CHEMISTRY 234 – ORGANIC CHEMISTRY LAB II

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

Tuesday or Wednesday, 1:10 – 4:00 pm

Prof.	John Hofferberth		
Office:	Tomsich Hall 312		
Office hours:	Mon Noon-3PM and Thu 1-3PM		
PBX:	5360		
email:	hofferberthj	*webpage:*	Moodle

Texts: Mayo; Pike and Trumper "*Microscale Organic Laboratory*" 5th ed.
Zubrick "The Organic Chem Lab Survival Manual," 6th ed.

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

Point Distribution:

6 Data Sets (100 pts each)	600
Lab Report Draft (20 pts Draft/20 pts Peer Edit Quality)	40
Lab Report	100
Exam	100
Notebook/Technique/Safety (12 weeks @ 3 pts/week)	36
Quizzes (6 points each)	60
Total	936

Academic Honesty: You are expected to follow the college policy for academic honesty. All materials submitted for credit must be your own work. *All academic infractions will be forwarded to the Chair and/or Academic Infractions Board and may result in a grade of F for the course, suspension or expulsion*.

Goals: Chemistry 234 builds your technical foundation in experimental organic chemistry. This semester will focus on a multistep convergent synthesis of hexaphenylbenzene. You will need to isolate products in high purity and yield not just for a data set, but also for use in subsequent synthetic steps. Careful planning and good experimental technique should allow you to complete the synthesis using only material you synthesize yourself.

Attendance: Organic chemistry builds on itself, and it is easy to get behind if you miss a lab period. Therefore, attendance to your assigned laboratory section is mandatory. Once lab sections are finalized, you may not switch during the semester. If you miss lab for an excused absence you must obtain permission from all instructors involved 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 an experiment. The class will begin with a 5 minute quiz germane to the experiment; if you are not present for the quiz, you will receive no credit. You may use your laboratory notebook during the quiz, so you should make relevant notes therein. These notes must 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; in the second week of an experiment, we will begin lab work promptly at 1:10 pm. You will 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.

Office Hours: If you are unable to attend my regularly scheduled office hours you may contact me for an appointment. If you would like to meet for the purpose or reviewing your written work, please email me a copy in advance of the meeting to allow me to provide careful feedback. I will provide verbal feedback during these meetings. I will not pre-grade your work.

Evening Analysis Sessions: The lab may be made available (7 - 9 PM) and 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 are allowed. As a guideline, if all you had was your sample and sample prep material, you may not do anything that 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. 2) Long pants and shoes that cover the entire foot must be worn at all times. If you are not properly clothed, you will not be admitted into lab. 3) No eating (gum included) or drinking in lab. 4) Be mentally alert to hazards and prepared for emergencies. If you are uncertain whether something is safe, consult the lab assistant or myself.

Reading: The location of an experiment in your lab 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 are not immediately proximate to the experiment in question. Also, some experiments will be performed in a manner different from that described in the text. Changes of this type are indicated on the schedule and background reading will also include materials placed on Moodle for that experiment. Read both the text and the Moodle materials in such cases.

Laboratory Notebooks: You are required to purchase and maintain a laboratory notebook; if you have one from the previous semester with sufficient pages, you may use it. Learning to keep an accurate and detailed lab notebook is critical as it is often the only source of information to help you remember what you actually did in lab when writing a report, interpreting spectra, testifying in court, etc. Although there is no single correct way to keep a notebook, *you will be expected to use a format mimicking that of the notebook page linked to the Moodle page for this class*. I will check your notebooks at the beginning and end of each lab (✓–, ✓, ✓+) and grade 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 in Mayo, pp. 30-32 or Zubrick, Chapter 2.

