocks. KEEP THE SHIELDS IN FRONT OF ANY GLASSWARE UNDER VACUUM!!
Liquid dinitrogen in an open dewar presents no hazards beyond frost bite, however, liquid dinitrogen condenses dioxygen at reduced pressure. **Should a trap cooled with liquid dinitrogen be left exposed to the air, dioxygen will condense. Liquid dioxygen is a deep blue color -- if you ever see a deep blue color in a trap, get HU immediately and follow his instructions.** If HU is not available, a general advice is to keep the vacuum on the system to pump the trap, and slowly warm up the trap (e.g., leave the trap on but not add more liquid N_2). LIQUID OXYGEN IN THE PRESENCE OF ORGANIC SOLVENTS PRESENTS AN EXTREME EXPLOSION HAZARD. We will always assume there are organic solvents in the trap.

II. PROCEDURE for vacuum lines:

1.1 Set-up

1.1.1 Always wear safety glasses whenever working in the hood area!

1.1.2 Examine vacuum manifold to ensure that it ready to be evacuated

1.1.2.1 Turn stopcocks to the horizontal position.

1.1.2.2 The liquid trap {looks like a giant glass finger} is empty and securely clamped in place.

1.1.3 Turn on vacuum pump with the switch located near the motor of the pump

1.1.3.1 Pump should become quiet within minutes indicating that there are no significant leaks

1.1.4 Connect the vacuum to the Schlenk line. This can be done by opening the corresponding stopcocks. You can notice the change in the reading of the vacuum monometer.

1.1.5 (optional) Check that the vacuum line is functioning correctly.

1.1.5.1 Test vacuum by placing thumb over one of the hoses descending from the manifold

1.1.5.2 Rotate the corresponding stopcock 90° CW such that the vacuum line is connected to your manifold.

1.1.5.3 Return stopcock to the starting position by rotating 90° CCW

1.1.6 Fill the Dewar with a small amount of Liquid N_2 (20%). Place the Dewar under vacuum trap. Adjust lab jack to appropriate height. Fill the trap with liquid N_2.

1.1.6.1 The Dewar is made of glass and is under vacuum. It will implode violently if the glass is shattered. Handle with care

1.1.6.2 Liquid N_2 is a cryogenic coolant and will cause burns to the skin if handled with bare hands

1.1.7 The vacuum line is now ready to be used.

1.2 **Shut Down**

1.2.1 Always wear safety glasses whenever working in the hood area!

1.2.2 Disconnect the pump from the manifold wither by turnoff a connection (if your vacuum line has one) or by turn off vacuum pump. **This is very important because we do not want to have the vacuum on while opening the Schlenk line to the air. To avoid condensing liquid O_2!**

1.2.3 Carefully lower the lab jack in order to remove the Dewar.

1.2.4 Open one of the stopcocks to relieve the vacuum in the line by rotating 90° CW and connect the Schlenk line to air. Valve can be left in open position

1.2.5 Remove the trap. **DO NOT add the contents of the Trap to a waste container unless it is > 0°C**

1.2.6 Vacuum trap can then be left in the hood to dry.

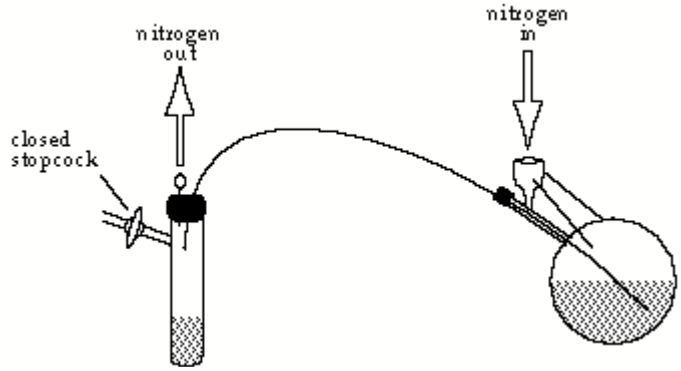
Vacuum Glassware (Schlenkware) is made with a side arm for evacuation of the apparatus and for the entering inert gas used to flush the apparatus.
Vacuum Grease should be used when assembling an apparatus for use on the double manifold. Grease should be removed using pentane, hexane, or petroleum ether, kimwipes, pipe cleaners and a pair of forceps.
A **septum** (plural "septa") is a stopper with a thin section in the middle to allow transfer of liquids in and out of the vessel with needles. Septa should always be folded down and wired when in use. *Never pump down on a septum capped flask-- always use ground glass stoppers.* Septa do not hold vacuum very well, even when they have not been pierced, and are best used only with a positive pressure of dinitrogen.
Exchanging a ground glass stopper for a septum or vice versa, requires a moderately strong flow of dinitrogen. When installing the septum under this positive nitrogen pressure, do so with a

small needle already piercing the septum. This allow the pressure to escape as you put on the septum and will allow you to purge out the small amount of air that is introduced with the septum installation.

Pump/fill cycles are used to establish an inert atmosphere in a vessel. The vessel is sealed, but attached to the line via pressure tubing. The vessel is evacuated by opening the double stopcock so that the vacuum manifold and the hose are connected, then filled with N_2 by moving the stopcock until the nitrogen manifold and the hose are connected. The nitrogen flow should be monitored via the silicon oil bubbler during this procedure. For best results, pump/fill cycles should be repeated three times.

A **cannula** is a long double ended needle. It is used to transfer liquids from one vessel to another. Cannula should be kept in the oven, and purged with dinitrogen while still warm. The other end of the cannula is inserted into the receiving flask, and the stopcock closed so that the only flow of dinitrogen is through the cannula. A needle is placed in the receiving septum to vent, and the cannula is pushed into the liquid to be transferred. *Do not transfer liquids through a cannula with vacuum -- the interface of the septum and needle will leak air.*

A **cannula filter** is a long needle with a piece of filter paper tightly wired onto a lipped glass attachment at one end. These can also be made with plain cannula.



Syringes are used to transfer liquids without exposing them to air. Make sure there is a positive nitrogen pressure in the vessel you are taking the liquid from. **This also applies to reagents sold with a septum cap (*e.g.* n-BuLi). These septa cannot withstand the vacuum you would create if you would take liquid without first inserting a nitrogen inlet.** An inert atmosphere should be established in a syringe by repeatedly drawing dinitrogen into the syringe and expelling it. *Do not pull hard on a syringe to create a vacuum -- the syringe will leak.* Allow the positive pressure of the dinitrogen flow to push the barrel out. By the same token, beware that the barrel is not forced all the way out of the syringe and broken. After measuring the correct volume with the syringe, bend the needle and take in a small amount of nitrogen from the vessel you removed the liquid from as a buffer. Quickly transfer the syringe to the receiving flask. First push out the buffer gas and then push down the plunger. Don't try to empty the small amount of liquid that remains in the tip of the syringe and the needle, as the graduation on the syringe is calibrated to exclude that volume. If the remaining content of the syringe is particularly sensitive (phosphines, strong reductants), take out a small amount of buffer gas again and immediately transfer the syringe to a quenching medium. Note that the atmosphere in the syringe will be inert for a very limited time during transfer. **Therefore, you should make sure to properly plan your experiment (donating flask, receiving flask and quenching flask) ahead of time and remove any unnecessary clutter in your fume hood, so that the transfers can occur without hinder.**