Syllabus for
CHM 2845 - 001
Organic Chemistry Laboratory II
Fall 2010

Course Description: CHM 2845 is the second-semester laboratory course in organic chemistry covering the chemistry of alcohols, ethers, aromatics, amines, and carbonyl compounds. The course strongly emphasizes spectroscopy (IR, NMR, MS) of organic compounds as well as expanding the student's experimental skills and reporting abilities.
Prerequisites: Prior or concurrent enrollment in CHM 2840, prior credit in CHM 2445.

Course Goals: (1) To increase knowledge and proficiency in standard organic laboratory techniques.
(2) To develop a deeper theoretical understanding of spectroscopic methods employed in organic chemistry, as well as increasing proficiency in interpretation of spectra
(3) To illustrate the concepts discussed in CHM 2840.
(4) To increase proficiency in writing formal laboratory reports.

Meeting Time: 2:00 P.M – 2:50 P.M. Tuesday

Meeting Place: Physical Science Building 4480 (lecture) Physical Science Building 4170 (laboratory)

Instructor: Dr. Ed M. Treadwell
Office: 4450 PSB Phone: 581-6229 E-mail: emtreadwell@eiu.edu
Office Hours: Mon 9–10 am, 2–3 pm (other hours gladly available by appointment)

Texts: (1) "Organic Laboratory Techniques" by Pavia, Kriz, Lampman, Engel
(2) "Introduction to Spectroscopy" by Pavia, Lampman, Kriz

Lab Manual: Available at Copy-X (on Lincoln Avenue by Chubbies and across from Bank One) 8:30-5 Mon-Fri for $3.15

Format:

This class will be a combination of lecture and laboratory periods (roughly 25% lecture and 75% laboratory). The lectures will cover IR, NMR, and MS spectral methods, and a problem set for each of major techniques will be given. Seven experiments will be preformed, with a brief pre-lab lecture and quiz proceeding the start of each experiment. A formal report (see below) for each experiment must be handed in by 2 P.M. on the date listed below. There will be a cumulative final exam given the last week of classes that will cover the lecture material on both the experiments performed and the spectroscopic techniques discussed. A notebook must be maintained and will be graded throughout the semester.

Course Policies:

1. **SAFETY IS VERY IMPORTANT. A list of safety rules is included in the handout, and you will be expected to obey these rules at all times. Violation of the rules can result in immediate dismissal from the laboratory.**
2. Goggles policy: **When you are in the lab, goggles must be worn at all times** except when you are walking in for the first time or leaving for the last time of the day. You will receive a verbal warning if you do not wear goggles. After your second verbal warning, you will **lose five points** from your lab score each time you do not wear goggles.
3. Please inform me of any preexisting medical conditions you have as soon as possible, or if you have a documented disability and wish to discuss academic accommodations.
4. You are responsible for all announcements made during class, whether you are present or not.
5. If you are absent because you were sick, you will be allowed to make up the lab/quiz if you provide an excuse that is *written* and *signed* by a medical official. If you must be absent for a lab period because of required travel with an athletic team, please arrange accommodations BEFORE you leave. Absences for emergency reasons will be judged on a case-by-case basis. Lack of a valid reason for an absence will result in a zero score on the quiz that was missed. If the first day of two-week experiment is missed without an acceptable excuse, you will NOT be allowed to perform the lab and will receive a zero on the lab report.

