

Syllabus for CHM 2445 - 003
Organic Chemistry Lab I
Fall 2008

Description: CHM 2445 is an introduction to experimental organic chemistry. The experiments performed will illustrate some of the reactions studied in the CHM 2440 as well as introduce common laboratory techniques. Spectroscopy will also be emphasized. The course is assigned one credit hour.
Prerequisite: CHM (1410, 1415 or 1510, 1515), concurrent enrollment or prior credit in CHM 2440.

Course Goals: (1) Learn and develop proficiency in the standard techniques used in the organic laboratory.
(2) Illustrate concepts learned in organic chemistry lecture courses.
(3) Develop writing skills necessary for formal laboratory reports.

Class Meeting: Time: 2:00 P. M. – 4:50 P. M. Thursday
Place: PSB 4180 (for pre-lab discussions and quizzes) PSB 4170 (for lab)

Instructor: Dr. Ed Treadwell Office: 4450 Physical Sciences Building
Phone: 581-6229 E-mail: emtreadwell@eiu.edu
Office Hours: Weds. 10:00 – 11:00 a.m. Thurs. 12:00 noon – 1:00 p.m.
(Other times gladly considered by arrangement.)

Text: “Introduction to Organic Laboratory Techniques” by Pavia, Lampman, Kriz, and Engel

Course Policies:

1. **Safety is very important and a list of safety rules can be found in the lab manual. Violation of these rules, or other unsafe practices, will not be tolerated and can result in expulsion from the laboratory session.**
2. Goggles policy: **Goggles must be worn at all times** except when you are walking in the lab for the first time or leaving it 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 overall lab score each time you are caught not properly wearing goggles. You will NOT be allowed in the lab without goggles, even for analysis days.
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. 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.
7. Beginning at 11 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. 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.
8. The class is cumulative. Questions about previous experiments or techniques may appear on any quiz.
9. 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 **2343208** and the password is **organic003**. Failure to submit online will incur a penalty of **2 points** from the report grade.
10. 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.
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.

Possible Points:

Item	Points
9 Reports @ 25 points each	225
9 Quizzes @ 10 points each	100
Lab Notebook	45
Lab Final	50
Total Points	410

Grades:

Final grades will be determined by the percentage of total possible points that you have earned over the semester. The standard scale of 90% for an A, 80% for a B, etc. is expected to be employed.

LABORATORY SCHEDULE

Date	Quiz	Experiment Reading	Report Due	Corresponding 2440 Text
Aug. 28		Orientation, Check In Reading: pg 1-9, 17-21, 36-41		
Sept. 4	Q1	Exp 1. – Recrystallization and melting points Reading: 128-149, 153-161, 107-109, 116-120, 121-126		2.11
Sept. 11	Q2	Exp 2. – Simple Distillation Reading: 201-209, 333-342, 346-348	Exp. 1	
Sept. 18		Exp 2. Cont'd – Fractional Distillation Reading: 220-232		
Sept. 25	Q3	Exp 3. – Caffeine Isolation Reading: 163-169, 177-182, 101-107	Exp. 2	
Oct. 2	Q4	Exp. 3. Cont'd – Sublimation and Analysis Reading: 269-273, 313-325		
Oct. 9		Exp 4. Identification of Unknowns Reading: 369-371, 385-406	Exp. 3	12.1-12.12
Oct. 16	Q5	Exp 5. – Menthol Stereoisomers Reading: 355-361	Exp. 4	5.2, 5.4, 5.7, 5.11, 5.12, 5.15
Oct. 23	Q6	Exp 6. – S_N1 / S_N2 Conditions	Exp. 5	6.8 – 6.16
Oct. 30	Q7	Exp 7. – Addition of Br₂ to an Alkene Reading: 79-99	Exp. 6	8.8
Nov. 6	Q8	Exp 8. - Addition of H₂O to an Alkene - Reaction		8.4
Nov. 13		Exp 8. Cont'd - Distillation / ¹H NMR Reading: 409-440	Exp. 7	13.1-13.8
Nov. 20		Exp 8. Cont'd - Distillation / ¹H NMR		
Dec. 4	Q9	Exp 9. Polymerization of Alkene	Exp. 8	8.16
Dec. 11		Lab final, Check-out	Exp. 9	

MAINTAINING A LABORATORY NOTEBOOK

Keeping a detailed, coherent laboratory notebook is an essential part of scientific research. 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. Not only is the notebook important as a permanent record of your research, but the notebook can be essential in understanding all the results, positive or negative. It is therefore extremely important that the notebook be complete and completed as the experiment is carried out. The experimental procedure section should ALWAYS be filled out BEFORE you leave the lab.

The Notebook:

Your lab notebook should be dedicated **exclusively to this course**, and have carbon-copy pages so that the second page (the copy) can be handed in at the end of each lab period.

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 it over with white-out.**

The front cover should contain your name, the name of the instructor and the course and section number.

