CHEM 233 – Organic Chemistry Lab I – Spring 2018

T, W or R 1:10 – 4:00, Tomsich 207 & 209

Instructors:	Vivian Ezeh	Mo Hunsen
	TOM 314 (ezehv)	TOM 310 (hunsenm)
Office hours:	M & W: 10 – 11 am	W: 11 am – 1 pm
	T & R: 10 – 11am	R: 9 – 11 am

Or by appointment

Course requirements:

Textbook: Mayo; Pike & Forbes "Microscale Organic Laboratory (MOL)" 5th ed. Zubrick "The Organic Chem Lab Survival Manual" 8th ed. Handouts and useful links on class Moodle site. You can use any edition of MOL and Zubrick for this course.

Required Material: The Hayden-McNeil Student Lab Notebook & goggles. If you have a lab notebook from a previous semester with many unused pages, you may use it.

Goals: CHEM 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 CHEM231 (Organic Lecture).

Grading: Grades are based upon your mastery of the materials and your demonstration of that ability on all assignments. Letter grades at the end of the semester will be assigned using the following scale: 100 – 90 % (A +/-); 89 – 80 % (B +/-); 79 – 70 % (C +/-); 69 – 60 % (D +/-); F: < 60%. The instructor reserves the right to assign whatever final grade is deemed appropriate.

Points Distribution:

ChemDraw Assignment	20
Reading Review (4 @ 10 pts each)	40
Notebook, Lab Hygiene and Safety	30
Data Sets (4 @ 50 pts each)	200
Unknown & oral lab report	50
Project proposal, presentation and progress	30
Draft Lab report & Peer editing	30
Lab Report	50
Exam	50
Total	**500**

ChemDraw Assignment: The aim of this assignment is to install ChemDraw software on your computer and to practice using it for drawing chemical structures. The instructions and appropriate links for obtaining this software can be found on the class Moodle page.

Reading Review: A five-minute quiz will be given at the beginning of each experiment (week of: 01/22, 02/05, 02/19 & 04/16) as a review of the assigned reading. The content of the quiz will be relevant to the experiment at hand and may include questions about technique as discussed in Zubrick, questions from MOL, spectral interpretation, or questions about material you should know in preparation for the experiment. These

suggested practice questions from MOL can be found at the end of the chapter describing the experiment: Exp 3B (12, 14, 17 & 20), Exp 4C (21, 22, 23 & 25), Exp 11A (66, 67, 68 & 70), Exp 5B (28, 29 30, 31 & 35), & Exp A2b (23, 24, 25 & 26)

Data Sets: At the end of 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 have 3 parts and will be due on the dates on the class schedule. Each data set will include the following items:

Electronic Data Set (EDS)**:** The EDS will have two sections and will be submitted on TurnItIn (via Moodle). A Word template file for the EDS is available on the course website. The first section will include essential summary information, *e.g.* product description, yield, % yield, and a list of analytical data and notebook pages to be separately submitted in hardcopy form (see below). The second section of the EDS will be an experimental procedure (an example is below). For each experiment you will write an experimental section in prose suitable for publication in an American Chemical Society (ACS) journal. General guidelines for scientific writing should be followed. Each EDS will be submitted through TurnItIn by 11:59 pm on the dates indicated on the schedule. Late electronic submissions will result in loss of following points from your EDS grade: 0.1% (1-10 min late), 1% (11-20 min late), 5% (21-30 min late), 10% (31-40 min late), 15% (41 mins – 24 hours) and additional 5 points for every 24 hours it is late.

Hardcopy Data Set (HDS): The HDS will have two components. The first will be all instrumental data collected for the experiment. The 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 Moodle site. All spectra should include the following: compound structure, compound name, compound ID number (VCD-01-009, initials – lab book number - page), 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. The second section of the HDS is your lab notebook pages. The HDS will be due at the beginning of lab after the TurnitIn assignment due date.

Labeled Product Vial: Place a small sample of your product into a vial labeled with the compound name, compound ID number, and your name. Place the vial in the submission rack at the beginning of lab the day the hardcopy data set is due.

Research Project: Propose a short project based on the reduction of cyclohexanone functional group. More information and guide will be provided the week of Jan 29th 2018. Keep in mind that your proposal (2 pages) is due on Friday, March 2nd 2018, a short presentation of proposal will be the week of March 19th 2018 and the experiment will be performed the weeks of March 26th & April 2nd 2018. The project will be reported in the form of a lab report.

Lab report: A lab report for your research project (approximately 4-6 pages, excluding spectra etc.) will be written by each student this semester. This lab report will be due on the approved final exam date and time for your lab period (T: 05/07 @ 6:30 pm; W: 05/11 @ 8:30 am; R: 05/08 @ 1:30 pm). The word-processed report include the following sections: Abstract, Introduction, Results and Discussion, Sample Calculations, Experimental, References and Supporting Information. Structures must be drawn with ChemDraw. *Chemical structures that are scanned, hand-drawn, copied from the web, etc. are not acceptable.*

Please refer to *A Brief Guide to Writing in Chemistry* for guidance in writing your report. Brief descriptions of 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). Abstracts must include an informative, legible, graphical summary (no larger than 3.25" x 1.75").