6. **Punctuality Policy**: There will be a **two point penalty** for every five minutes you arrive after 8:00 A.M. effected throughout the semester. After four occurrences, the penalty will increase to five points for every five minutes.
7. In order to complete the experiments in the designated time frame, it is important not only to be in class on time, but to be prepared to carry out the experiment. You should be familiar with the day's procedure before entering the lab.
8. Beginning at 8 A.M. on the day they are due, reports turned in after the deadline will incur a 5 point per late-day penalty unless they were late because of an excused absence. Lab reports will NOT be accepted beyond a week past the due date. Your course grade will be **lowered by one letter grade** if you have one unexcused absence or fail to turn in one lab report. You will **receive a course grade of "F"** if you have two or more unexcused absences or fail to turn in two or more lab reports.
9. The class is cumulative. Questions about previous experiments or techniques may appear on any quiz.
10. Lab reports must be done with word-processing software. Directions for writing reports and details on grading rubric are given on the last pages of the syllabus. The "Turn It In" program will be used to assist in identifying cases of plagiarism. You should set up an account at http://www.turnitin.com; the class id number is **3375908** and the password is **OrgTwo**. Failure to submit online will incur a penalty of **2 points per day** from the report grade.
11. EIU's policy on academic integrity (as described in the EIU Undergraduate Catalog and Student Conduct Code) applies to all parts of the course. Copying background information, results, answers, and calculations from someone else's lab report constitutes cheating and is grounds for an **"F"** in the course.
12. A carbon-copy laboratory notebook with at least 50 pages will be required for this class. You can continue to use your notebook from CHM2445 if it still has enough pages left.
13. There is a $10 lab fee for chemicals and supplies. Breaking glassware in the kits will result in additional charges.
14. Some of the chemicals you will be using can leave permanent stains or holes on your clothes should you spill them on yourself. For that reason, I would recommend that you wear a set of "old clothes" to lab that you won't mind throwing away if you have to, and to wash these clothes separately from the rest of your non-lab clothes.

Itemized Point Distribution:

Item		Points
Prelab Quizzes for Expts 1-7 @ 8 pts each (~ 12% of total grade)		56
Lab Reports for Expts 1-7 @ 25 pts each (~ 52% of total grade)		175
Problem Sets (3 @ 15-20 pts each) (~ 15% of total grade)		50
Cumulative Exam (30 points) (~ 9% of total grade)		30
Lab Notebook (42 points) (~ 12% of total grade)		42
Total		***370***

Grades:

Grades will be assigned on the percentage of total points earned, with the following approximate grade point ranges:

90 – 100 = A 80 – 89 = B 70 – 79 = C
60 – 69 = D < 60 = F

Schedule for Lectures and Experiments

Date	Topic or Experiment	Tie-In*	Items Due **
Aug. 24	Course introduction, check in IR discussion		
31	*Experiment 1: Dehydration of 2-Methyl-cyclohexanol*	7.10, 8.4	
Sept. 7	*Experiment 1: Dehydration Reaction cont'd* Product Analysis NMR discussion part 1		IR homework
Sept. 14	*Experiment 2: Reduction Reaction*	10.11	Exp. 1 report due Sept 17th
21	*Experiment 3: Diels-Alder Reaction* Preparation of Diene	7.9, 6.19-20	NMR homework 1
28	*Experiment 3: Diels-Alder Reaction* Diels-Alder Reaction NMR discussion part 2	15.11-12	Exp. 2 report
Oct. 5	*Experiment 4: Electrophilic Aromatic Nitrations* Performing reaction	17.1, 17.3, 17.6-9	Exp. 3 report
12	*Experiment 4: Electrophilic Aromatic Nitrations* Product Analysis		
19	*Experiment 5: Grignard Reaction* Performing reaction	10.8-10	NMR homework 2
26	*Experiment 5: Grignard Reaction* Quenching reaction and Product Analysis MS discussion		Exp. 4 report
Nov. 2	*Experiment 6: Flavone Synthesis and Bioactivity* Steps 1-2	17.15, 21.5	Exp. 5 report
9	*Experiment 6: Flavone Synthesis and Bioactivity* Step 3	22.2,22.3	MS homework
16	*Experiment 6: Flavone Synthesis and Bioactivity* Product Analysis and Bioactivity results		
Nov. 30	*Experiment 7: Aldol Reaction*	22.9	Exp. 6 report
Dec. 1	Cumulative Lab Exam; Check out		Exp. 7 report

MAINTAINING A LABORATORY NOTEBOOK

Keeping a detailed, coherent laboratory notebook is an essential part of scientific research. Not only is the notebook important as a permanent record of your research, but the notebook can be essential in understanding low yields and unexpected results. A good lab notebook should allow anyone, using only your notebook and whatever references contained therein, to repeat EXACTLY your experiment and obtain the same overall results as you obtained. It is therefore extremely important that the notebook be complete AND completed as the experiment is carried out. The experimental procedure section should NEVER be filled out after you leave the lab.

