

# SPRING 2008 SYLLABUS

## CHEM 344 ORGANIC II HONORS LABORATORY: SYNTHESIS AND CHARACTERIZATION

Senior Instructor: Dr. Christine V. Rich  
Room 307, Chemistry Bldg  
Phone: 852-7814  
E-mail: christine.rich@louisville.edu  
Office Hours: Monday 1 pm or by appt.

Text: *The Organic Chem Lab Survival Manual*, any edition  
Author: James W. Zubrick  
Packets from Chem. Stockroom; LL38

Your very capable Teaching Assistants this semester are:

Section 04 Thursday at 9:30: Phil Stoller ([pmstol01@louisville.edu](mailto:pmstol01@louisville.edu))  
Section 05 Friday at 12:00: Rebecca Bottom ([rlbott02@louisville.edu](mailto:rlbott02@louisville.edu))  
Section 07 Friday at 1:00: Wes Badger ([wrbadg01@louisville.edu](mailto:wrbadg01@louisville.edu))  
Section 08 Thursday at 8:30: Carol Patel ([ckpate01@louisville.edu](mailto:ckpate01@louisville.edu))

They will provide you with their office hours and other necessary information in the first lab session. Feel free to contact any one of them for help/questions. I'm confident they all know their organic chemistry. However, I would suggest that any grading questions be directed to your TA. If you have a complaint about the way your lab reports are graded, don't hassle the TA...come directly to me.

The tentative schedule of experiments is shown below. I may rearrange the ordering of the experiments if doing so better aligns the session with your lecture progress. Notice that there are no assigned readings this time since most of the bench techniques are already familiar to you. When you read over the experiment, be sure to consult Zubrick on your own if you need a "refresher". \*\*Don't forget to answer any questions posed in the packet in your final reports.

### SESSION

### EXPERIMENT

Thurs, Jan 10 Fri, Jan 11	Check-in; Safety Instruction; Lab protocol; pre-lab lecture for the Redox Sequence
Thurs, Jan 17 Fri, Jan 18 <b>and</b> & Thurs, Jan 24 Fri, Jan 25	<u>An Organic Redox Sequence</u> : (1S)-[endo]-borneol to (1S)-camphor to diastereomeric(1S)-isoborneol: Microscale synthesis and purification; IR characterizations and <sup>1</sup> H NMR determination of diastereomeric product distribution. (Packet procedure adapted by Dr. K Grant Taylor from Pavia, Lampman, Kriz & Engel). 200 pts
Thurs, Jan 31 Fri, Feb 1	<u>Electrophilic Aromatic Substitution</u> : Nitration of p-dichlorobenzene. Microscale synthesis and purification; tlc, mp, and IR characterization. (Packet procedure adapted from Mayo, Pike, and Trumper). 100 pts
Thurs, Feb 7 Fri, Feb 8	<u>Electrophilic Aromatic Substitution II</u> : Microscale Bromination of Acetanilide. Which isomer predominates? Characterization by tlc, mp, and <sup>1</sup> H NMR spectroscopy. Using splitting patterns to determine structural features. (Packet procedure adapted from Palleros and Schoffstall, Gaddis, and Druelinger). <i>Save your product for a multi-step sequence later in the semester.</i> 100 pts
Thurs, Feb 14 Fri, Feb 15	<u>A Substitution Puzzle in Electrophilic Aromatic Substitution</u> : Using <sup>1</sup> H NMR chemical shift calculations to determine site of substitution. Miniscale synthesis and purification of N-acetyl-4-ethoxy-3-nitroaniline from phenacetin. <sup>1</sup> H NMR determination of structure by comparison of spectral pattern with chemical shift calculations. (Packet procedure adapted from Palleros). 100 pts
Thurs, Feb 21	<u>A Stereoselective Wittig reaction</u> : Microscale synthesis and purification of 9- (2-

- Fri, Feb 22 phenylethenyl)-anthracene. Is it *cis* or *trans*? Using  $^1\text{H}$  NMR coupling constants to determine stereochemistry. (Packet procedure adapted from Schoffstall, Gaddis, Druelinger). 100 pts
- Thurs, Feb 28 Grignard Synthesis of Malachite Green: Microscale synthesis of triphenylmethane dye requiring anhydrous conditions. (Packet procedure adapted from Mohrig, Hammond, Morrill, and Neckers). 100 pts
- Thurs, March 6 What was in that aldol?: Crossed aldol reaction from two unknowns; determination of starting materials and product structure from spectroscopic data and mp.

### **Spring Break March 10<sup>th</sup> thru 16<sup>th</sup>. Enjoy! Sleep! Tan! Whatever!**

- Thurs, March 20 The Suzuki Coupling Reaction: 21<sup>st</sup> Century Chemistry Microscale synthesis, chromatographic purification and NMR characterization of the Pd-catalyzed Suzuki coupling of *p*-bromoacetanilide with phenylboronic acid.
- Fri, March 21 and Thurs, March 27 Microscale column chromatography revisited and  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR in structural analysis. Researching reactions in the literature. (Procedure developed by Devin Pantess and Christine Rich). 200 pts
- Fri, March 28
- Thurs, April 3 Microscale Asymmetric Synthesis: Enzyme-catalyzed Reduction of 1-phenyl-1,2-propanedione. Simultaneous or sequential reduction pathway?
- Fri, April 4 and Thurs, April 10 Conformational analysis using  $^1\text{H}$  NMR: Is this the *erythro* or *threo* product?
- April 11 Karplus curve correlations in determining stereochemistry. Finishing up the enzymatic reduction; Scheduled NMR times for different teams. (Procedure adapted from Palleros). 200 pts **Final Check-out**

### **Some helpful comments and instructions:**

**Safety:** Is still a #1 priority! Lab safety glasses and appropriate dress are mandatory. Long hair must be tied back. Gloves are always available and sometimes will be made mandatory. More so than in Org I, we will be handling toxic and/or mutagenic reagents. Please pay attention to any handling and disposal instructions given by me or your TA.