The back of the front cover, or 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 should be dated.

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

- A – the date, experiment number, title of the experiment, and name (at top of page)**
- B – a brief purpose stating what is to be done in the experiment, and what data will be collected/how data will be utilized**
- C – the reaction equation using structural formulas, or the structure of the compounds studied**
- D – 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 (that is, the name of the chemicals used) should be completed prior to the experiment (since the amounts you actually measure may not be exactly the amounts specified in the manual).**
- E – the reference for the experimental procedure**

As the experiment is carried out, the steps taken should be entered in nearly **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 (mass, mp, IR, GC, NMR, etc)** should be mentioned, though the analysis of the characterization need not be included.

Here is an example for your lab notes:

How to Write Reports for CHM 2445

In the first few experiments, you will only need to write certain parts of the report. But by experiment 4, you will be required to write a complete report. Irregardless, the guidelines below apply no matter if you are writing a full or partial report.

1. Report Format

Each report should be handed in with a cover sheet that contains 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 5 pages (not including spectra or the cover page) in reasonable font size (10 or greater) with 1.5 spacing and 1" margins all around unless otherwise stated. The report should be in the same font throughout, and should be on fresh paper (nothing on the reverse side). Failure to adhere to these formats shall incur in loss of credit.

The report should be written in complete sentences using the passive past tense, and without the use of pronouns. The report should not contain grammatical or spelling mistakes, and care must be taken to use superscripts/subscripts as appropriate for chemical formulae.

Use the headings below to divide the remainder of your report into the following four sections. Note that the examples correlate to the experiment attached at the end.

Purpose

Describe exactly what you were trying to do or determine, including the chemical names of the pertinent compounds and the means of purification and analysis, as applicable. Also list any new techniques learned in the experiment. The purpose should be no longer than 3 sentences, and avoid starting it with "*The purpose of this experiment is...*"

Example: "*Trimyrustin was extracted with ether from ground nutmeg, and after recrystallization from acetone, analyzed by melting point analysis. Heating at reflux and use of a rotary evaporator were introduced in this experiment.*"

For Experiments 2 and 5, you should draw the structures of the chemicals studied (but only the chemicals studied, not the solvents or drying agents). Structures should be computer-generated and pasted into your document before printing. You can either use ChemDraw (available only on the computers in the Chemistry computer lab) or ISIS Draw (a free download can be found, after registration, at <http://www.mdli.com/downloads/downloadable/index.jsp>)

For Experiments 6-9, the reaction should be clearly and neatly written below the purpose. Do not paste/copy the reaction drawn in the lab manual into your report.

Experimental

This section should allow the reader to repeat EXACTLY what you did in this experiment. Succinctly describe the details of the steps and the observations you made. Record all experimental conditions such as the order of mixing of reagents and the time durations for various steps. Record all observations such as changes in solubility and color, as well as describing the appearance (physical state, color, size, odor, etc.) of all crude and purified products. If a solution is used, the concentration should be included when it is first mentioned. If a spectrum is obtained, a description of how the sample was prepared should be included. All raw data should be included in the experimental, and all analysis of the data should be included in the discussion. It is permissible to have the data appear twice, once in the experimental and once in the discussion, if necessary. If the steps for a certain technique has already been fully described in a previous

report, you do not need to repeat them (for instance, in experiment 7, you can simply write “the crude product was recrystallized from 5mL of ethanol and 20mL of water” and not mention each step, such as putting the solid in an Erlenmeyer, adding solvent, etc..)

Remember that for all items measured, the units must be included. SIGNIFICANT FIGURES are significant - the balances in the lab read to the 0.01g or 0.001g, and the masses in your table should reflect this.

For the reaction experiments (Experiments 6-9) you should include a table of reagents, that includes the name of the chemical, the mass/volume used, the molecular weight, the moles, the equivalents, and the density/molarity as appropriate. Care should be paid to report the calculated values with the appropriate amount of significant figures.

Proper use of subscripts and superscripts is required for full credit. Write in complete sentences using the past tense, in a passive voice. Blatant copying of the procedure given in the lab manual will be considered plagiarism and dealt with severely.

One last comment - you should be writing in the passive past tense, meaning you should be giving a historical account of what you did, not dictating a recipe for somebody else to follow sometime in the future. Please try to avoid excessive use of words such as “next”, “then”, “after this”, etc, and/or starting a sentence repeating the end of the last sentence (for instance, “... solution was heated to reflux for 40 minutes. After refluxing for 40 minutes,...”)

