

# Chemistry 30476 – Organic Chemistry Laboratory – Spring 2009

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Dr. Earley's Schedule				
	M	T	W	R
9:30-12:20pm		Organic Lab		
10:00-11:00pm	Office Hrs.	Organic Lab	Office Hrs.	
11:00-12:15pm	Fund. Chem.	Organic Lab	Fund. Chem.	
12:15-1:00pm	Office Hrs.		Office Hrs.	
1:00-2:00pm		Office Hrs.		Office Hrs.
2:00-2:50pm		Basic Organic		Basic Organic
3:00-4:00pm		Office Hrs.		
5:00-5:30pm	Office Hrs.		Office Hrs.	
5:30-6:45pm	Chem. World		Chem. World	
7:00-9:50pm	Chem. World Lab			

*Additional office hours available by appointment*

## Required Materials

- Required: *Course Pack (handouts), Organic Laboratory Notebook, Safety Goggles, Calculator.*

## Prerequisites

Students are expected to have successfully completed the equivalent of one semester (two credit hours) 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<sup>nd</sup> 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.

## Attendance

Attendance in laboratory is required. 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.

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 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		190 pts
Best 3 Prelab Quizzes	3 x 5 pts	15 pts
Laboratory Exam	1 x 20 pts	20 pts
<b>Total</b>		<b>225 pts</b>

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%
Points	202-225	198-201	184-197	180-183	176-179	162-175	157-161	135-156	<135

## 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 complete procedures 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. 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.

## Student email accounts

All students enrolled in the Kent State University system are given a FlashLine account. FlashLine is the internet 'portal' that provides access to e-mail, Web for Students, WebCT/Vista, and a number of other resources. University policy states that email is a valid method of transmitting information to students, so it is important to check your e-mail periodically.

## Computers and Internet Usage

I will occasionally make reference to material that is available either locally or on the Internet throughout the semester. All of this material will be accessible using any modern Internet browser (Microsoft Internet Explorer, Netscape Navigator, Mozilla, Firefox, Opera, etc.) While it is not required that you take advantage of this information, some of this material can be quite helpful. The address for my Web site is:

<http://www.personal.kent.edu/~cearley>.

## Final Grades and Web for Students

Final grades for students are NOT mailed to students at the end of every semester. The ONLY way for students to find their grades is to look at Web-For-Students (<http://wfs.kent.edu> or through Flashline). I will not be able to give final course grades out over the phone, by e-mail, etc.

## 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 material 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 call) and we can set up additional time to meet.

## Course Withdrawal

If you are considering withdrawing from this or any other course, you are encouraged to consult with a staff member in the Student Services Office, 134 Main Hall. Withdrawal from a course can affect financial aid, student status, or progress within your major. For withdrawal deadlines, please refer to [http://www.registrars.kent.edu/home/TermUpdate/sche\\_adj.htm](http://www.registrars.kent.edu/home/TermUpdate/sche_adj.htm).

## Academic Honesty

Use of the intellectual properties of others without attributing it to them is considered a serious academic offense. Cheating or plagiarism will result in a failing grade for the work or for the entire course. Repeat offenses result in dismissal from the University. University guidelines require that all infractions be reported to the Student Conduct Officer on our campus.

## Students with Disabilities

Kent State University recognizes its responsibility for creating an institution atmosphere in which students with disabilities can succeed. In accordance with University Policy Subpart E...104.44, if you have a documented disability, you may request accommodations to obtain equal access in this class. Please contact the disability coordinator on campus, Kelly Kulick in Student Accessibility Services, located in the Student Success Center, lower level of the Campus Center, phone (330)244-5047, or [kkulick@kent.edu](mailto:kkulick@kent.edu). After your eligibility for accommodations is determined, you will be given a letter which, when presented to instructors, will help us know best how to assist you.

## Campus Security

If you need to reach Campus Security, their phone number is 330-705-0430.

## Grievance

The "Kent State University Digest of Rules and Regulations" (available online at <http://www.kent.edu/regional/>) should be consulted for information on grievance procedures, statement of non-discrimination, and additional information on official policies.

## Classes Canceled - Campus Closings

Announcements of class cancellations and/or campus closings will be made on the campus home page. In the case of an emergency, weather-related or otherwise, please check the web page at stark.kent.edu for information on the buildings and times of the closing. While information may be broadcast by radio and television, this should be confirmed by the web page, which is the official announcement of the campus and which will be the information used to determine issues related to student attendance, rescheduling of tests, and other concerns.

## Recycling

The KSU Stark Campus has made a commitment to recycling. Please take a few seconds to separate your trash. Aluminum cans and plastic and glass bottles may be placed in the blue recycling bins, and all types of paper may be placed in the blue recycling bins. All other waste may be placed in the black, brown or gray trash cans.

## General Information for Laboratory Reports

Unless otherwise noted, all laboratory reports should be modeled after a scientific paper. All sections must be included and appropriately labeled, your report should include all data obtained (yield, % yield, melting point, physical state of product, ...), etc. These reports must be typed. 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 your laboratory 'textbook' is strongly encouraged before writing these reports.

Late reports will be accepted, but will be penalized 1 point per day late. All laboratory reports must be turned in on or before May 8 (before the start of finals week).