The Notebook:

Your lab notebook should be dedicated exclusively to this course, and be a bound notebook (either a spiral notebook or a composition book).

All entries should be made in nonerasable ink, and should be legible. Any mistakes should be crossed out with a single line, not by scratching out completely the information or by covering with white-out.

All pages written on should be number consecutively.

The front cover should contain your name and the course number.

The first page should be used for a table of contents, with each experiment listed by title and correlated to the appropriate pages in the notebook. The table of contents should be continually updated throughout the semester, not filled in at the end of the semester.

Experiment Entries:

Each experiment should start on a separate page, and each page dated.

Before the lab for a particular experiment starts, the following items should already be entered:

A – the title of the experiment

B – the reaction drawn out using structural formulas, or the structure of the compounds studied

C – a table of reagents. There should be column headings for reagent, the amount used, the molecular weight, the moles used, and the equivalents used. Only the reagent column should be completed prior to the experiment (since the amounts you use may not be exactly the amounts specified in the manual).

D – the reference for the experimental procedure

As the experiment is carried out, the steps taken should be entered in complete sentences in the lab notebook. Note that it is not essential to completely rewrite the given experimental procedure, nor to describe the individual steps in any techniques. Be sure to include the amounts used, the order of addition, times, temperatures, and OBSERVATIONS as appropriate. Remember that I should be able to repeat your experiment exactly from your notebook. Any characterization of the product (mp, IR, GC, NMR, etc) should be mentioned, though the analysis of the characterization need not be included. If you are only taking a melting point/spectrum of your product on a different day than the rest of the experiment, the entry can be on the same page as the rest of the experiment, but with the new date written by the information.

CHM 2845 LAB REPORTS

1. Report Format

Each report is to be handed in with a cover sheet containing the experiment number, the name of the experiment, your name, and the date. Your name should not appear anywhere else in the report. Lab reports should be no longer than 6 pages (not including spectra, calculations page, or the cover page) , double spaced, and in reasonable font size (10 or larger) unless otherwise stated. Failure to adhere to the page limit shall incur in loss of credit. The reports should be grammatically correct, and the use of pronouns anywhere in the report is forbidden.

Use the headings below to divide the remainder of your report into the following **five sections**:

Purpose

(*The goal of this section is to succinctly give the reader the idea of what you are attempting, how it will be carried out, and how the results will be evaluated*)

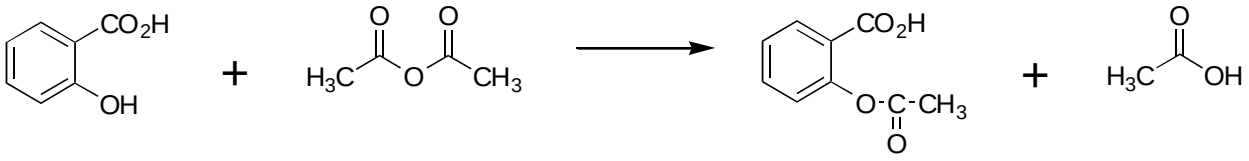
The purpose should briefly answer three basic questions – what compound(s) are being made in the lab from what starting materials and reactions; how will the product be purified; and how will the identity/purity of the product(s) be determined. Additionally, if the experiment is designed to determine the regio- or stereo-chemistry of a process, the purpose should reflect this. The purpose should be no longer than 4 sentences.

