

# How Do I...?

**A manual of Organic Chemistry Laboratory Processes**

**Written for the Honors Organic Chemistry Lab students at**

**The University of Arizona**

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## How do I ....?

### **Preface**

One of the challenges of organic chemistry labs is trying to figure out what techniques to use and when to use them! If you learn how to do a particular technique, such as refluxing a solution, and do not understand why or when it is used, the labs you are doing may not make sense. In other words, without knowing the processes used, the techniques just become things to do.

The purpose of this short manual is to provide you with a way to learn how to use some of the processes used by an Organic Chemist as you need them, hence the title How do I...?. As you begin each experiment, you will be asked to determine the procedures that you will use and to prove whether or not you have obtained the product(s) that you were expecting. How are you going to know how to do this since you probably have not previously used many of the needed techniques? If you know the processes involved, you can learn to recognize when each technique needs to be used.

We hope that you find this manual useful in your exploration of Organic Chemistry.

**Good Luck!**

### **Section 1-Getting Started on an Experiment**

Most experiments can be broken down in to three parts as shown in the diagram to the right.

In Part 1, the Sample preparation/calculation stage, you determine what to do to get your starting materials to react. You might ask yourself questions such as: how many grams of the starting materials do I need, what Molarity are the solutions, how many

milliequivalents of each substance do I need, what form do each of the starting materials need to be in (solid, liquid, gas), do I make solutions of the solids, what are the physical constants (melting point, color, mwt, etc) or solubility behavior of my substances?

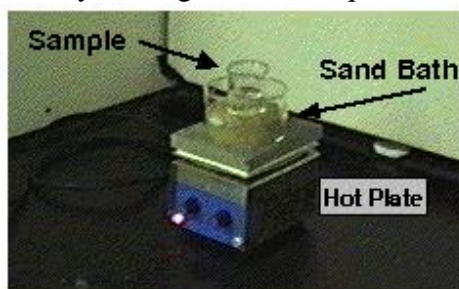
In Part 2, the manipulation stage, you determine what to do with the materials you have assembled for this lab. Do I reflux them for an hour? Do they need to be separated on the basis of their densities or their solubility in water? Do I need to wash the materials at some stage in the reaction? Do I separate by chromatography (flash, or column, or TLC). See the Lab Techniques booklet for specific ideas and help.

In Part 3, the Identification stage, you attempt to characterize your product(s) by selecting appropriate techniques. Some of the techniques are: Infrared or UV/VIS spectroscopy, melting/boiling points, mixed melting points, NMR, chromatographic methods, and physical characteristics.

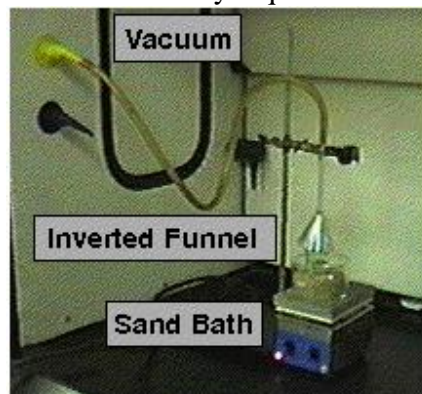
The more thought that you put into the initial design of your experiment, the easier it will be for you to anticipate potential problem areas. This should help you overcome any problems with less worry and aggravation, as well as hopefully learn something interesting!

## Section 2-Setting up the laboratory equipment

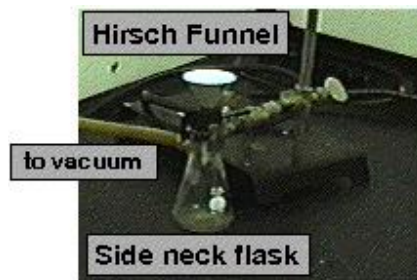
A Sand Bath is used for carefully heating a volatile liquid without using a flame.



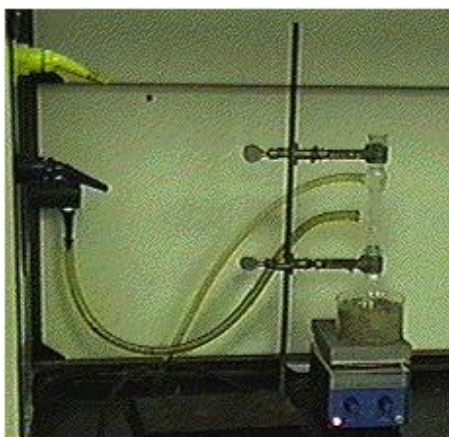
Concentrating a liquid under a vacuum usually requires a sand bath.



Suction filtering requires a Hirsch or Buchner funnel. There is a small piece of filter paper inside the funnel.



Refluxing is used to heat a substance for extended periods of time without having the solvent evaporate.



