

Chemistry 211L
Organic Chemistry Laboratory
Fall, 2000

1-5 p.m. 207 Kennedy Hall (pre-lab) & 408 Kennedy Hall (lab work)

Dr. Richard Redfearn Office – 409 Kennedy Hall

843-3960 (o), 272-3472 (h); redfearn@rhodes.edu

Description: This semester, you will focus on learning some of the techniques necessary to do experimental and synthetic organic chemistry. The experiments performed will correspond to the material we cover in class as closely as possible.

Goals: To be successful in this course, the goals that you should work towards are:

1. Becoming familiar with and gaining competence in the various synthetic techniques.
2. Understanding how the experiments relate to the concepts covered in the lecture.

Laboratory manual: Pavia, Lampman, Kriz, and Engel. Introduction to Organic Laboratory Techniques, A Microscale Approach, 3rd ed. Reference books in the library are The Merck Index and The CRC Handbook of Chemistry and Physics and the Aldrich catalogue (Sigma-Aldrich Chemical Company, Inc., on reserve in the library). These books are also available in 405 Kennedy. You will also need a laboratory notebook with pre-numbered duplicate white/yellow pages (available in the bookstore).

Evaluation: Your laboratory work and reports must be your own and are to be pledged. Although it's permissible to discuss the labs with your colleagues, you may not show your completed report to another person nor just tell them an answer. The final grade will be based on the lab reports or write-ups (100 points each). I will drop your lowest lab grade and only count the highest ten. The grading scale is:

A	930-1000 points
A-	900-929
B+	870-899
B	830-869
B-	800-829
C+	770-799
C	730-769
C-	700-729
D+	630-699
D	560-629
D-	500-559

Policies: The lab and the class are closely intertwined. Consider the lab period to be *your* time to prove to yourself that the concepts we discuss in the lecture are actually useful for understanding and prediction of natural phenomena. Therefore, the first hour or so of lab will consist of a discussion covering this connection and the scope of the experiment.

You *must* be prepared when you come to lab. I expect you to read and *understand* the material before you arrive. The pre-lab report is to be turned in at the very beginning of the lab lecture (**1:00 p.m.**) on the day that lab is to be done. Please don't be late to class- if you are late, the report will be considered late. You will not be admitted to the lab without a completed pre-lab report. The lab write-up is due at the beginning of the lab lecture (**1:00 pm**) in the week following completion of the experiment. Tardiness of either report will result in a lowered grade for the report (10% per week for a maximum penalty of 30%). You must have done the lab to receive credit for a lab report. Unexcused absences will result in a loss of that lab day, with no possibility of a make-up lab.

If you absolutely *must* miss a lab, please let me know ahead of time by email or phone. We will discuss the circumstances around your absence, and I will decide whether a make-up lab is warranted.

Lab Notebook and Reports: In general, your lab notebook should be written well enough that another scientist could *understand* and *reproduce* your work. Write in ink, and put your name and the date on every page. Mistakes are removed by drawing a single line through them, not by scribbling through or using whiteout. You must have recorded the data in your notebook during lab. For example, the statement "I obtained 250 mg of product" is not acceptable. The procedure section should be recorded in your notebook on consecutive numbered pages, and you must not be missing any pages. The pre-lab report should also be written in your notebook. The final write-up may be written on regular paper or word-processed. The lab report will be graded according to the following outline, with the pre-lab and procedure sections contributing 25 points each and the final write-up accounting for the remaining 50 points.

Lab Safety: The most important assignment you will have in any laboratory will be to work without injuring yourself or causing injury to others. This also means that you should not be exposed to any chemical compounds above the permissible exposure limits set by OSHA or other governmental agency. The safety equipment in 408 Kennedy Hall has been checked for safe operation and its use will be demonstrated by your instructor. Material Safety Data Sheets (MSDS) for all materials are available for review of safe handling and disposal procedures. Your apparel in the lab will follow these guidelines:

1. Safety glasses with side shields or goggles are mandatory at all times.
2. No open-toe shoes. Feet must be completely covered. Tennis shoes or other soft uppers are OK.
3. Legs should be covered. If the air conditioning in the lab is down (on a hot day) – this rule could be relaxed to allow shorts or skirts.
4. Preferably, arms should be covered. Short-sleeved shirts are OK.
5. No dangling jewelry that could get caught in equipment.

