

# Organic Chemistry Laboratory

## CHEM 2463

Summer Quarter 2013

**INSTRUCTOR:** Ronald S. Nohr

Graduate Teaching Assistant: Michael Holden

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

### **General:**

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. 11-20 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.

(Print) NAME

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. 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.

**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. 41 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	50 points
Lab reports, 100 pts/exp	800 points
Products, 10 pts/exp	50 points
Lab notebook, other than BYB Assignments	70 points
Subjective	30 points

## Schedule of Laboratory Experiments

June 17      **Check In**

June 18      **Experiment 1      Borohydride Reduction of Vanillin to Vanillyl Alcohol**  
Read pp. 123-130 in the lab text. Do the "Before You Begin" assignment. Your TA must approve your procedure before you begin the experiment. Do not do the IR spectrum. Include "Exercises" 1 and 6 in your lab report. Turn in your product with your report.

June 19      **Experiment 2      Synthesis of Triphenylmethanol**  
This is a two day experiment. Read pp. 133-142 in the lab text. Do the "Before You Begin" assignments. Do Part A of the experiment. Include "Exercises" 3, 5, and 6 in your lab report.

June 20      **Complete Experiment 2**

June 24      **Experiment 3      Wittig Synthesis of 1,4-Diphenyl-1,3-butadiene**  
Read pp. 169-175 in the lab text. Do Part B of the experiment. Do the "Before You Begin" assignments. Include "Exercises" 1 and 5 in your lab report. Turn in the product with your lab report.

June 25      **Experiment 4      Preparation of the Insect Repellent *N,N*-Diethyl-*m*-toluamide**  
Read pp. 177-186 in the lab text. Do the "Before You Begin" assignments. Analyze the product by gas chromatography and IR spectroscopy. Include "Exercises" 2 and 5 in your lab report. Turn in the product with your lab report.

June 26      **Complete Experiment 4**  
Begin Experiment 5 first. You can complete Experiment 4 during the two 1 hr. reflux periods of Experiment 5.

June 26      **Experiment 5      Synthesis of Dimedone and Measurement of Its Tautomeric Equilibrium Constant**  
Read pp. 187-196 in the lab text. Do the "Before You Begin" assignments. You will be provided with an NMR spectrum of dimedone. Include "Exercises" 2, 5, and 9 in

your lab report.

**July 1 Complete Experiment 5**

**July 2 Experiment 6 Simulated Identification of an Unknown**

Read about qualitative organic analysis on pp. 207-252 in the lab text. This lab is a computer simulation of the real experiment you will do next week. You will be assigned two unknown organic compounds to identify on the computer. In order to identify each compound you must tell the computer which test to run on the unknown. The computer provides you with the result of the test. You then have to decide what test to run next. You continue this process until you have identified the compound. Since the "tests" on the computer can be done easily, this experiment allows you to hone your skills before undertaking the real thing. Make sure to interpret the IR and NMR spectra of your unknown if they are available. Record the results of each test in your lab notebook just as though this is a real experiment done in the lab. Your report should include a complete write-up of the identification of one unknown, just as though the procedures were actually done in the lab. Also include the identity of the other unknown that you were assigned.

**July 3 Experiment 7 Identification of an Unknown**

Read pp. 207-252 in the lab text. This time you will be given a sample of a real unknown to identify. It will be an alcohol, aldehyde, amine, carboxylic acid, ketone, or phenol [one of the compounds listed in the tables in Appendix V (pp.395-404)]. Purify your unknown by distillation and determine its boiling point. Do not do the Ignition Test. Obtain the IR and NMR spectrum of your unknown. Using the IR spectrum, you should be able to tentatively identify the functional group present in your unknown. Based on this information, you can decide which on the Classification Tests listed on p. 214 and 215 to run. You must do at least one positive and definitive Classification Test. Then prepare a solid derivative of your unknown as described on p. 234 and following pages. Turn in the derivative with your lab report

**July 5 Clean Up, Check Out**

You must check out of lab at this time, turn in your breakage card and pay for any equipment you have broken or you will receive an I as your lab grade. **Do not forget to turn in your lab notebook.**