Organic Chemistry Laboratory
CHEM 2462
Major's Section

Spring Quarter, 2006

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Text: "Microscale and Miniscale Organic Chemistry Laboratory Experiments", Second Edition, by Schoffstall, Gaddis, and Druelinger.

General: Lab is scheduled from 2:00 to 5:30 on M, Tu, W, Th or 6:00 to 9:30 Tu. Please come, on time, to your assigned lab section. Attendance will be taken. Each lab will begin with a brief introduction to the experiment to be done that day.

The schedule of experiments for the quarter is attached. Please read the assigned sections in your lab book **before** coming to lab and do the "Prelab Assignments" that are specified in this syllabus. You will not be allowed to begin the experiment until the TA has verified that you have completed the "Prelab Assignments" and you have prepared your lab notebook for the experiment. If you read the assignments before lab and plan your time well, you will find it easy to complete the experiments in the allotted time or less. Planning ahead will also enable you to use your time most effectively. For example, an experiment might require that a reaction mixture be heated for one hour. The best use of your time would be to get the reaction started immediately. Then, while the reaction is being heated, you could distill the product from the preceding experiment, take melting points, wash glassware, or even study. In addition, you will find it advantageous to clean your glassware before you leave for the day. When you come for the next lab, your glassware will be clean, dry, and ready to use.

Safety: READ PP. 3-5 IN THE LAB TEXT.

The organic laboratory is a potentially dangerous place. However, no accidents need occur **if you are careful and are constantly aware of what you are doing and why.** Read the directions given in each experiment carefully and in advance. Pay particular attention to any special safety precautions that are given for an experiment. Make sure you understand each step of the experimental procedure and any potential dangers. If you have questions, ask! Use your common sense and, above all, **THINK!**

The following safety rules will be in effect at all times:

1. Safety glasses must be worn <u>at all times</u>.

2. Wear proper attire; gloves and lab coats when necessary; shoes (no sandals); no shorts.

3. No flames or smoking allowed in the lab.

4. No food or drink allowed in the lab.

5. No horseplay allowed in the lab.

6. Keep all work areas clean and orderly. This includes your bench, the balance table and the hoods. Clean up all chemical spills immediately.

7. Use proper disposal procedures, as specified by your TA, for all chemicals and solutions.

8. Be careful to avoid contaminating the reagents. Close all containers snugly after use.

9. Be sure to read the labels on chemical containers carefully. Many chemical names are very similar.

10. Most organic chemicals are toxic to some extent. Treat them all with respect. Avoid getting them on your skin or clothing and avoid extensive breathing of their fumes. Work in the hoods when the experimental directions so instruct.

11. No students are allowed in the lab unless the TA is present. Only students assigned to that lab section are allowed in the lab.

12. No chemicals, glassware or equipment are to be removed from the lab. No unauthorized experiments are to be done.

Notebooks: READ PP. 6-7 IN THE LAB TEXT.

You must have a **bound** lab notebook (not spiral). You should use the style described in the lab text. Steps 1, 2, 3, 4, 6, and 8 on p. 6 are to be completed before lab in your lab notebook. You will not be allowed to begin the experiment until this material is complete. All data, observations, etc. must be entered directly in your notebook at the time the information is obtained in the lab. Your notebook will not be graded on neatness, only on completeness.

Reports: Read pp. 6-7 in the lab text for a general procedure. Examine the sample lab report that is available on Blackboard. The report for each experiment must be typewritten and all structures must be done using a structure drawing program such as Isis/Draw. For preparative experiments (when you make a compound) these reports should include:

Your name and the date;
Title of the experiment;
Introduction
section with a brief description of the objective or purpose of the experiment and a chemical equation if applicable
Table
includes the reactants, reagents and products of the experiment with their molecular weights, amounts, etc.
Procedure
an outline of the experimental procedure
Results and Discussion
includes pertinent observations, data ,graphs, and spectra, and a brief discussion, analyzing your experimental data (If the experiment did not work well, comment on why this might be.)
Answers to Questions

Excluding the data, calculations, and graphs, the report should be a maximum of two pages long. See the sample lab report for more details. Investigative experiments should include your name and the date, the title, introduction (an equation may or may not be appropriate), procedure, results and discussion, and answers to assigned questions.

Unless you are told otherwise, each report is due at the beginning of the next lab after the completion of the experiment. Reports will be penalized 30% for lateness. Reports more than two periods late will not be accepted.

Products: For preparative experiments, submit the product in a clean, labeled test tube along with the report. The label should have your name and the structure, weight, percent yield and mp or bp range of the product.

Grading: Your grade will be based on a total of 1120 points, distributed as follows:

Prelab Assignments, 10 pts./exp.	60 points
Lab reports and products (if applicable) 100 pts/exp.	1000 points
Lab notebook, other than Prelab Assignments	40 points
Subjective	20 points

Schedule of Laboratory Experiments
Spring Quarter, 2006

Week of Mar. 27

Check In

Experiment 1 Synthesis of an Alkyl Halide from an Alcohol

Read pp. 221-224 in the lab book. You will do Experiment 4.1. Do Prelab Assignments 1, 2, 5, and 6 on p. 222. Answer questions 2 and 4 on p. 224 in your lab report.

