Chemistry 30476 – Organic Chemistry Laboratory – Spring 2017

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Dr. Earley's Schedule				
	M	**T**	**W**	**R**
9:00am				
9:30am				
10:00am			Organic	
10:30am			Lab.	
11:00am		*Office*		*Office*
11:30am		:		:
12:00pm		*Hours*	*Office*	*Hours*
12:30pm		Basic	*Hours*	Basic
1:00pm		Org II		Org II
1:30pm		*Office Hrs*		*Office Hrs*
2:00pm		Molec.		Molec.
2:30pm		of		of
3:00pm		Life		Life
3:30pm		*Office Hrs*		*Office Hrs*
4:00pm				

Additional office hours available by appointment

Required Materials

- Required: *Organic Laboratory Notebook, Safety Goggles, Calculator, Handouts (posted on Blackboard Learn).*

Prerequisites

Students are expected to have successfully completed the equivalent of one semester (one credit hour) of Organic Chemistry Laboratory (typically CHEM 30475). In addition, students must either have successfully completed or concurrently be enrolled in either Basic Organic Chemistry II (CHEM 20482), Organic Chemistry II (CHEM 30482), or an equivalent 2^{nd} semester Organic Chemistry lecture course.

Course Description

This course is a continuation of Organic Chemistry Laboratory I (CHEM 30475). In this semester, a greater emphasis is placed on multi-step organic synthesis and analysis of products using instrumental (HPLC) and spectroscopic (UV/Vis, IR, and NMR) techniques.

Learning Outcomes

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

1. Recall and demonstrate all important aspects of laboratory safety including correct handling and disposal of a wide variety of corrosive and organic/inorganic materials.
2. Carry out multi-step organic reactions.
3. Analyze and identify products of organic reactions using techniques introduced and learned in Organic Chemistry Laboratory I (CHEM 30475)

Attendance

Attendance in laboratory is required. Students who are habitually late for lab may receive a deduction on their lab report scores. 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.

Grading

Grades for this course will be based on laboratory reports and quizzes. 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 exam worth 20 points each will be given. These quizzes will focus on basic information from previous experiments or information relevant to that day's experiment that students should know before coming to lab. The lowest quiz score will be dropped. The exam will cover all material presented over the course of the semester. Dates for all laboratory experiments and the exam are given in the lecture outline below. Grades will be based on the scale shown below. Grades will not be curved or arbitrarily adjusted in any manner, and extra credit will not be given.

Grading Scale		
Laboratory Reports		210 pts
Best 3 Prelab Quizzes	3 x 5 pts	15 pts
Laboratory Exam	1 x 20 pts	20 pts
Total		**245 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. Copies of all notebook pages related to each laboratory report must be turned in by the due date for each report. For several labs, calculations (typically grams $\leftrightarrow$ moles) are required, so it is recommended that a calculator be brought to lab.

Quizzes and Exams

Quizzes and exams will be designed primarily to test understanding of the chemical reactions performed in lab. These will not focus on the details of experimental procedures (*How many milliliters of dichloromethane were used to extract caffeine from tea leaves?*), but rather will emphasize general principles (*Why was dichloromethane used to extract the caffeine?*). There is no final examination for this laboratory course.

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 have access to 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.

Office Hours

Office hours are listed near the top of this syllabus. If you would like to meet with me outside of these normal times, see me before or after class (or email or call) and we can set up additional time to meet.

University Policy/General Information

I have posted a page containing various University policy statements (Academic honesty, Students with disabilities, etc.) and other general information (email accounts, posting of grades, etc.) on my website. This information should be considered as part of this syllabus and is available at:

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

General Information for Laboratory Reports

All laboratory reports must be submitted as Adobe PDF files through the Assignment page on Blackboard Learn. Details for using this site to submit these reports will be discussed in the first laboratory period. Unless otherwise noted, these reports should be modeled after a complete scientific paper. All sections (Introduction, Experimental, Results and Discussion) must be included and appropriately labeled, your report should include all data obtained (yield, %yield, melting point, physical state of product, ...), etc. Spelling, grammar, etc. will be taken into account when these are graded. Students having trouble are encouraged to make use of the Writing Center. A review of Chapter 3 of the "Background Information" handout is strongly encouraged before writing these reports.

While some experiments may be done in pairs, all laboratory reports are to be done individually. Late reports will be accepted, but will be penalized 1 point per day late. All laboratory reports must be turned in on or before the last Friday of the semester (May 5), before the start of finals week .

Requirements for each of the individual reports are included in your laboratory 'textbook' and summarized below. Be sure to include all requested information from *both* sources in your reports. In some cases, more specific guidelines for reports will be provided in laboratory. Copies of IR spectra obtained in lab should be included in these reports. For some experiments, ^{1}H and/or ^{13}C NMR spectra may be collected and/or provided. Analysis of IR and/or NMR data should be included in the "Results and Discussion" section of your reports. Review the appropriate sections of your labbook or textbook for help with analysis of these spectra.

