

**Chemistry 339**  
**Organic Syntheses**  
**Spring 2017**

**Instructor:** Dr. Jessica M. Hoover  
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**Teaching Assistant:**  
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**Classes:** Lecture: Th 1:30 – 2:20 pm in Clark Hall Rm. 200  
Lab: T, Th 2:30 – 5:15 pm in CRL Annex Rm. 480

**Office Hours:** Tuesday 1:30 – 2:20 pm and by appointment (Room 459 CRL)

**Course Overview:** In this course you will learn advanced organic synthesis techniques including the handling of air sensitive reagents and reactions, vacuum distillation, and silica column chromatography. You will perform several classic named reactions, such as a Diels-Alder reaction, a Parikh-Doering oxidation, a Wittig reaction, and a Friedlander synthesis. In addition to the laboratory techniques, you will learn to read and interpret experimental procedures from the chemical literature, search the chemical literature to devise multi-step synthetic procedures for a particular structural target, assess the potential safety concerns of a procedure, and to plan and carry out multi-step synthetic sequences in the lab safely and efficiently.

The lecture component of the course will be used as a discussion section to address challenges encountered in the laboratory in the previous week and to introduce new techniques and relevant safety information for upcoming experiments.

## Tentative Laboratory Schedule

Week	Date	Experiment
Week 1	T 1/10 Th 1/12	<i>No Lab</i> Check-in and Expt. 1. Manifold
Week 2	T 1/17 Th 1/19	Expt. 2. Quinoline Synth. Expt. 2. Quinoline Synth.
Week 3	T 1/24 Th 1/26	Expt. 2. Quinoline Synth. Expt. 3. Nitrophenylethyne
Week 4	T 1/31 Th 2/2	Expt. 3. Nitrophenylethyne Expt. 3. Nitrophenylethyne
Week 5	T 2/7 Th 2/9	Expt. 3. Nitrophenylethyne Expt. 3. Nitrophenylethyne
Week 6	T 2/14 Th 2/16	Expt. 4. Benzoxazole Expt. 4. Benzoxazole
Week 7	T 2/21 Th 2/23	Expt. 4. Benzoxazole Expt. 2/4. Coupling Reaction
Week 8	T 2/28 Th 3/2	Expt. 2/4. Coupling Reaction Expt. 2/4. Coupling Reaction
Week 9	T 3/7 Th 3/9	<i>Spring Break, No Class</i> <i>Spring Break, No Class</i>
Week 10	T 3/14 Th 3/16	Expt. 5. Dihydronaphthalene Expt. 5. Dihydronaphthalene
Week 11	T 3/21 Th 3/23	Expt. 5. Dihydronaphthalene Expt. 5. Dihydronaphthalene
Week 12	T 3/28 Th 3/30	Expt. 5. Dihydronaphthalene Expt. 5. Dihydronaphthalene
Week 13	T 4/4 Th 4/6	Expt. 5. Dihydronaphthalene Expt. 5. Dihydronaphthalene
Week 14	T 4/11 Th 4/13	Expt. 5. Dihydronaphthalene Expt. 5. Dihydronaphthalene
Week 15	T 4/18 Th 4/20	Expt. 6. Vinyl Quinoline Synth. Expt. 6. Vinyl Quinoline Synth.
Week 16	T 4/25 Th 4/27	Expt. 6. Vinyl Quinoline Synth. Lab cleanup and check-out
Week 17	W 5/3	Final Exam, 8 am – 10 am

Assignment	Points (125/100)
Experiment 1	ungraded
Experiment 2	10
Experiment 3	10
Experiment 4	10
Experiment 5 (A, B, and C)	30
Experiment 6	10
Experiment 6 Procedure	25
Final Exam	25
Lab/TA Points	5

*\* All experiment reports are due at 1:30pm and products at 2:30 pm on April 27<sup>th</sup> \**

**Laboratory Experiments:** The purpose of this lab course is to learn to plan and execute multi-step organic syntheses. This involves learning advanced techniques of organic synthesis and learning to search and read the chemical literature.

