Subject: **Message #1**
From: koreeda@umich.edu
Date: May 3, 2011 9:06:35 AM GMT-04:00
To: 216S11-st@umich.edu **Cc:** 216S11-GSIs@umich.edu

If you are no longer in 216 this spring term, please let me know ASAP. I will remove your address from the e-mail group.

No CTools site for Chem 216.

We have the course web site set up for Chem 216 [http://www.umich.edu/~chem216]
I just posted a number of items to the course web site. But, I will bring copies of those to the class this morning.

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Pre-lab write up will be required starting from Tuesday, 5/10 (2nd half of Experiment 1).
I will post today on the course web site examples of pre-lab guidelines and lab reports.

In addition to those two office hour times shown in the course syllabus, I should generally be available in my office between 10 AM and 8 PM on weekdays. But, it may be wise for you to look up my weekly schedule posted on the course web site and send e-mail to check to see if I am in my office.

I will write about a database search later this week.
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A few safety issues: You should do ALL experiments in the HOOD. Please wear disposable gloves when you handle chemicals. As there are four different sizes (S, M, L, XL), you need to choose the size that fits your hands best. All items contaminated with chemicals such as disposable gloves, paper towels (if you use to wipe off chemicals), pipettes, TLC plates, weighing pans, CAN NOT be disposed into a trashcan. All of these should be put into a solid waste bucket in the hood.
You are not allowed to wear sandals in the lab. If you come in wearing sandals or those that do not cover your toes, you will be sent back home to come back with toe-covered shoes. Also, if possible, please try not to wear shorts. Cover your body as much as you can! Please wear safety goggles as soon as you go into the lab.
Please use a common sense when you take out chemicals from each of the reagent bottles. If you need to weigh 500 mg of aniline on a balance, you don't take out 30 mL of aniline into a beaker. Knowing its gravity, you know it should be less than one mL. Once you take out any chemical from a reagent bottle, it should not be returned back into the bottle.
#Since many of you seem to have difficulty in using those blue or green color-coded pipetters as I do, I suggest you use disposable 1-mL syringes (w/o needles) to take up a small volume of liquid reagents.
In general, it is far more accurate to weigh samples rather than measure out liquid samples by volume. Try to weigh a chemical even if it is a liquid. Also, it is essential that you swirl well the reaction mixture whenever you add a reagent to the solution.

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1. Experiment 1 consists of two parts. For the first half lab this Thursday (5/5), all of you make acetanilide from aniline following the procedure given on page 6 of the lab manual. During the second lab (on Tuesday, 5/10), you will start acetylation of an unknown aniline, i.e., a substituted aniline such as 2,4-dichloroaniline. You will also carry out during the first lab session, recrystallization of the (crude) acetanilide you have prepared. Use hot water; **don't use ethanol for the recrystallization of acetanilide**. First, pour about 15 mL of **HOT** (i.e., boiling) water into a 25-mL Erlenmeyer flask that contains your solid acetanilide product and then keep adding dropwise **HOT** water until all acetanilide is dissolved. Be conservative in adding hot water. Please refresh your memory by reading pp 119-127 of the textbook on recrystallization as well as my one page note on recrystallization. One golden rule about recrystallization: **Don't use a beaker for recrystallization. Use an Erlenmeyer flask** (of an appropriate size, not like a 125-mL flask!). After completely dissolving acetanilide in hot water (maybe a total of 18-20 mL of hot water?), you need to remove any insoluble impurities, including tiny particles, dusts, etc., from this hot solution. This can be achieved by gravity filtration to an Erlenmeyer flask using a glass funnel (use a powder funnel that has a wide stem; glass powder funnels are in a blue-colored box near the blackboard in the lab) and fluted filter paper that covers inside the funnel up to the rim. Hot filtration using a regular glass funnel with a much narrower stem takes a longer time than that using the

aforementioned glass powder funnel. This slow gravity filtration using a regular filter funnel often results in the formation of significant amounts of crystals inside the stem of the filter funnel. If any crystals form inside the funnel or the stem of the funnel (even if you use a powder funnel) during this filtration, you need to use hot (i.e., boiling) water just enough to dissolve those crystals down into the solution. This filtration process should be done while the whole apparatus is kept hot.

This clear, hot filtrate solution thus obtained then needs to be cooled down to room temperature. The Erlenmeyer flask that contains this hot solution should be covered/capped (use a cork wrapped by a glassine or weighing paper) so that none of the hot solvent will evaporate. Please make sure to cool the hot, saturated solution **slowly** down to room temperature (it will take at least **20 minutes** or so) so that you can induce "natural" crystallization (not forcing crystals to come out from the solution by immediately dipping the flask in an ice bath). The crystals thus obtained should be collected by **suction filtratio**n with a Hirsch funnel and a tiny piece of round filter paper that covers the inside bottom of the funnel. Don't use a Buchner funnel in this case, as you don't have that much sample. The filter paper needs to be made wet with the solvent used for recrystallization (in this case water) and then vacuum should be applied. Make sure that the filter paper is firmly adhered to the inside bottom of the Hirsch funnel. If the filter paper is not made wet with the solvent and firmly held at the bottom of the Hirsch funnel by applying vacuum, the filter paper will float up to the top inside the funnel when you pour the crystal-containing solution, thus letting precious purified crystals go through the funnel into the filter flask.

2. Check the purity of your acetanilide by TLC analysis (see my note on TLC and read pp 165-172 of the textbook). You should check against aniline. **Dissolve a tiny, tiny drop of aniline in 1-2 mL of dichloromethane** (or methylene chloride) and spot a small volume of this solution using a capillary on a TLC plate. Do not try to spot aniline itself. That would be way, way too much. You only need microgram (not milligram) quantities of a sample for TLC. For the rest of the term, you are expected to check TLC of a reaction product whenever you run a reaction, even if the lab manual may not say so. This also applies to the IR spectra.

Then, take IR spectra of both aniline and acetanilide and compare the major differences in the two spectra. Please check with your GSI to see if the quality of your IR spectrum is acceptable or not.

3. You will be provided with a vial that contains an unknown aniline during the lab session on Thursday, May 6. Make sure to write down in your lab notebook **your unknown sample number**. If your unknown aniline is a solid, take its melting point. If you have time during the first lab session, run an IR spectrum of your unknown aniline.

On Tuesday, May 11, please go to the blackboard and jot down observations on your unknown aniline (color, smell, solid (mp) or liquid, etc.) and see if someone else in your lab section might have the same compound as you do. If someone else seems to have the same unknown aniline, go ahead to confirm if you two have the same compound or not. Think about the way(s) of achieving such a goal.....

The IR spec of most of these unknown aniline compounds can be found in "The Aldrich Library of FT IR Spectra" (see page 9 of the lab manual about these books). Alternatively, you might find the IR spec on the following web site.

SDBS site: http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng

As soon as you open this web site and agreeing with whatever they want you to, you will get a screen that says "SDBS Compounds and Spectral Search." I suggest you type in molecular formula of your compound (i.e., you unknown aniline, not its acetylated derivative) rather than search by the name of a compound (you just type in like C6H7N w/o using subscript, etc.; CxHy first, followed by other atoms in an alphabetical order). You may often find more than 10 hits, then open the list of these compounds and find the one you want by guessing the structure from their names. Then, click on the compound you want, leading to the list of available spectroscopic data.

If you compare carefully the finger print region (1433 - 500 cm^{-1}) of the two IR spectra (i.e., yours and the one from SDBS), you can easily conclude if those two are identical or not.

I will talk about TLC, IR, and pKa today in the afternoon.

Masato