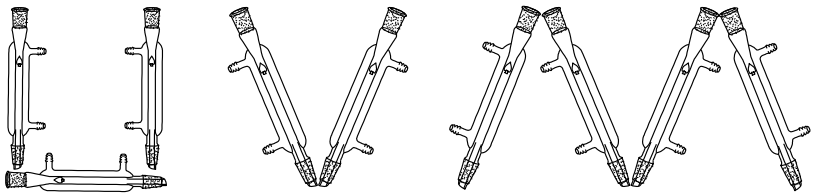

Advanced Synthesis Techniques

Chemistry 114

Spring 2019

Instructors: Rory Waterman 656-0278 Discovery W331 Rory.Waterman@uvm.edu
Adam Whalley 656-8246 Discovery W118 Adam.Whalley@uvm.edu
Teaching Assistant: Adam Dyer Adam.M.Dyer@uvm.edu
Lecture Time: 9:40 am – 10:30 am M, L/L-A A162
Lab Time: 2:50 pm – 6:50 pm TR, Discovery W408
Office hours: By appointment or for quick questions just stop by.

Texts: Blue lab notebook with page numbers (Bookstore item#: 77571)

No textbooks are required!

Useful Resources:

American Chemical Society Guidelines for Chemical Laboratory Safety in Academic Institutions: https://www.acs.org/content/dam/acsorg/about/governance/committees/chemicalsafety/publications/acs-safety-guidelines-academic.pdf

Basis of Recognize-Assess-Minimize-Prepare safety protocol and resources about it: https://www.acs.org/content/acs/en/chemical-safety/ramp.html

Encyclopedia of Reagents for Organic Synthesis (eEROS) https://onlinelibrary.wiley.com/doi/book/10.1002/047084289X
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.

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

Synthesis and Technique in Inorganic Chemistry, 3rd ed. G. S. Girolami, T. B. Rauchfuss, and R. J. Angelici ISBN: 0935702482

The Manipulation of Air-Sensitive Compounds, 2nd ed. D. F. Shriver and M. A. Drezdzon ISBN: 0070571554

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: We 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 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 preform will highlight some fundamental concepts in both organic and inorganic 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.
5. The students will learn to present scientific data clearly and effectively through both written and verbal communication.

Safety: This is an experimental laboratory—safety in this environment is of paramount importance. Each student will complete a contract that outlines the key safety responsibilities of the course and student.

Class meetings: Our lecture time is a place where we review safety consideration of upcoming experiment, discuss relevant concepts for the experiments, and prepare for lab work. Because we meet in the same space as the actual laboratory, always come dressed appropriately.

Grading: Lab report write-ups (80%); Work approach (20%)

The majority of your grade (80%) 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 lab work 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 R_f 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 preformed, 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 → 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 1:00 pm 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 (20%): 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.

Attendance: Due to the multi-step nature of the experiments to be preformed 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.