

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. Yutan Getzler
Office: Tomsich Hall 308
Office hours: Monday & Wednesday 10 am to noon; Tuesday, 10 to 11 am.
PBX: 5304
email: getzlery webpage: <http://chemistry.kenyon.edu/faculty/getzler.htm>

Texts: Mayo; Pike and Trumper "Microscale Organic Laboratory" 5th (or any, really) ed.
Zubrick "The Organic Chem Lab Survival Manual," 6th (or any, really) ed.
Handouts: Found at P:/Class/Chemistry/Chem 234

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

Point Distribution:

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6 Data Sets (100 pts each)	600
Lab Report Draft	40
Lab Report	150
Final Exam	100
Notebook (13 weeks @ 3 pts/week)	39
Quizzes (10 points each)	70
<hr/> Total	<hr/> 999

Academic Honesty: You are expected to follow the college policy for academic honesty (<http://www.kenyon.edu/x11747.xml>). All materials submitted for credit must be your own work. All academic infraction will be forwarded to the 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, granted only by the Dean of Students, 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 at hand and it ends at 1:15 pm sharp; 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. 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.

Evening Analysis Sessions: The lab will be open, as needed, Sunday or Thursday nights from 7 – 9 pm, 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. However, if you find me in *any* chemistry lab without my goggles, you are entitled to 10 points. 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.

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, *for this course you must precisely follow the format on the attached page*. I will check your notebooks at the end of each lab (✓-, ✓, ✓+) and grade them in 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 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 are due at the beginning of lab on the dates indicated on the schedule of experiments, generally two weeks following completion. Late sets will be penalized 5 points per day. Each data set will include the following items:

1. Product Cards: 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, ¹H NMR spectrum, ¹³C NMR spectrum, IR spectrum, product vial, and lab notebook pages’).

2. Experimental Procedure: For each experiment you will write an experimental section in prose suitable for publication in an ACS journal (links to representative papers may be found on the course webpage). General guidelines for scientific writing should be followed. All will be submitted through TurnItIn and will be due at 11:59 pm on the day preceding your lab section. Late submissions will be penalized 1% per minute.

3. Annotated Analytical Data: All of your analytical 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) and method of sample preparation (*i.e.* KBr pellet, thin film, CDCl₃, etc). For IR, only major features are labeled. For NMR, every peak must be accounted for.

4. Labeled Product Vial: Place a small sample of your product into a vial labeled with the compound name, compound ID number, and your name. Please tape the vial to the upper left corner of the product card.

5. Lab Notebook Carbons: Attach the yellow copies of your laboratory notes for the experiment.

Laboratory Reports: A combined lab report for experiments for the convergent synthesis of hexaphenyl benzene (approximately 10 typewritten pages, excluding attached spectra) 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 in Fischman 009 and the computer lab in Sam Mather 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.*

To allow for feedback, a draft will be due on 3/25, prior to completing the final experiment.

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. Spectra are included as Supplemental Information and are referenced in the text. You should interpret and discuss your data in terms of what you learned from them, and how the data are consistent (or not) with the 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.*

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.

Final Exam: You may attend any one of three times for the final exam: May 6th, 7th or 9th at 1:30 pm.

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.

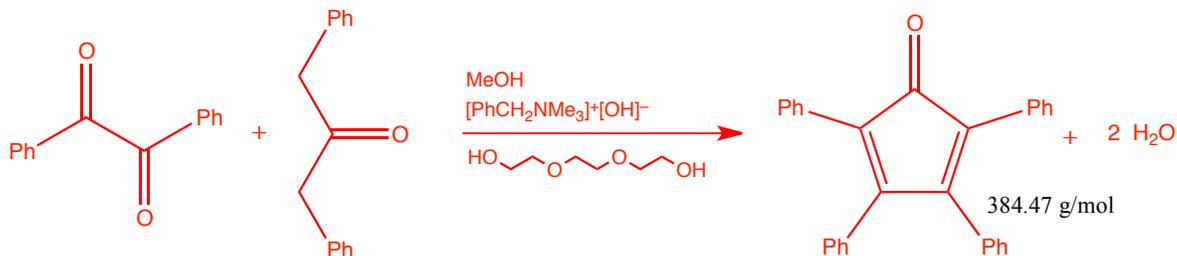
Withdraw Late: Co-requisite for this course is CHEM 232. However, withdrawing late (WL) from this lab course does not involve also withdrawing from the associated lecture course – they are separate courses with separate grades.

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

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

EXAMPLE LAB NOTEBOOK PAGE

01/17/2007



(cf: Mayo; Pike; Trumper, 436 – 437)

chemical	Benzil	1,3-diphenylacetone	triethylene glycol	$[\text{BnNMe}_3][\text{OH}]$
source	Aldrich, 98%	Aldrich, 98%	Akros, reagent	stockroom
purification	UAR*	UAR	UAR	UAR
MW	210.23	210.27		153.22
d(g/ml)	N/A (solid)			40% in MeOH
amount	0.401 g	0.406 g	2 ml	0.4 ml
mmol	1.91	1.93		1.
eq.	1.00	1.01		0.5

$(0.4 \text{ ml BnNMe}_3\text{OH soln}) \cdot (0.4 \text{ g BnNMe}_3\text{OH} / 1 \text{ ml BnNMe}_3\text{OH soln}) \cdot (1 \text{ mol BnNMe}_3\text{OH} / 153 \text{ g BnNMe}_3\text{OH}) = 1 \text{ mmol BnNMe}_3\text{OH}$

- benzil, diphenylacetone and triethylene glycol added to 5 ml conical vial (equipped w/air-condenser + spin vane)
- heated until sol'n homogeneous (sand bath $\sim 150^\circ\text{C}$, ~ 10 min)
- added 0.4 ml benzyltrimethylammonium hydroxide sol'n
- as sol'n cooled, deep purple/brown x-tals began to precipitate
- poured rxn into 15 ml Erlenmeyer, rinsed remaining material into Erlenmeyer w/ ~ 5 ml cold MeOH (reagent)
spilled some of sol'n, lost some x-tals
- cooled flask in ice bath (~ 10 min)
- isolated w/Hirsch funnel, rinsed 3 x w/small minimum cold MeOH
- x-tals are mottled, dark purple
- sample (YDG-075A) left to dry until next lab

1/25/07

yield: 0.80 g (2.1 mmol, $>100\%$!?!?)

mp: 200 – 219 (lit: 220–221)

damn! – must re-xtalize

- in 15 ml Erlenmeyer, dissolved YDG-075A in min. hot acetone & added MeOH until ppt began to appear
- added touch more acetone, covered w/parafilm & placed in ice bath for 1.5 hrs
- x-tals (isolated as above) are unblemished dark purple
- covered funnel w/kimwipe & drew air through for $\sim 1/2$ hr; x-tals look dry \rightarrow YDG-4-075B

yield: 0.43 g (1.1 mmol, 48 %)

mp: 218 – 220 (lit: 220–221)

IR – KBr pellet (see attached spectrum with relevant peaks labeled) }
NMR – ^1H CDCl_3 (see attached spectrum with *all* peaks labeled) } all look good!

* UAR = used as received