Introduction: This is a statement describing the theoretical background, purpose and goals of your work. Give the reader a reason to care. 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 that 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 must show *one* sample calculation for each type of calculation (i.e. one each for % recovery, theoretical yield, % yield, % selectivity, etc.) that you performed for the experiment. Significant figures matter.

Experimental: This is a description of what you did in the laboratory according to your notebook and not what is described in *MOL*. *The experimental is written in the third person, the past tense, and in the passive voice.*

References: These are the sources of information that were used in the report (*MOL*, Zubrick, CRC Handbook of Chemistry and Physics, *Science, Journal of Organic Chemistry*, etc.). This is a critical and oft overlooked section of a lab report. On what are you basing your statements? A book, a journal article, a website (be careful!), your own imagination? All references should be according to the *ACS Style Guide*, using the *Acc. Chem. Res.* style with full article titles.[1] You may find Table 14-2 particularly useful.

Supporting Information: Hard copies of your annotated spectra must be submitted along with your lab report on the final exam date and time for your section.

Peer editing workshop: We will hold a lab report workshop on the last week of labs (week of April 30th 2018). To participate in the workshop and get credit for the assignment, you must submit a draft copy via TurnItIn by 11:59 pm on April 20th 2018, then bring a hardcopy of your draft and all annotated spectral data to class.

Notebook, lab hygiene and safety: 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, interpreting spectra, testifying in court, etc. Although there is no single correct way to keep a notebook, the attached notebook example is a good format to follow. I will check your notebooks at the end of each lab and they will be graded 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 understand the outcome of the experiment and reproduce your work, using only your notebook as a guide. Other useful references can be found *MOL*, pp. 40-44 or in Zubrick, Chapter 2. You are also responsible for keeping your lab space clean and using personal safety equipment.

Unknown Lab & Oral Report: In this assignment, you will use spectroscopic methods and solubility/miscibility observations to determine the identity of an organic compound. Your analysis will include molecular formula, proposed structure, solubility/miscibility test interpretation and analysis of spectra. Oral presentation for this assignment will take place during the week of March 19th 2018. The presentation will include analysis of annotated spectra and observations documented in your notebook.

Exam: A cumulative exam will take place on exam date & time assigned to each section (T: 05/07 @ 6:30 pm; W: 05/11 @ 8:30 am; R: 05/08 @ 1:30 pm). The content of the exam will be relevant to all completed experiments and may include questions about technique as discussed in Zubrick, questions from MOL and spectral interpretation.

Important class policies:

Attendance: Attendance at all class meeting is mandatory. We will meet in Tomsich 207 at 1:10 pm for pre-lab lecture and proceed to Tomsich 209 for laboratory work. If you will miss lab for an excused absence such as a family or medical emergency or scheduled sporting event, you must obtain permission from all instructors involved to attend an alternate lab section.

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(1) Dodd, J. S.; Solla, L.; Bérard, P. M. References. In *The ACS Style Guide: Effective Communication of Scientific Information* [Online]; Coghill, A. M.; Garson, L. R., Eds.; Oxford University Press: 1996; Chapter 14, pp. 287-341. http://pubs.acs.org/doi/abs/10.1021/bk-2006-STYG.ch014 (accessed August 25, 2015).

Evening Analysis: The lab will be open, as needed, Sunday or Thursday nights from 7 – 9 pm, staffed by a teaching assistant. You may perform analytical techniques such as melting point analysis and the various spectroscopies. No other experiments are allowed.

Safety: When in Tomsich 209, remember to 1) Wear safety goggles at all times. 2) Wear long pants and shoes that cover the entire foot. 3) Do not eat, drink or chew gum. 4) Be mentally alert to hazards and prepared for emergencies. Ask me if you are not sure if something is safe.

Withdraw Late: CHEM 231 and 233 are separate courses with separate grades. If you withdraw from CHEM 233 you will not be automatically withdrawn from CHEM 231.

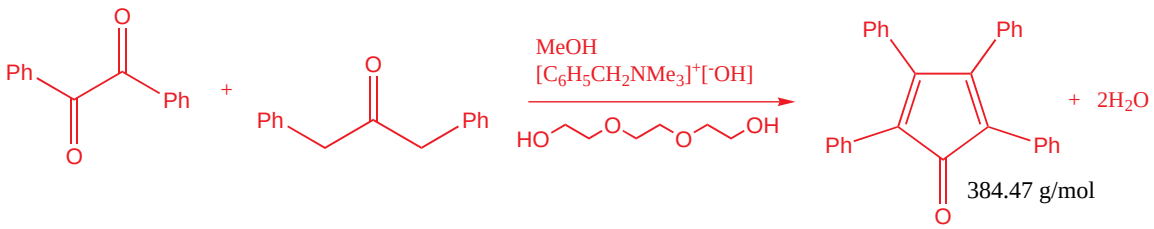
Academic accommodation: Students who anticipate they may need accommodation in this course because of the impact of a learning, physical or psychological disability are encouraged to contact Erin Salva (salvae@kenyon.edu, 740-427-5453), Director of Student Accessibility and Support Services. Early contact will help avoid unnecessary inconvenience and delays.

