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Proper Handling and Manipulation of Air- and Water- Sensitive Chemicals

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Corresponding Author:	Robert Rioux UNITED STATES
Corresponding Author Secondary Information:	
Corresponding Author's Institution:	
Corresponding Author's Secondary Institution:	
First Author:	Robert Rioux
First Author Secondary Information:	
Order of Authors:	Robert Rioux
Order of Authors Secondary Information:	

PI Name: Robert M Rioux & Ajay Sathe & Zhifeng Chen

Science Education Title: Proper handling/manipulation of air- and water- sensitive chemicals (i.e., using a schlenk line)

Overview: The use of reagents sensitive to oxidation or moisture necessitates the use of air-free techniques. A Schlenk line is routinely used glass apparatus to perform air and moisture free manipulations in a chemical laboratory.[1]The Schlenk line is widely utilized by many chemists since it allows them conduct air or water sensitive reactions even without the expense and restriction of a glove box. The Schlenk line can be configured to inert gas atmosphere such as Nitrogen and Argon or expose the glassware to vacuum. Another way to achieve air and moisture free involves the use of glovebox. The major difference between Schlenk line and glove box is that purge and refill applies directly to the reaction vessels whereas purge and refill applies to the airlock instead of the glovebox. Besides, in glovebox, conventional laboratory equipment can be set up with the large inert space however gloves have to be used to handle experiment and the glovebox itself is also expensive.

Principles: A Schlenk line consists of two glass tubes connected together using several ports. One of the tubes is connected to a source of vacuum, and the other is connected to an inert gas (typically nitrogen, but other inert gases can be used). The ports connecting the two tubes are equipped with two way valves which allow for the selection of either vacuum or inert gas atmosphere at the port outlet. A liquid seal is used at the end of the inert gas tube, to maintain the inert atmosphere in the event of backflow while refilling evacuated glassware.

Procedure:

1. General procedure for carrying out a reaction under an inert atmosphere

1.1 Evacuating the glassware

1.1.1 Start the vacuum pump and fill the dewar in which the schlenk line trap is immersed in with liquid nitrogen.

1.1.2 Use oven dried or flame dried glassware to ensure that there is residual moisture adsorbed on the walls of the glassware.

1.1.3 Seal the flask using either ground glass adapters, or rubber septa. It is highly recommended that vacuum grease is used to avoid seizing of the ground glass joints.

1.1.4 Connect the sealed glassware to the Schlenk line using an appropriate adapter/connection.

1.1.5 Turn the two way valve to open the line to vacuum.

1.1.6 Evacuate the system and let the flask cool to room temperature

1.2 Purging with inert gas

1.2.1 Make sure the inert gas line is open and sufficient flow is seen through the bubbler. (A typical flow for operation of the schlenk line is about one bubble per second. The flow should be increased while initially purging the system after evacuation.)

1.2.2 Once the flask has cooled to room temperature, turn the two way valve slowly to open the line to inert gas, taking care that some gas still manages to reach the bubbler (Use caution so as to not change over to inert gas very quickly as it can lead to the liquid sealant from the bubbler to back fill into the schlenk line.)

1.2.3 Once the inert gas flow has stabilized back to its original state (as observed on the bubbler), switch the two way valve back to vacuum, and repeat the above procedure two more times.

1.3 Adding reagents

1.3.1 Once the flask has been evacuated and backfilled with inert gas a total of three times, it is now ready for use in handling air/moisture sensitive reagents. The inert gas flow can be readjusted to one bubble per second to save the consumption of inert gas.

1.3.2 The reagents are typically added from a septum sealed bottle or from a solvent dispensing station.

1.3.3 Use an oven dried cannula or needles to transfer air sensitive reagents into the flask. (Make sure the flask is under an inert atmosphere, and not under vacuum as addition of chemicals to a flask under vacuum can damage the vacuum source.)

1.4 Quenching the reaction

1.4.1 Once the reaction is complete, carefully quench the reaction using an appropriate reagent.

1.4.2 The quenching step regularly results in the generation of gases, make sure that the two way valve is open to the inert gas so that there is no pressure build-up in the glassware.

