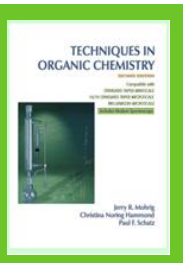
Chem 6A



AN INTRODUCTION TO ORGANIC LAB

TECHNIQUES, COLLIGATIVE PROPERTIES, & SPECTROSCOPY

Course Syllabus

1. TA responsibilities.

Safety: For safety, TAs permit a maximum of eighteen students per section and require their students to wear lab goggles and closed-toed-shoes.

Instruction and score: For each experiment, TAs give a brief review of the theory and procedure for the daily activities, give a quiz, grade the pre-lab, and in-lab parts, and assign technique grades as necessary. After each experiment, TAs will request a photocopy of the entire experiment (pre-lab, in-lab and post-lab write-up). TAs are to complete their grading prior to the next lab so students have adequate feedback. During each experiment, TAs may assign up to 5 points for technique. All TAs will use the same spreadsheet to track their sections. Each grade sheet will be normalized to a similar average and letter grade spread.

Absence and make-up: TAs do not have the authority to cancel class or permit a student to skip a lab. If a student does not attend, TAs should inform them to contact the instructor immediately with their reason. There are NO Make-Up Labs. If valid (doctor's note, family emergency) a pass will be given for the experiment and quiz that week. Only one pass will be granted per quarter. If you miss more than one experiment due to emergency, it is best to file for an incomplete or retroactive withdrawal than make-up content of the course. 3 absences result in a failing grade.

2. General procedures for students in each lab. Each class begins with a 10 minute quiz in which the questions come from a quiz bank online. The TAs will give a ~15 minute overview of the experiment, including the theory behind the practice. While you are taking the quiz, the TA will grade the pre-lab of your notebook in red ink, assigning a score with the date and their initials. Before leaving lab, your TA will again initial the in-lab portion of your notebook to verify your record of the experimental procedure and observations. You should work **independently** on ALL the experiments unless directed.

Course Outline

EIGHT EXPERIMENTS

Exp. 1 Density and Melting Point

Exp. 2 Distillation

Exp. 3 Extraction

Exp. 4 Isolation and Sublimation of Caffeine

Exp. 5 (Rxn 1: photoisomerization, TLC)

Exp. 6 (Rxn 2: E1 dehydration, GC)

Exp. 7 (Rxn 3: carbonyl reduction, IR)

Exp. 8 (Rxn 4: ether synthesis, MS)

Spring 2009

Instructor: Justin A. Russak jrussak@chem.ucsb.edu

Timeline

6A labs are on Tuesdays, Thursdays, and Fridays. Where applicable, each section will perform the same experiment the same week unless holidays interfere.

Week 1

Lecture 1: March 30th 2009 5pm
Check in and Lab 1

Week 2

Lecture 2
Lab 2

Week 3

Lecture 3
Lab 3

Week 4

Lecture 4
Lab 4

Week 5

Lecture 5
Lab 5

Week 6

Lecture 6
Lab 6

Week 7

Lecture 7
Lab 7

Week 8

Lecture 8
Lab 8 and Checkout

Week 9

Lecture 9
No Lab

Week 10

No Lecture or Lab

Final EXAM

Tuesday 9 June 2009
7:30-10:30 pm

3. Lab Reports.
General: The laboratory notebook is a record of all work performed in the laboratory. It is a legal document that gives testimony of the performed work. *It should be concise, written clearly and neatly so as to allow a future experimentalist to reproduce the experiment.* Your ability to keep a good notebook can also affect your technique grades.

Notebook: Students should use a non-perforated notebook. Do *not use* a spiral/loose-leaf notebook, and *never remove* pages from your notebook. The notebook should begin with a **table of contents** (see page 3 marginal note), including the title of every experiment and the corresponding page number, listed in sequential order. All entries should be written **in black ink**, whether done in advance of the experiment or while making observations. You should *never type* reports. All **pages** are to be numbered sequentially. The lab record (pre-, in- and post-labs) is written on only **right hand pages** of the notebook. This leaves the left-hand pages for notes and extraneous calculations.

