

**Organic Chemistry Laboratory**  
**CHEM 2462**

**Spring Quarter, 2008**

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**Text:** "Microscale Operational Organic Chemistry", by John W. Lehman

**General:** Lab is scheduled from 2:00 to 5:30 on M, Tu, W, or 6:00 to 9:30 on 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 experiment **before** coming to lab and do the "Before You Begin" assignments. You will not be allowed to begin the experiment until the TA has verified that you have completed the "Before You Begin" assignments and you have prepared your lab notebook for the experiment. If you read the experiments 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. 14-23 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 at all times.
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 Appendix II (pp. 792-796) in the lab text.**

Notebooks are to be kept in the same manner as last quarter. You must have a **bound** lab notebook (not spiral). You should use the style described in the lab text. The "Before You Begin" assignments must be completed in your lab notebook before lab. 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 Appendix III (pp. 792-796) in the lab text.** We will use a slightly modified version of the description in the text. The report for each experiment must be typewritten and all structures must be done using a structure drawing program such as ChemSketch. The report should include:

**Title of the experiment**  
**Your name and the date**

### **Introduction**

This section has a statement of the problem and any applicable chemical equations. See the beginning of the discussion section on p. 795.

### **Observations**

### **Data**

### **Calculations**

### **Results**

### **Discussion**

### **Conclusions**

### **Exercises**

Include the answers to any Exercises that are assigned in the syllabus.

Excluding the data, calculations, and graphs, the report should be a maximum of two pages long. The Discussion section can be considerably briefer than that of the sample lab report in the text.

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:** The syllabus specifies that the product should be turned in for several experiments. Submit the product in a clean, labeled test tube along with the report. The label should use the format on p. 44 of the lab text.

**Grading:** Your grade will be based on a total of 1000 points, distributed as follows:

Before You Begin Assignments, 10 pts/exp	80 points
Lab reports, 80 pts/exp	800 points
Products, 10 pts/exp	70 points
Lab notebook, other than BYB Assignments	40 points
Subjective	10 points

## **Schedule of Laboratory Experiments Spring Quarter, 2008**

### **Week of Mar. 24 Check In**

#### **Experiment 1      Dehydration of 2-Methylcyclohexanol**

Read Experiment 21 on pp. 162-171 in the lab text. Do the "before You Begin" assignments. Work in pairs. Run the reaction using 2-methyl-cyclohexanol as described on p. 169. Analyze the two distillation fractions by gas chromatography. Each partner should inject one of the fractions. Each partner should turn in one of the fractions with his/her lab report. Include "Exercises" 3a, 4, 5, and 6 in your lab report. The report is due during the week of Mar. 31.

### **Week of Mar. 31**

#### **Experiment 2      Preparation of Camphor**

Read Experiment 7 on pp. 69-74 in the lab text. Do the "Before You Begin" assignments. Do Part A of the experiment. Include "Exercises" 1, 2, and 4 in your lab report. Turn in the product with your lab report. The report is due during the week of April 7.

### **Week of April 7**

#### **Experiment 3      Stereochemistry of Bromine Addition**

Read Experiment 23 on pp. 180-185 in the lab text. Do the "Before You Begin" assignments. Include "Exercises" 1, 2, 3, and 7 in your lab report. Turn in the product with your lab report. The report is due during the week of April 14.

### **Week of April 14**

#### **Experiment 4      Hydration of a Difunctional Alkyne**

Read experiment 24 on pp. 187-194 in the lab text. Do the "Before You Begin" assignments. Analyze the product by IR spectroscopy. Include "Exercises" 2 and 6 in your lab report. Turn in the product with your lab report. The report is due during the week of April 21.

### **Week of April 21**

#### **Experiment 5      Investigation of a Chemical Bond by Infrared Spectroscopy**

Read experiment 13 on pp. 110-116 in the lab text. Do the "Before You Begin" assignments. Work in groups of three. The report is due during the week of April 28.

### **Week of April 28**

#### **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  $^1\text{H}$ -NMR spectra of the other compound to identify it. The report is due during the week of May 5.

**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-butenic 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)

**Week of May 5****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. The report is due during the week of May 12.

**Experiment 8      Preparation of Tropylium Fluoborate**

Read Experiment 34 on 267-275. Do Part B of the experiment. Do "Before You Begin" assignment 1. Turn in the product with your lab report. The report and the product are due during the week of May 12.

**Week of May 12****Experiment 9      Determination of the Structure of a Natural Product in Anise Oil**

Read experiment 37 on pp. 291-297 in the lab text. Do the "Before You Begin" assignment. Include "Exercise" 1 in your lab report. Turn in the product with your lab report. The report and the product are due during the week of May 19.

**Week of May 19****Experiment 10      Friedel-Crafts Acylation of Anisole**

Read Experiment 36 on pp. 283-290 in the lab text. Do the "Before You Begin" assignments. Include "Exercises" 1, 2, 3, and 4 in your lab report. Turn in the product with your lab report. The report and the product are due during the week of May 27. Turn in your lab notebook for grading along with your last report.

**May 27, 28, or June 2****Clean Up and Check Out**

Make sure all of your glassware is clean and the contents of your drawer are complete. Then check out of the lab. If you do not check out, you will get an incomplete.