Introduction to the Schlenk Line

In inorganic chemistry, we often handle air-sensitive reagents and chemicals. In order to safely and effectively handle air-sensitive materials, two main types of techniques are used: Schlenk line techniques and glove box techniques. In this introductory activity, you will be introduced to Schlenk line techniques using water. While water is obviously not air-sensitive, it is a safe and environmentally-friendly substance on which to practice these techniques.

Set-ups can vary, but all Schlenk lines operate according to the following principles: The Schlenk line consists of two separate glass pipes. An inert gas (usually nitrogen or argon) flows through one pipe until it reaches an oil bubbler. The second pipe is connected to the vacuum line. This pipe terminates in a cold trap, so that any solvents evaporated under vacuum can be condensed into the cold trap rather than contaminate the vacuum pump. Each of the pipes is connected to multiple tubes via stopcocks. These tubes can in turn be attached to special flasks known as Schlenk flasks. When the stopcock is open to the inert gas pipe, the attached tube (and a flask attached to it) will be under a positive inert gas flow. When the stopcock is open to vacuum, the attached tube (and a flask attached to it) will be under vacuum. Using the Schlenk line, you can work in an inert gas atmosphere, excluding air from your system.

1. Using the Schlenk line to remove solvent under vacuum
   1. Make sure all stopcocks on the Schlenk line are closed.
   2. Open the nitrogen valve so that it passes through the bubbler at a rate of ~1 bubble per second.
   3. Turn on the vacuum pump. It will sound loud at first but then should quiet down. Listen for any sustained gurgling noise, which indicates there is a leak. The pressure reading on the manometer should decrease. It is very important to verify that there are no leaks before proceeding to the next step. If there are leaks present, you can condense O2, which is an explosion hazard.
   4. Liquid nitrogen is a cryogen. Make sure to wear appropriate personal protective equipment when working with it. Carefully fill a dewar with liquid nitrogen and place the trap in the dewar. This prevents any solvent that is evaporated from entering the pump. Do not open the Schlenk line to air after this, as you risk condensing O2, which is explosive.
   5. Fill a 100 mL Schlenk flask with approximately 50 mL of water. Place a septum on the flask. Connect the flask to one of the hoses on the Schlenk line.
   6. Slowly open the stopcock connecting the hose you are using to the vacuum. You want to prevent solvent from bumping. The vacuum will hum loudly at first and the pressure reading on the manometer will momentarily increase, but make sure the vacuum quiets down and the pressure reading on the manometer decreases again quickly. If a gurgling noise persists, quickly close the valve to the vacuum. Inspect the flask and address any leaks before opening the flask to vacuum again.
   7. Touch your flask. It should feel cool due to evaporation of the solvent.
   8. Close the stopcock to the vacuum.
   9. Refill the flask with nitrogen by very slowly opening the valve to nitrogen. (Do this slowly to prevent the pump oil from bumping.)
   10. Watch the bubbler to make sure the flow rate returns to ~1 bubble per second.
2. Preparing a flask for air-sensitive synthesis
   1. For very air sensitive chemistry, it is necessary to exclude as much air as possible from the flask. On a Schlenk line, this can be accomplished by alternatively placing your flask under vacuum/N2 for 3-5 cycles. This will remove air from the flask and replace it with N2.
   2. Place a septum on a second Schlenk flask and connect it to a second hose on the Schlenk line.
   3. Open the stopcock connected to the second flask to vacuum and keep the flask under vacuum for three minutes. Refill the flask with nitrogen by very slowly turning the stopcock 180° to open the flask to nitrogen. Watch the bubbler to make sure the flow rate returns to ~1 bubble per second.
   4. Repeat step d two more times, ending with your flask under N2.
   5. Note: If you have a flask that already contains an air-sensitive substance and want to connect it to your Schlenk line, you should remove any air from the tubing by connecting your flask to the tubing, keeping the Schlenk flask stopcock closed, and doing 3-5 cycles of N2/vacuum before opening the Schlenk flask stopcock to the tubing.
3. Transferring a reagent via syringe
   1. Place a 12”-24” needle on a 10 mL plastic or glass syringe.
   2. Remove any air from the needle and syringe: With the plunger completely depressed, place the needle through the septum of the flask containing the water into the headspace of the first flask (above the water).
   3. If you have a glass syringe, the N2 pressure should push the plunger back, filling the syringe with N2. If you have a plastic syringe, you will need to pull the plunger back to fill the syringe with N2.
   4. Remove the needle from the flask and push down the plunger completely, expelling the N2 from the syringe.
   5. Repeat steps d-f two more times.
   6. The syringe is now air-free and we can use it to transfer our reagent. Place the syringe in the flask again, and lower the needle until it is in the water. We wish to transfer 5 mL. To ensure that the correct amount is transferred, allow slightly more than 5 mL of water into the syringe. Next, pull the needle into the headspace of the flask. Turn the syringe upside down, and gently depress the plunger to expel any gas bubbles and excess solvent. With the needle in the headspace, pull back the plunger slightly so that the first 1-2 mL of the syringe is filled with N2.
   7. Pull the needle out of the flask, and quickly transfer the water to the second flask.
4. Transferring a reagent via cannula (a double-tipped needle)
   1. To transfer more than 50 mL of solvent, it is better to use a cannula (a double-tipped) needle.
   2. Place the cannula into the headspace of the first flask and keep in for approximately 30 seconds to flush it with nitrogen.
   3. Place the other end of the cannula needle into the second flask.
   4. Push the end of the cannula needle in the first flask down until it is in the solvent.
   5. Place the receiving flask under a slight vacuum by briefly opening the valve to vacuum and then closing it again. At this point, the pressure differential should cause the solvent to flow from the first flask into the second flask. When approximately half the solvent has been transferred, pull the needle up into the headspaces of both flasks.
   6. Open the second flask to nitrogen. Remove the needle from the second flask, and then from the first flask.
5. Shutting down the vacuum
   1. Close all the valves on the Schlenk line and remove your flasks.
   2. Remove the liquid nitrogen dewar from the cold trap.
   3. Shut off the vacuum pump.
   4. Quickly open a valve on the Schlenk line to the vacuum line to vent the system.
   5. Shut off the nitrogen.