Section 5.4 **Title:** Degassing Solvents **Revision Date:** 11/01/19
Prepared By: Sungho Park 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:
Degassing of solvents for oxygen-sensitive chemistry

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)
Organic solvents	Consult relevant SDSs for more details

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

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 _X_ 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.).*

Receive training before degassing solvents.

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 excess organic solvents in the appropriate waste carboys.

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. New lab members should consult with senior members before degassing solvents and may wish for informal training or supervision. The freeze-pump-thaw technique should be demonstrated by an experienced lab member, and new members should be supervised the first time they perform this technique.

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: _____

Degassing Solvents

Introduction

Degassing solvents with an inert gas serves to remove the oxygen dissolved in the solvent. There are three methods available: (1) sparging with a needle, (2) quick liquid-phase vacuum cycles, or (3) freeze-pump-thaw cycles,. Sparging and liquid-phase vacuum cycles replace the dissolved gases in the solvent with the inert gas (often N_2 or Ar). Freeze-pump-thaw completely removes dissolved gases.

Method 1 (sparging with a needle)

1. Collect solvent in a Schlenk/Straus/solvent storage flask and seal the flask with a ground glass joint. Cycle the flask under inert gas. Under a dynamic inflow of inert gas through the Schlenk adapter, replace the ground glass joint with a septum. Purge the septum ("burp") for several seconds before folding it over. (Alternatively, you can seal the flask with a septum in the first place.)
2. Attach a needle hot from the oven to another outlet on your line and let it purge with a dynamic flow of inert gas until it cools.
3. Stick the needle tip through the septum into your flask containing the solvent. Do not immerse it yet. Then, stick a disposable needle into the septum. Be careful not to block the top of the disposable needle with your finger. The disposable needle should be a smaller diameter (larger gauge) than the long needle.
4. If you are sparging a particularly volatile solvent, such as diethyl ether or pentane, it is helpful to immerse the flask in an ice bath to reduce evaporation of the solvent during the sparging process.
5. Close the Schlenk adapter on the flask. Immerse the needle tip as far into the solvent as possible. Bubbles should start appearing. A good rule of thumb is to sparge for 1 minute per 5 mL.
6. When done, raise the needle above the solution surface and reopen the Schlenk adapter. Remove the disposable needle. Pull out the sparge needle and turn off the inert gas flow on that outlet.

Method 2 (liquid-phase vacuum cycles)

1. Make sure that your line has a LN_2 cooling trap. For particularly volatile solvents (Et_2O, pentane, CH_2Cl_2, etc.), a double trap setup is recommended.
2. Collect the solvent to be degassed in a solvent storage flask or in a Straus flask. Do not fill the flask more than 3/4 full. Use a greased inlet adapter to connect the flask to your Schlenk line.
3. With the threaded stopper on the flask closed, open your line to vacuum.
4. While swirling the flask rapidly to aid in liquid-gas mixing, open the threaded stopper and continue swirling until the solvent is bubbling vigorously. Close the threaded stopper.
5. Close your line to vacuum and open it to N_2.
6. Open the threaded stopper to refill the flask with N_2. Wait until the bubbler on your line resumes bubbling to ensure the flask has been fully repressurized. Close the threaded stopper.
7. Close your line to N_2 and open it to vacuum.

8. Repeat steps 4-7 between two and four additional times, for a total of three to five vacuum cycles.
9. If bringing the solvent into the glovebox, skip the final N_2 refill and leave the flask under a static vacuum. It can be brought into the glovebox in this condition.
10. Turn off the vacuum to your line and thaw the trap to remove accumulated solvent.

Method 3 (freeze-pump-thaw cycles)

1. Make sure your line has a LN_2 cooling your trap. Prepare an extra dewar filled with LN_2 or another suitable cooling bath for your solvent, such as dry ice and acetone. Make sure it is the appropriate size for your flask. Make sure your flask is sealed using a greased ground glass joint. Make sure your flask is at most half-filled (too large of a volume can make this method challenging). It is best to use a solvent storage flask or a Straus flask for freeze-pump-thaw procedures.
2. Under a dynamic inflow of inert gas, immerse your flask into your extra LN_2 dewar. Wait for it to freeze (swirling the solution helps).
3. Close off the inert gas flow and evacuate the flask for ten minutes (while still immersed in LN_2).
4. Close off the Schlenk adapter so that you now have static vacuum in your frozen flask. Remove it from the LN_2 bath and begin to thaw. Your frozen solvent may look like it's "bubbling" as it thaws. It's important that the flask does not warm up too quickly, as this can cause it to break. It is best to allow the flask to warm slowly in air at first. Once the outside of the flask has warmed for a few minutes, the entire flask can be submerged in a cool water bath to help the solvent fully thaw.
5. Once thawed, reimmerse the flask in LN_2. When frozen, open the Schlenk adapter to dynamic vacuum again for ten minutes. Repeat until thawing yields no bubbling (usually 3-4 cycles are sufficient).
6. After your last thaw, close off the vacuum and backfill the line with inert gas. Open the Schlenk adapter so that your flask is under a dynamic inflow of inert gas. If you're taking the solvent into the glovebox, you should leave it under static vac and thaw it completely to room temperature.

General notes:

Sparging and liquid-phase vacuum cycles are equally effective and much easier than freeze-pump-thaw. The biggest advantage to FPT is that it results in less loss of the solvent, making it the preferred method for very low boiling or expensive solvents, such as deuterated NMR solvents. Freeze-pump-thaw cycles should also be used for corrosive solvents (e.g. acetic acid) to prevent corrosion of the needle.