Requirements for each of the individual reports are included in your laboratory 'textbook' and summarized below (following the Tentative Schedule). 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 with these reports. For some experiments, ideal  $^1\text{H}$  and/or  $^{13}\text{C}$  NMR spectra may be provided. Analysis of IR and/or NMR data should be included in the "Results and Discussion" section of your reports. Review sections 5.1 and 5.2 for help with analysis of these spectra.

### Example of presenting data in the "Experimental Section"

*... 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. The following dates should be noted:

- <sup>(S)</sup>Last day to receive any tuition refund is February 13.
- <sup>(W)</sup>Last day to withdraw from a course is April 3.

Date	T
Jan. 20	Check-in, <b>6.9</b> Which Nitrogen?
Jan. 27	<b>6.4.1</b> Nitration of Methyl Benzoate
Feb. 3	<b>6.4.2</b> Friedel-Crafts Alkylation
Feb. 10 <sup>(S)</sup>	<b>6.5</b> Selective Reduction of Nitroacetophenone
Feb. 17	<b>6.5</b> Selective Reduction (cont.)
Feb. 24	<b>6.6</b> Synthesis of Vanillin
Mar. 3	<b>6.6</b> Synthesis of Vanillin (cont.)
Mar. 10	<b>6.11</b> Prep. of Cyalume
Mar. 17	<b>6.2</b> Grignard Reaction
Mar. 24	<b>SPRING BREAK</b>
Mar. 31 <sup>(W)</sup>	<b>6.7</b> Preparation of a Barbiturate
Apr. 7	<b>6.7</b> Prep. of a Barbiturate (cont.)
Apr. 14	<b>6.8</b> Synthesis of Lidocaine
Apr. 21	<b>6.8</b> Synth. of Lidocaine (cont.)
Apr. 28	<b>6.10</b> Protein Folding
May 5	<b>Exam, Makeup Lab. Period</b>

### Specific Requirements for Laboratory Reports

In addition to the information listed below, be sure to include all information requested in your laboratory manual.

Copies of IR spectra obtained in lab should be included with these reports. For some experiments, ideal  $^1\text{H}$  and/or  $^{13}\text{C}$  NMR spectra may be provided. Analysis of IR and/or NMR data should be included in the "Results and Discussion" section of your reports. Review sections 5.1 and 5.2 for help with analysis of these spectra.

In laboratory reports containing an "Experimental Section", it is expected that data related to the yield and purity of the compound be included. 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).*

#### Which Nitrogen? (15 pts)

Your report for this experiment should include the "Experimental" and a "Results and Discussion" section. You must include an analysis of the data given to you (results of quantum mechanical calculations and  $^1\text{H}$  NMR data given in your laboratory 'textbook'). *Due Feb. 3.*

#### Aromatic Substitution (30 pts)

*Nitration of Methyl Benzoate*

*Friedel-Crafts Alkylation*

For these experiments, standard data for synthesis reactions should be reported (yield, %yield, melting point, ...). The data obtained in lab should be included in the experimental section. Any discussion or interpretation of this data belongs in the Results and Discussion section. 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 notebook. 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. 17.*

#### Reduction Reactions (25 pts)

*Selective Reduction of Nitroacetophenone*

The 'Introduction' of your report should comment on the different reducing agents used in the reactions performed, and mention the importance and/or relevance of selectivity in practical organic synthetic reactions. The emphasis of the 'Results and Discussion' section should be on the characterization data (melting point and an analysis of the IR spectra) which provides evidence for both the structure and the purity of each of the substances obtained. Since characterization of the products obtained is such an important part of this report, you should include drawings of each of the molecules obtained (hand-drawn is fine). *Due Mar. 3.*

**Synthesis of Vanillin** (25 pts)

The emphasis on this report will be on characterization of the products obtained. You should include enough information on the yield and characterization data obtained to convince the reader that you have obtained the products claimed. *Due Mar. 17.*

**Preparation of a Cyalume** (10 pts)

No formal report is required for this experiment. You are simply required to turn in a copy of your laboratory notebook pages, which should include the yield, %yield, and melting point of your product, and indicate whether or not it undergoes chemiluminescence. *Due Mar. 17.*

**Grignard Reaction** (20 pts)

*Synthesis of Crystal Violet or Malachite Green*

Because this product is not isolated, you cannot include the standard data (yield, %yield, melting point, ...) normally present. Your lab report should include  $\lambda_{max}$  for your dye, and an explanation of how this value correlates to the color of the dye. In addition, include a diagram showing the mechanism of the Grignard reaction. *Due Apr. 7.*

**Preparation of a Barbiturate** (25 pts)

Again, the emphasis on this report will be on characterization of the products obtained. You should include enough information on the yield and characterization data obtained to convince the reader that you have obtained the products claimed. The mechanism for key reaction steps should be included in your report. *Due Apr. 21.*

**Synthesis of Lidocaine** (25 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 5.*

**Protein Folding** (15 pts)

Prepare a table in your laboratory notebook containing columns for your observations (before and after heating) and  $\lambda_{max}$  for each solution. The copy of your notebook pages that you turn in at the end of class will be your laboratory report. *Due Apr. 28.*