Chemistry 30475 – Organic Chemistry Laboratory – Fall 2017

Dr. Clarke W. Earley
Kent State University Stark Campus
421 Main Hall
Phone: (330) 244-3421
email: cearley@kent.edu
Web site: https://delta.stark.kent.edu/chemistry/

Dr. Earley's Schedule				
	M	**T**	**W**	**R**
9:00am		↑		
9:30am		Organic		
10:00am		Chemistry		
10:30am		I		
11:00am		Lab		
11:30am		↓		
12:00pm		↑		
12:30pm		*Office*		
1:00pm		*Hours*		
1:30pm	*Office Hrs*	↓	*Office Hrs*	*Office Hrs*
2:00pm	Organic	Organic	Organic	Organic
2:30pm	I	I	I	I
3:00pm	*Office Hrs*	*Office Hrs*	*Office Hrs*	*Office Hrs*
3:30pm	Molecules		Molecules	
4:00pm	of Life		of Life	

Additional office hours available by appointment

Required Materials

- **Manuals**: You must download two files from Blackboard Learn titled "Background" and "Experiments". It will be necessary to print a copy of the "Experiments" manual and bring this to lab. While you will need to refer to information in the "Background" manual, this reference does not need to be brought to lab.
- **Required Supplies**: *Organic Laboratory Notebook, Safety Goggles*

Prerequisites

Students are expected to have successfully completed the equivalent of one year of college-level General Chemistry, which will typically be CHEM 10060 and 10061. In addition, students must either have successfully completed or concurrently be enrolled in either Basic Organic Chemistry (CHEM 20481), Organic Chemistry (CHEM 30481), or an equivalent course.

Attendance

Attendance in laboratory is required. Unless you have made other arrangements with me, you may not turn in a laboratory report if you were not present to complete that experiment. While a small amount of flexibility is possible to allow makeup experiments, this should only be used for extraordinary situations. Safety goggles are required and must be worn at all times while in the laboratory. If you are unable to attend laboratory during the regularly scheduled time, you must contact Dr. Earley BEFORE lab. Excused absences will only be given for legitimate, documented excuses. Students are expected to be present at the start of each laboratory period. Students repeatedly arriving to lab late will receive a deduction on their laboratory report scores.

Course Description

Catalog Description: *Practical experience in synthetic methods and separation techniques. Spectroscopy applied to organic chemical problems. Multistep synthesis.*

This course provides an introduction to basic laboratory techniques and synthetic procedures of use to practicing organic chemists. Both theoretical and practical applications of spectroscopic (IR, NMR, and UV/Vis) and chromatographic techniques will be included.

Learning Outcomes

Upon successful completion of this course, students will be able to:

1. Recall all important aspects of laboratory safety including correct handling and disposal of a wide variety of corrosive and organic/inorganic materials.
2. Carry out fundamental laboratory procedures including melting point and boiling point determination, recording of infrared spectra, crystallization, sublimation, distillation (both simple and fractional techniques), extraction, thin-layer chromatographic analysis, preparative silica gel column chromatography, and use of gas chromatography to analyze a product mixture.
3. Use fundamental techniques learned above to carry out, analyze, and work up organic reactions.
4. Relate theoretical concepts to laboratory practice.

Grading

Grades for this course will be based on laboratory reports, quizzes, and one exam. Specific guidelines for each of the laboratory reports are given below. Point values and due dates for each of these laboratory reports are given in the "Tentative Schedule" section near the end of this syllabus. Late laboratory reports will be accepted, but will be penalized 1 point per weekday (Mon-Fri) late.

Copies of relevant laboratory notebook pages must be turned in at the end of each laboratory period and will be considered part of each report. These notebooks are not expected to be models of neatness and beauty, but must be legible and include all data obtained in lab (quantities of reagents, yields, mp, etc.).

In addition to the laboratory reports, four (4) unannounced prelab quizzes worth 5 points each and one final examination worth 25 points will be given. These quizzes will focus on basic information relevant to that day's experiment that students should know before coming to lab and/or on material from previous labs. The lowest quiz score will be dropped. Dates for all laboratory experiments and the exam are given in the lecture outline below.

Grades will be based on the scale shown below and will not be curved or arbitrarily adjusted in any manner. Extra credit will not be given.

