Section 5.2 **Title:** Schlenk Techniques **Revision Date:** 11/01/19
Prepared By: Jesse Guillet and Michael Roy **P.I.:** Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
___ Carcinogen ___ Reproductive Toxin ___ High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:
Instructions for using a Schlenk

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*
Daniels Chemistry - All Berry group labs

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Dependent on chemistry	Consult relevant SDSs for more details

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*
Sharps (needles)

Exposure Controls: *(Check all that apply)*
PPE: _X_ Safety Glasses ___ Face Shield ___Chemical Splash Goggles
___Chemical Apron _X_ Gloves (Nitrile) _X_ Lab Coat
___Respirator (type) ___Other:

Engineering Controls:
_X_ Fume Hood ___Biosafety Cabinet ___ Glove box
___ Vented gas cabinet ___Other:

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

N/A

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Dispose of the reagents involved as appropriate. Consult SDSs for more details.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

No formal training or documentation is required. This procedure should be demonstrated by experienced lab members. New members should talk through their procedures with experienced lab members.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry Signature: _____ Date: _____

Schlenk Techniques

Overview:
Schlenk techniques provide chemists with the ability to manipulate chemicals under a controlled atmosphere free from water and O_2. The central apparatus in Schlenk technique is the Schlenk manifold, which consists of two lines: one filled with inert gas (most often N_2) and the other evacuated with a vacuum pump. Mastery of Schlenk techniques takes time and practice.

The typical UW Schlenk line. The upper line connects to a vacuum pump via a solvent trap. The lower line is filled with N_2 through one of the top ports. The four lower ports allow for N_2 or vacuum to be applied to the connected glassware.

Inert gas:
The inert gas used in a Schlenk line can be provided by a gas cylinder or through plumbed gas lines. Standard use in the Berry group involves "house" N_2, which is available through gas taps in all fume hoods. The N_2 is fed first through a bubbler to provide an indication of flow rate and then through a tall column of Drierite to ensure absolute dryness of the gas. The N_2 then flows into the Schlenk manifold (pictured above) before exiting through a one-way gas bubbler. One-way bubblers are preferred because, in the event of an under-pressurization, they prevent the backflow of oil from the bubbler into the Schlenk manifold.

Vacuum:
A vacuum pump is connected to the upper line of the Schlenk manifold *via* a solvent trap. In the Berry group, the vacuum trap is a double-trap system with an optional bypass - i.e. vacuum flow can be directed through one or both solvent traps. Under normal operation, a single solvent trap is sufficient. When removing a larger quantity of solvent, or working with a more volatile solvent, using both solvent traps provides additional protection for the vacuum pump.

Schlenk Glassware:
There are many ways of connecting glassware to a Schlenk manifold. Most Schlenk lines employ rubber tubing to connect the taps on the manifold to other glassware. Standard ground-glass joints can be connected with the appropriate size of inlet adaptor, but several other types of connection exist.

Schlenk flask: Available in either the pear-shape (pictured) or a traditional round-bottom, Schlenk flasks are the most common piece of glassware for running reactions on a Schlenk line. Schlenk flasks connect to the rubber tubing of a Schlenk line through a sidearm with either a ground-glass stopcock or a threaded Teflon adaptor. When the ground-glass joint is sealed, the stopcock or threaded adaptor allow for the flask to be connected to the Schlenk line or sealed completely.

Solvent storage (Straus) flask: Available as round-bottom or long tube flasks, these flasks are suitable for storing liquids and solutions, as well as degassing solvents. These flasks connect to the Schlenk line using an inlet adaptor, and the threaded Teflon stopper can block the sidearm from the rest of the flask, sealing the contents of the flask from exposure. This type of flask should be used when bringing solvents or solutions into a glovebox, as they are designed to withstand the pressure difference as the glovebox antechamber is evacuated.

Other connections exist, but will generally employ a ground-glass stopcock, a threaded Teflon adaptor, or a threaded Teflon stopper, as pictured. When examining a new piece of glassware for use in Schlenk chemistry, always examine the flask to ensure that you understand how gas will flow in and out of the item and that you can create effective seals where needed.

Images from Chemglass.com

Preparing a Schlenk line: Before using a Schlenk line, ensure that the inert gas flow is uninterrupted from the source, through the bubbler and drying column, and out the exhaust bubbler. Ensure that the vacuum manifold is securely connected and sealed from the atmosphere. A newly setup line should have N_2 flow through overnight to ensure that all air is removed.

General tips

- SOPs exist for several specific operations, including:
    - Removing solvent under vacuum
    - Air-free filtration
    - Using cannulas and syringes

- Oven-dried glassware can be cooled in a desiccator for convenience or connected immediately to a Schlenk line to be dried under vacuum / inert gas.
- Inspect all Schlenk manifold joints, glassware, and tubing to ensure that your system won't leak.

Common Schlenk operations

Cycling a flask under N_2: Often, flasks and solid reagents are prepared in air and then connected to a Schlenk line for subsequent air-free handling. Connect a flask to the Schlenk line using the appropriate connection (inlet adaptor, threaded Teflon adaptor, etc.). Ensure the flask is sealed from the atmosphere (be sure to grease all ground-glass joints). Open the Schlenk adaptor on the flask (if present) so that the tubing and flask are open to each other. Open the vacuum tap on the Schlenk manifold. If you have a powder in the flask, open the tap slowly to prevent the powder from flying up into the line. Evacuate the flask for 5-10 minutes, depending on the size of the flask. Close the vacuum tap, then slowly open the inert gas tap. Wait until bubbling at the exhaust bubbler resumes, indicating the flask has reached standard pressure. Repeat the evacuation and refill steps at least two additional times.
Note: If the contents of your flask are already under inert gas, the evacuation/refill steps should be performed with the Schlenk adaptor closed (i.e. only evacuating and refilling the rubber tubing between the manifold and flask). Due to the small volume of the tubing, the evacuation cycles can be much shorter (~30 seconds).

Reflux under N_2: Prepare your reaction flask as above (if necessary), and add solvent using the appropriate transfer technique (e.g. cannula transfer). Your flask should be equipped with a reflux condenser, which connects to the Schlenk line *via* an inlet adaptor. When you are ready to heat your reaction, ensure that the inlet adaptor on the reflux condenser is open to N_2, then close off any other Schlenk adaptors in your reflux setup. This ensures that solvent will not escape into the rubber tubing or Schlenk manifold. Continue to carry our your reflux as you normally would.

Drying solids under vacuum: See the SOP for removing solvents under vacuum. If you wish to continue drying a solid powder to remove trace solvent, place your solid in the desired container. Often, screw-cap vials are used for the final storage of solids. Screw-cap vials can be connected to Schlenk lines through a threaded Teflon adaptor and an inlet adaptor, and several such adaptor setups are available in the lab. If you believe the solid contains a reasonable amount of solvent, set up liquid nitrogen at your solvent trap as described in the SOP for removing solvents under vacuum. Slowly open the vacuum tap on the Schlenk manifold, being careful not to pull powder into the line. If the sample may be air sensitive, evacuate and refill 3+ times before exposing the sample to prolonged vacuum. You may also wish to heat your sample gently if you are removing high boiling solvents such as water or toluene.