Organic Chemistry Laboratory
CHEM 2460 2
Majors Lab Section

Spring, 2002

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Text: "Introduction to Organic Laboratory Techniques: A Microscale Approach", Third Edition, by Pavia, Lampman, Kriz, and Engel.

General: Lab is scheduled from 2:00 to 5:30 on Monday. Please come on time. 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. **Also read the "Required Reading" sections that are indicated for each experiment.** This will enable you to follow the introduction more easily. If you read ahead 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, etc. 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. 4-20 IN YOUR 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 the "Special Instructions" that are described for each 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 at all times (no contact lenses).

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. 20-26 IN YOUR LAB TEXT.

You must have a **bound** lab notebook. You should use the style described in your lab text. All data, observations, etc. must be entered directly in your notebook. Your notebook will not be graded on neatness, only on completeness.

Prelabs: Before coming to the lab, you must do a prelab write-up in your notebook. This write-up should be very similar to that shown on p. 24 of your lab text up to the "Data and Observations" part. Use a "Table of Reagents" (rather than a "Table of Physical Constants") that includes the weights and/or volumes and moles of the required reagents along with their physical constants. For non-synthesis experiments, the write-up should contain a brief outline of what will be done in that experiment. You

must have your prelab write-up initialed by your TA before you can get your starting materials from the stockroom or begin the experiment.

Reports: The report for each experiment must be typewritten. These reports should include:

Your name and the date and title of the experiment;
A brief description of the objective or purpose of the experiment;
A chemical equation if applicable;
A description of the experimental procedure including pertinent observations, data (actual weights, volumes, mp, etc.), graphs, and spectra;
All of your calculations, e.g. for the theoretical and percent yields;
A brief discussion, analyzing your experimental data (If the experiment did not work well, comment on why this might be.);
Answers to any assigned questions.

Excluding the data, calculations, graphs, and answers to the assigned questions, the report should be a maximum of two pages long.

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 synthesis experiments, submit the product in a clean, labeled vial 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 1000 points, distributed as follows:

Pre-lab write-ups, 10 pts./exp.	60 points
Lab reports and products (if applicable) 100 pts./exp.	900 points
Lab notebook, other than pre-lab write-ups	40 points

Schedule of Laboratory Experiments
Spring Quarter, 2002
Majors Lab Section

Mar. 25
Experiment 1 An Oxidation-Reduction Scheme

Read pp. 266-277 in your lab text. Do Experiment 28. This is a two-week experiment. Do Part A during the first week and Part B during the second week. Determine the percentages of borneol and isoborneol by gas chromatography.

Apr. 8
Experiment 2 Bromination of Stilbene

Read pp. 409-412 in your lecture text. The procedure for this experiment is attached to this syllabus.

Apr. 15
Experiment 3 Catalytic Hydrogenation

Read pp. 244-248 in your lab text. Do Experiment 25.

Apr. 22
Experiment 4 Determination of a Conformational Equilibrium

There is no prelab for this experiment. The procedure for this experiment is attached to this syllabus.

Experiment 5 Unknown Identification by Spectroscopy

There is no pre-lab for this experiment. 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. The report for this experiment is due during the week of Mar. 6.

IR Unknown: You will be given an unknown compound 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: You will be given an unknown 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)

Apr. 29
Experiment 6 The Addition of HBr to Unsymmetrical Alkenes

Read pp. 403-408 in your lecture text. The procedure for this experiment is attached to this syllabus. 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.

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

Read pp. 608-627 in your lecture text. The procedure for this experiment is attached to this syllabus. The report for this experiment is due during the week of May 13.

May 6

Experiment 8 **Determination of Spice Components by GC/MS**

Read pp. 487-492. Do Experiments 57A and 57B, except use a scale that is three times larger than that described in the book. A larger scale distillation apparatus will be provided (see Figure 8.11 on p. 629). You will use the GC/MS instrument to analyze the compounds that you isolate.

May 13

Experiment 9 **The Grignard Reaction**

Read pp. 292-299. You will do a procedure very similar to Experiments 31 and 31A, except on a larger scale. The procedure for this experiment is attached to this syllabus. This is a two-week experiment.

June 3

Finish any experiments, clean up, and check out.

You must check out of the lab at this time or you will receive an incomplete in the lab. Monday, May 27, is Memorial Day, and there is no lab, so you should check out on June 3.