Contents: Reports consist of three components. The pre-lab **(Part I)** is completed before coming to lab. During lab, TA lecture notes are then written into the notebook (left-side) and the actual in-lab procedure **(Part 2a)** is completed on the right-side. The post-lab experimental analysis **(Part 2b)** is completed after the experiment is done.
Part I will be initialed, dated and graded by the TA at the beginning of each experiment.

Part 2a is written during the lab. It is comprised of the actual experimental procedure and experimental observations. Write what you are doing or observing, right after you

Grading

Notebooks [25%]
part I (30 pts)
part II (70 pts)

Technique [5%]
0-5 points per experiment

Lab Quizzes [10%]

Midterms [20%]
(2 midterms at 10% each)

Final Exam [40%]

Curved Course
Average set to B- / B break

do/see it so you don't forget. Sometimes drawing a picture is effective, especially when you perform TLC analysis. Before leaving lab you must get your TAs initials to verify you have composed an experimental procedure and have maintained experimental observations during lab.

Part 2b, the final post lab write-up, is written after lab.

Submission: ***<lab report for each lab>*** A photocopy of **all parts of the experiment,** including spectra when applicable, are submitted to the TAs box for grading. TA boxes are located next to the chemistry stock room. Reports will be due by Friday for students who have lab on Tuesday, by Sunday if your lab is on Thursday, and by Monday if your lab is on Friday. *Late work will be deducted 10% for each day it is late.* Students that fail to turn in 3 lab reports will automatically fail the course.

Detail of notebook preparation:

---Pre-Lab [Part 1]--- (30%) : need to be done before the beginning of lab

A. Title, date, name, student perm number and TA overseeing the experiment

B. Purpose/Objective

Give a brief introduction to the purpose of the experiment and the approach to be used. Demonstrate that you understand the objective and the key concepts of the experiment. **Do not copy directly from the laboratory manual.** Usually, one paragraph will be adequate (less than 1/2 a page). Use only the **third person**, **present tense**, **passive voice** when writing the introduction. For example, using third person, do not say I, we, or our. Speak as if it is happening right now (present tense), but use a passive voice for your verbs.

Incorrect Example: "I am preparing cyclohexanol from cyclohexanone by hydride reduction."

Correct Example: "Cyclohexanol is prepared from cyclohexanone by hydride reduction."

Notice the correct example says "is prepared" which uses the present tense and passive voice. It is implied that you are the one performing the experiment so it is redundant to speak in 1st person, which is why you don't see any grammatical person in the correct example.

C. Reaction Diagram (Labs 5-8)

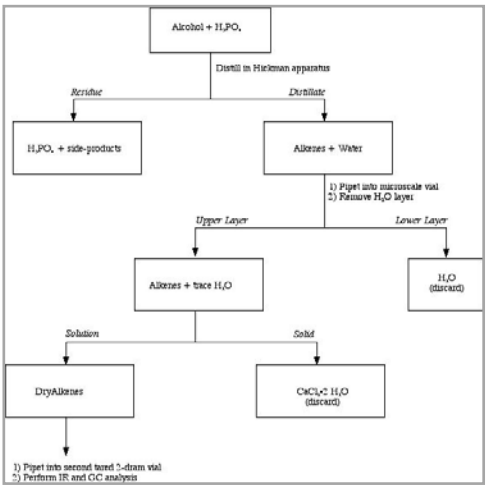
This is the balanced, fully labeled chemical equation. Each structure should have its chemical name underneath. Usually, conditions like temperature and the solvent used are indicated above or below the reaction arrow.

D. Table of Reagents and Products (All labs)

A table of reagents needs to be **completed** before lab starts. Construct a table similar to the one provided in each experiment. Any theoretical value needed to calculate your answers or to compare your results needs to be present. Of course, this is going to require you to read the lab and figure out what exactly you need. For example, in Experiment 1, you will measure the density of given liquids. It is imperative that you find these density values and put them in your table of reagents prior to the lab for your comparison. In lab comparison can greatly avoid making an error.

