CHEMISTRY 234 – ORGANIC CHEMISTRY LAB II

This syllabus is subject to change pending notification verbally or via the email list.

Tuesday, Wednesday & Thursday, 1:10 – 4:00 pm

Prof's.	Kristin Hofferberth
Office:	Tomsich Hall 308
Office hours:	Tues -Thurs: 12:30 – 1 pm and 4 – 5 pm or by appointment
PBX:	5360
email:	hofferberthk
webpage:	*http://chemistry.kenyon.edu/faculty/hofferberth.htm*

Texts: Mayo; Pike and Trumper "*Microscale Organic Laboratory*" 4th edition
Zubrick "The Organic Chem Lab Survival Manual," 6th edition

Required Material: "Organic Chemistry Laboratory Notebook" – Chemical Education Resources, Inc. (CER)

Point Distribution:

7 Data Sets (100 pts each)	700
1 Lab Report with Data Set	200
Final Exam	100
Notebook (11 weeks @ 2 pts/week)	22
Quizzes (10 points each)	70
Total	992

Goals: Chemistry 234 builds your technical foundation in experimental organic chemistry. This semester will focus on the seven-step convergent synthesis of hexaphenylbenzene. You products will need to be isolated in high purity and yield not only for the product card, but to be used in the next step of the synthesis. If you successfully complete this synthesis before the end of the semester, you may check out of lab early or attempt two other short syntheses for extra credit. Careful planning and good experimental technique should allow you to complete the synthesis using only material you synthesize yourself.

Attendance: Organic chemistry continually builds upon itself, and it is quite easy to get behind if you miss a particular lab period. Also, the labs are often quite full. Therefore, attendance to your assigned laboratory section is mandatory. Once lab sections are finalized, you may not switch lab sections during the semester. If you miss lab due to an excused absence such as a family or medical emergency or scheduled sporting event, you must obtain permission from your instructor to attend an alternate lab section.

Course Meeting Time: We will meet in Tomsich 207 at 1:10 pm for a 20 – 30 minute pre-lab lecture during the first week of a particular experiment. The lab will begin with a 5 minute quiz germane to the experiment at hand (see below). The quiz always ends at 1:15 pm sharp and if you are not present for the quiz, you will receive no credit. You will have only your laboratory notebook to help you during the quiz, so you should make relevant notes therein. It is vital that these notes be clearly separate from what you write *during* lab. Planning your lab work ahead of time will increase your efficiency in lab. Following recitation, laboratory work will commence in Tomsich 209. If we are in the second week of an experiment, you may begin working promptly at 1:10 pm in Tomsich 209. You should confine your lab work to the scheduled hours. No extra time will be given if you are unable to complete an experiment due to a clear lack of pre-lab preparation or a lack of focus or efficiency during lab.

Analysis Party: The lab will be open Tuesday and Thursday night during the Math Science Skills Center from 7 – 9 pm and will be staffed by an undergraduate assistant. During this time you may perform analytical techniques such as melting point analysis and the various spectroscopies. No other experiments may be performed. As a guideline, if all you had was your sample and sample prep material, you can not do anything which would require opening your drawer.

Safety: The safety rules for the course are stated in Mayo, Chapter 2 and in Zubrick, Chapter 1. The most important rules are: 1) Wear safety goggles at all times – being in the lab without goggles will cost you 5 points

per incident. However, if you find an instructor in *any* lab without goggles, you are entitled to 10 points ; 2) Long pants and shoes that cover the entire foot must be worn at all times; 3) No eating or drinking; 4) Be mentally alert to hazards and prepared for emergencies. If you are uncertain whether something is safe, consult with me or the laboratory assistant.

Reading: The location of an experiment in your laboratory text is listed on your schedule. At the beginning of each experiment *Prior Reading* is listed. I will assume you have read this material as well as any relevant discussions and introductions, even if they do not immediately proceed the experiment in question.

Laboratory Notebooks: You are required to purchase and maintain a laboratory notebook; if you have one from the previous semester with many remaining pages, you may use it. Learning to keep an accurate and detailed lab notebook is critical as it is often your only source of information to help you remember what you actually did in lab when writing a lab report, trying to interpret spectra, etc. The notebook for this course contains white pages for your original record of work and yellow pages which are copies of the white pages. Although there is no single correct way to keep a notebook, *for this course you must precisely follow of the sample given on the course webpage*. I will check your notebooks before the end of lab each day (`check -, check, check +`) and they will be graded again in more detail when handed in. The most important rules are: 1) Your lab notebook is your scratch paper – observations, data and calculations should be recorded directly into your notebook at the time the observations or measurements are made; 2) You should write with indelible ink; 3) After you are finished with your experiment, your lab notebook should contain sufficient information for another investigator, familiar with the field, to be able to reproduce your work, using only your notebook as a guide. Other useful references can be found Mayo, pp. 30-32 or in Zubrick, Chapter 2.