Laboratory time is limited and it is important that you use your time efficiently. There will be times when you will have to wait to use a piece of equipment. Try to find something else that needs to be done (use a different instrument, or perform a different part of an experiment) while you're waiting. We will also be sharing the vacuum & nitrogen manifolds and it will be important to plan and coordinate your experiments. Tasks like running a column, collecting NMR data, or recrystallizing a product can be done anytime, but setting a reaction up on the vacuum line will need to be done on a day when your group is scheduled to use the manifolds. Learning to plan your experiments and use your time efficiently is a critical laboratory skill, and one that you will be graded on in this course.

There are more experiments and syntheses assigned in this course than you can (and are expected to) complete! **It is important that you focus your efforts on the quality of your work, not the quantity.**

**Laboratory Reports:** Each of the laboratory reports will be due at the end of the semester. For each of Experiments 2-6, you will hand in a brief but formal laboratory report that consists of: (1) a ChemDraw scheme of the synthesis you completed, (2) a detailed experimental procedure for each step completed that is modeled after a scientific journal (including a ChemDraw structure of the intermediate or product made, your isolated yield, and your spectroscopic characterization data), (3) 2 copies of the  $^1\text{H}$  NMR data of any intermediates you made, (4) 2 copies of the  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, and IR spectral data of any final product(s) you made, and (5) your isolated compound(s) in a vial labeled with your name, the date, the name of the product, and the structure of the product. For Experiment 6, you will also be required to plan your own synthetic procedure. **Your proposed synthesis will be due in lecture on Thursday March 2<sup>nd</sup>.**

Your laboratory reports and your final compounds will be graded on quality (purity, spectral data, laboratory technique used, writing, etc.) not quantity. It will be better for you to complete three steps of a synthesis to isolate a clean crystalline intermediate than to complete five steps and isolate a brown goop!

**Laboratory Safety:** Appropriate laboratory attire will be enforced. Safety goggles and a lab coat must be worn at all times in the lab. Closed-toe shoes and long pants or long skirts are required. Eating or drinking in the lab is forbidden.

There are a number of hazards in this (and any) laboratory. Safety is an important aspect of this class, and you should think about safety as you read through your laboratory manual, and you plan your experiments, and as your work in the lab. The most important safety rule is to **think!** Think about what you are doing at any given time while in the laboratory. If there is anything that doesn't seem right or doesn't make sense to you, stop what you are doing and ask the instructor or your TA.

**Laboratory Notebook:** You must keep a good notebook in the course. Use a bound 8"x10" laboratory notebook that pages cannot be removed from. Update your notebook before each laboratory session (not experiment) with a description of the experiment you will perform. You should revise this procedure as you work, to reflect what you're actually doing. Your laboratory notebook is the place for recording your plan of attack, all of your experimental results, measurements, observations, and conclusions. It is a record of what you did and it should be written and revised as you are working. The teaching assistant and/or the instructor may check your notebook at anytime during the semester.

**Final Exam:** The final exam will be held on **Wednesday May 3<sup>rd</sup> from 8 am – 10 am**. This final exam will test any and all material covered throughout the course, including but not limited to, laboratory techniques, relevant organic reaction mechanisms, details of synthetic steps and purifications conducted in the laboratory. A make-up exam will be given only for valid reasons at the instructor's discretion and may be of a format very different from the regularly scheduled one.

**Academic Dishonesty:** According to the Office of the Provost "*Academic dishonesty includes the following: plagiarism; cheating and dishonest practices in connection with examinations, papers, and projects; and forgery, misrepresentations, and fraud.*"  
([http://provost.wvu.edu/academic\\_affairs/undergraduate/university\\_policies/academic\\_integrity](http://provost.wvu.edu/academic_affairs/undergraduate/university_policies/academic_integrity)).

Academic dishonesty on any assignment will result in a grade of zero for that assignment. Laboratory reports that are plagiarized will be given a grade of zero. This includes copying parts or all of your report from published materials or other students. Any material taken directly from another source should be referenced appropriately. Academic dishonesty on an exam will result in a zero on that exam.

**Social Justice Statement:** West Virginia University is committed to social justice. I concur with that commitment and expect to maintain a positive learning environment based upon open communication, mutual respect, and non-discrimination. West Virginia University does not discriminate on the basis of race, sex, age, disability, veteran status, religion, sexual orientation, color or national origin. Any suggestions as to how to further such a position and open environment in this class will be appreciated and given serious consideration. If you are a person with a disability and anticipate needing any type of accommodation in order to participate in this class, please advise me and make appropriate arrangements with Disability Services (293-6700).