



Advanced Organic Laboratory Chemistry 146 Fall 2014

Instructor: Adam Whalley 656-8246 Cook A330 Adam.Whalley@uvm.edu

Teaching Assistant: Corinne Sadlowski 656-4011 Cook A322 Corinne.Sadlowski@uvm.edu

Lab Time: 1:00pm – 5:00pm T & Th, Cook A305

Instructor office hours: By appointment or for quick questions just stop by.

T.A. office hours: By appointment or for quick questions just stop by.

Texts: Blue lab notebook with page numbers; same as required for Chem. 35 (Bookstore item#: 77571)
No textbooks are required!

Books that will be useful:

Encyclopedia of Reagents for Organic Synthesis (EROS) [QD 77.E35 in reference section] This series includes: information about safety concerns; methods of preparation/purification; physical data; a survey of uses for each reagent. This should be consulted each time you handle a new reagent.

Spectrometric identification of organic compounds R.M. Silverstein, F.X. Webster *In Whalley's Office*

Organic Structural Spectroscopy J. Lambert, H. Shurvell, D. Lightner, R. Cooks *In Library*

High-Resolution NMR Techniques in Organic Chemistry T. Claridge *In Library*

Other books that may be useful:

Advanced Organic Chemistry, Part A, 4th ed. F.A. Carey and R.J. Sundberg ISBN: 0-306-46243-5

Advanced Organic Chemistry, Part B, 4th ed. F.A. Carey and R.J. Sundberg ISBN: 0-306-46245-1

The Art of Writing Reasonable Organic Reaction Mechanisms R.B. Grossman

Writing Reaction Mechanisms in Organic Chemistry A. Miller ISBN: 0-12-496711-6

Advanced Organic Chemistry, J. March, 4th or 5th edition ISBN: 0-471-58589-0

Stereochemistry of Organic Compounds, E.L. Eliel, S.H. Wilen ISBN: 0-471-01670-5

Course Purpose: I have designed this course to try to achieve several goals. The first, obviously, is to introduce you to some advanced laboratory techniques that are essential in modern synthetic organic chemistry. The second is to provide you with hands on experience with NMR, and to introduce you to more advanced NMR techniques and interpretation. Third, the experiments we perform will highlight some fundamental concepts in organic chemistry that you have likely seen in your course work. Unlike previous labs, many of the experiments that we do will be multi-step sequences in which the material you produce in one lab will be used in the next.

This course will address each of the following departmental learning goals for chemistry majors:

1. Students will demonstrate general knowledge in chemistry and will be able to apply chemical and physical principles in the solution of qualitative and quantitative chemical problems.
2. Students will understand the interplay of observational data, hypotheses, and hypothesis-driven experimentation through application of the scientific method.
3. Students will become proficient in chemical laboratory techniques and be able to apply these to practical and current problems in research.
4. Students will be able to read and critically evaluate the chemical and scientific literature.

- The students will learn to present scientific data clearly and effectively through both written and verbal communication.

Grading: Lab report write-ups (85%); Work approach (5%); Other assignments (10%)

The majority of your grade (85%) will be based on the lab write-ups. These will be due one week from the completion of the lab work. This is an advanced lab and your write-ups should reflect this. The lab report will be comprised of several sections and separate handouts will demonstrate these to you in more depth. The following is a quick summary:

Prelab: The T.A. will verify that a prelab has been satisfactorily completed before any lab work will begin. If the prelab is not satisfactorily completed it will be assigned a grade of zero for its portion of the overall grade for this lab and the student will not begin labwork until the prelab is complete to the T.A.'s satisfaction. At a minimum the prelab must include: the date; a schematic drawing of the reaction to be done; a table showing the molecular weight, density (for liquids), number of moles, number of grams, and number of mL to be used for each substance; a list of potential hazards and precautions to be taken; a brief experimental procedure written in your own words that you will follow.

