

Chemistry 211L
Organic Chemistry Laboratory
Fall, 1999

Tuesday and Wednesday, 1-5 p.m., 207K
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Thursday, 1-5 pm, 207K
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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.

Please note that the lab and the class are closely intertwined. Consider that the lab day is *your* time to prove to yourself that the concepts we discuss in the lecture are actually true. Note that the first hour or so of lab will usually consist of an interactive discussion covering the scope of the experiment: we do not jump right into the lab in organic.

Goals: 1) To become familiar with and competent in the various synthetic techniques;
2) To understand how the experiments relate to the concepts covered in the lecture.

Laboratory manual: Pavia, Lampman, Kriz, and Engel. Introduction to Organic Laboratory Techniques, 3rd ed. Reference books you may find useful include The Merck Index and The CRC Handbook of Chemistry and Physics (available in the reference section of the library) and an Aldrich catalogue (on reserve in the library). These books are also available in 405K. 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. You may not work together in any way on any experiment unless directed otherwise. The grading scale is:

A	100-93%	C	76-73%
A-	92-90%	C-	72-70%
B+	89-87%	D+	69-63%
B	86-83%	D	62-56%
B-	82-80%	D-	55-50%
C+	79-77%		

Policies: You *must* be prepared when you come to lab. We expect you to have read and understood the material before you get there. 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 final lab report is due in its entirety 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%/week for a maximum penalty of 40%). 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.

You may NEVER work alone in the lab!!

Lab Reports: In general, your report should be well-written enough that another scientist could understand *and reproduce* your work. Please write your pre-lab and data sections in your notebook in ink, and put your name on every page. Mistakes are best removed by drawing a solid line through them, not by using white-out. You must have recorded all relevant data in your notebook during lab. For example, the statement "I obtained 250 mg of product" is not acceptable if no weights are also recorded.

The final write-up must be word-processed. You may of course neatly and legibly draw in any chemical structures by hand- accuracy is more important than learning to use a chemical drawing program (although this is available if needed).

Outline for Lab Reports:

I. Pre-lab (a copy of this will be turned in before the lab begins, ~25 points):

- i. Title of lab.
- ii. Purpose- a brief statement will usually suffice. What technique and/or concept is being studied?
- iii. Reaction to be performed. *Include a detailed mechanism.*
- iv. Table of Reagents- 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 necess.	if necess.	x
Reagent	x	x	x	if necess.	if necess.	x
Product	x	x	x	x	if necess.	x
			(expected)			
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 necessary for the experiment. Note that only reagents needed for the actual reaction should be filled in- the data for chemicals used after the reaction is complete (such as aqueous 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. Procedure (~25 points):

- 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 (~50 points):

- i. Products/Unknowns- Identify any unknowns and products, along with proof of its identity.
- ii. Conclusions- What do you think of the technique and/or experiment? Analyze the results. Were they what you expected? Why or why not? Rationalize the outcome. You should *always* have conclusions!!

iii. Questions- Answer the assigned questions.

Schedule of Experiments:

- Week 1
Aug. 31, Sep. 1, 2 ***Molecular Models: Conformational Investigations.*** No pre-lab or final lab report. Read pp. 2-8; 17-26; 33-36 in Pavia, paying careful attention to the determination of percent yield.
- Week 2
7, 8, 9
that ***Molecular Models: Fun with Stereochemistry!*** No pre-lab or Sep. final lab report. Bring the Chapter 3 class notes with you. Pledge that

you have read the Pavia material assigned above.
- Week 3
Sep. 14, 15, 16 Laboratory check in; ***Carbon-13 NMR of Hydrocarbons*** (Week 3 handout; no pre-lab) Also, begin ***Exp. 30A: The Resolution of (\pm)- α -Phenethylamine.*** Read pp. 285-286 and 288-290. (We will not be doing any NMR experiments). Pre-lab: Include i-v, but a mechanism is unnecessary. Note also that the reaction scheme shown is incomplete: the amine•tartrate salt is not the final product (read the text).
- Week 4
Sep. 21, 22, 23 ***Melting Points, Boiling Points, and Crystallization: Purification of Solids.*** Pre-lab: Read pp. 556-584. For a pre-lab, photocopy the procedure on page 574 and make notes in the margins as you read. This amended photocopy will be collected and graded. Note that we will be using the microscale procedure and we will not be removing insoluble impurities. Write-up: Answer questions on Week 4 handout.
- Week 5
Sep. 28, 29, 30 ***Exp. 23A: The Resolution of (+)- α -Phenethylamine.*** For lab: Read pp. 600-609 and 731-737. Write-up: See Week 5 handout; include conclusions with question #3.
- Week 6
Oct. 5, 6, 7 ***Preparation of meso- and rac-Stilbene dibromide.*** Read pp. 202-204 in Brown text; Week 6 handout. Pre-lab: Include i-v, including a good mechanism for either reaction. Write-up: See Week 6 handout.
- Week 7
Oct. 12, 13, 14 ***Markovnikov and Anti-Markovnikov Addition to 1-Methylcyclohexene.*** Read pp. 204-212 in Brown text; Week 7 handout. Pre-lab: Include i-v, with a table of reagents for each reaction. Also answer question 1a on page 784. Write-up: See Week 7 handout.

Week 8
Oct. 26, 27, 28

Free Radical Halogenation of Hydrocarbons. Read pp. 246-256 in Brown text; Week 8 handout. Pre-lab: Include i, ii, v. Write an equation for both of the reactions you will perform, showing all possible products and naming each compound. Write a mechanism for *one* of the above reactions. Write-up: Answer the questions on the handout.

Get assigned 1-butanol, 2-butanol, or 2-methyl-2-propanol today.

Week 9
Nov. 2, 3, 4

Exp. 18: Reactivities of Some Alkyl Halides; Exp. 19: Competing Nucleophiles. Read pp. 190-191 and 194-200. Pre-lab: For Exp. 19, include i-v on the alcohol assigned the previous week. For Exp. 18, include i-ii; write a generic equation and mechanism for the reaction of an alkyl halide with NaI and with AgNO₃. Write-up: See Week 9 handout.
Get assigned 1-butanol, 2-butanol, 1-bromobutane or 2-bromobutane today.

Week 10
Nov. 9, 10, 11

Exp. 22: Elimination Reactions: Dehydration and Dehydrohalogenation Read pp. 215-221. Pre-lab: Include i-v, with a table of reagents completed for the alkane assigned to you last week. Write-up: See Week 10 handout.

Week 11
Nov. 16, 17, 18

Kinetic Investigation: Formation of Sweet Esters. Week 11 handout. Pre-lab: Include i-iv; a mechanism is not necessary. Write-up: See Week 11 handout.

Week 12
Nov. 30, Dec. 1, 2

Check-out (only if necessary). There most likely will be a somewhat lengthy discussion today.

Dec. 8

Last possible day to turn in lab reports.