**Notebooks:** I'm still somewhat flexible. If you prefer having 2 composition books so that while the TA or I grade one experiment, you can be using the other notebook to prepare for the next session, that's still okay. Handwritten reports should be done in ink (I'll allow pencil for calculations only). It really makes more sense though to use a computer to prepare reports and then hand in a hard-copy. Why? Since the format of a synthesis lab doesn't change, the computer approach allows you to create/reuse a template for reporting. Spiral notebooks or loose leaf papers are not acceptable!! Be sure your report is complete and turned in by the due date set by your TA (usually 1 week after completion of the experiment). Late reports are docked 10 pts per day. The TA or I will discuss the format of the lab report with you in class. There is also a sample lab report posted outside my office and in lab 214.

**Lab Preparation:** We'll use the same tongue-in-cheek lab manual since it provides most of the technical info you need. I'll cover procedures not in Zubrick as necessary in the pre-lab lectures. As in 343, the actual experiments will be provided in a packet that can be purchased from Tracy in the chemistry stockroom. If necessary, review the appropriate section describing these techniques in the lab manual so you'll be prepared when you come to lab. BE SURE to come to lab prepared to discuss and run the experiment. If we notice that you are continually clueless about the day's experiment, you will lose points on that particular lab. I have a variety of other, less light-hearted, lab texts available if you would like additional information about a technique or an experiment. Don't be bashful about coming to my office and signing out these supplementary texts.

We'll be emphasizing spectroscopic characterizations much more in Org II lab...especially NMR as a tool for analyzing product structures. This means that we'll go beyond simple spectral analysis. For instance, we'll see how peak integration can be used to quantitate product distribution, how J-values can be measured and used to verify stereochemistry, and how chemical shift calculations can be used to distinguish structural

isomers. I guess what I'm saying is that higherlevel texts and discussions may be in order. Of course, I'll lecture these concepts, but for the curious there are also a number of texts available in my office.

**Grading:** We'll work on a 10 pt scale (100-90 = A, 89-80 = B, etc). Each single-session experiment is worth 100 points; the 3 multi-session labs (eg, the redox sequence) count for 200 pts each. You may drop your lowest lab grade (or half of a 200 pt lab grade)... if you do them all...which means your grade will be based on a total of 1100 points. If you drop part of a multi-session lab, be sure to consult with the TA regarding what will be needed in the way of a lab report. If you must miss a lab session due to illness or some other unavoidable circumstance, that might become your "drop". If you would like to propose something else, come talk to me. Usually we can work out an arrangement agreeable to both of us. If you earn the following number of points, you are guaranteed the letter grade shown:

1100-990	90% - 100%	A
989-880	80%-89%	B
879-770	70%-79%	C
769-660	60%-69%	D
below 659 pts	below 60%	F

Remember, yields are still not my primary concern!!! Be honest. If you have a low yield or unusual results, it's more important to me that you figure it out and tell my why/how this might have occurred. More so than in Org I, the TA and I will take into account how well you work in the lab. (translation: yourlab preparation and lab skills will also contribute to your grade) The experiments are designed to give you much more "freedom" to draw on what you should already have learned. Naturally, if you really don't know... ask! Independence isn't the same as reckless abandon. We expect to see you practicing good techniques for routine tasks like transferring product, filtrations, tlc, etc. Again, the TA and I reserve the right to consider your performance (and preparedness) in lab when assigning a grade to your lab report. If we see something really wonderful, it's possible to score better than 100%! Downside: if we see you doing something really "stupid" or dangerous, you can lose points even though your final lab report is very good. Often there are questions posed within the experimental procedure. They contribute to that lab's final grade so be sure to answer them in your lab report. Sometimes you are asked to come to lab prepared to answer a particular question or propose an explanation –be sure to do that or points may be deducted.

\*\*Grade averages for each section are normalized before assigning final grades to account for minor discrepancies in how hard/easy an individual TA tends to grade lab reports.

**MISCELLANEOUS INFO:** This semester we'll continue with microscale chemistry. We still need to share things like micro beakers, Ehrlenmeyers, centrifuge tubes, etc. Please take good care of this stuff and be considerate about cleaning it after use...others need to use it too. Also, please be careful to return all community microscale equipment at the end of the session so other sections can be assured of having an adequate supply. I think the extra effort here is worthwhile. Not only is our microscale emphasis an environmentally sound approach to bench chemistry (reduced reaction scale = less chemical usage = less chemical waste generated = less student exposure to toxic and explosive chemicals = safer lab conditions), but also microscale experiments enjoy another benefit .... shorter reaction times!

**Problems?** Both the TAs and I have office hours. Most of you already know it's okay to just drop by and catch me in my office for quick questions or explanations.