Experiment 1 —

Properties of Organic Compounds

Pre-lab preparation In your notebook, (1) In one or two sentences, state the purpose of this experiment. (2) Draw skeletal structures of all the organic compounds you will be working with in this lab. Include lone pairs and formal charges, if any. A reasonable place to find structures and data for simple organic compounds is Wikipedia. Other good options, especially for less common compounds are www.chemspider.com and chemical suppliers www.sigmaaldrich.com and www.acros.com. Also record the densities, melting points, and boiling points of the compounds whose densities you will be measuring in the first part. (3) Classify the liquids on p 3 according to their functional groups (see the list inside the front cover of Hornback, and Chs 5 and 12). (4) Remember, you will need to turn in a copy of your pre-lab write-up at the beginning of the pre-lab lecture period. Pre-lab lecture meets in Merrill 430; please be on time. Also remember to dress properly for lab — in particular, no shorts or open-toed shoes!

This is a series of experiments that will introduce you to a few common organic lab techniques and properties of simple organic compounds. Be sure to record all data and observations in your notebook as you go. Although you will be working in pairs, remember that data recording and lab write-ups need to be done independently. That includes the pre-lab part — comparing data with your friends *after* you've gathered it up is fine; but everyone needs to do this exercise individually.

Pasteur pipet practice and density measurements. The object of this experiment is to give you a chance to practice using Pasteur pipets and to transfer small amounts of liquids. These pipets are "disposable". It's not worth your time to clean them, so just use them and pitch them, but remember that they must go in the glass waste, *never* in the regular trash! Of course, if a pipet has been used only for a volatile solvent like diethyl ether or dichloromethane, you can reuse it after you're *sure* the solvent has *completely* evaporated. Note that the glass part of the pipet is the disposable bit, *not* the latex bulb! Please don't pitch the bulb into the glass waste.

Attach a latex bulb to a pipet and draw water from a small beaker or Erlenmeyer flask into the pipet. Keep the pipet upright, don't invert it — you don't want the liquid running into the bulb! Practice drawing and expelling water dropwise until you feel comfortable with this technique. Learn to do this with one hand. Professional chemists can make drops in the shape of rings, cubes, or even tiny animals. But if you're not this good just yet, don't get discouraged, just keep practicing.

Organic chemists often estimate small volumes of liquids using pipets. Pipet water in and out of your small graduated cylinder until you have a good idea what 1 ml looks like in a pipet. Also practice drawing up half a ml and 1-1/2 ml.

When greater accuracy is required, a glass syringe with a teflon plunger is normally the organic chemist's weapon of choice, but these are expensive. Plastic syringes are fine for aqueous solutions and some organic solvents, but many solvents will dissolve the plastic barrel or degrade the plunger. Our variant — the best of both worlds — is a little contraption that we'll call a "syringette". We'll use a bit of latex tubing to mate a glass pipet to the calibrated plastic syringe barrel. The barrel calibrations will let you determine volume, but the liquid will only be in contact with the glass pipet. Clever, eh?

Assemble a syringette and practice drawing up and expelling 1 ml of water. You have to be careful not to draw up air and make sure that the seal between the pipet and syringe barrel is secure. How reliable is the volume measurement?

Now use either a simple pipet bulb or the syringette assembly to determine the following: (a) the number of drops in a milliliter and (b) the density of the following solvents: (1) water, (2) diethyl ether, and (3) dichloromethane. Be sure to record the data in your notebook. In a few sentences, briefly discuss how your measured densities compare with the known values. Did you encounter any problems with the two organic solvents? Why?

You can reuse the ether and dichloromethane for the next part, but when you're ready to dispose of these, be sure to dump them in the waste containers in the fume hood. (*Always keep* *your safety goggles on when disposing of waste!)* Be sure to dump all the dichloromethane in "halogenated organic waste"; everything else can go in the main waste container.

Solubility tests — liquids. Combine about ½ ml of each of the following compounds with about ½ ml of water. Note whether each organic compound is miscible with water or insoluble in water (or perhaps somewhere between these extremes, though this may be difficult to determine with such a qualitative technique). If insoluble, which liquid layer is the organic solvent (upper or lower)?

ethyl acetate	hexane	1-butanol	acetone
tert-butyl alcohol	cyclopentanol	tetrahydrofuran	acetonitrile
methanol	diethyl ether	dichloromethane	ethanol
cyclopentanone	1-propanol	dimethylsulfoxide	toluene
1-hexanol	pentyl acetate	1-pentanol	2-butanone

The set of liquids includes a number of alcohols as well as a few members of other compound classes. How do you account for the variation in solubility within the series of alcohols, in terms of molecular structure? Was there a variation in solubility within other groups of compounds containing the same functional group? What structural features are important in making organic compounds soluble in water? (Your answers to these questions should go directly in your notebook — this is in leiu of a formal discussion section.)

Many organic liquids are miscible with each other, but not all are. See how many pairs of mutually insoluble compounds you can find within this set of 20 organic liquids. Now it's important to keep in mind that your time is valuable, so let's not mindlessly plod through all 200 possible combinations. *You get to mix any **6** pairs of solvents, no more.* So choose wisely. There may be a prize for the group who finds the most insoluble pairs, so you may want to avoid drawing attention to your positive results — let the other groups figure it out for themselves.

Think about what structural features might cause mutual insolubility. Are functional groups most important? Polarized bonds? Overall shapes (straight-chain vs branched vs cyclic)?

Water solubility? Halogens? Hmmmm... After you've tested six pairs of solvents and recorded your results, move on.

Solubility tests — solids. For this part of the lab you will be testing the solubilities of four solids — potassium bromide, benzoic acid, ethyl *p*-aminobenzoate, and triphenylmethanol (made and purified by Chem 22 students last spring!) — in five solvents — hexane, diethyl ether, water, 5% aqueous HCl, and 5% aq. NaOH.

Place 20 - 30 mg of KBr into each of 5 small test tubes. These are qualitative tests, so the exact amount of solid isn't important. Weigh out one portion to see what the amount looks like, then just eyeball the rest. Obtain 5 samples of each of the other three solids as well. Add about ¼ ml of solvent, gently shake the tube to encourage the solid to dissolve if it wants to. Although we're mainly trying to distinguish soluble from insoluble, you may find something that's slightly soluble. If you're not sure, you can try adding a little more solvent. You can also find out roughly how much dissolved by placing a drop of the solution on a watch glass and letting the solvent evaporate.

Briefly explain, in terms of the structures of the solutes and solvents, (1) the variation in solubilities among the solvents ether, hexane, and (neutral) water, and (2) the variation in solubilities of some solids in aqueous acid and base, as compared to neutral water. That is, if certain compounds dissolved in acid or in base, what structural feature(s) were responsible for that, and why? If acidity/basicity didn't affect the solubility of some solids, explain why not.

The duplicate copies of your notebook pages containing your data and observations and the answers to the questions in this write-up constitute your lab report — please turn it in to your TA when you're finished. There is no out-of-lab report required for this experiment.