

Chemistry 211L
Organic Chemistry Laboratory
Fall, 2002

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

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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). 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

The 100 points for the lab report grade will be divided into 25 points for pre-lab and notebook information, and 75 points for the final lab report (see below, **Lab Notebook and Reports**).

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. You will not be admitted to the lab without the completed pre-lab information recorded in your notebook, *and* all or part of the points allotted to the pre-lab may be lost if this occurs. The final lab report is due at the beginning of the lab lecture (**1:00 pm**) in the week following completion of the experiment. Please don't be late to class- if you are late, the report will be considered late. Tardiness of a 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. Lab reports that are "in progress" at **1:00 pm** on your lab day will be considered late and will receive the 10% penalty. 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. The procedure section should be recorded in your notebook on consecutive numbered pages, and you must not be missing any pages. The pre-lab information should be written in your notebook. The lab report will be graded according to the following outline, with the pre-lab section (including your observations taken during the experiment) contributing 25 points and the final report accounting for the remaining 75 points.

About the "pre-lab" information - you will record the information and data required in the pre-lab report (see **Outline for Lab Reports** below), but these will remain in your lab notebook. The pre-lab contribution to your grade will be evaluated twice during the semester, during the week of 10/8, then at the end of the semester. You are required to record the information required in the pre-lab before the lab period! If there is any evidence that the pre-lab information was recorded after the fact, this could result in loss of all or part of the points allotted to this section, and the incident may be brought before the Honor Council. Your instructor may choose to check for compliance in writing the pre-lab information weekly, before the lab period, or spot-check for compliance at any time. There is a sample pre-lab on the Academic Volume, in the Redfearn folder – we expect you to look at it and use it as a model for writing your own pre-labs. Since we are not grading the pre-labs until midway through the semester it is essential that you model your pre-lab after the pre-lab on the Academic Volume. If you lose points for the same error on all your pre-labs, it is because you have not paid close enough attention to the "model" pre-lab. Do not expect to retrieve points for a mistake made that has been carried through all the pre-labs.

The final lab report should be a polished document, written in a word processing program and printed in a standard format. More information about pre-lab and final reports can be found below in the **Outline for Lab Reports** section.

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.
6. If you have long hair – please be sure to have it contained so that it does not hang down into equipment.

Outline for Lab Reports:

I. Pre-lab (to be kept in your lab notebook and evaluated twice during the semester):

- 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 detailed mechanism information if the experiment involved a chemical reaction or reactions.*
- 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 needed" 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.

Note: A sample pre-lab will be posted on the Academic Volume (Redfearn/Loprete).

II. Detailed procedure and observations (to be recorded during the experimentation):

- 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 copies of your data with your lab report.

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

- i. Cover page: title of experiment, date, your name, pledge.
- ii. Discussion section: purpose, reactions, mechanisms.
- iii. Detailed procedure, or reference to the procedure if already outlined elsewhere.
- iv. Results - Products/Unknowns: Identify any unknowns and products, along with proof of their identity. Give the yields of products in this section. Include all data, including printouts of spectra.
- v. 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, try to explain why.
- vi. Questions: Answer any assigned questions.

Note: A sample final report will be posted on the Academic Volume (Redfearn/Loprete).

We expect you to examine the sample pre-lab information and sample final lab reports. Therefore, you should be familiar with the format and types of information in the pre-lab information and the final lab report.

Schedule of Experiments:

Expt. 1	Molecular models: conformational investigations
9/3-4-5	No lab report. Laboratory check-in. Receive Expt. 1 instructions; pre-lab handout and reading assignments for Expt. 2
Expt. 2	Molecular models using ChemDraw and Chem3D – in Buckman 212
9/10-11-12	Lab report for Expt. 1 due. Receive pre-lab handout and reading assignments for Expt. 3.
Expt. 3	Melting points, boiling points, crystallization –microscale method
9/17-18-19	Lab report for Expt. 2 due. Receive pre-lab handout and reading assignments for Expt. 4. 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. 4	The resolution of (\pm)-α-Phenethylamine
9/24-25-26	Lab report for Expt. 3 due. Receive pre-lab handout and reading assignments for Expt. 5.

Expt. 5	Preparation of <i>meso</i>- and racemic 1,2-dibromo-1,2-diphenylethane
10/1-2-3	Lab report for Expt. 4 due. Receive pre-lab handout and reading assignments for Expt. 6. 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. 6	Markovnikov and anti-Markovnikov addition to an alkene: hydroboration-oxidation and oxymercuration of 1-methylcyclohexene
10/8-9-10	Lab report for Expt. 5 due. Receive pre-lab handout and reading assignments for Expt. 7.
Expt. 7	Free radical halogenation of hydrocarbons
10/15-16-17	Lab report for Expt. 6 due. Notebooks to be submitted for pre-lab evaluations. Receive pre-lab handout and reading assignments for Expt. 8.
Expt. 8	Reactivities of some alkyl halides; competing nucleophiles
10/29-30-31	Lab report for Expt. 7 due. Receive pre-lab handout and reading assignments for Expt. 9.
Expt. 9	Elimination reactions: dehydration and dehalogenation; dehydration of 1- and 2-butanol; dehydrobromination of 1- and 2-bromobutane
11/5-6-7	Lab report for Expt. 8 due. Receive pre-lab handout and reading assignments for Expt. 10.
Expt. 10a	An oxidation-reduction scheme: borneol, camphor, isborneol. Part 1.
11/12-13-14	Lab report for Expt. 9 due. No new pre-lab handout and reading assignment.
Expt. 10b	An oxidation-reduction scheme: borneol, camphor, isborneol. Part 2.
11/19-20-21 12/3-4-5	Lab report for Expt. 10 due - week of 12/3-4-5. Note: the lab days 12/3-4-5 can be utilized for the data collection part of this two-step experiment if necessary. BUT all late reports and notebooks must be submitted by 12/11 to be accepted. (Thursday, 12/12 is a Reading Day.)
Check-out	Laboratory check-out (if necessary)
12/3-4-5 12/10-11	Lab report for Expt. 10 due. Notebooks to be submitted for pre-lab evaluations. Dec. 11 is the last day to turn in late lab reports and notebooks unless special permission is granted by the instructor. Late penalties will apply.