CHEMISTRY 373 – ADVANCED ORGANIC CHEMISTRY LAB
FALL 2019

Tuesdays 1:10 – 4:00 pm, TOM 001 & 301

Instructor: Professor Mo Hunsen

Tomsich Hall 310
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Office Telephone: 427-5091
Office Hours: W 11:30 am – 1:30 pm, R 12-2 pm, or by drop-in/appointment. You are welcome, encouraged, and expected to meet with me throughout the semester.

Texts: No textbook is required. We will use primary articles throughout the course. You might find organic chemistry lab books you have used previously to brush up some techniques, i.e. Mayo, Dana W., Pike, Ronald M. and Trumper, Peter K. "Microscale Organic Laboratory: With Multistep and Multiscale Syntheses," any edition; and Zubrick, James W. "The Organic Chem Lab Survival Manual," any edition.

Required Material: Lab Notebook, three-ring binder, & goggles.

Point Distribution:

A. Preparation of reactive carbohydrates	20%
B. Preparation of acid chlorides and amides	20%
C. Staudinger reactions	20%
D. Huisgen cycloaddition reactions	20%
E. Purity of samples, safety, report, annotated spectra	20%
Note: Progress & demonstrated effort is important.	

Rules for the Course:

Goals: Chemistry 373 provides a technical foundation for first-hand experimental work in organic synthesis. The course emphasizes techniques and skills that are used in research projects requiring macroscale and microscale synthesis of organic compounds. The techniques and reactions involved integrate and illustrate some of the chemistry in chemical biology (Chem 401, Spring 2020). We will independence and teamwork in design and safe implementation of multistep experimental procedures. You will practice detailed recordkeeping in a laboratory notebook and of spectral data.

***Learning Goals*:** Students will understand:

1. safe laboratory practices including on how to appropriately dispose of chemicals
2. effective team working practices
3. effective use of literature for experimental procedures and physical & spectroscopic data
4. maintaining detailed notes on a lab notebook
5. detailed characterization of products using physical and spectroscopic data (e.g. mp, IR, NMR, GC-MS)
6. how to formulate and solve syntheses problems individually and as a member of a group
7. effective ways of searching for primary articles in organic chemistry; and evaluating and using these information to write concise & clear adapted procedures, summary of findings, & reports.

Attendance: Advanced organic chemistry lab is a experimental science that continually builds upon itself, and it is quite easy to get behind if you miss a particular lab period. Therefore, attendance is mandatory. This lab is

designed to give you a true research experience. Several of the reactions you will carryout will need to carried out overnight – please work with your teammate to make sure that work up and characterization is completed at the appropriate times. I will rarely if ever formally present material. However, during that time I will be available to answer your questions and assist you with technique. You are to come to lab having thoroughly read the relevant material. I expect you to ask question during the week, prior to any new experiment.

Course Meeting Time: We will meet in Tomsich Hall 001 or 101, if needed, for a pre-lab discussion during the first week of a particular experiment. This meeting should last about 30 minutes. We will go over your adapted protocol to discuss safety and the scales of the reaction. You will have only your laboratory notebook to help you during the experiment, so you will need to prepare a preliminary write-up of the details of the reaction at the appropriate scale. Planning your lab work ahead of time will increase your efficiency in lab. We will work both in TOM 001 & TOM 301. Please make sure that at least one other student is available near you when you carryout reactions.

Sample Analysis: You may perform analytical techniques such as melting point analysis and the various spectroscopies in TOM 209, TOM 311, TOM 001, & TOM 105.

Late Work*:* There is a built in buffer time to catch up and complete experiments. You will depend on each other for some of the intermediates – so let's do our best effort to make sure failed experiments are repeated promptly after a discussion with me and team members.

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** – 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; 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. In many of the experiments you will be working a macroscale reaction and with more hazardous compounds than previously. All flasks must be labeled with their contents in an unobscurable manner. Finally, while you will often work outside of class time, you may never work in the lab by yourself – always make sure someone is with you. Waste containers must be labeled with the full name of the chemical and the approximate amount present. Abbreviations or chemical notation such as EtOH or CH2Cl2 are not acceptable.

Laboratory Notebooks: You are required to purchase and maintain a laboratory notebook. 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 when writing a lab report in the days ahead. The notebook for this course contains white pages and carbon copies of the white pages. After you have finished for the day, your notebook will be signed and dated by me or the laboratory teaching assistant and the relevant pages will be submitted with the lab report. Your lab notebooks will stay in TOM 301, I will regularly chech your notebook. There is no one right style for writing in a laboratory notebook; everyone has his or her own style. A good template is in Mayo and in Zubrick, Chapter 2. For this course I expect you to follow the format shown on the attached page. The most important rules are: 1. Your lab notebook is your scratch paper -- data and observations should be recorded directly into your notebook at the time the observations or measurements are made; 2. The writing should be done 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.

Report: *Guidelines on what is expected in the form of a report will be provided as a handout.*

Exam*:* In lieu of a final exam, we will meetat a time of your choosing during finals week, to do a final cleanup of the lab.

Grading*:* Your performance will be evaluated based upon the following absolute scale: A (+/-) = 100-90%; B (+/-) = 89-80%; C (+/-) = 79-70%; D (+/-) = 69-60%; F <60%.

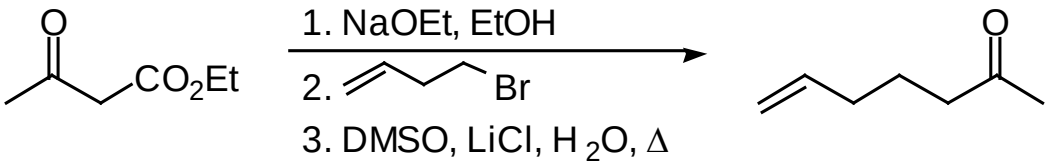
Academic Integrity: At Kenyon we expect all students, always, to submit work that represents the highest standards of academic integrity. It is the responsibility of each student to learn and practice the proper ways of documenting and acknowledging those whose ideas and words they have drawn upon (see Academic Honesty and Questions of Plagiarism in the Course Catalog). Ignorance and carelessness are not excuses for academic dishonesty. If you are uncertain about the expectations for this class, please ask for clarification.

***Electronic Device Policy*:** Active participation and full engagement is critical for your success in this course. As such all electronic devices should be turned off before the beginning of every lecture.

Section 504 of the Rehabilitation Act of 1973 and the Americans with Disabilities Act of 1990*:* Students who anticipate they may need accommodations in this course because of the impact of a learning, physical, or psychological disability are encouraged to meet with me privately early in the semester to discuss their concerns. In addition, students must contact Erin Salva, Director of Student Accessibility and Support Services (740-427-5453 or salvae@kenyon.edu), as soon as possible, to verify their eligibility for reasonable academic accommodations.

Title IX Responsibilities*:* As a faculty member, I am deeply invested in the well-being of each student I teach. I am here to assist you with your work in this course. If you come to me with non-course-related concerns, I will do my best to help. It is important for you to know that all faculty members are mandated reporters of any incidents of harassment, discrimination, and intimate partner violence and stalking. Meaning, I cannot keep information involving sexual harassment, sexual misconduct, interpersonal violence, any other form of harassment or discrimination based on a protected characteristic confidential. The Health and Counseling Center, the College chaplains, and the staff at New Directions Domestic Abuse Shelter & Rape Crisis are confidential resources.

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 □L, 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-hepten-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 is subject to change at my discretion. I will notify you of any changes in class and/or by email.