In general, it is expected that the data obtained in lab should be briefly summarized in the "Experimental Section". Any discussion or interpretation of this data belongs in the Results and Discussion section. An example of how this may be presented is shown below.

> *... The product was crystallized from toluene to yield 1.378 g (8.29 mmol, 69.3% yield) of cubic, yellow crystals (m.p. 128.5-130.0°C).*

Schedule

All dates listed below are tentative and are subject to change.

In addition to the information listed below, be sure to include all information requested in your laboratory manual. Laboratory reports are due by 11:59pm on the dates indicated below.

- Jan 18 - Diels-Alder Reaction (1.2)
- Jan 25 - Computational Chemistry: Conjugation (1.3)

Dienes and Conjugation Report (30 pts) - The experimental section for this report should be divided into three subsections (preparation of ionic liquid, Diels-Alder reaction, and computational procedure). In the Results and Discussion section, you should first discuss the computational results. For this, you will need to include a table of calculated energies for both your compound and the relevant calculations performed by your classmates. Discuss how the computed energies relate to the relative stability of each compound and compare this with expectations. For the Diels-Alder reaction, you should report the yield, %yield, and an analysis of the IR data. Include a copy of the IR spectrum and a drawing of the product of this reaction. *Due Feb 8.*

- Feb 1 - Nitration of Methyl Benzoate (1.1.1)
- Feb 8 - Friedel-Crafts Alkylation of *p*-dimethoxybenzne (1.1.2)

Electrophilic Aromatic Substitution Report (30 pts) - For these experiments, standard data for synthesis reactions should be reported (yield, %yield, melting point, ...). The Results and Discussion section for this paper should also include an analysis of the spectra obtained in lab and those provided in your laboratory textbook. Finally, show the mechanism for both of these reactions and discuss how the choice of experimental reaction conditions lead to either mono- or di-substituted products. *Due Feb 22.*

- Feb 15 - Synthesis of cinnamaldehyde (handout)
- Feb 22 - Reduction of cinnamaldehyde to cinnamyl alcohol (handout)

Cinnamaldehyde: Synthesis and Reduction (30 pts) - This is a multi-step synthesis of cinnamyl alcohol. In the first step, a crossed-aldol condensation is used to synthesize cinnamaldehyde. In the second step, a selective reduction is performed on this product to produce the final alcohol. The introduction section of your report should talk about uses and fragrances of the two compounds synthesized. The emphasis of the 'Results and Discussion' section should be on the characterization data (particularly analysis of the IR spectra) which provides evidence that you successfully synthesized these two products and an indication of the purity of each of these. Since characterization of the products obtained is such an important part of this report, you should include drawings of each of the molecules obtained. *Due Mar 8.*

- Mar 1 - Aldol Condensation (1.6)
- Mar 8 - Aldol Condensation (cont.)

Aldol Reactions Report (30 pts) - This report should be modeled after a full scientific paper. The purpose of this paper is to describe the synthesis of the product and provide sufficient analysis to convince the reader that the claimed products were obtained. Show the mechanism for these reactions using the curved arrow formalism. *Due Mar 22.*

- Mar 15- Synthesis of Methyl Diantilis (1.5)

- Mar 22 - Synthesis of Methyl Diantilis (cont.)

  Methyl Diantilis Report (30 pts) - Synthesis of this compound is a multi-step reaction. Since the starting material and all intermediates have multiple functional groups, each of the reaction steps must be selective. Your introduction should cover these topics. Somewhere in your report, you will also need to include the mechanisms for the reactions performed. In your Results and Discussion section, you should include the yield and %yield of each step, and the overall yield and % yield. The IR and ^{1}H NMR spectra should also be fully analyzed. It is also important to report how the characterization data obtained supports the claim that the correct products were formed. *Due Apr 12.*

- Mar 29 - **SPRING BREAK**

- Apr 5 - Prep of Malachite Green or Crystal Violet (1.7)

  Dyes Report (15 pts) - This report will only include the Experimental Section for this experiment. Although the yield is not reported, the success of this experiment can easily be determined by the color of the product formed. *Due Apr 19.*

- Apr 12 - Preparation of *N*-(2,6-dimethylphenyl)chloroacetamide (1.8)
- Apr 19 - Preparation of 2-(diethylamino)-*N*-(2,6-dimethylphenyl)acetamide (cont.)

  Lidocaine Report (30 pts) - The Introduction of this report should comment on the nature or importance of drugs. In the Results and Discussion section, comment on the relative rates (and the observations made in lab to support this) of the acyl substitution vs. the S_N2 substitution. Characterization data supporting the proposed structure of the products obtained should be explained. *Due May 3.*

- Apr 26 - Prep of Cyalume (1.9)

  Cyalume Report (15 pts) - This report will only include the Experimental Section for this experiment. You should include the yield, %yield, and melting point of your product, indicate whether or not it undergoes chemiluminescence and the color of this light. *Due Friday, May 5.*

- May 3 - **Laboratory Exam**
