

Section 5.8 Title: Use of Sublimation Apparatuses
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Revision Date: 11/01/19
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:

General protocol for using a sublimator to purify reagents or separate reaction mixtures.

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)
Compounds to be sublimed	Consult relevant SDSs for more details
Dry ice	Frostbite risk
Acetone or isopropanol (for dry ice)	Frostbite risk when cold; flammable and toxic

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

Implosion hazard (reduced pressures)

Exposure Controls: *(Check all that apply)*

PPE: Safety Glasses Face Shield Chemical Splash Goggles
 Chemical Apron Gloves (Nitrile) Lab Coat
 Respirator (type) Other:

Engineering Controls:

Fume Hood Biosafety Cabinet Glove box
 Vented gas cabinet Other: A blast shield should be used when subliming thermally unstable materials.

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: _____

Use of Sublimation Apparatuses

Sublimators have several uses in the Berry Lab. We often use them to remove excess ligand in melt reactions or purify material. Sublimation occurs when a solid becomes a vapor without entering the liquid phase. When we use a sublimator, material in the bottom of the sublimator is heated into the gas phase and then condenses as a solid on a cold finger. What is left on the cold finger is the pure product (or in the case of melt reactions, the excess ligand.)

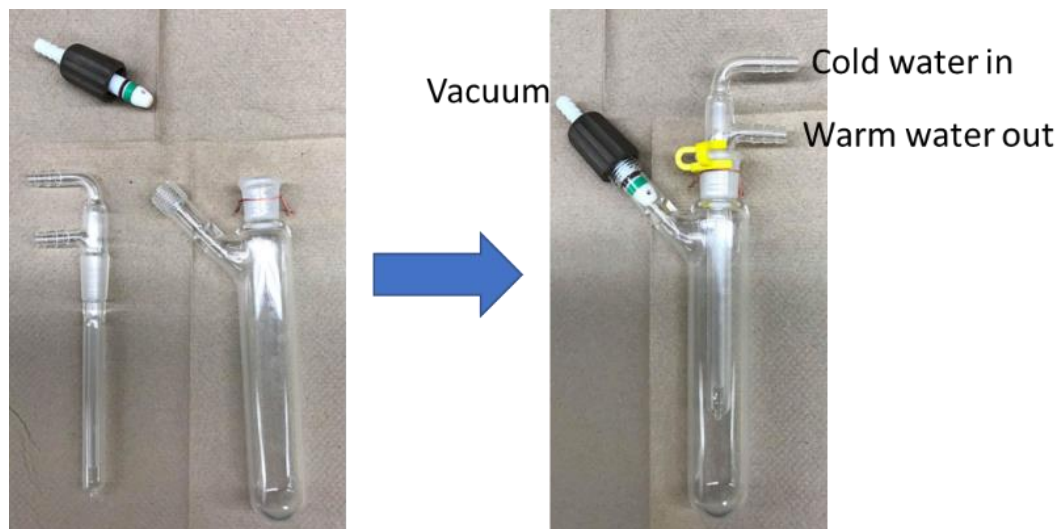
The Berry Labs have four sizes of sublimators. The smallest is a cold finger that you can fit into a Schlenk tube. This size is appropriate for very small volumes of compound. The next two are medium sizes. These are good for hundreds of milligrams to several grams. For large gram scale quantities, we own a large sublimator. Aside from size, the main difference is the cooling mechanisms. Aside from the largest sublimator, which is cooled by dry ice and acetone, the other sublimators are chilled with running water.

The larger of the two medium sublimators is also equipped with a reaction-top. This allows us to do a reaction in the sublimator itself, without the cold finger, and then replace the reaction top with the cold finger for sublimation.

The Smallest Sublimators

The smallest sublimators are effectively cold fingers with 14/20 ground glass joints that fit into Schlenk tubes.

Parts: Cooling finger, Schlenk Tube of your choice, 14/20 clamp, vacuum adapter.