Academic Honesty: All work turned in for credit must adhere to the principles of academic integrity (see Academic Honesty and Questions of Plagiarism in the Course Catalog). Copying colleague's texts, not citing source materials are examples of incidences that could potentially violate academic integrity. Potential violations will be forwarded to the Academic Infractions Board for adjudication, as is required by University policy. If the ethical implication of any situation is not clear, do ask me for clarification.

Bias/Discrimination/Harassment: Kenyon College seeks to provide an environment that is free of bias, discrimination, and harassment. If you have been the victim of sexual harassment/misconduct/assault we encourage you to report this. If you report this to a faculty member, she or he must notify our college's Title IX coordinator about the basic facts of the incident (you may choose whether you or anyone involved is identified by name). For more information about your options at Kenyon, please go to: http://www.kenyon.edu/directories/offices-services/title-ix/sexual-assault-and-harassment/

EXAMPLE OF A LAB NOTEBOOK PAGE

01/17/2006



(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

* UAR = used as received

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 soln homogeneous (sand bath ~150 °C, ~10 min)
- added 0.4 ml benzyltrimethylamonium hydroxide soln
- as soln 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 soln, 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 (A – sample before purification step)) left to dry until next lab

1/25/06

yield: 0.80 g (2.1 mmol, >100 %!!?)
mp: 200 –219 (lit: 220–221)
must re-xtalize
- 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 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 → VCD-01-009B

yield: 0.43 g (1.1 mmol, 48 %)
mp: 218 –220 (lit: 220–221)

IR – KBr pellet (see attached spectrum with relevant peaks labeled) } all look good!

NMR – ^1H $CDCl_3$ (see attached spectrum with *all* peaks labeled)

Sample Experimental

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_2O (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 $MgSO_4$, 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-heptene-2-one as a colorless liquid.

Notice the use of common abbreviations (i.e. 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_2O" instead of

“water”, because these formulae can only describe these particular compounds. However, “C_2H_6O” cannot be written for ethanol, since another compound, dimethyl ether, has the same formula. Therefore, the word “ethanol” is written instead.

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

CHEM 233: **ORGANIC Lab Schedule – FALL, 2018**

Week of	Experiment	*MOL (5e)* Experiment **(Zubrick 8e Ch's)**	Data set due
1/15	*Check–In, Distillation Intro, ChemDraw*	**(1, 2)**	ChemDraw Item 1/19
1/22 & 1/29	*Experiment [3B] Fractional Distillation* Methods: Distillation, Refractive Index (RI), & Gas Chromatography (GC) Changes: Scale = 2x, run complete experiment twice **Note: Start working on your project proposal!**	132-135 **(5, 11, 20, 29, 30)**	T: 2/6, W: 2/7, R: 2/8
2/05 & 2/12	*Experiment [4C] Solvent Extraction* Methods: Liquid-Liquid Extraction & Melting Point (mp) & ^{1}H NMR (amine) Changes: Scale = 4x	147-151 **(10, 12, 15, 16)**	T: 2/20, W: 2/21, R: 2/22
2/19	*Experiment [11A] Isolation and Characterization of Usnic Acid* Methods: Recrystallization, mp, IR Change: Scale = 2x, no specific rotation	224 – 229 **(13)**	T: 2/27, W: 2/28, R: 2/29
2/26	*Spectroscopic Identification of an Unknown* Methods: IR, ^{1}H NMR & solubility/miscibility, RI or mp	638 – 639 & handout	Unknown – week of 3/19 Proposal – 03/02
	Spring Break! 3/5 – 3/16		
3/19	*Research project presentation & discussion. Preparation for your project: TLC of starting materials*	**(27)**	
3/26, 4/02 & 4/09	*Project & Experiment [5B] cis- and trans-4-tert-Butylcyclohexanol* Methods: mp, IR, & ^{1}H NMR Change: Scale = 4x	158-163 **(32, 33)** **Student handouts**	Draft – 4/20
4/16 & 4/23	*Experiment [A2$_b$] Bromination of (E)-Stilbene: meso-Stilbene Dibromide* Methods: Green synthesis, IR, ^{1}H NMR Semi-microscale	444-449 & handout **(32, 33)**	T: 5/1, W: 5/2, R: 5/3 *Up till exam date*
04/30	*Peer Edit Workshop*		

Notes: You may find it useful to read relevant sections of Zubrick multiple times. All Electronic data sets will be submitted through TurnItIn and will be due at 11:59 pm on the days indicated on the schedule (T = Tuesday section, W = Wednesday section & R = Thursday section).