

CHEMISTRY 25  
Organic Chemistry Laboratory  
Fall 2011

*Discussion Instructors*

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**Office Hours** Dr. Kellen-Yuen: T= 2:30-3:30 pm; W= 10-11:30 am; R= 1:30-2:30 pm  
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**Text-Required:** *Chemistry 25 Laboratory Manual printout (sold in bookstore)*  
**Texts-Recommended:** *Organic Laboratory Techniques, 3<sup>rd</sup> ed.*, Fessenden, Fessenden and Feist  
(ISBN 0 534-37981-8)  
Any standard Organic Textbook

**Required Materials:** Bound, lined, laboratory-grade notebook (preferably with page numbers), calculator, gloves and chemistry-grade laboratory goggles. A lab coat is highly recommended.

**Prerequisites:** Successful completion of Chem 1A, 1B, and 24 (C- or higher) and completion of or concurrent enrollment in Chem 124—NO EXCEPTIONS.

**Course Description:** This class is designed to give students hands-on experience with basic techniques used for the preparation, separation, purification and identification of organic compounds.

**Learning Objectives:** Students will learn basic organic laboratory techniques, which will be utilized to prepare, separate, purify and identify organic compounds. Students will also learn some instrumental techniques (e.g. gas chromatography, infrared spectrometry, and nuclear magnetic resonance spectrometry). Students will also be required to keep a laboratory-grade notebook, to write discussions summarizing their laboratory findings, and to familiarize themselves with safety information sources.

**Student Responsibilities:** Before starting any lab work, a student must familiarize him/herself with lab procedures, chemicals, equipment, hazards, safety procedures, and disposal procedures associated with the work being performed. If the instructor feels that a student is unprepared the student will be asked to leave. The student must also complete the pre-lab assignment or he/she will not be allowed to work in lab. There are no make-up labs. Students are required to properly clean their work area before leaving lab or their grade will be lowered.

**Attire:** Students will always come to the lab in clothing appropriate to a chemistry lab. This means long sleeves, long pants and shoes that cover the toes. This is the best way to protect yourself in the case of a chemical spill. A lab coat or lab apron is a wise investment since it also protects your clothing. **STUDENTS WILL WEAR CHEMICAL-GRADE LABORATORY GOGGLES AT ALL TIMES IN THE LABORATORY. THERE ARE NO EXCEPTIONS.** Any student who must be repeatedly warned to put on his/her goggles will be told to leave the class. **Contact lenses should never be worn in a chemistry laboratory.**

**Experiments:** The experiments to be performed this semester are listed below in the approximate order in which they will be carried out, along with the point values for each experiment. Points for each experiment are based on your pre-lab, laboratory report write-ups, and in-lab questions.

**Points    Experiment and Reading Assignment**

- 25    Melting Points, with Unknown  
Read: **Text:** pp. 1-22, 213-221, 39-48, and **Lab Manual:** **Experiment #1** and Appendix IV
- 50    Recrystallization of Unknown  
Read: **Text:** pp. 23-38, and **Lab Manual:** **Experiment #2**
- 50    Distillation (simple and fractional) and Gas Chromatography (GC)  
Read: **Text:** pp. 71-75, 77-92, 93-97, 107-110, 141-152; **Lab Manual:** **Experiment #4** and Appendix I
- 50    Dehydration  
Read: **Text:** pp. 141-152 and **Lab Manual:** **Experiment #9** and Appendix I;
- 50    Spectroscopy  
Read: **Text:** p. 163-194, and **Lab Manual:** **Experiment #3** and Appendix III
- 50    Extraction  
Read: **Text:** pp. 49-76, 163-178, 206-7, **Lab Manual:** **Experiment #6**, Appendix II and III
- 50    Column Chromatography  
Read: **Text:** pp. 119-131, 133-140, 153-162 and **Lab Manual:** **Experiment #5**
- 50    Redox Reactions of Aromatic Aldehydes  
Read: **Lab Manual:** **Experiment #14**, Appendix II and III
- 50    Grignard Synthesis of Benzoic Acids  
Read: **Lab Manual:** **Experiment #10**, Appendix II and III
- 50    Microwave Reactions of Ketones—Imine/Indole  
Read: **Lab Manual:** **Experiment #15**, Appendix II and III