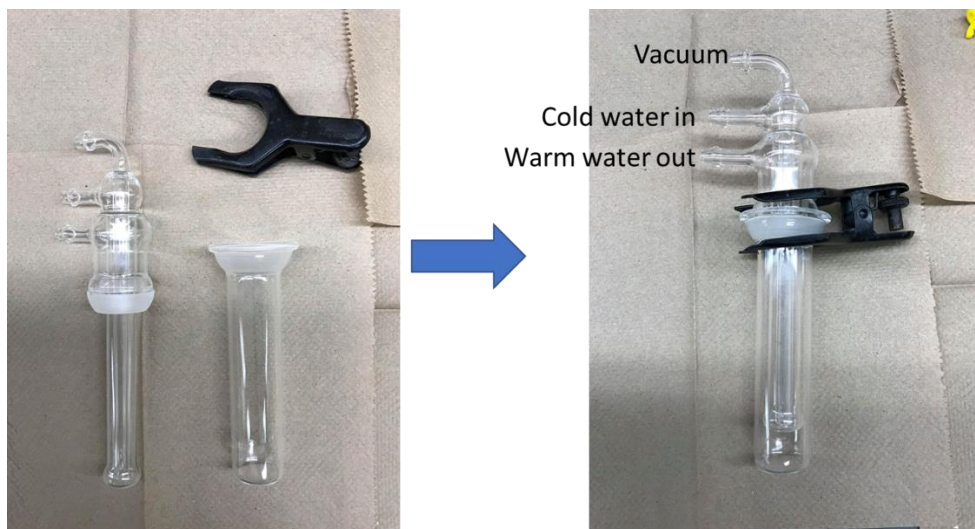


Medium Sublimator Number #1

Cooled by water, ideal for ~100 mg of solid

Parts: Bottom containment vessel, cold finger, metal clamp

Note: The ball-and-socket joint for this sublimator requires vacuum grease to form a seal.

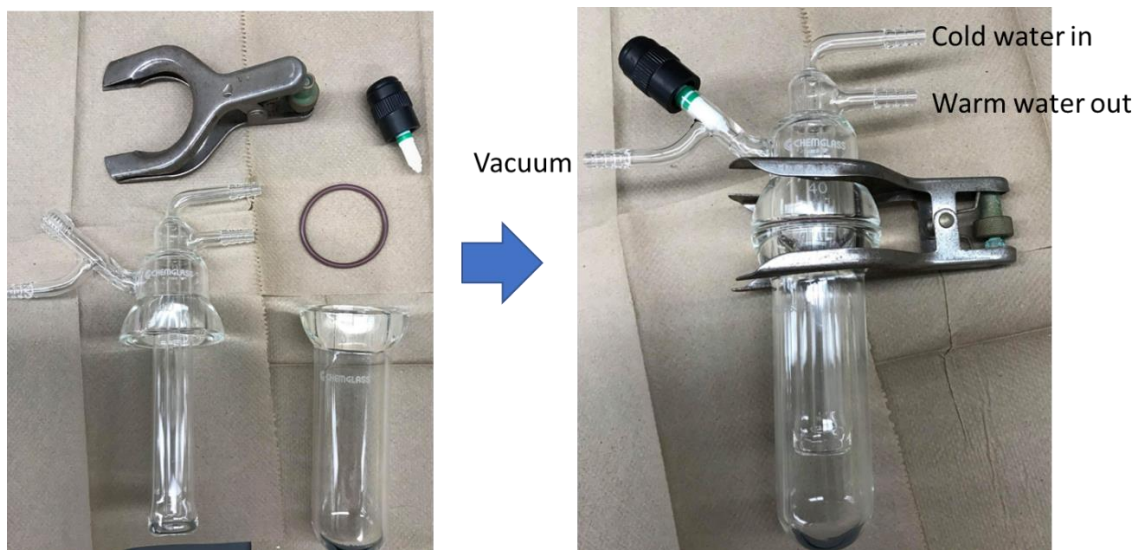


Medium Sublimator Number #2

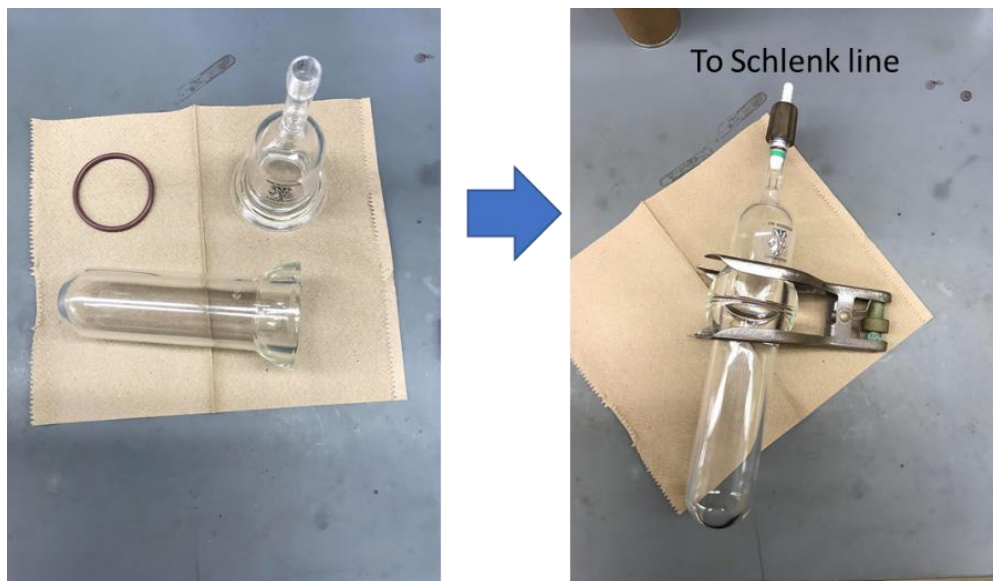
Cooled by water, ideal for 500 mg to 5 g of solid

Parts: Bottom containment vessel, cool finger, O-ring, metal clamp, vacuum adapter.

