Experiment 0. Introduction To The Organic Laboratory

Reading assignment: ***Techniques in Organic Chemistry*** 2nd ed pages 2-15, 44-46, 116-127.
3rd ed pages 3-13, 41-46, 174-182.

Topics and Techniques

i) Use of Pasteur, plastic and filter pipettes
ii) Approximate volumes
iii) Calibration of a melting point instrument
iv) Melting points and mixed melting points

Introduction

The following preliminary experiments are designed to introduce you to the organic chemistry laboratory. The experiments are relatively easy, and should not take more than a few hours. You must improve upon your pre-laboratory preparation and organization if you need more than 6 hours (two laboratory sessions) for this experiment. Some equipment you may have not seen or used prior to this laboratory course. If you have questions about specific laboratory equipment or the chemistry, make sure you take a few minutes out to ask your TA. Before you come to the laboratory, you need to describe all of the procedures that you plan to do during the course of that days experiment in a laboratory notebook.
During the experiment you must record all of your observations and turn in a carbon copy duplicate of that days work to your TA. Finally, you will complete the write up of your experiment in your lab notebook and turn in your report to your TA. **Your TA will provide you with more information on the design or the format of the pre-lab and lab report write-ups and as well, the format the TA will use to grade your report sometime during the second week**.

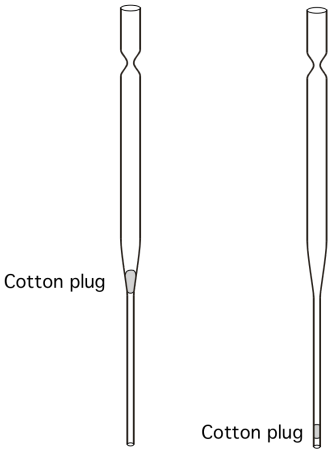
Part A: Proper techniques using Pasteur pipettes and plastic pipettes.

Throughout this laboratory course, you will often handle solvents and samples using the Pasteur pipettes and plastic pipettes. You will be asked to transfer solvents using the correct technique described below. **One of the most frequent accidents in the lab are cuts from the Pasteur pipettes sticking up in the locker**. **Please be careful with the handling and storage of glass pipettes**.

Record the weight of an empty, clean and dry 10 mL graduated cylinder. This is what we call the "tare" weight of the cylinder. Attach a 1 mL latex bulb to the end of a 5 3/4 inch Pasteur pipette and draw water into the pipette. Be careful so you do not draw liquid up into the rubber bulb. Also, it is very important that you keep your Pasteur pipette in a vertical position or you may lose several drops or the entire sample in the pipette. **Your TA will demo the proper technique of using a Pasteur pipette**. Practice drawing and expelling water until you feel comfortable with this technique.
Add 50 drops of water, one drop at a time at, into the graduated cylinder. Record the volume of water transferred and weigh the graduated cylinder with the water in it. Record the difference in the weight of the cylinder containing water and the tare weight of the cylinder. Calculate the average weight of 1 drop of water and the average volume of a drop of water coming out of a 5 3/4 inch Pasteur pipette.
What is the average number of drops in 1 mL of H_2O?
Repeat the exercise with a plastic pipette. Keep these values in mind for you will use these values in later experiments in order to estimate sample or solvent volumes.

Part B: Preparation of filter pipettes.

There are two types of filter pipettes for separating small amounts of liquid from solid particles (for example: a solution in a test tube containing a few crystals). The pipettes are prepared by inserting a small piece of cotton into the pipette. In the first type a plug is placed at the neck of the pipette. To use this pipette solution is added from the top of the pipette. Then solids are trapped inside the pipette while the solution is collected at the bottom. Therefore this type of pipette is usually used when the solids are a waste. In the second type the cotton plug is placed at the tip of the pipette. The pipette is used to suck a solution up the pipette while leaving the solids in the test tube.



Prepare two of each type of filter pipettes. **The TA will demonstrate the preparation of both types of filter pipettes**. The cotton wad should be moderately snug in the pipette, snug enough to where the cotton plug will not float when solvent is drawn into the filter pipette, and not too snug to where the solvent flow is completely restricted. After preparation, rinse the filter pipette with 1 mL of methanol followed by 1 mL of hexane and allow to dry. The filter pipettes will be used in later experiments.

Part C: Determination of a melting point of an unknown compound.

Taking a melting point is very easy and a common method used to establish the qualitative purity and possible identity of an organic compound. A very distinct melting point along with a narrow melting point range provides an indication that the sample is pure. The presence of other organic compounds (impurities), lower the melting point and broaden the melting point range. **Your TA will demonstrate the proper technique in preparing a sample for melting point determination and the appropriate use of the melting point apparatus**.

You will be provided a small amount of an organic substance chosen from the Table 0.1 below. You will not be given the name of the sample, so the substance will be "**unknown**" to you. Prepare two identical samples and determine their melting points. Use a proper rate of heating (1-2 °C/min.) during the final 10 degrees prior to melting. Report the melting point as a melting point range. The melting point ranges should not differ more than 1-2 ºC. You will be taking melting points of "unknowns" several times during chem 143A, so it is important that you become very familiar with the proper technique. Narrow the identity of your unknown down to 2 or 3 possibilities using the observed melting point of your "unknown" and the melting point data given in the Table 0 below.

Table 0.1: Possible Unknowns for Part C

Compound	Melting point (°C)
Benzhydrol	66-68
Phenyl benzoate	68-71
Phenylacetic acid	77-78.5
Vanillin	81-83
o-Anisic acid	100-102
o-Toluic acid	103-105
Fluorene	114-116
Cholesteryl acetate	113-116
Acetanilide	114-116

Part D: Calibration of a melting point instrument.
In order for you to compare melting point data with literature values, the melting point instrument or thermometer must be calibrated. The calibration procedure involves taking a melting point of a known or "standard compound". The difference or "correction" between the observed melting point for the standard compound and the literature value can be applied to your sample or "unknown". This procedure will help assure you that your experimentally observed melting point accurately represents the melting point of your sample or unknown. **In order to shorten this portion of the experiment**, use your melting point data for your unknown in part C above. Now choose and take the melting point of a "standard" (provided in Table 0.2) that has a melting point closest to your unknown. Report both the observed and corrected melting point for your standard and unknown.

Table 0.2: **Standard chemicals for Thermometer Calibration**

Standard Compound	Melting Point (^{o}C)
Benzhydrol	66-68
Vanillin	81-83
Acetanilide	114-116
Benzamide	128-129
Salicylic acid	158-159
Succinic Acid	185-188
Dimethylglyoxime	239-241

Part E: Mixed melting points.
In part C you narrowed the number of possibilities for the identification of your unknown to two or three compounds. Now you will employ the technique of obtaining "mixed melting points" to identify your unknown. The idea behind this procedure is that if two compounds (e.g.; your unknown and a known compound) are "mixed" together and a melting point taken, a change in the melting point temperature and range for the solid mixture will not be observed if the unknown and known are identical compounds. However, if your unknown and a known compound are **not identical**, then the melting

point temperature of the solid mixture will be lower and the melting point range more broad. Mix your "unknown" with one of the known compounds given in Table 0.1 above and report the melting point range. Repeat this process with the same or other "knowns" from Table 0.1 as many times as necessary so as to be confident in your mixed melting point data. Identify the unknown compound that was issued to you from your melting point and mixed melting point data.

NOTE: The Material Safety Data Sheet for each of these compounds is available on the web at:
http://chem-courses.ucsd.edu/Uglabs/