Data Sets: After finishing each experiment you will prepare a data set. Data sets are your proof that you have completed the experiment and will be the primary basis of your grade. Data sets have 3 parts and will be due as described below. Late sets will be penalized 5 points per day. Each data set will include the following items:

1. Electronic Data Set (EDS): The EDS will have two sections and will be submitted on TurnItIn (via Moodle). A Microsoft Word template file for the EDS is linked to the Moodle page for the course. The first section will include essential summary information including: product description, yield and % yield and a list of analytical data and notebook pages to be separately submitted in hardcopy form (see below). The second section of the EDS will be an experimental procedure. For each experiment you will draw a reaction scheme (ChemDraw) and write an experimental section in prose suitable for publication in an ACS journal (links to representative papers may be found on the course webpage). Annotated spectral data (NMR and IR, see below for instructions on annotataion) in electronic format should be included in the EDS (see template file for details). General guidelines for scientific writing should be followed. Each EDS will be submitted through TurnItIn and will be due at 11:59 pm the day before your lab section meets. Due dates are indicated on the schedule.

2. Hardcopy Data Set (HDS): The HDS will have two components. The first will be all analytical data collected for the experiment that is not in electronic format. The data should be interpreted and clearly annotated. Annotation includes carefully drawing the structure of the compound under analysis and clearly correlating spectral signals to that structure. Links to sample annotated spectra are on the course webpage. All spectra should have the following information on them: compound structure, compound name, compound ID number (YDG-04-009, initials – lab book number - page), your name, your class number, the instrumental details (NMR carrier frequency) and method of sample preparation (*i.e.* KBr pellet, thin film, $CDCl_3$, etc). For IR, only major features are labeled. For NMR, every peak must be accounted for. The second section of the HDS is your lab notebook carbons. The HDS will be due at the beginning of lab on the day the lab is due.

3. Labeled Product Vial: Place a small sample (2-5 mg) of your product into a vial labeled with the compound name, compound ID number, and your name. Place the vial in the submission rack at the beginning of lab the day the data set is due.

Laboratory Report: A combined lab report for experiments for the convergent synthesis of hexaphenyl benzene (max length: 2400 words) 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, Experimental, and References. All structures must be drawn using ChemDraw which is available on publicly accessible computers or as a free download http://scistore.cambridgesoft.com/sitelicense.cfm?sid=94. *Chemical structures which are scanned, hand-drawn, copied from the web, etc. are not acceptable.* The document should be written in a single column format with 12 pt font and double spaced.

Please refer to *A Brief Guide to Writing in Chemistry* (on Moodle) 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. Briefly (1 or 2 paragraphs) describe the goal of the overall experiment (a retrosynthesis and with discussion) and then focus on two of the reactions performed by you this semester by describing them in detail with both prose, a general example of the reaction, a mechanism scheme, etc.

Results and Discussion: This includes your data (results) and the interpretation/explanation of your data (discussion). Results of synthesis projects are typically reported both visually as forward synthetic reaction schemes (with reagents and yields) and in prose describing each reaction and its outcome. Spectra are included as Supplemental Information and can be referenced in the text when appropriate. You should interpret and discuss your data in terms of what you learned from each reaction, and how the data are consistent (or not) with the expected outcome and/or principles taught in this and other courses. Typically, this is the main body of text in your report.

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*. These will be edited and improved versions of the experimental sections submitted in the EDSs.

References: Sources of information that was 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. On what are you basing your statements? A book, a journal article, your own imagination? Please format references as endnotes in the ACS style (http://library.williams.edu/citing/styles/acs.php). Please format in-text citations as superscript numbers only.

Quizzes: As noted above, a 5-minute quiz will be given at the beginning of each experiment (on the first week of two-week experiments, excepting the first experiment of the semester). The content of the quiz will be germane to the experiment at hand and may include questions about technique as discussed in Zubrick, suggested questions assigned from Mayo, spectral interpretation, or questions about material you should know in preparation for the experiment.

Grading: Your performance will be evaluated over the entire semester based upon the following absolute 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.

Exam: An exam covering the experiments in the hexaphenylbenzene synthesis will be given on the date indicated on the syllabus.

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.