Grading Scale

Laboratory Reports		195 pts
Best 3 Prelab Quizzes	3 x 5 pts	15 pts
Laboratory Exam	20 pts	20 pts
Total		**230 pts**

Grade	**A**	**B+**	**B**	**B-**	**C+**	**C**	**C-**	**D**	**F**
%	90-100%	88-89%	82-88%	80-81%	78-79%	72-78%	70-71%	60-70%	<60%

Laboratory Procedures

Bound laboratory notebooks containing carbonless, removable pages are required and can be purchased from the bookstore. To encourage the most efficient use of lab time, students are generally required to write important information (balanced equations, names and amounts of reagents required, theoretical yields, ...) in these notebooks before attending lab. Near the beginning of each lab period, I will check to verify that these procedures have been written out, and this information will become part of the laboratory report grade. Copies of these notebook pages must be turned in at the end of each laboratory period.

Quizzes and Exams

Quizzes and the exam will be designed primarily to test understanding of the chemical reactions performed in lab. These will NOT focus on experimental procedures (*ex. How many milliliters of dichloromethane were used to extract caffeine from tea leaves?*), but rather will emphasize general principles (*ex. Why was dichloromethane used to extract the caffeine?*).

Pregnancy

There are very few studies on the potential hazards of most of the chemicals we use in the laboratory toward unborn children. Organic compounds, particularly volatile organic solvents, have the potential of causing harm. While you will be taught how to access safety data sheets for all of the compounds we use in lab, in most cases this will yield little to no useful information for pregnant mothers. *The safest option is to not be in the laboratory if you are pregnant.* If you choose to stay in the laboratory, be aware that there are potential risks for your child.

University Policy/General Information

Information on various University policies (Academic honesty, Students with disabilities, etc.) and other general information (email accounts, posting of grades, etc.) is posted on the course website. This information should be considered as part of this syllabus and is available at:

https://delta.stark.kent.edu/chemistry/KSU/UniversityPolicy

General Information for Laboratory Reports

Laboratory reports will generally summarize either a single experiment or a series of related laboratory experiments. In general, complete sentences must be used for all reports. Spelling, grammar, etc. will be taken into account when these are graded. Students having trouble are encouraged to make use of the Writing Center.

The format of these reports will vary considerably depending on the laboratory activities covered and the point in the semester. In general, laboratory reports will start out as fairly simple summaries, but should advance toward the goal of producing a formal report in a format suitable for submission to a scientific journal. **Chapter 3 of the "Background" manual contains important additional information to assist with preparation of your reports.**

Requirements for each of the individual reports are summarized below. In addition to these requirements, more specific guidelines for reports may be provided in the experimental procedure in your laboratory manual or in the pre-laboratory lecture.

Tentative Schedule and Requirements for Laboratory Reports

All dates listed below are tentative and are subject to change. Note that lab does not meet during final exams week.

Introduction

Date		Report Due
Aug 29	Lab Safety, Check-in, Melting Points (1.3)	Sep 12
Sep 5	Scientific Literature (1.1), Drawing Molecules	Sep 12

Note: Lab will NOT meet on Tuesday, September 5.

- **Melting Points** (15 pts) - Write a report modeled after the "Results and Discussion" section of a scientific paper using the guidelines in Chapter 3 of your "Background" manual. (Do not include experimental procedures, introduction, etc.). Summarize all of the requested data, and include a graph showing your results. Including a table to summarize data is appropriate in this section of a scientific paper, and this should also be part of your report.
- **Scientific Literature** (5 pts) - Everyone will be assigned a compound during the first laboratory period. For your report, you need to turn in a copy of the first page (or the full article) of a recent scientific paper describing the synthesis of this compound or a closely related derivative. Alternatively, you can send me a copy of the article via e-mail.
- **Drawing Molecules** (5 pts) - Create computer-generated drawings of 2,2-dimethyl-4-hexanone and 4-aminobenzoic acid.

Laboratory Techniques

Date		Report Due
Sep 12	Column Chromatography (2.1)	Sep 19
Sep 19	Trimyristin from Nutmeg (2.2)	Oct 3
Sep 25	Fractional Distillation (2.3)	Oct 10

- **Column Chromatography** (15 pts) - Write a report modeled after the "Results and Discussion" section of a scientific paper. The emphasis of your report should be on the success of your procedure for separating and purifying the two dyes used. Include a table of R_f values obtained from the TLC experiment and discuss why you choose the solvent for column chromatography. For the column chromatography, report the colors of the fractions obtained and include a table summarizing the UV/Visible spectroscopy results.
- **Trimyristin from Nutmeg** (15 pts) - Write a report modeled after the "Results and Discussion" section of a scientific paper. The emphasis of this report is to explain the steps used to isolate the product. This should include a discussion of the structure and properties of the isolated product, with an emphasis on the observed/expected solubility of the compound in the solvents used in this experiment. You should also comment on the amount of fat in the nutmeg and the results of the crystallization.
- **Fractional Distillation** (15 pts) - Write a report modeled after the "Results and Discussion" section of a scientific paper. The emphasis of this report should be on the success of your distillation for separating the two solvents. In addition to reporting the absolute volumes and %recovery for each solvent, you will need to include your IR spectra and explain how this relates to the purity of your product.