### Section 3-Calculating quantities to be used

When the amount of reactant required for a reaction is to be calculated you must first determine what mole quantities of the substances are equivalent to each other. This equivalence is determined from the balanced chemical equation for the reaction that you are performing. Once the required ratio of moles has been determined, the quantities that can be measured in the laboratory must be calculated. These quantities are mass or volume. The organic substances that you will be using are usually in one of three forms: a pure solid, a pure liquid, or a solution with a known molarity. The mass or volume can be calculated as follows:

#### Solid

grams to be measured = moles required x molecular weight

#### Liquid

volume = moles needed x molecular weight. / density note: the density of a substance is generally given in grams per milliliter and can be found in the CRC or the Merck Index

#### Solution

volume = moles needed / molarity note: molarity (M) is a concentration unit of moles per liter or millimoles per milliliter.

**Here are a few examples to show what we mean.**

### **Solid**

Determine the mass of p-dichlorobenzene required to have 2.5 mmoles available for use.

### **Liquid**

Determine the volume of a furan solution that contains 0.010 mol of pure furan.

### **Solution**

How many grams of maleic anhydride are needed to prepare a 5.00 M solution of maleic anhydride in 25.0 mL of methylene chloride? Keep in mind that any of the equations used in these examples can be rearranged to solve for a different variable. (For example, solving for molecular weight by using the Liquid equation.)

## **Section 4-Techniques for separating different types of mixtures**

Your most common separations will be separating solids from liquids and liquids from liquids.

For separating liquids from liquids a general rule is that like dissolves like. If the solute is very polar, a very polar solvent will be needed; likewise a nonpolar solute will need a nonpolar solvent. Often two liquids with different polarities can be separated by using a separatory funnel. A micro-pipet may also be used to remove one layer from another.

### **Comparison of some Common Solvents**

After an organic solvent has been used with an aqueous solution, it will be wet. A drying agent, such as anhydrous sodium sulfate, is used to remove water from the organic layer. After adding a spatula tip of the agent, stir or shake occasionally and let set for 10 - 15 minutes. If clumping occurs, it means that you should add more drying agent.

Another technique for separating liquids from liquids is Column Chromatography.

This semester we will be using both solid-liquid and gas-liquid chromatographic methods.

When preparing the solid-liquid column, be sure to place a small plug of glass wool at the bottom of the syringe (or other column), then carefully add the adsorbent (usually as a slurry which is prepared by using as small amount of solvent as possible) in a fashion that

does not allow the slurry to move through the glass wool plug at the end of the column or forming an air pocket in the column.

Distillation is also another technique for separating one liquid from another. The set-up is similar to refluxing..

If you need to separate a Solid from a Liquid, you can first try centrifuging or filtering.

Centrifuging for 2 - 3 minutes. is a good amount of time to start with. Continue to centrifuge until the liquid on top is clear. Remember to balance the centrifuge by placing a centrifuge tube containing a volume of water equal to the volume of liquid being centrifuged on the opposite side of the centrifuge.

When filtering you might use a regular gravity funnel and filter paper or filter by suction using either a Buchner funnel (large surface area) or a Hirsch funnel (small surface area).

### **Section 5-How and When to use Thin Layer Chromatography (TLC)**

What is it? Thin Layer Chromatography is a technique for quickly separating and analyzing a small amount of material. The material travels up the adsorbent on the glass plate. The distance depends upon its solubility in the solvent.

Why use it? TLC is often used to follow a reaction. Spotting at various times throughout the course of the reaction will allow you to determine purity at various times.

When compared to a known sample, TLC can be used to identify an unknown.

How to use it: Spotting the plate. Draw a line about 1 cm above the bottom of the plate. Using a capillary tube which you have dipped into your liquid sample, allow a drop of liquid to spot the plate. Repeating will increase the concentration of the substances being tested. But keep the spot from getting too large.

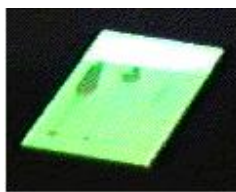


Developing Jar Pour a thin layer your chosen solvent into the jar, stand the plate in the jar and seal the lid.



Leave the jar sealed until the liquid solvent front comes within about 2 cm of the top of the TLC plate.

Visualizing the Spots Shining ultraviolet light on the TLC plate is a good first step. Place your TLC plate under the UV lamp and if spots show up, circle their position on the plate with a pencil.



If this technique does not work, then your Lab Instructor or TA can suggest other techniques for you to try (such as Iodine vapor).

## Section 6-Recrystallization

**Getting Started** When recrystallizing, you may have to initially warm the solution. If you have a volatile liquid, using a sand bath is far safer than an open flame.

When using a sand bath, remember to **TURN ON** the hot plate under the sand bath at the start of class, or about 30 minutes before you really need to use it.

When using a sand bath, it is wise to set the thermostat at the #2 setting so that the sand does not initially become hotter than 50 - 60 °C. If you need it to be hotter, then you can slowly adjust the thermostat setting. A good rule of thumb is to have the sand bath at a temperature that is below that of the substance that has the lowest melting or boiling point.

