

CHEMISTRY 233 - ORGANIC CHEMISTRY LAB I

Fall 2009

Tuesday or Thursday, 1:10-4:00 PM

Professor Mo Hunsen

Office: Tomsich 310

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Office Hours: T 10 – 11 AM; R 11 AM – 1 PM, or by appointment

Office Telephone: 427-5091

Texts: Mayo, Dana W.; Pike, Ronald M. and Trumper, Peter K. "Microscale Organic Laboratory: With Multistep and Multiscale Syntheses," 4th edition

Zubrick, James W. "The Organic Chem Lab Survival Manual," 6th edition

Required Material:

"Organic Chemistry Laboratory Notebook" - Chemical Education Resources, Inc. (CER)

Point Distribution:

5 Product Card Reports @ 50 points each	250
1 Full Laboratory Reports @ 75 points each	75
Final Exam	115
Quizzes, Notebook, and Laboratory Technique	60

Total	500

Rules for the Course:

Goals: Chemistry 233 provides a technical foundation for first-hand experimental work in organic chemistry. The course emphasizes techniques and skills that will be used in other courses, especially Chemistry 234 and any research projects requiring material manipulations. The techniques and reactions involved integrate and illustrate Chemistry 231 (the lecture course) material.

Attendance: Organic chemistry is a science that continually builds upon itself, and it is quite easy to get behind if you miss a particular lab period. Therefore, attendance to your assigned laboratory section is mandatory. I strongly discourage you from switching lab sections during the semester. If it is absolutely imperative that you perform an experiment during another lab section for a particular week, you must obtain permission from me and the instructor of the section you are attempting to switch into.

Course Meeting Time: We will meet in Tomsich Hall 207 at 1:10 pm for a pre-lab discussion during the first week of a particular experiment. This meeting should last 20-30 minutes and may begin with a 5 minute quiz which will be germane to the experiment at hand. You will have only your laboratory notebook to help you during the quiz, so you will need to prepare a preliminary write-up in your notebook. Planning your lab work ahead of time will increase your efficiency in lab. Afterwards, we will walk down to Tomsich Hall 209 and start working. If we are in the second (and last) week of an experiment, you can begin working promptly at 1:10 pm in Tomsich Hall 209. You should confine your lab work to the scheduled hours. If you need to make physical or "dry" measurements (masses, melting points, IR and NMR spectra, refractive indices, etc.) outside of the scheduled lab time, there will be a lab teaching assistant available on Sundays 6-8 PM in Tomsich 207/209.

Rules for the course, continued:

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 -- I will only warn you only once this semester before making point deductions; 2. 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 not sure whether something is safe or not, please feel free to consult with me or the laboratory assistant.

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 for your original record of work and yellow pages which are 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 yellow pages will be submitted with the lab report.

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, pp. 30-32 or in Zubrick, Chapter 2. 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.

Product Cards: The card should be filled out with all of the pertinent information in ink. Attach an experimental, all the spectra, chromatograms, etc. to the card. In addition, the entire remaining product (the material not used for physical measurements, spectra, or GC) should be submitted in a vial clearly labeled with your name and its contents. Answers to the questions should be submitted on a separate sheet of paper. (see Laboratory Schedule).

Laboratory Reports: A typewritten laboratory report is due at 1:10 pm at the stated date (see Laboratory schedule). Late lab reports will be penalized as follows:

up to one week late:	5 points
between one week and two weeks late:	10 points
between two weeks and three weeks late:	20 points

No report will be accepted for grading which is more than three weeks late, and a numerical score of zero will be given for that experiment. No report will be accepted for grading after the final exam date. Use the template I kept on P:\DATA\CHEM\CHEM233 (This template is normally used to submit articles to the journal Organic letters) to write your report. I have also kept two sample articles in the same directory

A report (approximately 3-6 typewritten pages excluding attached spectra and chromatograms) should be written in your own words and should be divided into the following sections: Abstract, Introduction, Results and Discussion, Sample Calculations, Experimental, Questions, References, and supplemental information.

Abstract: A brief summary of your results. The abstract will vary from 1-5 sentences, but never exceeds a single paragraph.

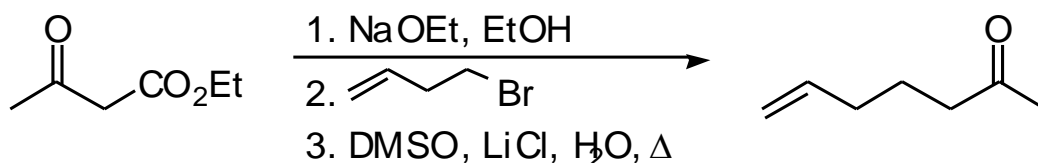
Introduction: A statement that describes the purpose and goals of the experiment. You should describe (in words, *balanced* chemical equations, mathematical equations, etc.) the *new* method(s) and/or chemical reaction(s) that you have investigated for this report.

Rules for the course, continued:

Results and Discussion: Your data (results) and the interpretation/explanation of your data (discussion). Your data are most effectively presented using tables, graphs, lists, etc. 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 course and in Chemistry 231 (the classroom course). Typically, this is the main body of text in your report.

Sample Calculations: Give a detailed account of how you arrived to 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, relative %s, etc.) that you performed for a particular experiment.

Experimental: 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. After a new method has been described in detail for a particular lab report, you can simply use the term to describe the method for any subsequent reports. The following page has a sample experimental write-up for the synthetic experiment shown below:



An example of an 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). H₂O (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 MgSO₄, 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 "H₂O" instead of "water," because these formulae can only describe these particular compounds. However, "C₂H₆O" cannot be written for ethanol, since another compound, dimethyl ether, has the same formula. Therefore, the word "ethanol" is written instead.

Questions: Tersely answer the questions (as indicated in the **Laboratory Schedule**) for each experiment.

References: Sources of information that was used in the report (Mayo *et. al.*, Zubrick, CRC Handbook of Chemistry and Physics, Aldrich Catalog Handbook of Fine Chemicals, etc.).

Supplemental information: This includes spectra, chromatograph, etc.

Rules for the course, continued:

After you are finished writing the text of your report, check it for spelling, awkward sentences, and sentence fragments; edit your report accordingly.

Oral Lab Report: For the "Spectroscopic Identification of an Unknown" lab, you should bring a half-page summary and present an oral report using the spectral data that you have acquired to convince me of the correctness of your structure.

Grading: I will be evaluating your performance 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 as outlined in the "Kenyon College Student Handbook 2009-2010". In a nutshell, all materials submitted for credit must be of your own work.

Section 504 of the Rehabilitation Act of 1973 and the Americans with Disabilities Act of 1990: If you have a disability, and in order to participate fully in this course you have a need for some type of accommodation, please feel free to discuss your concerns with me in private and be sure to contact Ms. Erin Salva at PBX 5453 or via e-mail at salvae@kenyon.edu.

This syllabus is subject to change at my discretion. I will notify you of any changes in class or by e-mail.