Example: *“Using a mortar and pestle, 3.97 g of McCormick brand nutmeg seeds was ground to a fine powder, and added to a 50 mL round bottom flask. A stirbar and 11 mL of diethyl ether were added, and the suspension heated at reflux for 38 minutes. During this time, the solution gradually developed a dark brown color, and the seed material shrunk in size and the edges appeared frayed. After cooling, the solution was cooled and gravity filtered into a tared 25 mL round bottom flask. The solvent was removed using a rotary evaporator to afford 0.67 g of a dark brown, pungent oil. To this oil was added 2.5 mL of acetone, and the flask was warmed on a hot plate until the acetone began to boil. The solution was poured immediately in a 50 mL Erlenmeyer flask, and upon cooling, large, yellow needles of trimyrisitin formed. The flask was cooled in an ice bath, and the crystals were collected by vacuum filtration and washed with 3 mL of cold acetone. After air drying for one week, the purified product weighed 0.38 g and melted at 53.5 – 54.8 °C. ”*

Discussion

Interpret your results and observations, writing in complete sentences. This is the most important part of the report, and should show that you gave critical thought to your results and should be in an organized fashion. You should be focusing on questions like: Did the experiment work? How well did it work? How do I know that it worked/didn't work? What could have been done better? Remember that for most of these experiments you want to comment on the identity **and** purity of the product, as well as the efficiency of the technique used. Note that often it is just as important to discuss what the compound isn't as what it is - you should be in part like a lawyer, who offers concrete proof both for his idea as well as against other competing ideas. Do not forget to include any literature data (known mp's/bp's), as they really strengthen an argument.

For some of the experiments, you should relate your results to the concepts learned in CHM 2440 – did your experiment give the expected results? What underlying principles are responsible for your results? Proper use of subscripts and superscripts is required for full credit. Be sure to comment on causes for low yields or impure products that is specific to your experiment (ie, do not write “the yield was low due to spillage if you did not spill the liquid”).

Within the discussion should be an interpretation of any spectra obtained. Be sure to label important peaks on the spectrum itself.

For IR Spectra

List the significant 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).

For NMR Spectra

The only type of NMR spectrum analyzed in this course has a specific name: ^1H NMR. Describing this spectrum any other way (such as 1H NMR or H-NMR) is unacceptable and will result in loss of credit.

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.

Conclusions

Come to a conclusion about whether you succeeded in achieving the purpose of the work. It should not be any longer than 4 sentences. State only what can be backed up with information you have collected. Items mentioned in the purpose should be summarized here (Did the distillation work? Were you able to obtain a high yield of your product?). Note that the conclusion usually includes numerical data. Please do not use this section to express your personal feelings about the experiment. A good start for writing the conclusion is to look at your purpose and evaluate the objectives included therein. The conclusion should not include any new thoughts or statements – only summarize items previously discussed.

Example: *“The extraction of nutmeg seeds afforded yellow needles of trimyristin in a 9.6% recovery after recrystallization from acetone. The trimyristin was determined to be very pure based on the narrow melting point range (53.5 – 54.8) and it’s close agreement to the literature value. A longer period of heating at reflux may have afforded a higher percent recovery.”*

Calculations page

Attach a page showing explicitly all your calculations, with enough accompanying text that it is clear what you are calculating. This can be handwritten.

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

3. 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.

4. Grading

Format:

12%

(Is the report written with the correct sections, and things placed in the proper section?)

Were subscripts, superscripts, symbols, and/or units done properly?

Do graphs/spectra have titles?)	
Grammar:	12%
(Is the report written in the right tense? Are there spelling/grammar mistakes?)	
Results and Interpretation:	36%
(Are the results reasonable? Have the results been thoroughly analyzed and explained? Has comparison to literature values been done? Have results been related to concepts discussed in lecture (theory))?	
Significant figures and Measurements:	12%
(Are values/calculated values reported to correct significant figures?)	
Completeness:	12%
(Has all the required data been included? Is the experimental section complete?)	
Overall quality:	16%
(Does the report read like it was carefully and thoughtfully written? Is the report presented in a clear and organized fashion? How insightful is the report?)	

Format Mistakes to Avoid in Writing Lab reports

1. Incorrect tense/multiple tenses. All sentences should be written in the past tense – these are things you did. Most of your verbs should end in “-ed”.
“... *and pour into a separatory funnel.*” (Not past tense)
2. Incomplete Sentences. Every sentence needs at the minimum a noun (a subject) and a verb (what action the subject does).
“*Stirred for one hour.*” (there is no noun – what is being stirred?)
3. Pronouns should not be used.
“*I dried the solution with MgSO₄ ...*”
4. Misspellings. Note this includes misspelling chemical names.
5. Sentences should not begin with a number.
“*10 minutes later...*” (Rerword, for example, “*After 10 minutes...*”)
6. Every numerical piece of data reported needs to include the appropriate units.
“*The peak at 1540 was indicative of...*” (should be “*The peak at 1540 **cm**⁻¹ was indicative of..*”)
7. Failure to use superscripts/subscripts properly
“*m*” instead of “*mL*”; “*C3H6*” instead of “*C₃H₆*”

You could lose ¼ a point for EACH violation of the above. A report that receives no points on the format section must be rewritten and resubmitted in order to receive a grade on the report.