

## Organic Chemistry Laboratory

### CHEM 2462

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**Text:** Organic Chemistry Lab, CHEM 2461, 2462, 2463, Prof. Joseph Hornback

**General:** Lab is scheduled from 12:00PM to 3:30 PM MTWTH. Each lab will begin with a brief introduction to the experiment of the day by your TA.

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 Lab Safety: General Guidelines** at the beginning of the lab text and the **Laboratory Safety** section on pp. 11-20. 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. 381-382) 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. 383-387) in the lab text. Reports should be done similar to last quarter. Your TA will describe exactly what is expected. The report for each experiment must be typewritten and all structures must be done using a structure drawing program such as ChemSketch.

**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. 41 of the lab text.

**Grading:** Your grade will be based of 1050 points distributed as follows:

Before You Begin Assignments		60 points
Lab. Reports	100 points/exp.	900 points
Products	10 pts./exp	50 points
Lab. Notebook		30 points
Subjective		10 points

## Schedule of Laboratory Experiments

**July 29 Check In**

**July 30 Experiment 1 Dehydration of 2-Methylcyclohexanol**

Read pp. 87-95 in the lab text. Do the "before You Begin" assignments. Work in pairs. Run the reaction using 2-methylcyclohexanol as described on p. 94. Do not do the dehydration of 4-methylcyclohexanol. 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.

**July 31 Experiment 2 Preparation of Camphor**

Read pp. 61-66 in the lab text. Do the "Before You Begin" assignments. Include "Exercises" 1, 2, and 4 in your lab report. Turn in the product with your lab report.

**August 1 Experiment 3 Stereochemistry of Bromine Addition**

Read pp. 97-102 in the lab text. Do the "Before You Begin" assignments. Do Part A of the experiment. Include "Exercises" 1, 2, 3, and 7a in your lab report. Turn in the product with your lab report.

**August 5 Experiment 4 Hydration of a Difunctional Alkyne**

Read pp. 105-112 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.

**August 6 Complete Experiment 4**

**August 7 Experiment 5 Investigation of a Chemical Bond by Infrared Spectroscopy**

Read pp. 73-79 in the lab text. Do the "Before You Begin" assignments. Work in groups of 4. The carbonyl compounds to be investigated are acetophenone, cyclohexanone, ethyl acetate, and *N,N*-dimethylformamide. Each member of the group should run the IR spectrum of one of the compounds. Xerox all of the IRs so that each group member has a set of four IRs to work with.

## August 8 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.

**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

**August 12 Experiment 7**

**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)

**August 13 Experiment 8 Preparation of Tropylium Fluoborate**

Read pp. 143-150 in the lab text. Do Part B of the experiment. Do "Before You Begin" assignment 1. Include "Exercise" 7 in your report. Turn in the product with your lab report.

**August 14 Experiment 9**

**Determination of the Structure of a Natural Product in  
Anise Oil**

Read pp. 161-167 in the lab text. Do the "Before You Begin" assignment. Include "Exercises" 1 and 7 in your lab report. Turn in the product with your lab report.

**August 15 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.