Separation and Characterization of Unknown Solids

Date		**Mini-Report Due**
Oct 3	Extraction of Unknown Solids (3.2)	Oct 17
Oct 10	Crystallization of Unknowns	Oct 24
Oct 17	Titration (3.3) and Melting Points	Oct 31
Oct 24	IR and NMR	*No report*

Unknown Solids Full Report Due November 7

At the end of this project, you will be required to submit a laboratory report summarizing all of the information obtained in the format of a full scientific paper. Information to help prepare this report is described in Chapter 2 of your "Experiments" manual and Chapter 3 of the "Background" manual. In addition to this formal report, summaries of data collected during the course of this project (mini-reports) as described below will need to be submitted.

It will be necessary to obtain IR spectra for both of your solid unknowns. ^{1}H NMR spectra will be provided for both of your unknown solids.

- **Liquid-Liquid Extraction** (5 pts) - Turn in a short (1 page) report summarizing the results of this experiment. This report should include the unknown code (ex. S25), the yield (mass) of each crude solid, the calculated total % recovery, and the melting points for each solid.
- **Crystallization** (5 pts) - This report should include the results of the solubility tests for each solid component, the yield (mass) of the crystallized compounds, and the % recovery for each (% recovery = $\frac{\text{mass}_{\text{crystals}}}{\text{mass}_{\text{crude solid}}}$ x 100%). A description of the form of the crystals obtained (blocks, needles, ...) should also be included. This mini-report is due two weeks after starting this experiment.
- **Titration and Melting Points** (5 pts) - Turn in a short (1 page) report summarizing the results from these experiments. For the melting points, simply include the melting point ranges for each solid. For the titration, include all experimental data (conc. of NaOH, mass of solid used, etc.).
- **Unknown Solids Lab Report** (40 pts) - This report should be in the format of a full scientific paper. Review chapter 3 of the Background Information manual. This report should include copies of all printouts (IR, NMR, etc.). The focus of this report should be on the identities of the two components in your unknown mixture. Pictures of the chemical structures of each of these must be included in your report. In addition, complete analysis of all of your IR and NMR data should be included and related to these structures. Interpretation of these spectra will be an important part of the characterization data reported in the Results and Discussion section of your report. Any discrepancies between this data and your proposed structures should be explained. Comparison of experimental and literature melting points should also be included.

Organic Reactions

Date		**Report Due**
Oct 31	S_N2 Reactions (4.1)	Nov 14
Nov 7	Preparation of Cyclohexene (4.2.1)	Dec 5
Nov 14	Preparation of Bromocyclohexane (4.2.2)	
Nov 21	Preparation of Cyclohexanone (4.2.3)	
Nov 28	Preparation of Acetyl-glucose (4.3)	Dec 8
Dec 5	Check-out, Final Exam	—

- **S_N2 Reactions** (15 pts) - Write a report modeled after the "Results and Discussion" section of a scientific paper. In your report you need to classify each of the alkyl halides used and tabulate the times it took for precipitation to occur for each set of reactions. These results should be analyzed to explain (i) how the nature of the alkyl group affected reactivity, (ii) how the nature of the leaving group affected reactivity, and (iii) how the concentration of reactants affected reactivity. Discuss how the observed results compared with those predicted. Note that when analyzing this data, some sets of data will make much more useful comparisons than other sets. For example, comparing 1-bromobutane with 2-bromobutane should be very instructive, but comparing 1-bromobutane with 2-chlorobutane would be much less useful.

- **Reactions of Cylohexanol** (40 pts) - Note that this report covers three separate experiments and should be written in the format of a full scientific paper. Your report should include all data obtained (yield, %yield, and melting points) and discuss characterization (including spectral analysis) and purity of the products obtained. The introduction should include background information on the types of reactions of alcohols used. Your discussion should indicate why these three experiments were combined into a single report and how the experimental conditions used led to synthesis of the different products obtained.

- **Preparation of Acetyl-glucose** (15 pts) - This report is to be modeled after the "Results and Discussion" section and should include the yield, % yield, and melting point of your product. In addition, you should show the mechanism for synthesis of this product and describe suitable reaction conditions that would allow conversion of the acetyl-glucose product back into α-D-glucopyranose.