Data Sets*:* At the completion of each experiment you will prepare a data set to be graded. Data sets are your proof that you have completed the experiment and will be the primary basis of your grade. The due dates for each data set are indicated on the schedule of experiments. Each data set will include the following items:

1. **Product Card** -- A product card is a summary card of the data collected and should be stapled to the front of your data set. Fill out all pertinent sections of the card and in the remarks section indicate the attachments that are stapled to the card (*e.g.* -- 'Included with this card: experimental section, ^{1}H NMR spectrum, ^{13}C NMR spectrum, IR spectrum, product vial, and lab notebook pages')

2. **Experimental Section** -- For each experiment you should write an experimental section in prose suitable for publication in an ACS organic chemistry journal (example experimental sections are linked to the course webpage). General guidelines for scientific writing should be followed. For a review of these guidelines refer to *A Brief Guide to Writing in Chemistry* (linked to the course webpage).

3. **Annotated Analytical Data --** All analytical data you turn in should be interpreted and clearly annotated. Annotation includes carefully drawing the structure of the compound under analysis and clearly correlating spectral signals with that structure. Examples of annotated spectra are linked to the course webpage. All spectra should have the following information on them: compound structure, compound name, compound ID number (I-JEH-017, lab book number - initials - page) and method of sample preparation (*i.e.* KBr pellet, thin film, $CDCl_3$, etc). For IR spectra, only major features need to be labeled. For NMR spectra, every peak must be accounted for.

4. **Labeled Vial Containing your Product --** Place your product material into a sample vial and label the vial with the compound name, compound ID number, and your name. The vial can be taped to the back of your product card.

5. **Carbon Copies of Pertinent Lab Book Pages --** After you have finished your notebook entry for a given experiment, tear out the carbon copies and turn them in with the data set.

Laboratory Report: A combined lab report for experiments [9] and [10] (approximately 3-6 typewritten pages, excluding attached data sets) will be written by each student this semester. The report is to be typewritten and should include the following sections: Abstract, Introduction, Results and

Discussion, Sample Calculations, Experimental, Questions, and References. You must also attach the Data Set for both experiments as an appendix to the report which will graded. All structures must be drawn using ChemDraw which is available on publicly accessible computers in Fischman 009 and the computer lab in Sam Mather or as a free download at (**https://accounts.cambridgesoft.com/login.cfm?serviceid=1**). *Chemical structures which are scanned, hand-drawn, copied from the web, etc are not acceptable.* Once you have received your own personal license key, access the software installer by mapping to the network drive: \\Potomac\Downloads, open the Software Packages folder, and open ChemDraw. Then choose an installer based on if your computer is a PC or a Mac.

The week following the completion of experiment [10] you will be required to bring a **complete draft** of your laboratory report to lab. We will discuss laboratory report writing and you will be asked to peer edit one of your classmates reports. The peer edited version of the report will be turned in with the final report the following week in lab (see lab schedule below).

Please refer to *A Brief Guide to Writing in Chemistry* for guidance in writing your report. A brief description of expectations for each section are included below:

Abstract: This is a summary of your results and the methods used to obtain them. It varies from 1-5 sentences, but never exceeds 110 words (approximately 8 lines).

Introduction: This is a statement describing the purpose and goals of the experiment. You should describe (in words, pictures, *balanced* chemical equations, mathematical equations, etc.) the *new* method(s) and/or chemical reaction(s) that you have investigated for this report.

Results and Discussion: This includes your data (results) and the interpretation/explanation of your data (discussion). Your data are most effectively presented using tables, graphs, lists, etc (spectral data should be included as appendices that are referenced in the text). You should interpret and discuss your data in terms of what you learned from them, and how the data reinforce or contradict the principles taught in this course and in Chemistry 231 (Organic lecture). Typically, this is the main body of text in your report.

Sample Calculations: This contains a detailed account of how you arrived at a certain number or result during a calculation. You should show *one* sample calculation for each type calculation (i.e. one each for % recovery, theoretical yield, % yield, optical rotation, etc.) that you performed for a particular experiment. As always, pay attention to significant figures.

Experimental: This is a description of what you actually did in the laboratory according to your notebook and not necessarily what is described in Mayo. *The experimental is written in the third person, the past tense, and in the passive voice*. Example experimental sections are given on the course webpage.

References: Sources of information that were used in the report (Mayo *et. al.*, Zubrick, CRC Handbook of Chemistry and Physics, *Science*, *Journal of Organic Chemistry*, etc.). This is an important and often overlooked section of a lab report. Please format references as endnotes in the ACS style (http://pubs.acs.org/books/references.shtml).