Title IX: Kenyon College and the instructor of this course seek to provide an environment that is free of bias, discrimination, and harassment. If you have been the victim of sexual harassment/misconduct/assault we encourage you to report this. If you report this to the instructor, he must notify our college's Title IX coordinator. For more information about your options at Kenyon, please go to: http://www.kenyon.edu/directories/offices-services/office-of-equal-opportunity/sexual-assault-and-harassment/

The instructor of this course will respect the privacy of all personal conversations with his students. In the case that he learns of activities or incidents that violate Title IX (sexual discrimination and/or harassment) or otherwise violate the law he is required by law to report such information to the Title IX coordinator or campus law enforcement, respectively.

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.

– This syllabus constructed over multiple years with contributions from Profs. Hunsen, Hofferberth, Hofferberth, and Getzler –

CHEM 234: ORGANIC LAB SCHEDULE

Week of	Experiment	Reference/Week Due*	Questions (Ch. 7)
8/28	Orientation, Safety and NMR training	**Week Due: 9/4**	
9/4 & 9/11	*Experiment [A1$_a$] The Benzoin Condensation of Benzaldehyde* Methods: Macroscale (Kem Kits), Reflux, Filtration, Recrystallization, mp, IR Scale: 2 ml benzaldehyde	Mayo pp. 429 **Week Due: 9/18**	1, 2, 3, 5
9/18 & 9/25	*Experiment [A2$_a$] HNO_3 Oxidation of Benzoin: Synthesis of Benzil* ***Note: Alternative Protocol (HNO_3 Oxidation) – see handout*** Methods: Reflux, Filtration, Recrystallization, mp, IR, TLC, Chromatography (if needed) Scale: $\geq$800 mg	Handout Mayo pp. 433 **Week Due: 10/9**	6, 8a, 9, 10
10/9	*Experiment [A3$_a$] Tetraphenylcyclopentadienone* via *Aldol Condensation* Methods: Filtration, Recrystallization (if needed), IR, mp, NMR Scale: >400 mg	Mayo pp. 438 **Week Due: 10/16**	12, 13, 14, 15
10/16 & 10/23	*Experiment [A2$_b$] A Greener Bromination of Stilbene* ***Note: Alternative Protocol (HBr/H_2O_2 Bromination) – see handout*** Methods: Reflux Filtration, mp, IR, Recrystallization (if needed) Scale: See Handout	Handout Mayo pp. 444 **Week Due: 10/30**	23, 24, 25, 26
10/30 & 11/6	*Experiment [A3$_b$] Dehydrohalogenation of* meso-*Stilbene Dibromide: Diphenylacetylene* Methods: Filtration, Recrystallization, mp, IR, NMR Scale: 400 mg	Mayo pp. 450 **Week Due: 11/13**	28, 29, 30
11/13 & 11/27	*Experiment [A4$_{ab}$] Hexaphenylbenzene* via *Diels-Alder* Methods: thermolysis, IR, TLC (3:1 hexanes:CH_2Cl_2)[#] Scale: whatever you've got! **Lab Report: Convergent Synthesis of Hexaphenylbenzene** (no data set)	Mayo pp. 453 **Draft: Week of 12/4** **Final: Week of 12/11** Word Limit: 2,400	32, 33, 34
12/4	***Peer Editing of Lab Report***		
12/11	Final Exam (Final Report Due Prior to Exam Time) Tuesday Section (234.01) Exam on Monday 12/12 6:30PM Wednesday Section (234.02) Exam on 12/15 Thursday 1:30PM		

*All Electronic Data Sets will be submitted through TurnItIn and will be due at 11:59 pm on the day preceding your lab section. For example, if you are in the *Tuesday* section, your first Experimental will be due at 11:59 on *Monday*, September 19th . Hardcopy Data Sets and product vials are due at the beginning of lab on the day the data set is due. For example, if you are in the *Tuesday* section, your first Hardcopy Data Set and vial are due on September 20th at the beginning of lab.

[#] TLC conditions developed by Lars Matkin ('12).