Observations and notes: A detailed description of what you actually did and what you observed. This should be in sufficient detail that anyone reading your notebook could repeat the reaction exactly as you did it. Examples of standard notes and observations include: the time something was added, how long it took to add it, how long materials were allowed to react, gas evolution, color change, precipitate formation, reproduction of TLC results including *rf* calculations, tare weights, weights or volumes of materials used, etc....

Post-lab write-up: This is where the majority of the grade will lie. This should be a formal (typed) detailed discussion of the experiment including a background discussion about the type of reaction performed, a discussion of the mechanism (computer drawing software must be used to draw all chemical structures in the post-lab [drawing software can be downloaded for free from the UVM Chemistry Department website]), spectral interpretation (Proton **and** Carbon NMR **and** MS data), and an interpretation of the results. *For indicated experiments this must also include a detailed experimental procedure written in journal format including all spectral data.*

Assignment deadlines: Part of your assignment will be to gather NMR or GC/MS data for the compounds you prepare. You must turn these spectra into the T.A. on the assigned date or face a 1/3 grade deduction (i.e. A \rightarrow A-). All assignments including post-lab write-ups must be handed in at the beginning of the lab period on the date they are due. Any assignment not handed in at the beginning of lab will be penalized by one letter grade if they are handed in by 1:00pm the following day (i.e. within 24 hrs of the deadline). If the assignment is handed in within the subsequent 24 hrs (i.e. within 48 hrs of the deadline) then the final grade will be penalized two letter grades. No assignment will be accepted after 48 hrs of the deadline.

Work Approach (5%): Work approach points will be assigned by the TA. These points should be considered something you have to earn, not something that is taken away as a punitive measure for making mistakes. How do you earn these points? Approach your laboratory work in a safe, effective and efficient manner while being a good class citizen. The actual yield of your product will not be considered, but your approach to safety in the laboratory as well as your cleanliness and ability to apply good chemistry practices in your work certainly will. People who leave class early rather than taking time to help tidy up communal laboratory space will find it difficult to earn these points, as will people who are careless in their work habits, use poor chemical practices, show unsafe behavior or use flawed laboratory techniques.

Other assignments (10%):

The following assignments will be worth a combined total of 10% of your final grade. Detailed instructions will be given at a later date:

1) *Chemical database searching*: Reaxsis provides electronic access to Beilstein organic chemistry database. SciFinder Scholar provides electronic access to Chemical Abstracts. These databases and their corresponding search engines are invaluable research tools for organic chemistry. You will be assigned a project that will introduce you to these tools.

2) *Graduate school search*: The goal here is to get you to think seriously about graduate school as a possible career path. You will be asked to identify at least 5 graduate programs that you would consider applying to, and to provide a quick summary of the research that interests you within those departments.

3) *NMR spectrum interpretation exercise*: You will be given an NMR spectrum to interpret. The structure will be provided, but all coupling constants must be calculated and all peaks must be assigned to the correct proton on the structure.

Attendance: Due to the multi-step nature of the experiments to be performed in this laboratory attendance is particularly important. If you miss a step you will not have the material available to you to perform the subsequent step. Therefore, any absence not prearranged with the instructor will result in a grade of zero for that particular multi-step sequence. This will have a major impact (>25%) on your final grade. *Religious Holidays: Students have the right to practice the religion of their choice. Each semester students should submit in writing to their instructors by the end of the second full week of classes their documented religious holiday schedule for the semester. Faculty must permit students who miss work for the purpose of religious observance to make up this work.*

Academic Conduct: Cheating or plagiarism will be considered grounds for failing the course. Cases of cheating or plagiarism may lead to further disciplinary actions including dismissal from the University according to the rules set forth in The University of Vermont's *Code of Academic Integrity*.