**Cooling** After warming the substance to dissolve it, you will often need to start the recrystallization by using an ice-bath. The colder the ice-bath the better. An ice-salt mixture or an ice-acetone mixture will produce a liquid mixture with a temperature below 0 °C.

Precipitation For some substances when recrystallization by cooling is used, a small amount of the original solvent will need to be added to the concentrated liquid to start precipitation.

If you have a pure sample, a small crystal can be added to the cooled solution to catalyze the crystallization process.

### **Section 7-Concentrate a liquid solution?**

Why? When the amount of recovered material is small the solution will need to be concentrated before proceeding with TLC or Recrystallization.

How? Three possibilities to use in this course are:

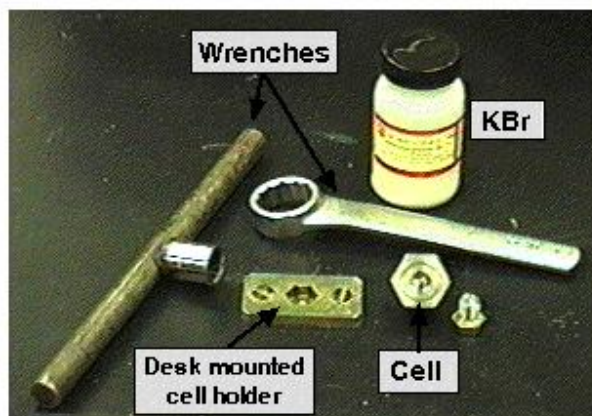
- Evaporation by heating (See Section 2- Sand Bath)
- Evaporation by heating over a vacuum (See Section 2 - Concentrating a liquid under a vacuum)
- Evaporation by using the Rotovap.

This is a quick technique for concentrating larger amounts of liquid. Please check with your Lab Instructor or TA for the availability.



### **Section 8-Setting up to use I.R. or GC or NMR spectroscopy?**

Infrared Spectroscopy. You can prepare your own samples for using the IR. You will need the following materials:

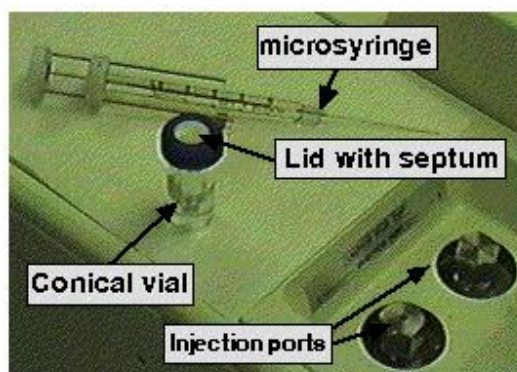


Generally these can be found in your lab, or you can ask the lab. coordinator in the storeroom.

Grind a small amount of KBr and your sample in a mortar and pestle. Place a small amount inside the cell, spread it around evenly and carefully screw in the second bolt until it is seated firmly.

Place the cell in the cell holder and tighten and hold for about 3-4 seconds. Remove both bolts and view your sample. It should be relatively uniform in material distribution.

Gas Chromatography You can also prepare your own samples for using the GC. You will need the following materials:



The conical vial and septum can be found in your locker. The microsyringe is found with the Gas Chromatography machine (or as directed by your lab instructor).

Place your sample of liquid to be tested in the conical vial. You can now draw out the 10 mL of sample that you will inject thru the injection port.

NMR Obtain an NMR sample tube from the storeroom. Place enough sample to fill the tube approximately 1/3 full. Give this tube to your lab instructor.

## Section 9 - Characterizing your compounds

Here are some suggestions for identifying the substances you are using: Solids:

- melting point
- mixed melting point
- Thin Layer Chromatography (TLC)

Liquids:

- gas chromatography
- Thin Layer Chromatography (TLC) (non-volatile substances)
- Column Chromatography

Instrumental Methods:

- Infrared-Helps identify functional groups
- UV/VIS-Compares absorption of light in the ultraviolet and visible wavelengths
- NMR-Helps indicate the number, type, and relative positions of protons in a molecule

The Merck Index will tell you if a substance has an IR, UV/VIS spectra or an NMR

## **Section 10-Finding useful references**

The best and first place to start looking for references is the course Internet Web Page.

Use Netscape or Mosaic\*, connect to the WWW laboratory at <http://mercury.aichem.arizona.edu/lab/>

Netscape and Mosaic are available at all computer labs on campus. If errors are obtained when viewing molecules in exp5, use the information page at <http://mercury.aichem.arizona.edu/lab/viewinfo.html> for help.

The next place to look is the booklet How Do I ...? that you are reading now! This booklet was created by the authors specifically to help you in this course.

Next, look at the Techniques booklet that you purchased. The more lab techniques that you become familiar with, the greater the probability is that you will be able to solve the puzzles of these labs!

These references are helpful:

- Aldrich catalog
- Merck Index
- Reference Librarian