CHEMISTRY 233 – ORGANIC CHEMISTRY LAB I

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

Wednesday, 1:10 – 4:00 pm

Instructor: Vivian Ezeh
Office: Tomsich Hall 208
Office hours: Tues, 9 am – 11 am; or by appointment
PBX:
email: ezehv

Texts: Mayo; Pike and Forbes "*Microscale Organic Laboratory*" 5th ed. (or any edition, really)
Zubrick "*The Organic Chem Lab Survival Manual*" 6th ed.

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

Point Distribution:

5 Data Sets (100 pts each) and 1 Unknown (70 pts)	570
NMR Section	80
Lab Report	150
Final Exam	100
Notebook (8 weeks @ 3 pts/week)	24
Laboratory Hygiene (9 weeks @ 3 pt/week)	27
Quizzes (10 points each)	50
Total	1001

Goals: Chemistry 233 complements Chemistry 231 (Organic lecture) and builds a good foundation in experimental organic chemistry. In this course you will learn organic chemistry reactions and laboratory techniques that you will find useful in future chemistry research projects. You will also learn to write experimental protocols in a manner suitable for publication in a scientific literature.

Attendance: Many experiments are linked and you will fall behind if you miss class. Also, lab sections are full. Therefore, attendance to your assigned section is mandatory. Once lab sections are finalized, you may not switch during the semester. If you miss lab for an excused absence 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. The quiz ends at 1:15 pm sharp; if you are not present, you will receive no credit. You may use your laboratory notebook 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.

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.

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 and gloves at all times – being in the lab without goggles will cost you **5 points** per incident. 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 or drinking in lab. 4) Be mentally alert

to hazards and prepared for emergencies. If you are uncertain whether something is safe, consult with me or the lab assistant.

Reading: The location of an experiment in your 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 precede the experiment in question.

Laboratory Notebooks: You will 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 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 shown 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) Start the record of a new experiment on a fresh page of your notebook and document every observation, data and calculations as soon as these observations or measurements are made. 2) Your lab notebook must contain sufficient information for another investigator familiar with the field, to reproduce your work using only your notebook as a guide. 3) You must write legibly with indelible ink. Other useful references can include Mayo, pp. 40-44, and 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. 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, ^{1}H NMR spectrum, ^{13}C NMR spectrum, IR spectrum, product vial, and lab notebook pages.').

2. Experimental Section: 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.

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 (VCD-01-009A or B, initials – lab book number – page {A = first isolated compound, B = purified compound}) and method of sample preparation (*i.e.* KBr pellet, thin film, $CDCl_3$, etc). For IR, only major features are labeled. For NMR, every peak must be accounted for.

4. Labeled Product Vial: Place your product material into a vial labeled with the compound name, compound ID number, and your name. The vial should be taped to the back of your product card.

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

Laboratory Reports: You will write a combined lab report for experiments [9] and [10] (approximately 3-6 typewritten pages, 12 pt font, excluding attached data). The report will be prepared on a word processor and will include the following sections: Abstract, Introduction, Results and Discussion, Sample Calculations, Experimental, and References. All structures must be drawn using ChemDraw which is available on publicly accessible computers in Fischman 009, in Sam Mather or as a free download (http://sitelicense.cambridgesoft.com/sitelicense.cfm). *Chemical structures that are scanned, hand-drawn, copied from the web, etc. are not acceptable.* The report is due as a Word or .pdf attachment by **11:59 pm** on **Dec 1st 2012**.

The week of the workshop you will be required to bring a complete draft of your report to lab. We will discuss report

writing and you will be asked to peer edit one of your classmates' reports. The peer edited version of the report will be turned in with the final report due **Dec 1st 2012 by 11:59 pm.**

You may find *A Brief Guide to Writing in Chemistry* helpful in writing your report. Brief descriptions of the 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 appendices which are referenced in the text. 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 and other courses. 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. As always, pay attention to significant figures.

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 (J. Org. Chem.)*, 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 (example: Flynn, A. B., *J. Chem. Educ.* **2012**, *89*, 1210)

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). 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.

Academic Honesty: You are expected to follow the college policy for academic honesty. All materials submitted for credit must be your own work. The complete policy is available online (http://www.kenyon.edu/x11747.xml).

Final Exam: We will vote on the scheduling of the final on November 14th. The options are December 19th either at 1:30 pm or 6:30 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 231. 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 chemistry lab fees; however, at the beginning of each year, you will need to sign a check-in sheet stating that you are accepting financial responsibility for any breakage or loss of lab drawer contents. Your student account will be assessed charges for lost or broken items at the end of the year.