Outline for Lab Reports:**I. Pre-lab (this section will be turned in before the lab begins):**

- i. Title of lab.
- ii. Purpose: A brief statement will usually suffice. What techniques and/or concepts are being studied?
- iii. Reaction to be performed. *Include a detailed mechanism.*
- iv. Table of reagents and other materials: Make a table listing the reagents you'll be using, such as the following:

Compound	Mol. Weight	Amount Used	#mMols	m.p. or b.p.	Density/other data	Hazards
Starting material	X	X	X	if needed	if needed	X
Reagent	X	X	X	if needed	if needed	X
Product	X	X	(expected)	X	if needed	X
Solvent		X		X		X

An "x" indicates that this column should be filled in; an "if necess." should only be filled in if the data is needed to calculate the amounts you'll need. Note that the table requires that reagents needed for the actual reaction only should be filled in - the data for work-up chemicals (such as sodium bicarbonate or extraction solvents) are unnecessary.

- v. Intended Procedure: A brief step-by-step outline of the procedure you will follow
- vi. Anything else specified in the syllabus

II. Detailed procedure:

- i. Clearly record what you did. Include the time between steps, when necessary
- ii. Data: Clearly label any collected data to avoid the possibility of confusion later. Make sure to turn in your data with your lab report.

III. Final write-up (detailed procedure is included herein):

- i. Products/Unknowns: Identify any unknowns and products, along with proof of their identity.
- ii. Conclusions: Think critically and analyze your results. Were they what you expected? Why or why not? Rationalize the outcome. If you obtained an unexpected result (zero yield of product, unexpected isomer of a product) – try to explain why.
- iii. Questions: Answer any assigned questions.

Schedule of Experiments:

Expt. 1	Molecular models: conformational investigations
8/29-30-31	No pre-lab or final lab report. Laboratory check-in.
Expt. 2	Molecular models: fun with stereochemistry
9/5-6-7	No pre-lab report. Assigned questions from Expt. 1 due. Receive pre-lab handout for Expt. 3 – assigned reading.
Expt. 3	Symmetry in organic chemical structures: evidence from ^{13}C NMR
9/12-13-14	Final lab report for Expt. 2 due. Pre-lab report for Expt. 3 due. Receive pre-lab handout and reading assignments for Expt. 4.
Expt. 4	Melting points, boiling points, crystallization -microscale method
9/19-20-21	Final lab report for Expt. 3 due. Pre-lab report for Expt. 4 due. Receive pre-lab handout and reading assignments for Expt. 5. Note: this will be the beginning of your “wet chemistry” study using the microscale method. Read carefully the assignment from the text regarding the microscale method and the determination of percent yield.
Expt. 5	The resolution of (+)-α-Phenethylamine
9/26-27-28	Final lab report for Expt. 4 due. Pre-lab report for Expt. 5 due. Receive pre-lab handout and reading assignments for Expt. 6.
Expt. 6	Preparation of <i>meso</i>- and racemic 1,2-dibromo-1,2-diphenylethane
10/3-4-5	Final lab report for Expt. 5 due. Pre-lab report for Expt. 6 due. Receive pre-lab handout and reading assignments for Expt. 7. Note: the preparation of <i>meso</i> -stilbene dibromide is not an alternate procedure, since you will be making both products. In your conclusions, discuss the purity of your products and the percent yield of each.

Expt. 7	Markovnikov and anti-Markovnikov addition to an alkene: hydroboration-oxidation and oxymercuration of 1-methylcyclohexene
10/10-11-12	Final lab report for Expt. 6 due. Pre-lab report for Expt. 7 due. Receive pre-lab handout and reading assignments for Expt. 8.
Expt. 8	Free radical halogenation of hydrocarbons
10/24-25-26	Final lab report for Expt. 7 due. Pre-lab report for Expt. 8 due. Receive pre-lab handout and reading assignments for Expt. 9.
Expt. 9	Reactivities of some alkyl halides; competing nucleophiles
10/31-11/1-2	Final lab report for Expt. 8 due. Pre-lab report for Expt. 9 due. Receive pre-lab handout and reading assignments for Expt. 10.
Expt. 10	Elimination reactions: dehydration and dehalogenation; dehydration of 1- and 2-butanol; dehydrobromination of 1- and 2-bromobutane
11/7-8-9	Final lab report for Expt. 9 due. Pre-lab report for Expt. 10 due. Receive pre-lab handout and reading assignments for Expt. 11.
Expt. 11	An oxidation-reduction scheme: borneol, camphor, isoborneol.
11/14-15-16	Final lab report for Expt. 10 due. Pre-lab report for Expt. 11 due.
Check-out	All work due and laboratory check-out
11/28-29-30	Nov. 30 is the last day to turn in lab reports unless special permission is granted by the instructor.