Organic Chemistry Laboratory
CHEM 2461

Winter 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 1000 points, distributed as follows:

Prelab Assignments, 10 pts./exp.	40 points
Lab reports and products (if applicable) 1000 pts/exp.	900 points
Lab notebook, other than Prelab Assignments	40 points
Subjective	20 points

Schedule of Laboratory Experiments
Winter Quarter, 2006

Week of Jan. 9
Check In

Experiment 1 Drawing Chemical Structures on the Computer

You will download a free program for drawing chemical structures from the Internet. You will learn to use this program during this lab. (The procedure for this experiment is available on BLACKBOARD.) This is the program that you will use it to draw any structures in lab reports for subsequent labs. Bring your laptop computer to this lab. (You may load the program before coming to lab if you so desire.) Access www.mdli.com on the Internet. Click "Downloads". You must register first. Then download the version of ISIS/Draw that is appropriate for your computer. Also download the appropriate Help file. While you are at this site, also download the Chime program that is appropriate for your computer. This program enables you to view molecules in three dimensions and will be very useful in class. Both of these programs are self-extracting. Click "run" when prompted and the programs will be installed on your computer.

Experiment 2 Scavenger Hunt

Read pp. 183 – 189 in the lab book. You will do Experiment 3.1, Parts A and B.

Week of Jan. 16

Make four calibrated Pasteur pipettes as described in Exercise B.3 on page 21.

Experiment 3 Identification of Unknowns by Melting Point and Boiling Point

Read pp. 22 – 32 in the lab book. You will do Exercise C.2 on p. 26 and Exercise D.2 on pp. 30 – 31. (You do not need to calibrate the thermometer.) Answer questions 1, 2, 3, and 4 on p. 27 and questions 1, 2, 3, 4, 6, 7, and 8 on pp. 31 – 32 in your lab report.

Week of Jan. 23

Experiment 4 Recrystallization of an Organic Solid

Read pp. 34 – 45 and 197 – 200 in the lab text. Do Prelab Assignments 1 (replace *trans*-stilbene with dimethyl terephthalate), 2, and 3 (one row only) on p. 197. You will be given an unknown compound (dimethyl terephthalate, 9-fluorenone, or benzoic acid). Determine a good solvent for recrystallization of the unknown as described in Experiment 3.4, Part A. Then recrystallize the unknown as described in part B. Identify your unknown by its melting point. Answer questions 1, 2, 3, 5, and 6 on p. 200 in your lab report.

Week of Jan. 30

Experiment 5 Separations Based on Acidity and Basicity

Read Technique I on pp. 72 – 80 and Experiment 3.5 on pp. 200 - 205 in the lab text. You Will do Experiment 3.5, Part B. Do Prelab Assignments 1 (no problems), 3, 4, and 5 on p. 202. Use the data provided in the following table to design a flow scheme for the separation of naphthalene, benzoic acid, and ethyl 4-aminobenzoate prior to coming to lab. Show your flow scheme to the TA. Once it has been approved, proceed to separate the compounds and determine the composition of your mixture. Answer questions 1, 2, 3, 4, and 5 on p. 204 in your lab report.

Compound	HCl	NaOH	CH_2Cl_2	Water
Naphthalene	i	i	s	i
Benzoic acid	i	s	s	i
Sodium benzoate	i	s	i	s
Ethyl 4-aminobenzoate	s	i	s	i
Ethyl 4-aminobenzoate hydrochloride	s	i	i	s

Week of Feb. 6

Experiment 6 Thin Layer Chromatography (TLC)

Read pp. 92 – 97 and 210 – 216 in the lab text. Do parts 1 – 4 of the Prelab Assignment. You will do Experiment 3.7. For part A, work in groups of four. Each person should determine the R_f values for the three compounds in <u>one</u> of the solvents. Combine this information and select a solvent or solvent mixture that will separate the compounds. Test your choice on a mixture of the three compounds. Then do Part B individually. Answer questions 3, 4, 5, 6, 9, and 10 on p. 215 in your lab report.

Week of Feb. 13

Experiment 7 Stereochemistry

Read pp. 173 – 177 in the lab text. The procedure for this experiment is available on BLACKBOARD. Bring your laptop computer to lab if you have one. Work with a partner.

Experiment 8 Gas Chromatography

Read pp. 83 – 91 in the lab text. Do Exercise J.3. Answer questions 3, 5, 11, and 12 on pp. 90 – 91 in your lab report.

Week of Feb. 20

Experiment 9 Synthesis of 1-Bromobutane

Read pp. 267 – 270 in the lab text. You will do Experiment 8.2, Part A. Do Prelab Assignments 1, 3, 4, and 5 on p. 268. Answer questions 2, 4, 5, and 6 on p. 270 in your lab report.

Week of Feb. 27

Experiment 10 Relative Rates of Nucleophilic Substitution Reactions

Read pp. 261 – 266 in the lab text. You will do Experiment 8.1. Do Prelab Assignments 1, 2, and 3 on p. 263. Work with a partner and do Parts A and B. Answer questions 1, 3, 4, 6, 7, and 8 on p. 266 in your lab report.

Week of Mar. 6

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.