E. Intended Procedure with Flow Chart (Labs 3-8)

Demonstrate that you are prepared for lab by giving a brief description of what you actually intend to do in lab experimentally; a "game plan" or checklist written in your own words. This can be written in paragraph form but preferred as a bulleted list. Do not copy directly from the laboratory manual. In addition to this, prepare a flow chart (see example). The flow chart is helpful in understanding the exact procedure of the lab. Notice that it begins with all reagents used and branches when you perform purification. Ultimately, the pure product should be at the end of the road and each branch that leads to a dead end is most likely going to be disposed along the way. You need a flow chart for labs 3-8.



Composition Notebook

Front of Notebook:

Name
Perm #
Spring 2009
Monday 8:00 am-12:00 pm
TAs Name

Table of Contents
(leave a few pages for this at the beginning)

Experiment 1 page XX
Density, melting point and refractive index
..... Quiz Score (0-100)
..... Pre-Lab (Part I) Score (0-30)
..... In- and Post-Labs Score (0-70)
..... Total Score (0-100)
..... Technique Score (0-10)

Experiment 2 page XX
Separation of water and ethanol (simple and fractional distillation)
..... Quiz Score (0-100)
..... Pre-Lab (Part I) Score (0-30)
..... In- and Post-Labs Score (0-70)
..... Total Score (0-100)
..... Technique Score (0-10)

Experiment 3 page XX
Acid, base and neutral separation (liquid-liquid extraction and drying)
..... Quiz Score (0-100)
..... Pre-Lab (Part I) Score (0-30)
..... In- and Post-Labs Score (0-70)
..... Total Score (0-100)
..... Technique Score (0-10)

---In-Lab-[Part 2a]-- (20%)

A. Actual Procedure and Observations

This is an account of what really was done. Do not regurgitate the laboratory manual. You need to write the procedure as it was exactly carried out. If the procedure has been modified or changed in any way from the original experiment, note the changes here. Remember that the procedure section should be sufficiently detailed, such that another student would be able to repeat the whole experiment based on your report. Keep the following points in mind:

(i) Use the **third person**, the **passive voice**, and the **past tense**. In this case, you have just performed it, so you need to use passive voice and past tense.

Correct: The solution was heated on a hot-plate for 30 minutes.

Incorrect: I heated the solution on a hot-plate for 30 minutes.

Incorrect: The solution is heated on a hot plate for 30 minutes.

(ii) Avoid the "recipe format". Recipes are written in a command tense, telling you to do something. This is not the way you should be writing your observations.

Incorrect: Heat the solution on a hot-plate for 30 minutes.

(iii) Incorporate your **observations** into the procedure.

(ex) The solution was heated on a hot-plate for 30 minutes during which time the color of the solution changed from red to green.

(iv) Observations should be **concisely** written. Avoid unnecessary detail.

(ex) The reaction mixture turned green and a precipitate formed. The precipitate was collected using a Buchner funnel and washed with water. The crude product, in the form of yellow crystals, had a mass of 15mg.

---Post-Lab-[Part 2b]-- (50%)

A. Results:

This is one of the most important sections of your report. Whenever possible, tabulate your data, such as the melting/boiling point with its range. Analyze the IR and/or NMR spectra, and any other observations or measurements. Include all the spectra, which will be provided on the course website, with your interpretations and peak assignments. Show clearly how you performed any calculations.

B. Discussion

This is the most important section of your report. This section should be completely based on your results (measured or calculated values) and observations. The values you recorded are your data and you need to consider the meaning of your data. You also need to show your understanding of the experiment. The discussion is written in the **passive voice** and **past tense** just like your observations. Often, you will need to cite references where you obtained any supplementary information.

First, your discussion should state what you've made and what it appears like. Was it as expected compared to the literature? e.g. white shiny crystalline solid.

Next, discuss the yield and purity of the product(s) you recovered/synthesized. Qualitatively assess the performance. A discussion should quote actual experimental values and not talk in vague terms.