**Experiment Points:** Your points for each experiment will be based on (1) a completed pre-lab written into lab notebook – see lab notebook section below; (2) lab report sheets which include results, calculations, spectral data, graphs, and purity/yield of chemicals synthesized in the lab; and (3) in-class post-lab questions answered when reports are turned-in. Synthesized compounds must be turned-in in a properly labeled vial (student name, date, compound name, weight, mp range). The pre-lab must be completed **BEFORE** the beginning of each experiment (at the time the lab begins, not 5 minutes later). *No student will be allowed to work in the lab without first completing the pre-lab.* The lab report sheets are due one week after the posted end date for that lab experiment. All lab reports are due at the **beginning** of the laboratory period in which they are due—**IF YOU COME TO LAB LATE, YOUR LAB REPORT IS LATE AS WELL.** Late materials will be accepted for grading only within one week of the due date or until graded material is returned to students (whichever comes first) unless prior permission of the instructor is obtained. Late material will lose 10% of its assigned value with each day it is late. Post-lab questions will be given on the day the lab report forms are collected and at the beginning of the lab period. If you come to lab late you will lose that time for answering the post-lab questions.

**Attendance:** Students are expected to attend all laboratory lectures (attendance will be taken). Students missing a lecture will lose 10% of the corresponding experiment grade for the lecture missed; students missing three lectures will fail the course.

Students who miss two or more lab sessions without a valid excuse will fail the class. **STUDENTS WILL WORK ONLY DURING THEIR ASSIGNED LAB TIME, AND MAY NOT WORK DURING ANOTHER LAB TIME** without prior written permission of all instructors affected by the scheduling change. Lab sections are full and cannot take overloads, therefore make-ups will be allowed only under extreme circumstances and will not be given for students who are simply slow in finishing their work. Students who are late to lab pose a safety hazard to their lab mates, since safety instructions are given in the first few minutes of the lab. Students who are chronically late (four or more times) will be asked to leave the lab and not allowed to make up the time. No materials, chemicals, or supplies may be removed from the lab without permission of the instructor.

**Exams:** There will be two exams given. The first will be given about half way through the course during the discussion/lecture hour. There will also be an exam given during finals week (see schedule below).

**Grading:** Your grade for the course will be based on your experiment points, exams, your laboratory notebook (including a hazard table), and your laboratory technique throughout the course of the semester (see below for instructions on keeping a laboratory notebook).

Lab book and technique	50
Hazard Table	50
Review Problems	25
Experimental points	425
Exams (2)	300
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Total	850 pts.

Letter grades are assigned based on a range of: A to A- = 90.0% and above, B+ to B- = 89.9-80.0%, C+ to C- = 79.9-70.0%, D = 69.9-60.0%, F = below 60.0%

**Cheating:** Cheating in any form is not tolerated in this class. A student caught cheating will receive a zero on that experiment/exam. If a student is caught a second time, the student will fail this course. Cheating includes: copying from another student's report (**yes, even lab partners must have their own unique answers**), **copying material or data from a previous report (your own or someone else's)**, turning in lab work done during a previous semester, reporting data for an experiment you did not perform, using extra materials during testing, programming data into a calculator, having other people take tests for you, altering exams after they have been graded, etc.

**Cleanliness in Lab:** Due to problems encountered in previous classes, students are warned that they are directly held responsible for the cleanliness of the lab, since messy labs pose hazards to the students and add costs to the department in terms of clean-up time and wasted chemicals. Therefore the instructors will periodically walk through ALL lab sections and can fine every student in the section up to 5 points per day if the laboratory room is found to be messy or unsafe. Students are encouraged to remind each other to clean up their areas to avoid losing points. If any student notices that one of their lab mates is not following the rules, they should report this to the lab instructor immediately. Instructors will require that all students clean up the lab before leaving for the day.

**Laboratory Notebook:** (See Fessenden and Fessenden pp. 8-22, 197-202 or any lab text for reference). The correct notebook for the lab is a hardcover, bound notebook containing lined pages. A loose-leaf or spiral notebook is not satisfactory because pages are easily removed and lost. A separate notebook should be used for each laboratory course. If the pages are not numbered, number them before using the book. Pages should never be stapled in or otherwise added to the laboratory book. Make sure you write your name, address, and telephone number on the inside cover, in case it is lost. Record your locker number and combination of your locker in your lab book or in some convenient place. Leave two pages at the front of the lab book for a table of contents. Then enter your experiments consecutively. All entries will be recorded in permanent ink, and data is to be entered as it is collected—never transferred from a “temporary paper”. NEVER ERASE IN A NOTEBOOK. If an error is made, ONE line is drawn through the mistake. DO NOT SCRATCH OUT ERRORS AND DO NOT RIP OUT PAGES FROM THE LAB BOOK. Points will be deducted for illegible notebooks; however we do understand that the only "perfect" lab book is one that has been copied. Make sure your work is neat and easily followed.

