Section 5.3 **Title:** Using Organic Solvents **Revision Date:** 11/01/19
Prepared By: John F. Berry 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:
Using organic solvents to perform common laboratory techniques.

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.*
N/A

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.*

Non-halogenated organic solvents are disposed of in the white carboys. Halogenated organic solvents are disposed of in the yellow carboys. Solvents contaminated with chemicals not suitable for the carboys (e.g. metal compounds) should be placed in segregated waste containers for disposal through EH&S. Contact the group CHO or disposal officer for questions concerning waste disposal and management.

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: _____

Using Organic Solvents

General Guidelines

Organic solvents can pose potential fire hazards, explosion hazards, and health hazards. Contact with organic solvents should be minimized by the use of the appropriate PPE and a fume hood.

Fire Hazards: Many solvents are highly flammable (halogenated solvents are exceptions). Flammability is generally correlated with the solvent vapor pressure. Note that solvent vapors are heavier than air and can thus spread easily. When working with organic solvents, make note of their autoignition temperatures, the temperatures above which the solvents will spontaneously combust in air. Diethyl ether (160 °C) and carbon disulfide (90 °C) have particularly low autoignition temperatures.

Explosion Hazards: Mixtures of solvent vapors and air pose a potential explosion hazard. After emptying a container of solvent, it is generally a good idea to store the empty vessel upside down; this is why our drying racks orient glassware pointed downwards.

Ethers can form explosive peroxides. These form upon extended exposure to oxygen and light. The peroxides have a higher boiling point than the ether itself and they can therefore accumulate in stills, posing a detonation risk.

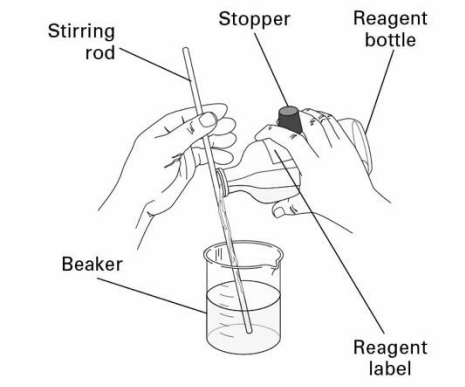
You can test for peroxides by exposing the ether to a mixture of $FeSO_4$ and KNCS. A red color indicates that the peroxide in an ether has oxidized Fe(II) to Fe(III). Peroxide test strips are also stored in the refrigerator.

Health Hazards: General health hazards associated with solvent exposure include toxicity to the nervous system, reproductive damage, liver and kidney damage, respiratory impairment, cancer, and dermatitis.

Acute exposure results from inhalation of large amounts of solvents in a short period of time, which can cause loss of consciousness. Chronic exposure to organic solvents in the work environment can produce a range of adverse effects.

Specific Procedures that Use Organic Solvents

In our group, organic solvents are used to dissolve substances, purification steps, washing, and crystallizations.

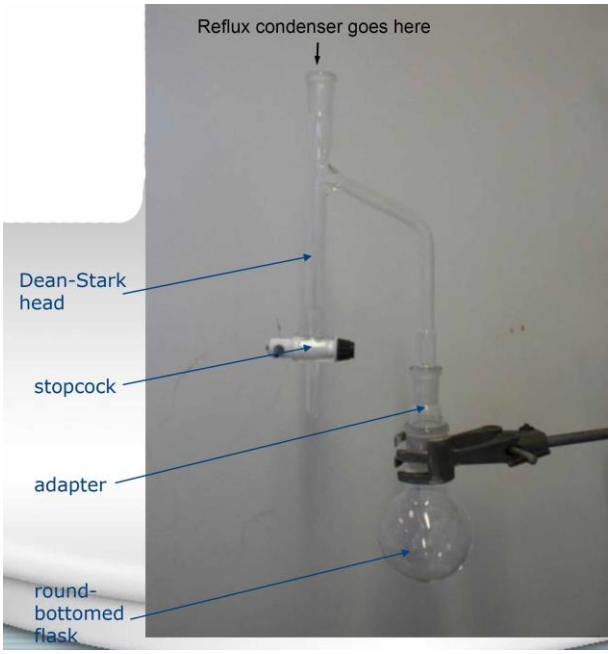


Making Solutions: When dissolving a substance, avoid pouring directly from the solvent bottle into your reaction flask. It is better to use smaller secondary containers. It is often helpful to use a glass rod or pipet to aid in solvent transfer (see figure).

Using Solvents in Purification: One of the most solvent-intensive purification techniques is column chromatography. To avoid spills while running a

column, use a secondary container and a funnel to charge the column with solvent (see figure). Use a test tube rack to collect fractions and analyze the fractions as soon as possible so that you can combine and work up those containing your product without excessive solvent evaporation as the tubes sit in your test tube rack. Try to estimate the correct amount of eluent that you will need to run a column so that you do not prepare an excessive amount.

Another important purification method that uses a lot of solvent is azeotropic distillation. Here, a Dean-Stark trap (see below) is used to separate an immiscible impurity (typically water) from a solution or suspension of your material.



Washing: When you wash a solid, the goal is to extract impurities into the solvent leaving behind a more pure solid product. Proper washing takes time. Stir your solid together with the washing solvent for ~ 30 min before collecting the solid by filtration or decantation.

Crystallization: There are many methods of coaxing crystals of a substance from solution. The method that is most solvent-intensive is liquid/liquid diffusion, in which a solution of a compound is layered with a cosolvent in which the desired compound is not soluble. Layering can be tricky to do. It is mainly based on the difference in density of the two solvents used, but even in cases where the densities differ, it can be difficult to achieve a good layer without causing some of the compound to precipitate right away. Here are some bits of advice on layering:

1. Control the rate of cosolvent addition. A layer can be disturbed if there are major changes in the *rate* of cosolvent addition. It can be difficult to achieve a constant rate of cosolvent addition when adding the cosolvent with a pipette or by syringe. A fail-safe method to control this rate is to add the cosolvent via cannula.
2. You can add a little bit extra solvent (~ 1 mL or so) to your solution before adding the cosolvent. This way, even if a precipitate is formed at the layer, the small crystallites can redissolve in the major solvent as they sink into the flask.
3. You can use solvent miscibility/immiscibility to your advantage. For example, suppose you want to layer an acetonitrile solution with diethyl ether. First add 1 – 2 mL of hexanes, which is immiscible with acetonitrile and will not mix with it. Then, layer the ether on top of the hexanes. The ether will diffuse first through the hexane, and eventually the cosolvent mixture will diffuse into the acetonitrile.