

CHEMISTRY 233 – ORGANIC CHEMISTRY LAB I

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

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

Prof. Yutan Getzler
Office: Tomsich Hall 308
Office hours: Tue – Thurs 10 am – 12 pm, or by appointment
PBX: 5304
email: getzlery

Texts: Mayo; Pike and Trumper "*Microscale Organic Laboratory*" 4th edition
Zubrick "*The Organic Chem Lab Survival Manual*," 5th edition

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

Point Distribution:

6 Product Cards & 2 Lab Reports (100 pts each)	800
Final Exam	100
Notebook (12 weeks @ 2 pts/week)	24
<u>Quizzes (10 points each)</u>	<u>80</u>
Total	1004

Goals: Chemistry 233 builds your technical foundation in experimental organic chemistry. The course emphasizes reactions, techniques and ideas that will be used in other courses and any research projects requiring physical manipulations of materials. This lab integrates and illustrates Chemistry 231 (Organic lecture).

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 me 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. 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.

Thursday Night Analysis Party: The lab will be open Thursday night from 7 – 10 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 me in *any* 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; 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 the format shown on the attached page*. I will check your notebooks before the end of lab each day (✓-, ✓, ✓+) 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.

Product Cards: The card should be filled out with all of the pertinent information in ink. Attach all the spectra, chromatograms, answers to assigned problems & lab carbons (but not pre-lab carbons!) to the card. In addition, the entire remaining product (the material not used for physical measurements, spectra, or GC) should be submitted in a vial clearly labeled with your name and its contents.

Spectra: All spectra should have the following information on them: compound structure, compound name, compound ID number (see below) method of sample preparation (*i.e.* KBr pellet, thin film, CDCl₃, etc). For IR spectra, only major features need to be labeled. For NMR spectra, every peak must be accounted for.

Laboratory Reports: A typewritten laboratory report is due at 1:10 pm for a particular lab two weeks after that lab is scheduled to be finished (see **Laboratory Schedule** for due dates). Late reports will be penalized at the rate of 5 points per day. The report (approximately 3-6 typewritten pages, excluding attached spectra, chromatograms & lab carbons), written in your own words, is required for only two labs this semester, **and thus I expect them to be of a very high quality**. Each written lab report should be divided into the following sections: Abstract, Introduction, Results and Discussion, Sample Calculations, Experimental, Questions, and References. You must also attach the lab carbons (but not the pre-lab carbons!) which will be graded. Excluding the questions, 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.

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

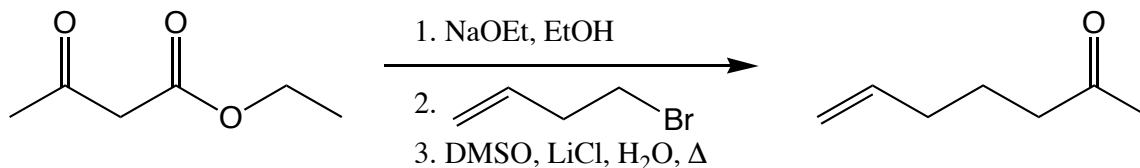
Introduction: This is a statement describing the purpose and goals of the experiment. You should describe (in words, *balanced* chemical equations, mathematical equations, etc.) the *new* method(s) and/or chemical reaction(s) that you have investigated for this report.

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. 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.

Laboratory Reports, continued:

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.* After a new method has been described in detail for a particular lab report, you can simply use the term to describe the method for any subsequent reports. Following is a sample experimental write-up for the synthetic procedure shown below:



6-Hepten-2-one. Na (25.0 mg, 1.09 mmol) was added to a solution of ethyl acetoacetate (YDG-073B, 130 mg, 1.00 mmol) in ethanol (1.0 mL) in a 3-mL conical vial equipped with a reflux condenser and a drying tube. The mixture was allowed to stir until all of the Na had dissolved. 4-Bromo-1-butene (150 mg, 1.11 mmol) was added, and the mixture was heated to reflux until it was no longer basic to litmus. The reaction mixture was cooled, filtered, and the solvent was removed by distillation. The resulting oil (153 mg, 83% crude) was dissolved in dimethyl sulfoxide (0.5 mL). H₂O (40 μL, 2.2 mmol) and LiCl (63.6 mg, 1.50 mmol) were added, and the solution was heated to reflux for 1 h. The resulting dark brown solution was diluted with saturated aqueous NaCl (1.5 mL), extracted with ether (3 x 0.5 mL), dried over excess MgSO₄, filtered, and the solvent removed by warming the flask under a stream of nitrogen in a warm sand bath. The resulting residue (YDG-076A) was distilled into a Hickman still head, and the fraction boiling in the range of 145-148 °C was collected to give 76.7 mg (68.5%) of 6-hepten-2-one as a colorless liquid (YDG-076B). IR (neat): $\nu_{\text{CO}} = 1711 \text{ cm}^{-1}$.

Notice the use of common abbreviations (*e.g.* volumes in mL; weights in mg or g; molar amounts in mmol; temperature in °C; time in s, min, h, or d; distance in mm or cm; etc.). Make use of them. Also notice that a simple chemical formula is used instead of the compound's name whenever possible. For example "Na" is written instead of "sodium," or "H₂O" instead of "water," because these formulae can only describe these particular compounds. However, "C₂H₆O" cannot be written for ethanol, since another compound, dimethyl ether, has the same formula. Therefore, the word "ethanol" is written instead. Also notice that the sample is named with the author's initials and notebook page

Questions: Tersely answer the questions (as indicated in the **Laboratory Schedule**) for each experiment.

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?

After you are finished writing the text of your report, check it for spelling, awkward sentences, and sentence fragments; edit your report accordingly. One good way to edit is to read what you have written out loud.

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.

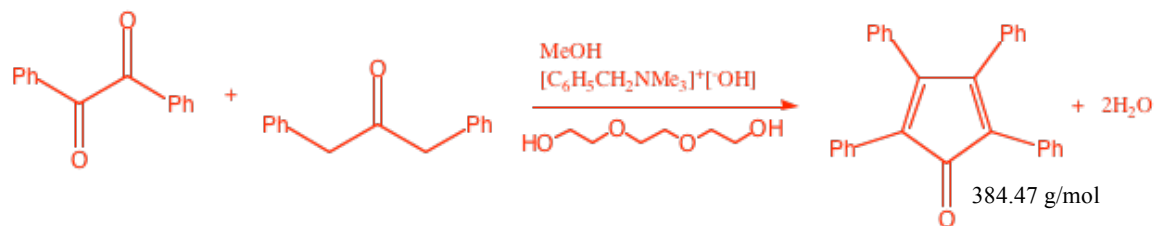
Academic Honesty: You are expected to follow the college policy for academic honesty (*Kenyon College Course of Study 2006-2007*, pp 26 – 29). All materials submitted for credit must be your own work. The complete policy is available for download (http://documents.kenyon.edu/courses/2006_2007/honesty.pdf).

Final Exam: Two sessions – Dec. 16, 1:30 pm & 6:30 pm. You may attend either but must tell me which.

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.

EXAMPLE LAB NOTEBOOK PAGE

01/17/2006



(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

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

- 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-4-075A) left to dry until next lab

1/25/06

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

mp: 200 – 219 (lit: 220–221)

damn! – must re-xtalize

- in 15 ml Erlenmeyer, dissolved YDG-4-075A 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