Organic Chemistry Laboratory II
Spring 2012

303 Sullivan Hall

LABORATORY INSTRUCTOR

Paul Johns E-mail: pjohns@francis.edu
Office: Sullivan 313
Office hours: Tuesday & Wednesday 9:30–10:30, Thursday 1:30–2:30, 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: Michael DeLyser and Lauren McConnell
Wednesday 1:15 – 5:00 p.m., Teaching Assistant: Seth Burkert and Travis Rosmus
Thursday 1:15 – 5:00 p.m., Teaching Assistant: Ben Schultz and Laura Smith

GOALS

The primary goals of the laboratory sessions are to teach experimental techniques and data interpretation. 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. Several different organic reactions will be performed. Techniques will be employed to identify synthesized products, including spectroscopic methods. 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 a step, 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.

GRADING POLICY

This semester contains 6 experiments. The grading for the semester will be as follows:

Experiment 4.1	110 pt
Experiment 12.2A	110 pt
Experiment 22.1	130 pt
Experiment 15.1A	110 pt
Green Chemistry (Microwave experiment)	110 pt
Experiment 20.1	130 pt
Lab Practical	150 pt
	850 pt

Grading for each experiment is based on:

Prelab Discussion	10 pt
Notebook	5 pt
Lab preparation, technique (subjective)	10 pt
Results (*e.g.*, product you synthesize)	25 pt
Report	60 pt (80 pt for 2 week exp)
	110 pt (130 pt)

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 is used prior to the experiment to organize the experiment and to summarize useful information. It is 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 permanent **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 is 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 contains:

1. Title
2. Objective
3. Reaction
4. Detailed procedure (step by step instructions). Illustrations of glassware are helpful
5. Data Table
6. Observations
7. Copies of spectra
8. All calculations

REPORTS

Short reports are due before the lab period, one week after completion of the experiment, unless specified. Write in past tense without pronouns. Late lab reports will be penalized 20% per day, up to 1 week at which point a zero will be assigned for the report. Please beware of plagiarism. Reports are to be written in the following order:

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: Draw **by hand** the chemical reaction performed, including compound names.
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. Summarize numerical data in **tables**.
   - 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.
   - Calculate the limiting reactant
   - 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 (in this order):
   - Restate the objective of the experiment.
   - How was the objective met? Briefly summarize the reaction conditions.
   - 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 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? Be specific on the source of error and how it affects the results
   - Identify the product, based on product characterization.
   - Answer assigned questions.
   - Compare your data with that of your lab partner or the class, when necessary.
   - Do the results make sense? Why or why not?
7. Conclusion:
   - In one sentence, under what conditions the reactants were converted to products.
   - State the percent yield and the outcome of all product characterizations.
8. References:
   - Reference all sources used in the report

LAB PRACTICAL
The lab practical is a written exam that covers laboratory technique and procedure. You will be given laboratory scenarios then be required to explain mistakes in the procedure or how a specific step in the procedure was performed. Questions in the lab practical will have foundation in the experiments performed throughout the semester.

EQUIPMENT 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. Specialized equipment will be used throughout the semester. Incomplete cleaning of this equipment can lead to contamination and/or damage to the instrument. 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 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, contamination, evaporation, or flask breakage. Grading is based on purity, color, crystallinity, melting point, and spectral data. Each product will be submitted in a Parafilm® enclosed test tube with a label printed neatly in ink. Product mass is taken prior to characterization. All information must be present on the test tube label, written or printed neatly in ink, to receive full credit. A sample form follows:

n-Butyl Bromide
b.p. 100-101.5 ºC
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 are to be kept outside the lab, leaving the bench tops and floor free. Safety goggles and appropriate clothing must be worn at all times when in the laboratory. You will be expelled from the laboratory if you fail to obey any safety rules.

LAB PREPARATION
You must come to lab prepared to perform the assigned experiment. Unprepared students may be expelled from the laboratory.

HEALTH ISSUES

With the exception of prelab 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 or sensitivity to magnetic fields, that make you sensitive to chemicals, please inform me immediately. Prior accommodations will be made.

SEQUENCE OF EXPERIMENTS

January 10–12	**Check-in, Safety**
17–19	**Synthesis of an Alkyl Halide from an Alcohol (Experiment 4.1)**
24–26	**Relating Color to Structure: Synthesis of Azo Dyes (Experiment 22.1 A, B.1) (Handout)**
January 31–February 2	**Relating Color to Structure: Synthesis of Azo Dyes (Experiment 22.1 C, D, E) (Handout)**
February 7–9	**Nitration of Methyl Benzoate (Experiment 12.2 A)**
14–16	**Experiment 12.2 A characterization**
21–23	**Stereoselective Reduction of Ketones (Experiment 15.1 A)**
February 28–March 1	**Green Chemistry: Hydrolysis of a Nitrile (Handout)**
March 6–9	**No Lab—Mid-semester break**
13–15	**Combinatorial Chemistry and the Synthesis of Fruity Esters (Experiment 20.1 A)**
20–22	**Combinatorial Chemistry and the Synthesis of Fruity Esters (Experiment 20.1 B)**
27–29	**Lab Practical**
April 3–5	**No Lab—Easter vacation (makeup week)**
10–12	**Synthesis worksheet**
17–19	**Check-out**