2. Basic design and set up of Schlenk line.

The design of Schlenk lines vary from labs but the keys feathers are the same. Figure 1 shows a schematic set up of the Schlenk line with key feathers.

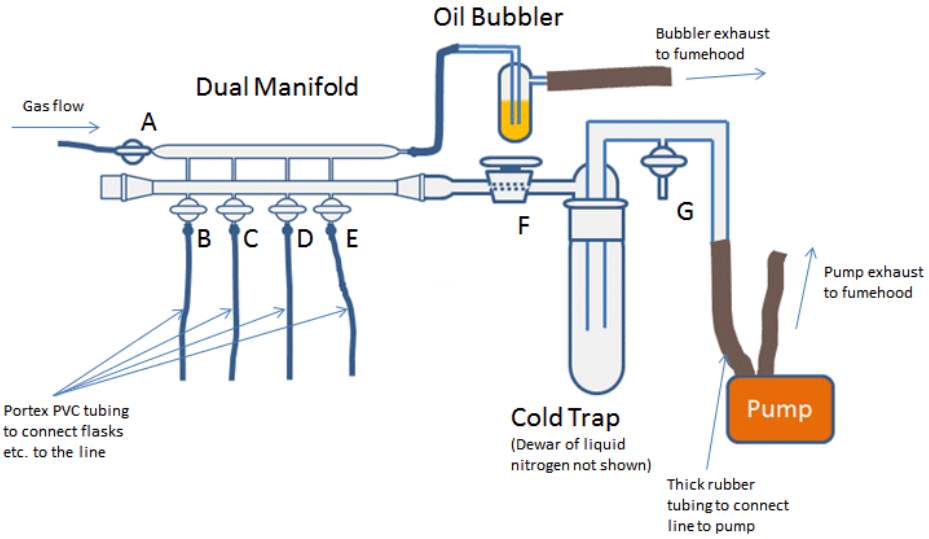

Figure 1. Schematic set up of a Schlenk line.

The dual manifold is consisted of two parallel glass tubes, which forms the main body of Schlenk line. Gas manifold connected to the inert gas supply and vacuum manifold connected to the vacuum line, which allows easily switches between inert gas and vacuum by tuning the taps. The gas exits the manifold through a visible oil bubbler in order to monitor the flow of the gas. The vacuum manifold is closed at one end and the other end is connected to a cold trop and a

vacuum pump. The cold trap is used to prevent volatile or corrosive solvents from damaging the pump.

3. Safety concerns with Schlenk line.

3.1 Liquid oxygen.

Liquid oxygen can be accumulated if a constant steam of air went into the vacuum line with cold trap and it can be indicated by a light blue liquid in the trap since liquid oxygen is light blue. Liquid oxygen is dangerous due to its violent reaction with organic solvent including the vacuum grease and the high pressure generated once vaporized in the confined space. Remember never open the vacuum line to air when the cold trap is in place.

3.2 Explosion.

3.2.1 Pressured gases. Make sure the system is open with the inert gas flowing. Explosion can occur if pressure is built up in case of closing system.

3.2.2 Violent reaction. If the reaction went out of control, a large volume of gas might generate quickly to cause explosion. Be aware of the reaction before operating in the Schlenk line.

3.2.3 Heating a closed system. In a closed system, the increase of the temperature will increase the pressure also. Be aware of any reactors under heating condition and make sure the line is open and there is bubbler for pressure relief.

3.3 Implosion.

Cracks in glassware will cause breaks under vacuum. Repair or replace the cracked glassware.

Summary: The Schlenk line is an extremely useful system, allowing the manipulation of air- and/or water-sensitive reagents without the expense and restriction of a glove box. It is most safely utilized inside a working fume-hood. Care should be taken to prevent pressure build-up in the schlenk line by ensuring proper venting through a gas bubbler.

References:

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2. Tips and Tricks for the Lab: Air-Sensitive Techniques, Sarah Millar, 2013, ChemistryViews at http://www.chemistryviews.org/details/education/3728881/Tips_and_Tricks_for_the_Lab_Air-Sensitive_Techniques_1.html