Week of Apr. 3

Experiment 2 Dehydration of an Alcohol

Read pp. 229-231 in the lab book. You will do Experiment 5.1A. Analyze the product mixture by gas chromatography and gc-ms. Do Prelab Assignments 1, 2, 3, and 5 on pp. 230-231. Answer questions 1, 2, and 3 on p. 233 in your lab report.

Week of Apr. 10

Experiment 3 Hydration of Norbornene

Read pp. 242-246 in the lab text. You will do Experiment 6.2B. Do Prelab Assignments 1 and 2 on p. 245. Purify the product by sublimation and identify it by its melting point. Answer questions 1 and 2 on p. 246 in your lab report.

Week of Apr. 17

Experiment 4 Bromination of Stilbene

Read pp. 413-418 in your lecture text. The procedure for this experiment is available on Blackboard.

Week of Apr. 24

Experiment 5 Hydrogenation of Allylbenzene

Read pp. 237-242 in the lab text. You will do Experiment 6.1B. Do Prelab Assignments 1, 2, and 3 on p. 239. Analyze the product by IR spectroscopy and gc-ms. Answer questions 2 and 5 on pp. 241-242 in your lab report.

Week of May 1

Experiment 6 Unknown Identification by Spectroscopy

You will be given two unknown samples. You will identify one of them using its IR spectrum. You will use both the IR and ^{1}H-NMR spectra of the other compound to identify it.

IR Unknown: Your unknown will be one of the compounds from the list below. Obtain the IR spectrum of your unknown and identify it. The report should contain an interpretation of the IR spectrum and the reasons behind your identification of the unknown.

t-butanol
benzyl alcohol
ethyl acetate
ethyl benzoate
acetophenone
3-pentanone
benzophenone
acetic acid
benzoic acid
toluene
benzene
o-nitrotoluene
cyclohexane
nitrobenzene
aniline
p-nitroaniline
cyclohexylamine
benzonitrile
acetonitrile
benzaldehyde

IR/NMR Unknown: Your unknown will be one of the compounds from the list below. Obtain an IR and a NMR spectrum of your unknown. The report should contain a complete interpretation of both the IR and NMR spectra.

Aldehydes
2-methylpropanal
4-nitrobenzaldehyde
(E)-3-phenyl-2-propenal
(cinnamaldehyde)

Acids
diphenylacetic acid
1,4-butanedioic acid
(succinic acid)
propanoic acid
(E)-2-butenoic acid
(crotonic acid)

Amines
4-methylaniline
dibutylamine
triethylamine

Esters
ethyl formate
ethyl acetate
methyl butyrate
3-methylbutyl acetate
methyl benzoate
diethyl phthalate
methyl m-nitrobenzoate
2-propenyl acetate

Ketones
3-methyl-2-butanone
3-pentanone
3,3-dimethyl-2-butanone
(pinacolone)
2,5-hexanedione
propiophenone
(ethyl phenyl ketone)
acetophenone
(methyl phenyl ketone)
4-heptanone

Alcohols
2-isopropyl-5-methylphenol
(thymol)
ethanol
3-methyl-1-butanol
2-phenylethanol
E-3-phenyl-2-propene-1-ol
2-propanol
2-propen-1-ol
1-propanol
benzyl alcohol
(phenylmethanol)
diphenylmethanol
(benzhydrol)

Experiment 7 **Identification of Unknowns by Gas Chromatography-Mass Spectrometry**

Read pp. 617-632 in your lecture text. The procedure for this experiment is available on Blackboard.

Week of May 8

Experiment 8 **The Addition of HBr to Unsymmetrical Alkenes**

Read pp. 406-412 in your lecture text. The procedure for this experiment is available on Blackboard. Each person should use a different one of the alkenes, 3-propen-2-one (methyl vinyl ketone), methyl 2-propenoate (methyl acrylate), vinyl acetate, or phenylethene (styrene). Obtain a ^{1}H-NMR spectrum of your product. You should discuss the regiochemistry of addition to all of the alkenes in your report.

Week of May 15

Experiment 9 **Friedel-Crafts Acylation**

Read pp. 310-314 in the lab text. You will do Experiment 12.3B. Do Prelab Assignments 1 and 5 on p. 311. Determine the composition of the product mixture by TLC analysis.

Week of May 22

Experiment 10 **Relative Rates of Bromination**

Read pp. 298-304 in the lab text. You will do Experiment 12.1. Work with a partner. Do Prelab Assignment 2 on p. 301. Answer questions 1, 3, 4, and 6 on p. 303 in your lab report.

Week of May 30

Clean Up and Check Out

You must check out of the lab or you will get an incomplete. Do not forget to give your lab notebook to your TA for grading.