*If you do not have a copy of the Aldrich Catalogue (i.e. Aldrich Handbook) request one at:
<http://www.sigmaaldrich.com/Brands/Aldrich.html>
This is an invaluable free resource.*

Laboratory Safety:

The organic laboratory is a very safe place to work if safety precautions are always observed. Caution, as well as careful thought and knowledge of the characteristics of what one is working with are necessary to avoid accidents and injuries. Knowledge and preparedness is key to safety. If you have thought in advance of potential mishaps and solutions to those mishaps you will be prepared to deal with them in a calm and controlled manner. Minor accidents can snowball into major catastrophe due to panic.

Potentially hazardous apparatus and flammable, toxic, and/or corrosive chemicals will be used in this course. We will guide you in the safe handling and use of these materials, but ultimately your own actions will dictate your level of safety. The following rules and procedures will be observed at all times.

Rules:

1. You must wear safety goggles or OSHA approved glasses in the laboratory. This rule will be strictly enforced at all times. Do not wear contact lenses.
3. Know the location of exits, safety showers and eye-wash fountains.
2. Avoid personal contact with chemicals. Many chemicals have an adverse physiological effect (e.g. narcosis, toxicity, allergenicity, etc.). It is best to wear protective gloves. If you spill any chemical on your skin, wash it off at once with soap and water and tell your TA. Do not inhale chemicals or put them in your mouth.
3. Performance of unauthorized experiments is not allowed.
4. Horseplay in the laboratory is strictly forbidden.
5. Drinking, eating, or smoking in the laboratory is prohibited.
6. Removal of chemicals and equipment from the laboratory is forbidden.
7. All accidents and injuries, however minor, must be reported to the instructor.
8. Extraneous sources of sound are not allowed.
9. Do not work in the laboratory while under the influence of drugs or alcohol.
10. Dress properly:
 - Do not wear open shoes or sandals.
 - Do not wear baggy clothes.
 - Long hair must be tied back.
11. Do not pipette by mouth.
12. When leaving the laboratory make sure all gas, air, water, steam, and electricity are turned off.
14. Protect your hands with gloves or a towel when pushing glass tubing or thermometers into stoppers or rubber tubing. Lubricate the hole to avoid glass breakage.

15. The working space, drawers, cabinet, and shelf above your bench should be neat and clean at all times.
16. The balances and balance area should be cleaned of any chemical spill.
17. Put glass in the broken glass disposal box; not in the trash.
18. Always point test tubes, flasks, and separatory funnels away from you *or other passers by*.
19. Carefully follow the instructions for proper waste disposal.

In case of accident

1. Fire. Personal safety is most important. Make sure everyone gets out of the room and the building. After the safety of all is assured, you may extinguish the fire. If a person's clothing catches fire, he or she needs help. Prevent the person from running. Put him or her under the safety shower and pull the chain. (It is less effective to smother flames with a fire blanket. Never spray a person with a carbon dioxide fire extinguisher.
2. Chemicals. If corrosive chemicals are spilled on clothing, immediate showering with the clothes on is the best remedy. If chemicals are spilled on the skin, wash them off with large volumes of water. If the chemical is pilled in the eyes, it should be washed immediately at the eye wash fountain. In all cases spills must be reported to the instructor.
3. Injuries. All injuries, no matter how minor must be treated immediately by a medical professional.

Housekeeping

The safety of a laboratory is directly related to the cleanliness of the laboratory. You will be held responsible for keeping this laboratory clean and un-cluttered. A part of your technique grade will be determined by your housekeeping efforts. If you finish your experimentation early you will be expected to start the clean-up process.

The following is a brief checklist:

- All equipment must be returned at the end of the lab period to whence it came.
- All glassware must be washed at the end of the lab period.
- All bench tops must be wiped down at the end of each lab period.
- All communal areas are the responsibility of the lab as a whole and must be cleaned and organized at the end of each lab.
- The chemical dispensing and waste areas must be kept clean and properly labeled.
- All gas, air, water, steam, and electricity must be turned off.