

Department of Chemistry
University of Missouri-St. Louis

Chemistry 263: Techniques of Organic Chemistry

Fall 1997

Lecture Tuesday 1:00-1:50 PM; Room B115

Laboratory Wednesday; Thursday 12:30-5:00 PM; Room B401

Instructor

Dr. David Garin

Office: 315f Benton Hall

Phone: 516-5311

Teaching Assistant

to be announced

Text and Supplies

J.W. Zubrick, **The Organic Chem Lab Survival Manual**; 4th ed, Wiley & Sons, 1997;

Laboratory Manual for Chemistry 263, August 1997 (available in book store);

Laboratory Notebook: **must be hard bound** (loose-leaf or spiral-bound notebooks are **not** acceptable)

Safety Glasses: These are available in the bookstore. Safety glasses must be worn in the laboratory at **all** times.

Course Description

Chemistry 263 is the first semester in the U.M.-St. Louis Organic Chemistry Laboratory sequence. The formal prerequisite for this course is Structural Organic Chemistry, Chemistry 261 (or an equivalent lecture course).

This course is designed to provide an introduction to the basic techniques and procedures of Organic Chemistry, thus furthering your understanding of the fundamentals of this science. It is assumed that you are acquainted with general techniques employed in the laboratory, such as weighing, measuring volumes, preparing solutions, determining temperatures, etc. You should also be familiar with the basics of Organic Chemistry including nomenclature, structural theory and the reactions of common functional groups. Familiarity with the spectroscopic properties of organic compounds would also be helpful.

Evaluation of Performance

Your final letter grade will be based on your accumulated point total on a 800 point scale in the following areas:

- I. Notebook grades (250 pts.).
- II. Grades on yields and unknowns (250 pts.).
- III. Homework, examinations, and other written assignments (200 pts.).
- IV. Laboratory performance, including instructor evaluations (100 pts.).

The examinations will cover material from the lectures, laboratory experiments, and reading assignments. You are responsible for the material covered in the reading assignments, even if not specifically covered in the lectures.

Letter grades are primarily assigned on the basis of your accumulated point total relative to the class performance as a whole. Therefore, the exact number of points or percentage required for each of the various letter grades varies and will not be known until near the end of the course.

LECTURE SCHEDULE

August 26

Introduction to Chemistry 263
Safety in the Laboratory
The Laboratory Notebook
Basic Laboratory Techniques: Heating and Filtration
Purification and Characterization of Organic Solids

Assigned Reading for this week: Laboratory Safety, Z pp. 1-9; Advance Preparation and Laboratory Records, Z pp. 12-24; Laboratory Glassware, Z pp. 46-58; also read the corresponding pages in the Manual, M pp 1-9.

September 2

Purification and Characterization of Organic Solids (continued)
Preparation of Aspirin

Assigned Reading: Aspirin, see Manual, pp 18-19; Techniques: Solvents and Methods of Heating Reaction Mixtures, Z pp. 172-178, 182-192; Technique: Crystallization and Filtration, Z pp. 122-138; also read Z pp 90-91 and 98-99; 102-115 (melting point) and the corresponding pages in the Manual, pp 10-17.

Homework: Manual, p. 20, questions 1-4. All homework assignments will be due during this week's lab session unless otherwise specified.

September 9

Acetaminophen; Extraction
Introduction to Chromatographic Techniques

Assigned Reading: Acetaminophen, see Manual pp 21-22; Technique: Extraction, The Separatory Funnel, Drying Agents, Z pp. 148-163, 170, 94-95, 374-376 and the corresponding pages in the Manual, pp 15-17.

Homework: Manual p. 17a, questions 1-9

September 16

Isolation of Caffeine from Tea (Manual pp 23-24)
Thin Layer Chromatography (we will use commercially prepared TLC plates, see Manual, p 23)

Assigned Reading: Chromatography, Z, pp. 252-267; Sublimation, Zpp. 242-243.

Homework: tba

September 23

Purification and Characterization of Liquids
Simple and Fractional Distillation; Steam Distillation

Assigned Reading: Z, pp. 194-220 and 352-372; see Manual, pp 28-37

September 30

Distillation theory; Simple and Fractional Distillation
Infrared Spectra of Organic Compounds, Group Frequencies; Banana Oil (see M, pp 26-27)

Assigned Reading: IR, Zubrich: 318-326, 333-338; see Manual pp 54-80 and Appendix

Homework: Manual, pp. 26-27, Questions 1-6

October 7

Infrared Analysis

Assigned Reading: as above;

Homework: Manual, p. 40, Questions 1-5

October 14

Introduction to Gas Chromatography/GC Methods for Analysis
Identification of Organic Unknowns by Methods of Qualitative Analysis
Functional Group Classification of Organic Compounds

Assigned Reading: GC analysis Z: pp. 290-299; Micro bp, neutralization equivalent (Z: pp 246-247; see Manual, p. 48)

