

# CHEMISTRY 234.03: – ORGANIC CHEMISTRY LAB II

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

Thursday, 1:10 – 4:00 pm

Prof. Yutan Getzler

Office: Tomsich Hall 308  
Office hours: Wed 9 am – 12 pm, Thurs 11 am – 12 pm, or by appointment.  
PBX: 5304  
email: getzlery

**Texts:** Mayo, Dana W.; Pike, Ronald M. and Trumper, Peter K. "Microscale Organic Laboratory: With Multistep and Multiscale Syntheses," 4th edition  
Zubrick, James W. "The Organic Chem Lab Survival Manual," 5th edition

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

## Point Distribution:

7 Lab Reports/Product Cards (100 point each)	700
Final Exam (5/3 1:10 – 3:40 pm)	200
<u>Quizzes (10 points each), safety, clean-up</u>	<u>100</u>
Total	1000

## Rules for the Course:

**Goals:** Chemistry 234 continues to build 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 material manipulations. The material covered in this lab integrates and illustrates Chemistry 231 and 232 (Organic lecture) material.

**Attendance:** Organic chemistry continually builds upon itself, and it is quite easy to get behind if you miss a particular lab period. Furthermore, enrollment is quite high this year. Therefore, attendance to your assigned laboratory section is mandatory. Once lab sections are finalized, you are strongly discouraged from switching lab sections during the semester. If it is absolutely imperative that you perform an experiment during another lab section for a particular week, you must obtain permission from myself and the instructor of the section you wish to attend. The Tuesday section is taught by Prof. Hunsen while Wednesday and Thursday sections are taught by myself.

**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 lecture will begin with a 5 minute quiz germane to the experiment at hand. 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 have relevant notes in your notebook. 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 (and last) week of an experiment, you can begin working promptly at 1:10 pm in Tomsich 209. You should confine, as much as possible, 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.

**Safety:** The safety rules for the course are stated in Mayo, Chapter 2 and in Zubrick, Chapter 1. In the laboratory, the most important rules are: 1) Wear safety goggles at all times -- I will warn you only once this semester before making point deductions; 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.

## Rules for the Course, continued:

**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. Although the notebook will only be graded when you hand the carbons in with your product card or lab report, I strongly encourage you to check with me periodically regarding the style and quality of your notebook. 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.

**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 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.

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

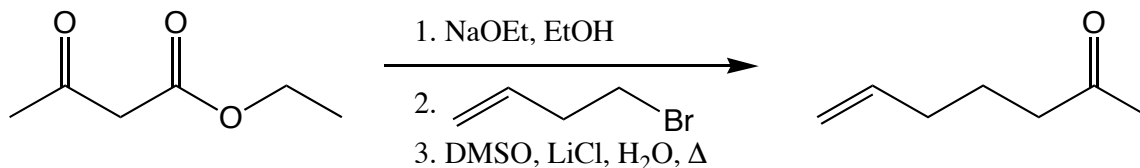
Introduction: This is a statement that describes the purpose and goals of the experiment. You should describe (in words, *balanced* chemical equations, mathematical equations, etc.) the *new* method(s) and/or chemical reaction(s) that you have investigated for this report.

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.

## Rules for the Course, continued:

**Experimental:** 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 experiment shown below:



**6-Hepten-2-one.** Na (25.0 mg, 1.09 mmol) was added to a solution of ethyl acetoacetate (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). H<sub>2</sub>O (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 MgSO<sub>4</sub>, filtered, and the solvent removed by warming the flask under a stream of nitrogen in a warm sand bath. The resulting residue 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-076C).

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 "H<sub>2</sub>O" instead of "water," because these formulae can only describe these particular compounds. However, "C<sub>2</sub>H<sub>6</sub>O" 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.

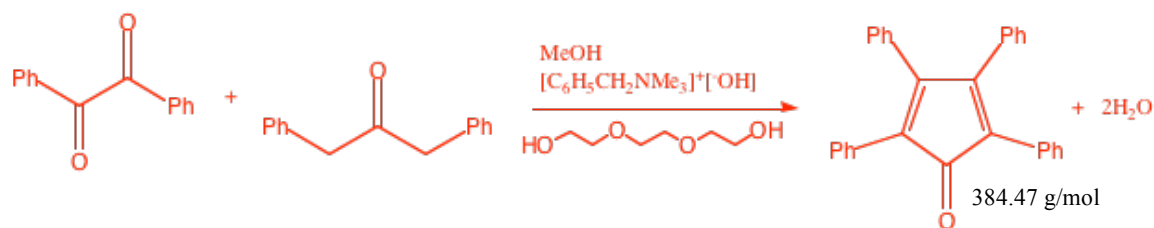
**Grading:** Your performance will be evaluated over the entire semester based upon the following absolute scale: 97% --> A+; 93% --> A; 90% --> A-; 87% --> B+; 83% --> B; 80% --> B-; 77% --> C+; 73% --> C; 70% --> C-; 67% --> D+; 63% --> D; 60% --> D-; <60% --> F.

**Academic Honesty:** You are expected to follow the college policy for academic honesty as outlined in the "Kenyon College Student Handbook 2003-2004" on page 26. All materials submitted for credit must be your own work. For more information, read the attached article.

**Final Exam:** The final exam will be given during the last day of lab and will be 1.5 hours long (5/3 1:10 – 3:40 pm). It will be open lab-notebook.

**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](mailto: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.

01/17/2006



(cf: Mayo; Pike; Trumper, 436 – 437)

chemical	benzil	1,3-diphenylacetone	triethylene glycol	[BnNMe <sub>3</sub> ][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 ~150 °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, &gt;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 ~1/2 hr; x-tals look dry → 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 – <sup>1</sup>H CDCl<sub>3</sub> (see attached spectrum with *all* peaks labeled) } all look good!

\* UAR = used as received