Examples:

"Aspirin was prepared by room temperature acetylation of salicylic acid using acetic anhydride, and the product was purified by recrystallization. The purity of the final product was evaluated on the basis of melting point and IR spectral analysis."

"5-Hexyne was reduced with hydrogen and a number of different metal catalysts to determine the ratio of cis to trans products and the overall effectiveness of each catalyst in the reaction. The organic extracts for each reaction were analyzed by gas chromatography, and the product ratios determined by integration of the relative peak areas."

All reactions must be drawn with a drawing program and electronically pasted into this section. The drawings should look clean and professional. An example of an acceptable drawing is below:



Experimental

(*The goal of this section is to give an account of how the experiment was carried out, and should include all observations made during the experiment. This is NOT the place for any interpretation of data or results)*

This section should start with a table of reagents – these are ONLY the chemicals that **react** to give the product (these are the chemicals where bonds are actually made/broken). Thus solvents or drying agents or items used in the work-up do not need to be included in this table. This table should not include products. If the experiment has more than one step, make a separate table for each step, where the product from the previous step is used as the starting material for the next table.

Example:

	mass	volume	MW	mol	equivalents	density
Salicylic acid	1.89 g	-	138.12 g/mol	0.0137	1.0	-
Acetic anhydride	5.5 g	5.1 mL	102.09 g/mol	0.054	3.9	1.087 g/mL

Succinctly describe the details of the work and the observations you made. Record all experimental conditions such as the order of mixing of reagents and the length of times used for various steps. Be sure that this is not a block copy of the given procedure, but an account of the experiment as you performed it with the actual amounts, times, etc. that you employed. Record all observations such as changes in solubility and color in response to things you do. If a solution is used, write out its concentration when you mention its use. **Proper use of subscripts (in chemical formulas) and superscripts (such as º C) is required for full credit**. **Write in complete sentences using the past tense, in a passive voice. Do not start any sentences with numbers**. Mention should be made of any spectra collected (as well as how they were collected or what solvent was used), but NO ANALYSIS of the spectra should appear in this section.

Example: "*To 1.89 g of salicyclic acid in a 125 mL Erlenmeyer flask was added 5.1 mL of acetic anhydride and 4 drops of concentrated sulfuric acid. The flask was gently shaken to dissolve the solid, and then heated for 15 minutes on a steam bath. During the heating process, the solution gradually darkened to a deep brown color. Upon removing the flask from the steam bath, small white crystals began to appear in the solution. After 48 mL of water was added to the flask, it was placed in an ice bath for 15 minutes and more crystals formed. The product was collected by vacuum filtration, and the solid washed with two 5 mL portions of cold water. The crude product was air-dried for 15 minutes, and weighed 1.64 grams.*

The crude product was recrystallized from 4 mL of ethyl acetate to give 1.21 grams of a shiny, white crystalline product. The melting point of the product was determined to be 138.5 – 140.0 ºC, and an IR spectrum (Nujol mull) was collected."

Discussion

(*The goal of this section is to analyze the results of the experiment and to discuss the efficiency of the procedure, the clarity of the results, and the overall quality of the lab. Additionally, any issues of regio- or stereo-selectivity should be mentioned and correlated with the theory learned in CHM 2840)*

If a reaction was done, begin by listing the actual yield (in grams), the theoretical yield (in grams), and the percent yield.

Next, interpret your results and observations, writing in complete sentences. This should be done in a sequential and coherent format. (For multi-step reactions, discuss the first step completely before discussing the subsequent step. Organize by step, not by type of analysis (do not talk about all the melting points first, then all the spectra, etc…) Talk about the melting point, spectra, and yield for the first step, then move onto the second step).

Proper use of subscripts and superscripts is required for full credit. Be sure to comment on both the identity and the purity of the products. Any literature data should be included in this section, such as the reported melting point. If applicable, comment on any possible causes for low yields or impure

products. An example of this would be: "*The low yield of product X may have been caused by insufficient cooling during the recrystallization step so that not all the material precipitated out of the solution." or "A longer reflux during the reaction might have improved the yield*."