Homework: handout

October 21

EXAM, part 1

October 28

Identification of Organic Unknowns by Methods of Qualitative Organic Analysis

Assigned Reading: Manual, pp 42-48

November 4

Multistep Synthesis

Assigned Reading: Manual, pp. 49-53

November 11

Multistep Synthesis

Functional Group Classification of Organic Compounds, Review

Assigned Reading: Manual, pp 42-53

November 18

Identification of Unknowns continued

Spectroscopic Methods for Identification

November 25

EXAM, part 2

December 2

NMR Spectroscopy; other topics

SCHEDULE OF EXPERIMENTS

week of August 25th

Introduction to the Organic Laboratory; Laboratory Safety; Laboratory Check-in;
Determination of Melting Points; Use of balances

week of September 1st

Preparation of Acetylsalicylic Acid (Aspirin); see Manual pp. 18-19

week of September 8th

Preparation of Acetaminophen; see; Manual pp. 21-22

week of September 15th

Isolation of Caffeine from Tea; we will do a modified sublimation (to be described) and a caffeine salicylate derivative; see Manual pp 23-24.

Your notebook will be collected at the end of the lab and the Aspirin and Acetaminophen experiments are due at this time

week of September 22nd

TLC Analysis of Analgesic Drugs (Unknown) ; see Manual p. 25

week of September 29th

Preparation of Isopentyl Acetate (Banana Oil); Infrared Spectrum (neat); see Manual pp. 26-27

Your notebook will be collected at the end of the lab and two additional experiments (caffeine and T.L.C.) are due at this time.

week of October 6th

Hydrolysis of Methyl Salicylate; see Manual pp. 38-39

week of October 13th

Demonstration of Fractional Distillation (see below*); Gas Chromatography, Unknowns - begin identification of unknown organic compounds using spectroscopy and other methods; Manual p. 41

week of October 20th

Analysis of simple and fractional distillation

Gas Chromatography

Identification of Organic Compounds; see Manual pp. 42-48

week of October 27th

Unknowns; Identification of a solid and liquid unknown

Start of Benzoin synthesis, see Manual pp 49-50

Your notebook will be collected at the end of the lab and banana oil, methyl salicylate, fractional distillation/glc experiments are due at this time.

week of November 3rd

Multistep Synthesis: Preparation of Tetraphenylcyclopentadienone

Step 1. Synthesis of Benzoin; see Manual, p. 49-50

Continue Identification of Unknowns

week of November 10th

Multistep Synthesis: Preparation of Tetraphenylcyclopentadienone

Step 2. Oxidation of Benzoin to Benzil; see Manual, p. 50-51

week of November 17th

Multistep Synthesis: Preparation of Tetraphenylcyclopentadienone

Step 3. Synthesis of Tetraphenylcyclopentadienone; see Manual p. 51

Reduction of Benzil; see Manual, p. 52

week of November 24th No labs this week due to THANKSGIVING vacation

week of December 1st

Catch up and Check-out

Notebooks are due at this time with all experiments completed including unknown reports.

December 10th is the last day of classes

*Simple Distillation and Fractional Distillation

The simple and fractional distillation experiments will be set up as demonstrations and each student will obtain samples of the distillate to analyze by GC. Store the fractions in tightly closed vials to prevent evaporation.

At the beginning of this period, sign up for use of the GC. In this experiment you will analyze the cyclohexane-methylcyclohexane mixture obtained from the simple and fractional distillation. You will measure the composition of the fractions obtained by methods to be discussed. After GC analysis, the cyclohexane-methylcyclohexane fractions may be recycled.

Disposal of Wastes

Solvent wastes: Solvent wastes are not to be poured down the drain. Instead receptacles will be available in the laboratory to contain the waste solvents that you generate. A carboy will be available for your wash acetone as well as receptacles for halogenated and non-halogenated solvents. Aqueous solutions that do not contain heavy metals can probably be discarded in the drain. Additional information will be provided during the laboratory period. If you are in doubt, ask the instructor for assistance.

Solid wastes: Most of the solid wastes you will generate during the semester are non-toxic and can be disposed of in the trash receptacles. Most of your products will be collected. Heavy metal precipitates and similar toxic solids will be collected in special receptacles that will be appropriately labeled. Additional information will be provided during the laboratory period. If you are in doubt, ask the instructor for assistance.

Broken glass: Special receptacles are available for broken glass (large cardboard boxes). DO NOT put anything into these glass receptacles except glass. There are different trash receptacles for paper, old labels, etc.

Cleaning Glassware

Washing glassware: When time allows, wash your dirty glassware with soap and water using the brushes in your kit and the soap at your sink. If a residue persists, try to dissolve it with a minimal amount of acetone, usually 5 mL or less. If that does not work, ask your instructor for suggestions.

Drying Wet glassware: The oven in the rear of the lab is set to approx. 80-100°C. Wet glassware will usually dry in that oven in 15-20 min. CAUTION: hot glassware can burn your fingers--use the glove(s) provided. In those cases where you need the glassware sooner, you can "rinse out" the residual water by adding approx. 5 mL of acetone, swirling to mix the water with the acetone, draining the wash acetone, then placing the glassware in the oven (5 min) or allowing it to drain dry at your desk. DO NOT blow air into the glassware.

IMPORTANT: Keep acetone use to a minimum.