Quizzes: A 5-minute quiz will be given at the beginning of each experiment (on the first week of two-week experiments). The content of the quiz will be germane to the experiment at hand and may include questions about technique as discussed in Zubrick, questions assigned about the experiment at hand in Mayo (see lab schedule), spectral interpretation questions, or questions related to material that should be written in your lab book in preparation for the experiment.

Grading: Your performance will be evaluated over the entire semester based upon the following scale: 97% --> A+; 93% --> A; 90% --> A-; 87% --> B+; 83% --> B; 80% --> B-; 77% --> C+; 73% --> C; 70% --> C-; 67% --> D+; 63% --> D; 60% --> D-; <60% --> F. The instructors reserve the right to alter the scale at the completion

of semester however, the scale will be no harder than that listed above.

Academic Honesty: You are expected to follow the college policy for academic honesty (*Kenyon College Course of Study 2006-2007*, pp 26 – 29). All materials submitted for credit must be your own work. The complete policy is available for download (http://documents.kenyon.edu/courses/2006_2007/honesty.pdf).

Final Exam: The final will be cumulative for the semester. You may use your laboratory notebook but will *not* be allowed to use a calculator. Two sessions – May 5, 1:30 pm & May 6, 6:30 pm. You may attend either but must sign up for a session the last week of lab.

Section 504 of the Rehabilitation Act of 1973 and the Americans with Disabilities Act of 1990: If you have a disability and need accommodation in order to fully participate in this class, please identify yourself to Erin Salva, Coordinator of Disability Services (PBX 5145, salvae@kenyon.edu). All information and documentation of disability is confidential. No accommodations of any kind will be given in this course without notification from the Coordinator of Disability Services.

Withdraw Late: Co-requisite for this course is CHEM 232. However, withdrawing late (WL) from this lab course does not involve also withdrawing from the associated lecture course – they are separate courses with separate grades.

Equipment Loss or Breakage: There are no up-front chemistry lab fees; however, at the beginning of each year, you will need to sign a check-in sheet stating that you are accepting financial responsibility for any breakage or loss of lab drawer contents. Your student account will be assessed charges for lost or broken items at the end of the year.

CHEM 234: ORGANIC LAB SCHEDULE

Week of	Experiment	Location in text (suggested questions)	Data set due on arrival to lab week of
1/14	*Spectroscopic Identification of an Unknown* Methods: Boiling Point, mp, RI, solubility, IR, & ^{1}H NMR	Handout	1/28
1/21 & 1/28	*Experiment [A1$_a$] The Benzoin Condensation of Benzaldehyde* Methods: Macroscale, Reflux, Filtration, Recrystallization, m.p., IR Scale: 2.0 mL benzaldehyde	Mayo pp. 421-430 (1, 2, 3, 5) Handout	2/11
2/4 & 2/11	*Experiment [A2$_a$] Nitric Acid Oxidation of Benzoin: Synthesis of Benzil* Methods: Recrystallization, Thin Layer Chromatography, Column Chromatography* * if needed	Mayo pp. 431-435 (6, 8a, 9, 10) Handout	2/25
2/18	*Experiment [A3$_a$] Tetraphenylcyclopentdadienone* via *Aldol Condensation* Methods: Filtration, Recrystallization*, NMR * if needed	Mayo pp. 435-438 (13, 14)	3/17
2/25 & 3/17	*Experiment [A1$_b$] (*E*)-Stilbene* via *Horner-Wadsworth-Emmons Modified Wittig* Methods: Macroscale, Reflux, Extraction, Recrystallization, m.p., IR Scale: 1.2 ml benzaldehyde	Mayo pp. 439-441 (18, 20, 21)	3/31
3/24	*Experiment [A2$_b$] A Greener Bromination of Stilbene* Methods: Reflux Filtration, m.p., Recrystallization*, IR * if needed	Mayo pp. 441-445 (23, 24, 26) Handout	4/7
3/31	*Experiment [A3$_b$] Dehydrohalogenation of* meso*-Stilbene Dibromide: Diphenylacetylene* Methods: Filtration, Recrystallization, m.p., IR, NMR	Mayo pp. 446-449 (28, 29, 30)	4/14
4/7	*Experiment [A4$_{ab}$] Hexaphenylbenzene* via *Diels-Alder* Methods: thermolysis, IR, TLC **Lab Report: Seven-Step Convergent Synthesis of Hexaphenylbenzene**	Mayo pp. 449-453 (32, 33)	Draft due 4/21 Final Report due 4/28
4/14	*Fischer Esterification*	Handout	no data set, submit lab carbons at end of lab
4/21	*Lab Report Workshop (bring a complete draft of your report to lab)*	**Final Report for Seven-Step Synthesis due week of 4/28**	
4/28	***Tying-Up Loose Ends & Lab Check Out***		

While specific chapters in the Zubrick text are not indicated, you are expected to use the text to learn good laboratory technique.