Within the discussion should be an interpretation of any spectra obtained. Be sure to label important peaks on the spectrum itself as well as write a title on each spectrum.

For IR Spectra

List the assignable IR absorptions in tabular format with the frequency in cm^{-1} and the stretch/bend responsible for the absorption. Also in the report, mention (in writing) the important IR absorptions that give strong evidence of the structure of your product, and the peaks absent from the IR spectrum corresponding to the starting material (thus suggesting your product is not simply unreacted starting material). Note that all the peaks in your table do not necessarily need to be mentioned in the text.

For NMR Spectra

It is important that you clearly state what type of NMR spectra you are analyzing – it should be either ^{1}H NMR spectra or ^{13}C NMR spectra. Failure to include the nucleus observed (simply writing NMR spectra) will result in loss of points. For each absorption in the NMR in tabular format, list: (1) the chemical shift in units of ppm, (2) the splitting, and (3) the integration. Describe (in words) your reasoning for assigning each absorption to the structure of the compound that was analyzed, and how the NMR spectrum gives strong evidence of the structure of your product.

Conclusion

(*The goal of this section is to provide a concise, overall evaluation of the success/failure of the experiment. I should be able to read <u>only</u> the purpose and conclusion sections of your report and have a good idea of how well the experiment worked.)*

Come to a conclusion about whether you succeed in achieving the purpose of the work. State only what can be backed up with information you have collected, and be sure to include the percent yield. Items mentioned in the purpose should be summarized here (Did the distillation work? Were you able to obtain a high yield of your product?). Please do not use this section to express your personal feelings about the experiment. The conclusion should not be longer than 8-10 sentences at most.

Example:

"*Acetylsalicylic acid was prepared in a 65% yield by acetylation of salicylic acid using acetic anhydride, and the sharp melting point (138.5 – 140.0 ºC) as well as the IR spectrum of the recrystallized product indicated that the product was very pure* ."

Calculations page

At the end of the report, you should include a page showing all your calculations (to determine moles of reageants, as well as to determine theoretical and percent yields). This page is the only page that CAN be handwritten, but it should be legible and there should be some identification for each calculation as to what the calculation is determining (ie, a label such as "calculation of theoretical yield" above the numbers).

2. All reports must be prepared with word processing software and printed on a printer.

The text must be prepared on a computer, and hand-written corrections are unacceptable. Correct use of symbols, subscripts, and superscripts is required. The chemistry computer lab has the Chemdraw® program already installed, and there are various free Web software programs (such as ISIS/Draw) that you can download and use to drawn structures.

3. Grading

Grading of lab reports will based on the items listed below, along with their approximate weight percentages.

Format (12 %): (Is the report written in the correct format? Do tables, figures, and spectra all have titles? Have subscripts/superscripts in formulae been correctly used? Do measurements have units?)

Grammar (12 %): (Is the text grammatically correct, and is everything spelled correctly?)

Results/Interpretations (36 %): (Are all the results and pertinent literature data included? Has each result been interpreted? Has the theory behind the experiment been discussed?)

Significant Figures (12 %):

Completeness (12 %): (Is all the relevant information presented? Are spectra fully interpreted?)

Quality (16 %): (Is the presentation of data clear? Is the report insightful and carefully done? Are the interpretations/conclusions well argued and believable? Is the product pure and obtained in respectable yield)

4. What to do with the returned report?

When you get your report back, in addition to the grade at the end, there will be numerous comments in each section. Do not worry if it seems like I wrote as many comments as you did words - I am not being "mean" or "vindictive" but rather want to give you suggestions for improving your writing. You should look at these comments, and learn from them, and perhaps even look back at your graded reports before you hand in your next report to make sure you're not repeating the same mistakes. Should you pervasively continue to ignore the comments and make the same mistakes, I will not hesitate to hand the report back to you, ungraded, and ask for a proper report before I grade it. This will result in a point loss as well.