Section 5.12 Title: Removing Solvent Under Vacuum **Revision Date:** 11/01/19
Prepared By: Mike Aristov 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:
Removal of solvent from using a Schlenk line and solvent trap.

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.*
Vacuum (implosion), cryogenic liquids

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.).*

Inspect all glassware for cracks or defects before exposing to vacuum.

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 solvents involved in appropriate carboy. Consult SDSs for more details. Excess liquid nitrogen is returned to the storage dewar.

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: _____

Removing Solvent Under Vacuum

1. Turn the vacuum pump on. Both solvent traps should be evacuated for at least 10 minutes before proceeding. Adjust the valves so that solvent vapors have to pass through both traps in series. This will ensure all vapors are caught.
2. The solution from which you wish to remove solvent should be in a Schlenk flask with a greased ground glass stopper and well-fitting, closed Schlenk valve and (ideally) a stir bar.
3. Attach the Schlenk valve of your flask to the tubing of the line.
4. Evacuate and backfill the tubing at least three times before opening the flaks to the line.
5. Add liquid nitrogen to two appropriately sized dewars. They should not be filled, as the solvent traps will take up a substantial amount of volume.
6. Submerge the two solvent traps of the Schlenk manifold in the liquid nitrogen containing dewars. Perform this task slowly, as liquid nitrogen is likely to splash over the sides of the dewar.
7. Allow the traps to cool and ensure the dewars are full of liquid nitrogen. A third dewar with additional liquid nitrogen should be available to refill the solvent trap dewars if necessary. This will be required if you are removing a large volume of solvent. It is helpful to wrap the tops of the dewars with insulating packing material to reduce nitrogen evaporation and water condensation.
8. Turn on the stirring to a medium to rapid pace, this will help reduce bumping (but may not stop it entirely).
9. Once the solvent traps are ready, gently open the flask to vacuum. Do not open rapidly or fully, as this may cause the solution to bump into the tubing. It is generally easier to control the vacuum level via the valve on the flask, rather than the valve on the Schlenk line. (Be cognizant of the volatility of your solvent. Ether will require much finer control of the vacuum than water will).
10. As the solvent evaporates, it will cool down. You may wish to place your Schlenk flask in a room-temperature water or oil bath to keep it from getting too cold.
11. Once most of the solvent is removed, fully open the Schlenk valve to ensure that your compound is fully dried. As the last few drops of solvent evaporate, it is likely your solid with start “popping” and coat the sides of your flasks. This seems to be largely unavoidable.
12. A good rule of thumb is to continue to pull vacuum on your flask until it returns to room temperature. This will ensure most of the trace solvent has been removed.
13. Once all solvent is removed, close the Schlenk valve. If desired, you can backfill the flask with dry N_2 from the Schlenk line.
14. Turn off the vacuum pump and relieve the vacuum on your line.
15. Remove the liquid nitrogen dewars immediately upon breaking vacuum. Be very careful to turn off the vacuum pump before opening the line, as liquid oxygen can condense in your solvent traps. Excess liquid nitrogen can be returned to the storage dewar.
16. Disconnect the solvent traps and allow the solvent to thaw. Dispose of the solvent in the appropriate carboy.
17. There will likely be solvent frozen on the inner glass tubes connected to the Schlenk line. If you have already removed the solvent traps, place a beaker under the manifold to catch the solvent as it thaws.

18. Unless you are working will water, do not try to speed up the thawing process by heating the frozen solvent with a heat gun. This can produce excessive vapors and potentially cause a fire.
19. If you are working with a more viscous solvent, wash the solvent traps with acetone. You can use an air hose to speed up the drying process of the traps. (Do not put wet traps back on the line, as it will destroy your pump oil.)

Trouble shooting tips/comments:

1. If removing large volume of solvent (>100 mL), use the secondary solvent trap, which is placed between your flask and the line. The vessel is cooled in a large dewar and should catch the majority of the solvent. If this solvent trap is used, only one trap on the Schlenk line is necessary.
2. The worst situation that can arise when pulling off solvent is a clog in the solvent traps, resulting in solvent freezing the trap shut. This will happen on the inner tube and may be impossible to spot without removing the liquid nitrogen traps.
    a. The easiest way to spot this situation is when the flask is under vacuum and no longer losing solvent. (Though be careful not to confuse this with losing solvent at a slower rate resulting from a colder flask).
    b. To check if this is occurring, back fill your flask under N_2 and close it off from the line. Then briefly open your line to air.
        i. If it pulls strongly and you notice your vacuum pump change sound, then you do not have a block in your line.
    c. If your line does not draw in air/you hear no change in your vacuum pump, then you have a clog in your trap.
    d. At this point turn off your vacuum. Remove the liquid nitrogen dewars from the traps and vent the vacuum line both before and after the clog. Letting the liquid nitrogen dewars stand could allow oxygen to condense in the trap, which is quite hazardous.
    e. Open up the solvent traps and let the solvent thaw and drain.
    f. Follow steps 16 through 18, rebuild your line, and continue removing solvent.
3. Be aware that a flask under vacuum that is also undergoing temperature changes is at risk for an implosion.
4. For high-boiling solvents, it can help to gently apply heat with the hot plate to aid in the drying process.
5. Be aware of how much solvent your solvent traps can hold and don’t try to evaporate large quantities (>75 mL), without stopping to empty out your traps periodically.
6. To fully dry a compound, you will likely need to pull vacuum on it overnight.
    g. Once your flask appears dry, follow steps 13-19 in the above procedure. At this point, there should be less than 0.1 mL of solvent stuck to an otherwise dry solid, and it should be safe to apply vacuum directly over night without liquid nitrogen cooled solvent traps.