

# Advanced Organic Laboratory (Chem 373) – Spring 2007

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

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

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## 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	450
Notebook & spectra (as of 3/2/07)	50
Individual independent project – to be determined	250
<u>Notebook &amp; spectra (as of 5/4/07)</u>	<u>250</u>
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 than 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 lose 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 – always make sure someone is in the lab with you.

**Waste:** Waste jugs must be labeled with the full name of the chemical and the approximate amount present. Abbreviations or chemical notation such as EtOH or CH<sub>2</sub>Cl<sub>2</sub> are not acceptable.

**Attendance:** Attendance during the scheduled lab time is required. I will rarely if ever present any material. However, during that time I will be available to answer your questions. Given this structure, I expect you to come to lab having thoroughly read the relevant material. You may need to read through it several times to

begin to understand it. I expect you will have questions, and I will be more than happy to answer them. 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<sub>2</sub>Cl<sub>2</sub>...). For IR, you need only indicate major bands (C=O, C-H, aromatic, CO<sub>2</sub>, etc...). For NMR, every peak must clearly be identified – this includes residual solvents (indicate which solvent), water, remaining starting material, etc. We will go over this with actual spectra from the class as they are taken.

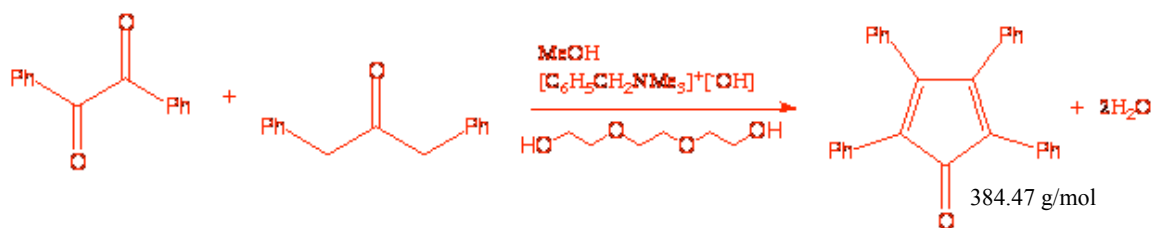
**Structure:** The first half of the semester will involve a very specific project geared towards the synthesis and use of an important catalyst for the resolution of terminal epoxides. In the second half of the semester you can use that catalyst to investigate other reactions, use the ligand you built 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 are expected to follow the college policy for academic honesty (*Kenyon College Course of Study 2006-2007*, pp 26 – 29; [http://documents.kenyon.edu/courses/2006\\_2007/honesty.pdf](http://documents.kenyon.edu/courses/2006_2007/honesty.pdf)). 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.

**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.

01/18/2007



(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-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)

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-075B

yield: 0.43 g (1.1 mmol, 48 %)

mp: 218 –220 (lit: 220–221)

IR – KBr pellet (see spectra book: YDG-075B)

NMR – <sup>1</sup>H & <sup>13</sup>C, CDCl<sub>3</sub> (see spectra book: YDG-075B)

} all look good!

\* UAR = used as received