

Organic Chemistry Laboratory

Fall 2011

303 Sullivan Hall

LABORATORY INSTRUCTOR

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Office hours: Monday, Tuesday, Wednesday 9:30–10:30, or by appointment

LABORATORY MANUAL

Schoffstall, A.M., Gaddis, B.A., and Druelinger, M.L. "Microscale and Miniscale Organic Chemistry Laboratory Experiments", 2nd Ed., McGraw-Hill, 2004.

LABORATORY PERIODS

Tuesday 1:15 – 5:00 p.m., Teaching Assistant: Travis Rosmus

Wednesday 1:15 – 5:00 p.m., Teaching Assistant: Seth Burkett

Thursday 1:15 – 5:00 p.m., Teaching Assistant: Selam Woldemeskel, Laura Smith

GOALS

The primary goals of the laboratory sessions are to teach experimental techniques and how to interpret data collected. Principles described in the lecture courses are illustrated in laboratory. An attempt has been made to select experiments which are not just recipes but offer some relevance to biology or everyday life.

Most of organic chemistry can be classified into structure determinations, reactions, mechanisms, synthesis of complex molecules, or theory. This course will illustrate techniques in all of these aspects. You will carry out several different organic reactions, employ techniques to identify products, and learn about spectroscopic methods used to identify them. It is intended that you will learn modern and relevant science and be graded on your technical and deductive skills. In the physical and biological sciences, learning to work with your hands is comparable in importance to the intellectual achievements.

TIPS FOR SUCCESS

1. Before coming to lab, have an outline in your mind of what should take place and have a "mental picture" of the equipment and materials to be used. Your lab notebook should contain an outline of the procedure (diagrams are helpful) and document all safety precautions and hazards.
2. Think about what is happening during the lab. There is a reason for every observation and every step in a procedure. If you don't understand, you are missing the point of the lab.
3. Work carefully; mistakes are often irreversible.
4. Work efficiently. You can often do two things at the same time. If your work is planned and you are thinking and working efficiently, you need not be rushed.
5. Choose a lab partner you feel comfortable working with and you trust.

NOTEBOOK

Scientific work can be no better than the records, which are kept of it. Consequently, you are required to keep a bound notebook as described on page 11 of the laboratory text. The lab notebook should be used prior to the experiment to organize the experiment and to summarize useful information. It should be used during the experiment to record quantities, describe observations (such as chemical appearance and change in state), and summarize operations carried out. All data are to be recorded in the notebook at the time they are obtained. There is no reason for recording on odd pieces of paper to be transcribed into the notebook later. Record your notes in ink (not pencil) and if corrections are necessary, cross out the incorrect information once then add the corrected data; never erase. Neatness is desirable, but it is less important than having an accurate and complete notebook. Notebooks should be available for inspection at any time.

The following general structure should be used for setting up the notebook:

1. Leave room at the beginning of the notebook for a Table of Contents, and keep it up to date.
2. Number the pages, if they are not already numbered.
3. Start new experiments on a fresh page; you may use both the left and right side pages.
4. Date your work (every page).

Each experiment should include:

1. Title
2. Objective
3. Detailed procedure (step by step instructions). Illustrations of glassware are helpful
4. Data Table
5. Observations
6. Calculations

GRADING POLICY

This semester contains 8 experiments, 1 post-laboratory discussion, and 2 spectral periods. The grading for the semester will be as follows:

Experiment 3.3 A	100 pt
Experiment 3.4 B	100 pt
Experiment 2 & 3	100 pt
Experiment 3.6 A or B	100 pt
Experiment 15	100 pt
Experiment 22	100 pt
Experiment 20	100 pt
Experiment 20 post lab discussion	25 pt
Experiment 7.1 A	100 pt
NMR	50 pt
spectral ID unknown	<u>50 pt</u>
	925 pt

Grading for each experiment is based on:

Prelab Discussion	10 %
Notebook	5 %
Lab preparation, technique (subjective)	10 %
Results (<i>e.g.</i> , product you synthesize)	25 %
Report	<u>50 %</u>
	100 %

REPORTS

Short reports (~3 pages) will be required. These must be submitted during the lab session following the completion of the experiment, unless specified. Late lab reports will be penalized gradually up to 1 week at which point a zero will be assigned for the report. Please beware of plagiarism. The reports should be written in the following general format:

1. Title: Experiment number and name, your name, your lab partner's name, course number and section, your instructor's name, and date due.
2. Purpose: Give a brief statement of the objective of the experiment.
3. Reaction: Write the chemical reaction performed in this experiment.
4. Procedure: Properly reference the procedure from the lab manual and state any significant deviations that would impact the outcome of the experiment.
5. Results and Observations:
 - Present your data and results as clearly and concisely as possible. Tables are often very useful here.
 - For the synthesis of a product, record the actual yield (in grams), the mole percent yield, and the physical properties (*e.g.* color, odor, m.p., b.p.) of the product.
 - Describe the starting material and note any changes in the solution at every step of the experiment.
 - Include starting material measurements required to calculate the percent yield.
 - Show all calculations.
 - Reference any literature data used for comparison or used in calculations (*e.g.* density, molecular weight).
 - If a value is less than one, put a zero in the ones place.
6. Discussion:
 - Restate the objective of the experiment.
 - How was the objective met? Briefly summarize the procedure.
 - Summarize the data.
 - Interpret the results obtained with respect to the purpose of the experiment (learning a new technique, demonstrating a principal, or synthesizing a compound). If the compound is characterized, how does your measurement compare to literature values? (www.sigmaaldrich.com and www.fishersci.com may be useful)
 - Discuss any nonhuman sources of error or improvements that could be made. Which steps in the procedure could lead to a product loss? Does the reaction go to completion? Was all of the reactant dissolved?
 - Answer the assigned critical thinking questions.
 - Compare your data with that of your lab partner or the class.
 - Do the results make sense? Why or why not?
7. Conclusion:
 - State in one sentence under what conditions the reactants were converted to products.
 - State the percent yield and the outcome of all tests performed on the product.

EQUIPMENTS AND CHEMICALS

Many experiments require special chemicals. Amounts of some chemicals are controlled. If bottles are removed from their central location, please return them promptly. Violation of this rule is unfair to your classmates and you may be penalized. Cleanliness in the lab is of paramount importance. A considerable part of your grade is subjective, and dirty equipment gives a bad impression, which will hurt your grade. All items checked out from the stockroom must be returned to the stockroom on the same day, unless you are given specific instruction to do otherwise. Many of the items are in short supply.

PRODUCTS

Products should be turned in as soon as the experiment is completed. Products that are not turned in promptly may be lost by spillage, flask breakage, evaporation, or contamination. Grading will be mainly on the basis of purity, as determined by the index of refraction and boiling point (for liquids) or the color, crystallinity and melting point (for solids), as well as spectral data when available. Each product should be collected in a parafilm enclosed test tube. A label should be written or printed neatly in ink. The reported product mass is taken prior to transfer to a test tube. A sample form follows:

n-Butyl Bromide
b.p. 100-101.5 °C
Yield 5.5 g
John Adams
date

MAKE-UP POLICY

Missed experiments can be made up only for valid reasons (*e.g.*, documented illness). In all other cases a zero will be assigned.

GRADES

Laboratory grades can be accessed through Blackboard. Any discrepancy between lab report grades and posted grades should be reported within 24 hours after receiving graded reports.

SAFETY

The safety practices outlined in the laboratory manual and on the dark yellow safety sheet should be closely followed. Bags should be kept outside the lab, leaving the bench tops and floor free. Safety goggles and appropriate clothing must be worn at all times when you are in the laboratory. You will be expelled from the laboratory if you fail to obey any safety rules.

HEALTH ISSUES

With the exception of prelab or postlab discussion sessions, all laboratory work must be performed while standing. If you have a health issue that precludes prolonged standing, please inform me immediately. Likewise, if you have any other medical conditions, such as pregnancy, that make you sensitive to chemicals, please inform me immediately. Prior accommodations will be made.

SEQUENCE OF EXPERIMENTS

August 30-September 1		Check-in, Introduction, Safety
September	6-8	Experiment 3.3 A Relationships between structure and physical properties
	13-15	Experiment 3.4 B Microscale recrystallization of an organic solid
	20-22	Experiment 2 & 3 (handout) Separating the components of "panacetin"
	27-29	Experiment 3.6 A or B Isolation of a natural product
October	4-6	Experiment 15 (handout) TLC analysis of drug components
	11-13	Nuclear Magnetic Resonance Spectroscopy (Handout)
	18-20	No lab (Fall Break)
	25-27	Identification of an unknown (NMR, IR, MS)
November	1-3	Minilab 22 (handout) The nylon rope trick
	8-10	Experiment 20 (handout) (do NMR) Reaction of iodoethane with sodium saccharin
	15-17	Experiment 20 postlab discussion/ make-up week
	22-24	No lab (Thanksgiving vacation)
November 29-December 1		Experiment 7.1 A Stereochemistry of alkenes
December	6-8	Check-out