Correct: The product obtained was found to be fairly pure as it had a mp of 110-112° C, a mp range of only 2° C. This result was 3 degrees below the literature value of 115 °C for 'compound X'. Because the observed mp is depressed in comparison to the literature mp, this shows that the product may not be completely pure.

Correct: The infrared spectrum of the alkene product (refer to page xx of your report) had the absorption bands of the expected alkene, 3050 cm^{-1} sp^2 C-H stretch and 1650 cm^{-1} C=C sharp absorption. No broad alcohol band was observed at 3300 cm^{-1}, indicating no reagent alcohol remains and that the reaction resulted in the conversion of the alcohol to the alkene product.

Incorrect: The product obtained was found to be pure. Data interpretation should demonstrate a clear understanding of the technique, the experiment, and the spectra.

Then, discuss any sources of error. Try to think of at least two sources of each. Sources of error include theoretical sources such as the reaction did not go to 100% completion, or practical sources such as the instrument or glassware used was not calibrated. Use this procedure for discussing error. First state the error: "The sample bottle indicated a 90/10 mixture by mass of urea to cinnamic acid. It is difficult to estimate that the actual portion of sample that was taken from this bottle for the experiment was the same as that listed on the bottle. It is not known how well the sample was mixed prior to the lab. If not mixed well, a different ratio could be represented in the small portion taken from the bottle." This clearly says what the possible error is. Then, state **exactly** how it could affect the results: "If the sample bottle was not correct, then the data plotted for melting point depression of each sample would be construed." If you can directly show proof that the error you postulated is consistent with your experimental observations, do so. Now, state how the problem could be addressed: "In order to prevent any possibility that the sample bottle differed from the actual sample taken, each pure chemical could be obtained and mixed in the exact mass ratio needed for the sample. Since the melting point sample is so small, this is not a likely answer. The only possible solution would be to vigorously mix the bottle prior to taking a sample to ensure adequate mixing of the solids."

Finally, mention at least one way to improve the experiment.

C. Witness

Give your signature and a pledge that all of the observations and conclusion herein are your own and that you believe them to be correct. Then have another person witness your pledge with their signature.

How to write a Post-Lab

Results: Label and title all attached flowcharts, spectra, graphs, etc… The results section is where you tabulate your data (save analysis for the discussion). You will list the data you collect with the appropriate units. You will report any calculated answers (% yield, % error, etc…). Please include calculations or, if there are many of the same, provide a sample calculation. Comment on IR spectra when applicable and select peaks of importance. Show sample calculations. Remember, there is a difference between **% recovery yield** calculations and **% Yield** calculations. In the latter, you must determine **limiting reagents** and a **theoretical yield**.

Discussion: an example

A clear colorless liquid with a slight alcohol odor, corrected bp 196-201 °C, and refractive index of 1.5262 (at 20 °C), was obtained from the reaction of…[also draw and name structure of product here]…

The yield of 1-phenylethanol was 13.2 g of clear, colorless liquid, and the % yield was 56%. The theoretical yield for the reaction was calculated to be 23.57 g, but this assumes that all the limiting reagent (acetophenone) reacted and that no byproducts formed (styrene). Thus, this was a fairly good yield for this reaction, which normally gives yields of product around 85% (ref: textbook pp#). The product appeared to be pure. According to the CRC Handbook the product should be a clear, colorless liquid, with a bp of 203 °C. The product obtained was clear and colorless with a (barometric pressure corrected) bp of 195-201 °C. The boiling point of the product was 2 °C below the literature value, indicating some impurity and/or error, and boiled over a range of 6 °C, which definitely means some impurities are still present. The refractive index of the product was 0.0010 below the literature value of 1.5272, indicating again that some slight impurities were present. The infrared spectrum for the product showed good purity. All the signals for an aromatic/aliphatic alcohol were present; O-H stretch @ 3350 cm^{-1}, aromatic C-H stretch @ 3080 cm^{-1} and alkane C-H stretch @ 2850-2950 cm^{-1}, C=C stretching @ 1600, 1500 and 1450 cm^{-1}, and C-O stretch for a alcohol @ 1077 cm^{-1}. No bands due to reasonable impurities were observed in the infrared spectrum.