The format for writing experiments in your laboratory notebook is as follows:

Date-date experimental work performed. NEVER FORGET TO DATE YOUR WORK—in the real world this can mean the difference between getting a patent and losing it to someone else.

Title – name of experiment

Reaction Equations – you should write a complete and balanced equation for any reaction taking place in the experiment performed. **Under each of the starting materials you should list the molecular weight of each compound, the amount you intend to use in the experiment, and space to write in the actual amount used and the number of moles this represents.**

Procedure Outline – the outline should include sufficient detail to carry out the experiment without referring to the original experiment in the book. DON'T COPY WORD FOR WORD, (a waste of time!!) and long paragraphs make it hard to quickly determine your next step.

**\*\*Your lab instructor will check your lab book for these four sections (this is your completed pre-lab assignment) before you are allowed to begin any experiment.**

Observations and Experimental Data – Observations include words describing the experiment: color changes, phase changes, nature of the products, sudden volcanic eruptions or lack thereof, observed temperatures, etc; what you did a little differently than the original procedure. Also give any factors that might have influenced the outcome of the experiment, or reasons why you chose one method over another (why one solvent and not another, particularly important in recrystallization and purification experiments). Include as data values you collected during the experiment in a form that makes it easy for you to follow, for example, in Experiment #1 a table with six columns would work well: (1) the name and (2) the structure of the compounds, (3) reagent bottle melting points, (4 & 5) experimental data (two trials), and (6) the average melting point. Data also includes things like dates, exact weights, product yields/calculations, and mp data.

Conclusions – a Brief analysis of the outcome of the experiment, for example: name and structure of the identified unknown, calculation for yield of a product (**always show calculations**), physical constants measured in an experiment, purity found by analysis, a comparison of observed versus literature values for physical properties written as a 2-3 sentence summary.

**Laboratory Safety:** (See Fessenden and Fessenden pp. 1-8, 213-221 or any lab text for reference). Since most organic compounds are potentially volatile, flammable and toxic either alone or in combination, a student must become familiar with the chemical and physical properties of all the substances with which they work. These properties can be found in the MSDS (Material Safety Data Sheet), which is available on the Internet web sites of many companies (Acros, Baker, Sigma-Aldrich) and Universities (Cornell, Vermont SIRC, Oklahoma State University). Cambridge Scientific sponsors a ChemFinder web site. The MSDS's are on file in the service center, but for your convenience it would probably be better to look them up on a computer for yourself. In the CSUS Library web page under the Chemistry databases you will find a link to many of these on-line MSDS sites.

You will devote the last 10-15 pages of your lab book to listing the following types of information about chemicals used in the lab. (You only need to list a chemical once, even if it is used in many different experiments - plan for about 80 chemical citations). Keep the MSDSs for each experiment on a separately labeled page for easy reference when checked by your instructor. Turn your book sideways and make a table with the following columns:

1. Chemical name
2. Structure (this is NOT a formula)
3. Melting point for solids / Boiling points for liquids
4. Density for liquids
5. An oral LD<sub>50</sub> (lethal dose with 50% kill rate)
6. Exposure Dangers – is it a carcinogen or potential carcinogen? Something worse?
7. Incompatibilities
8. Other Notes (if you find something else you want to remember about the compound)

**Special Needs:** Students requiring special accommodations should see the instructor ASAP. Students who are pregnant should consult with the instructor and their physician before undertaking any lab work.

#### Tentative Laboratory Work Schedule

Week of:	Lecture		First Lab (M/T)	Second Lab (W/R0)
8/29	Intro/MP		<b>No Lab</b>	Orientation/Review
9/5	MP		<b>No lab-Labor Day (Monday)</b>	Check in / Melting Point
9/12	Recrystallization		Melting Point	Recrystallization
9/19	Distillation		Recrystallization	Distillation and GC
9/26	Dehydration		Distillation and GC	Dehydration
10/3	Spectroscopy		Dehydration	Spectroscopy
10/10	Extraction		Spectroscopy	Extraction
10/17	Chromatography		Extraction	Column Chromatography
10/24	<b>Exam 1</b>		<b>No Lab</b>	Column Chromatography
10/31	Redox		CC and Redox	Redox
11/7	Grignard		Redox	Grignard
11/14	Advanced NMR		Grignard	Grignard
11/21	Imine/Indole		<b>No Lab</b>	<b>No Lab-Thanksgiving</b>
11/28	Review		Imine/Indole	Imine/Indole
12/5			Clean-up and Check-out	

**Final Exam:** Monday, Dec. 12<sup>th</sup> 12:45 pm – 2:45 pm for Monday lecture class  
 Thursday, Dec. 15<sup>th</sup> 12:45 pm – 2:45 pm for Tuesday lecture class