Advanced Organic Laboratory (Chem 373) – Spring 2006

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

Instructor availability: Monday 1:10 – 4:00 pm, Tomsich 001

Prof. Yutan Getzler

Office:	Tomsich 308
Office hours:	Wed 9 am – 12 pm, Thurs 11 am – 12 pm, or by appointment.
PBX:	5304
email:	getzlery
website:	http://chem.kenyon.edu/faculty/getzler.htm

Required Texts:

J. Am. Chem. Soc., *Angew. Chem. Int. Ed., Tetrahedron, Science, etc…*

Potentially Useful Texts:

Zubrick – The Organic Chem Lab Survival Manual
Mayo, Pike & Trumper – Microscale Organic Laboratory

Required Material: bound laboratory notebook, three-ring binder (for spectra)

Point Distribution:

Percent purity & percent enantiomeric excess of epoxide	400
Notebook & spectra (as of 3/3/06)	100
Individual independent project – to be determined	400
Notebook & spectra (as of 5/5/06)	100
Total	1000

Goals: The primary goal of this course is to foster independence in design and safe implementation of multi-step experimental procedures to achieve a stated objective. Supporting this are many other goals including meticulous recordkeeping in a laboratory notebook and of spectral data. New experimental techniques will be introduced including macro-scale synthesis and, perhaps, the handling of air-sensitive compounds. You will also need to be able to find relevant information using databases such as SciFinder and Science Citation Index. Once that information has been located, you will need to be comfortable accessing both electronic and print journal articles as well as their supplemental information sections.

Safety: This is a synthetic laboratory. In many of these experiments you will be working both on a larger scale and with more hazardous compounds then you have before. Thus you must always wear appropriate protective clothing. Safety glasses, long pants and shoes that cover the entire foot are required at all time. If I see you in the lab without any of these three items, you will loose 5 points from your final score. To be fair, if you see me in the lab without any of these three items, I will add 10 points to your final score. I strongly recommend that you also wear gloves and a lab coat at all times. These items provide important temporary barriers which can and should be quickly removed when (not if) you accidentally spill a chemical. Another important aspect of safety is proper labeling. All flasks must be labeled with their contents in an unobscurable manner. Reactions must be labeled with a reaction ID tag. Finally, while you will often work outside of class time, you should never work in the lab by yourself – make sure some in is lab with you.

Attendance: Attendance during the scheduled lab time is required. During that time I may demonstrate new techniques or highlight theoretical background. I will be in the lab throughout the scheduled time in the event that you have further questions. You are also expected to put in as much time as is needed outside of class to complete the projects.

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 your only source of information to help you remember what you actually did in lab. While there is no single correct way to keep a lab notebook, for this course you must precisely follow the format shown on the attached page. Although it will only be graded twice during the semester, 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 in Zubrick, Chapter 2.

Spectra: For any given spectrum, write the compound index number (*e.g.* YDG–4–075A) and structural formula clearly at the top. The method of sample preparation should also be clearly indicated (*i.e.* KBr pellet, Nujol mull, neat, CD_2Cl_2…). For IR, you need only indicate major bands (C=O, C-H, aromatic, CO_2, etc…). For NMR, ever peak must clearly be identified – this includes residual solvents (indicate which solvent), water, remaining starting material, etc. We will go over this with actually spectra from the class as they are taken.

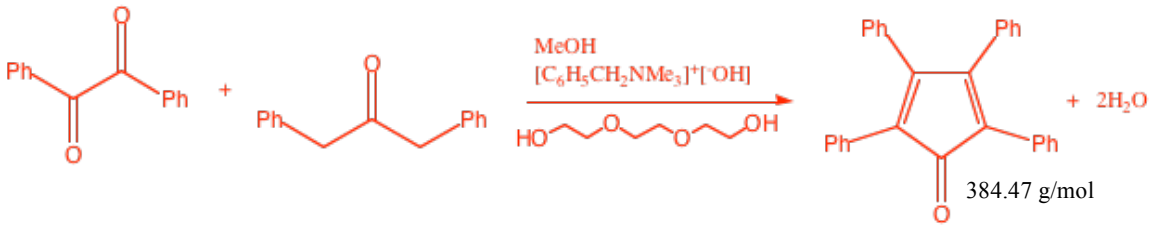
Structure: The first half of the semester will involve a very specific project geared towards the synthesis and use of an important catalyst for the resolution of terminal epoxides. In the second half of the semester you can use that catalyst to investigate other reactions, use the ligand you built to make other catalysts or work on an entirely different catalytic system.

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 must follow the college policy for academic honesty as outlined in the "Kenyon College Course of Study 2005-2006," pp 26-29. All materials submitted for credit must be your own work. http://www1.kenyon.edu/academics/cos/2005-06/downloads/acad_honesty.pdf

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.

01/18/2006



(cf: Mayo; Pike; Trumper, 436 – 437)

chemical	benzil	1,3-diphenylacetone	triethylene glycol	$[BnNMe_3][OH]$
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 ml $BnNMe_3OH$ soln)*(0.4 g $BnNMe_3OH$/1 ml $BnNMe_3OH$ soln)*(1 mol $BnNMe_3OH$/153 g $BnNMe_3OH$) = 1 mmol $BnNMe_3OH$

- 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 benzyltrimethylamonium 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)
crap… 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 spectra book: YDG-4-075B)
NMR – ^{1}H & ^{13}C, $CDCl_3$ (see spectra book: YDG-4-075B) } all look good!

* UAR = used as received