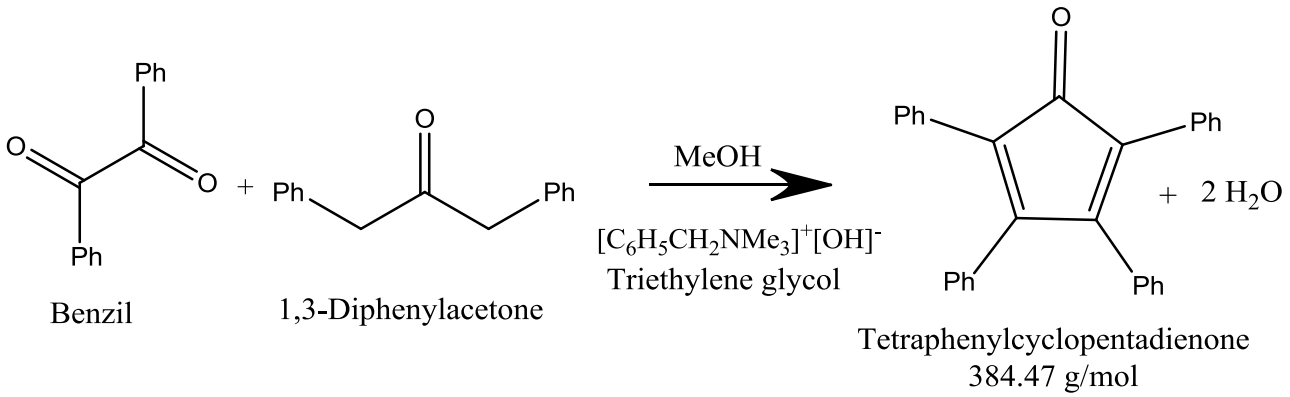
– This syllabus constructed over multiple years with contributions from Profs. Hunsen, Hofferberth, Hofferberth, and Getzler –

EXAMPLE LAB NOTEBOOK PAGE

1) Date: 01/17/2006

2) Title: Synthesis of Tetraphenylcyclopentadienone

3) Reaction scheme:



Benzil 1,3-Diphenylacetone

Tetraphenylcyclopentadienone
384.47 g/mol

4) Reference in ACS format: (Mayo, D. W.; Pike, R. M.; Forbes, D. C., *Microscale Organic Laboratory*, Wiley: New York, 2011; pp 439 – 441.)

5) Useful information and calculation:

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

5) Sample calculation (Number of moles of $BnNMe_3OH$)

(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 $BnNMe_3OH$

6) Observation, measurement and calculations as they are made

- benzil, diphenylacetone and triethylene glycol added to 5 ml conical vial (equipped w/air-condenser + spin vane)
- heated until solution is homogeneous (sand bath ~150 °C, ~10 min)
- added 0.4 ml benzyltrimethylamonium hydroxide solution
- as solution cooled, deep purple/brown x-tals(crystals) began to precipitate
- poured rxn into 15 ml Erlenmeyer, rinsed remaining material into Erlenmeyer w/~5 ml *cold* MeOH (reagent)
  spilled some of solution, 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 (VCD-01-009A) left to dry until next lab

* UAR = used as received

7) Date: 1/25/06

yield: 0.80 g (2.1 mmol, >100% !!?-high yield could be due to impurities or excess solvent)
mp: 200 –219 ^{o}C (lit: 220–221 ^{o}C)
recrystallization to purify the product
- in 15 ml Erlenmeyer, dissolved VCD-01-009A 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 h
- x-tals (isolated as above) are unblemished dark purple
- covered funnel w/kimwipe & drew air through for ~1/2 hr; x-tals look dry → VCD-01-009B

yield: 0.43 g (1.1 mmol, 48 %)
mp: 218 –220 ^{o}C (lit: 220–221 ^{o}C)

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

CHEM 233: ORGANIC LAB SCHEDULE – FALL, 2012

Lab Date	Experiment	Location in text (suggested questions)	Data Set Due
9/5	*ACS Standardized Exam, Check–In, Solvent Extraction Intro*		
9/12 9/19	*Experiment [4C] Solvent Extraction* Methods: Liquid-Liquid Extraction & Melting Point (mp) Changes: Scale = 2x	Mayo pp. 147 – 150 (21, 22, 23, 25) New Zubrick Ch. 5, 6, 7, 10, 12, 15, 16, 17	10/3
9/26 10/03	NMR Workshop: Texts listed in approximate order in which they should be read and in approximate order of increasing complexity.	U. Alberta Website, Theory Section Zubrick Ch: 35 Volhardt & Schore: Ch. 10 Mayo pp. 561 - 593	All assignments for credit in class
10/17	*Unknowns* Schedule 10 min oral session w/instructor to report the identity of your unknown.		10/24
10/24	*Experiment [9] E1 Elimination - Dehydration of 2-Butanol* Methods: Gas Trapping, ^{1}H NMR Change: See handout	Mayo pp. 209 – 217 (57, 58) New Zubrick Ch: 8, 19, 23, 24 Handout	11/7
10/31 11/7	*Experiment [10] E2 Elimination - Dehydrohalogenation of 2-Bromobutane* Methods: Anhydrous Technique, Gas Trapping, ^{1}H NMR Change: Scale = 5x **Lab Report: Elimination Reactions – include Exp [9]**	Mayo pp. 217 – 224 (60, 62, 64, 65) New Zubrick Ch. 9 Handout	11/28
11/14	*Lab Report Workshop*	**Combined reports for [9] and [10] due 12/1**	
11/28 12/5	*Experiment [5B] cis- and trans-4-tert-Butylcyclohexanol* Methods: TLC, mp, IR, ^{1}H NMR Change: Scale = 4x	Mayo pp. 158 – 163 (28, 29, 30, 31, 33, 35) New Zubrick Ch: 27, 28, 34	12/12
12/12	*Experiment [11A] Isolation and Characterization of Usnic Acid* Methods: Recrystallization, mp, IR Change: Scale = 2x, no specific rotation	Mayo pp. 224 – 229 (66, 67, 68, 70) New Zubrick Ch. 13	12/14, 4 pm

Notes: All questions are from Mayo, Chapter 6. You may find it useful to read relevant sections of Zubrick multiple times.