Note: the metal clamp and O-ring are the same sizes as the ones on our Schlenk line.
Vacuum grease is not needed to seal this sublimator.



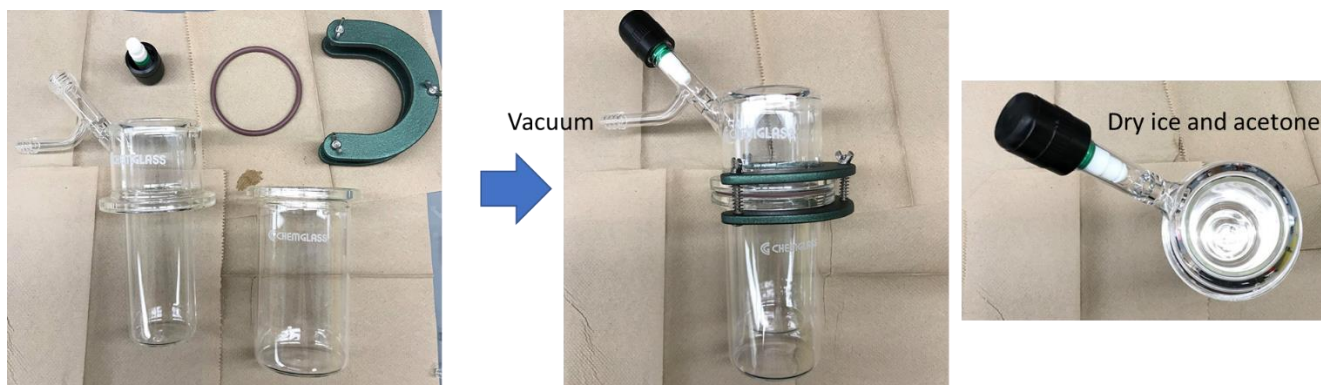
“Reaction Top”



The Large Sublimator

Cooled by dry ice and acetone (or another suitable cooling bath), ideal for gram sized amounts of compound

Parts: Bottom containment vessel, cooling finger, O – ring, green clamp, vacuum adapter.



To Use a Sublimator

1. Select the appropriate size sublimator for your reaction.
2. Add your crude material to the larger bottom portion of the sublimator. It should not touch the cold finger. If it does, the sublimation will not yield clean material. There should be space between your crude material and the cold finger.
3. For the smaller three sublimators, do a test run on the cold finger to ensure water runs through the unit and does not leak. It is easier to fix this now rather than when everything is set up.

4. For the large sublimator, ensure you have added dry ice and acetone. This can be done before or after joining the upper and lower portions of the sublimator.
5. Assemble the top and bottom of the sublimator together. The smaller two sublimators are held together with grease and ground glass. The larger two sublimators are held together with an O-ring. All sublimators should be held together with a clamp. The medium sublimator uses a silver clamp the same size as those on the vacuum tramps. The largest sublimator uses a green half – ring clamp. All sublimator equipment is located in the North – West corner of the lab in a drawer labeled “Sublimators”
6. Do a final check that hoses are secure, and that you can pull vacuum on the sublimator. It is easier to make adjustments now rather than when the sublimator is in the oil bath.
7. Lower your sublimator into the oil bath that should be resting at room temperature. *Never* place a sublimator into a hot oil bath. This is dangerous to the glass. In addition, this often leads to the sublimation of impurities along with the desired product, defeating the purpose.
8. Slowly increase the temperature on the oil bath to your desired temperature. Consult literature to determine the appropriate temperature range and whether to apply active or static vacuum.
9. It is not a good idea to leave a sublimation unattended (with the exception of overnight melt reaction purifications). If a sublimation is left long enough, it is possible to sublime all the contents at the bottom, including impurities, defeating the purpose.
 - a. Note: The largest sublimator needs to be topped off with dry ice. Be extremely careful when doing so as to not get acetone into the hot oil bath.
 - b. Note: When using a sublimator in purifying a melt reaction, pull vacuum for an extended period of time to ensure all ligand is on the cold finger and all of the product is at the bottom of the vessel.
10. When there is little product left at the bottom of the vessel, slowly remove the sublimator from the oil bath and very slowly, open the N₂ to your system. This only applies if the sublimation was performed under static vacuum. If there is any chance your reaction may have evolved gas, do this behind a blast shield.
11. Collect your material from the cold finger and clean all of the sublimator parts.

Example of a Sublimator in Use.

This sublimator is being used to purify ligand off of a melt reaction.