The boiling point of the product was 2 °C below the literature value, however an uncalibrated thermometer was used to take this reading. This may account for why the temperature reading was low, but does not explain why the product boiled over a range of 6 °C. The refractometer used in this experiment was uncalibrated. This is a practical source of error for the experiment. And might partly account for why the RI was 0.0010 below the literature value of 1.5272.

Discussion of error:…

Witness: (an example)

To the best of my knowledge all of the conclusions and observations herein are true [signed], [witnessed], dated.

D. References:

You should reference any literature used in your report, i.e. melting points, spectral data, etc. Use an acceptable scientific journal style/format for your references. Be consistent.

4. Technique Score. During each experiment, technique scores will be assigned, worth 0-5 points. The technique score can be very subjective. Your TA can give you a low score if you are wearing inappropriate clothing, have a messy area, use incorrect disposal techniques, are extremely inefficient in lab, have an unorganized, unkempt notebook, are not wearing your safety goggles, or are unprepared for lab. Failure to listen, learn, and comply will be reflected in your technique grade. Your TA can give you an outstanding score if you clean up a dirty area such as the balance table, ask good questions, have a very neat, organized notebook, or are efficient during lab. TAs should strive to have everyone finish the lab course with an average technique score. Therefore, if you receive a low score, you should make use of opportunities to zero it out with a high score.

5. Grading. (see page 2 marginal note for course breakdown) Grades are based on technique; in class quizzes; notebooks; midterms; and the final exam. *TAs will be entering eight quiz scores, eight notebook scores (total of Part 1+ Part 2a/b), and eight technique scores.* There is no extra credit and TAs *do not* have the ability to assign extra work. All TAs will use the same grading schemes and the same Excel® spread sheet. The grade sheet is emailed to the instructor every week and posted on the Grade Checker website. See the Chem Dept website for the link. **Reports that are more than 10 days late or turned in after dead week will not be accepted.** The course is curved at the end of the course…not after every exam. Each TAs sections will be normalized to account for any differences in the grading. So, students with more difficult TAs will get more of a curve than those with easy TAs. This serves to minimize any unnecessary grade loss for a student who does comparable just because of the way the TA graded. Basically, the average of each TAs students will be compared to the class average and the difference added (if TAs average is below class average) or subtracted (if TAs average is above). The class average is set to the B/B- break.

To give students an idea of how they are doing in the class along the way, a curve will be applied on the current work performed each week and can be viewed on Grade Checker. It should be remembered that the curve will be applied at the end of the course, and the curve applied along the way is only an indicator of the grade at that present time.

6. Laboratory Safety. Please read Chapter 1 of your text regarding safety. Dealing with chemicals is a serious responsibility and should be taken as so. You must always wear safety goggles while in the lab. Even though you may not be performing an experiment at the time, someone else may be and there is that potential for an accident. Closed toed shoes are the only appropriate footwear for the lab. You will not be allowed to do the lab without them. A cotton lab coat is not required but highly advised. Glass waste goes in a separate container, not the trash. Needles go into a separate container, not the trash. Gloves are available, but you may want to purchase some rubber dishwashing gloves that can be reused and stored in your drawer. If you have long hair you should pull it back so it is not in the way of anything dangerous. Safety showers and eye washes are available in the case of accidents. The eye wash fountains at the front of the lab are for flushing the eyes with water after an accident. Follow your TAs instructions at all times regarding safety.

7. Rules for the Disposal of Reaction Wastes. We must be conscience of our environment and never dispose of hazardous waste improperly. Always put waste in the proper container and ask your TA if you are unsure what to do.

8. Laboratory Instruments: There are many expensive pieces of equipment used in the organic laboratory. Please use them properly and seek help if you are not sure what to do.

Get a pair of safety goggles

You can purchase a pair of rubber dishwashing gloves to use in the lab or use disposable ones from the stockroom.