University of Rhode Island; Department of Chemistry
CHM 425: Qualitative Organic Analysis Laboratory
Fall 2022; TR 2:00-5:00pm
Beaupre 235

Course Instructor	Matt Kiesewetter 325F Beaupre mkiesewetter@uri.edu
Office Hours	email for appointment
Teaching Assistant	Kassie Picard
Textbook	Experimental procedures and supplemental information will be provided to you, but the textbook for the accompanying lecture course may be useful for preparing laboratory reports. (optional) Grossman, *The Art of Writing Reasonable Organic Reaction Mechanisms*, 2nd Ed (ISBN 0-387-95468-6)
Course Goals	CHM 425 is a pathway to independent research. The ultimate goal of the course is to provide students a basis by which they will be able to conduct a chemical reaction that they have never performed before. The 'real world' of organic chemistry involves performing reactions and making compounds that are unknown. This course will introduce students to a variety of chemical techniques that they may implement in their professional development and careers.
Course Policies	Evaluations are directly related to work performed in lab, and attendance is required. There are no makeup labs. A missed presentation or late report will be scored as zero, no exceptions. Expectations for lab reports, presentations and conduct in lab are detailed in a separate handout. All graded work should be performed individually with the exception of presentations on which collective grades will be issued. Cheating or plagiarism on a graded assignment will result in a zero for that evaluation and possible referral to the Dean and failure of the course. Students are expected to follow the University policy of ACADEMIC HONESTY and all other University policies.
Community Health	Facemasks will not be required during the laboratory session but may be worn by individuals. HOWEVER, the instructor, TA or university may deem that facemasks become required at some point during the semester. If this event arises, students will be informed via email. If a student is experiencing COVID-19 symptoms or symptoms of illness, they should not come to class, report to Health Services and get a COVID test. Students who miss lab/presentations as a result of a positive COVID test will receive alternate assignments or be excused from the experiment. Should the University shift to instruction online, alternative assignments and scheduling will be required.
Grading	Student grades will be based on 2 in-class group presentations of their experimental results (100 pts each), 5 brief reports (100 pts each) and 1

experimental section (200 pts). Brief lab reports will be due at the beginning of the lab 1 week after the experiment is completed. Final grades will be determined by a scale no stricter than >90% A, >80% B, >70% C, >60% D. Students may request a re-grade on any evaluation for up to 2 days from when the evaluations are returned in class. If a student must be absent for a legitimate reason, missing an experiment or presentation, their grade will be based on their other lab evaluations.

Tentative Schedule

Meeting	Date	Topic
1	9/8	Course introduction; check-in
2	9/13	Diels-Alder Cycloaddition
3	9/15	Diels-Alder Cycloaddition
4	9/20	Diastereoselective Reduction of a Ketone
5	9/22	Diastereoselective Reduction of a Ketone
6	9/27	Synthesis and COSY of an Unknown Ester
7	9/29	Synthesis and COSY of an Unknown Ester
8	10/4	Aldol Condensation
9	10/6	Aldol Condensation
10	10/11	Polystyrene
11	10/13	Presentation preparation*
12	10/18	*Experiment Section 1 Presentations*
13	10/20	Stilbene Synthesis by Wittig
14	10/25	Stilbene Synthesis by Wittig
15	10/27	Stilbene Synthesis by Metathesis Reaction
16	11/1	*Experiment Section 2 Presentations*
17	11/3	**No lab**
18	11/8	**Election Day- no class**
19	11/10	Synthesis of a Pyrethrin
20	11/15	Synthesis of a Pyrethrin
21	11/17	Synthesis of a Pyrethrin
22	11/22	Synthesis of a Pyrethrin
23	11/25	**Thanksgiving Day – no class**
24	11/29	Synthesis of a Pyrethrin
25	12/1	Synthesis of a Pyrethrin
26	12/6	Synthesis of a Pyrethrin
27	12/8	Synthesis of a Pyrethrin
28	12/13	Synthesis of a Pyrethrin

*This time can be used to meet with your presentation group and prepare for your presentations, but there may not be a formal meeting of the course.

Experiment Sections

Section 1 – Mechanism and Analysis

1. Diels-Alder Cycloaddition
2. Diastereoselective Reduction of a Ketone
3. Synthesis and COSY Analysis of an Unknown Ester
4. Aldol Condensation

Section 2 – Synthesis and Catalysis

5. Polystyrene
6. Stilbene Synthesis by Wittig Reaction
7. Stilbene Synthesis by Metathesis Reaction

Section 3 – Capstone Project

8. Synthesis of a Pyrethrin

Evaluation Rubrics

Presentations (2 @ 100 pts each)

Oral presentations are designed to familiarize students with speaking publicly about science. Students are expected to share presentation responsibilities with all group members. Individual students will be allowed to be evaluated outside the group setting in special circumstances upon request *prior* to group presentations.

Evaluation will be based on correctly and completely addressing these criteria:

1. The questions posed in the lab experiment.
2. Including relevant background information.
3. Presentation of results.
4. Discussing sources of error and suggesting improvements.
5. Answering questions asked during the presentation.
6. Critical analysis of results.
7. Clarity of presentation.

Visuals for the presentations should be prepared in Powerpoint or similar.

Brief Lab Report (5 @ 100 pts each)

Brief lab reports are due at the beginning of the lab period 1 week after an experiment is completed. Reports are to be performed alone with your own data unless instructed otherwise. Reports should be type written and use Chemdraw (or similar) for figures. Spectra can be assigned by hand.

Evaluation will be based on correctly and completely providing these sections:

1. *Purpose* (5 pts): a purpose of the experiment is given
2. *Reaction scheme* (5 pts): a reaction scheme is given, if applicable
3. *Data* (25 pts): relevant data is given in table or list form
4. *Spectra* (25 pts): relevant spectra are provided and assigned
5. *Conclusion* (20 pts): briefly discuss the meaning of your results, answer all questions posed in the lab experiment, give suggestions for improving the experiment/yield/etc
6. *Laboratory Notebook* (10 pts): hand in copies of your lab notebook pages with your report
7. *Lab Technique and Citizenship* (10 pts): Students maintain a clean and respectable learning environment. Minor violations include: coming to lab unprepared to perform an experiment; maintain an untidy work space and does not return the lab to its starting condition; fail to follow the lab safety policies.

Laboratory notebook and record keeping
All students are expected to maintain a laboratory notebook which will be handed in after each experiment. See notes on laboratory notebook keeping below.

Evaluation Criteria (all, half or no credit):
1. Student comes to lab prepared to conduct the experiment.
    a. A reaction scheme is included, if appropriate.
    b. Quantities of reagents are noted.
2. Amounts of reagents are given in mass/volume, moles and molarity.
3. A clear description of their actions during the experiment are given.
4. Existence and identity of spectroscopic/characterization are correctly noted.
5. All relevant information is recorded.
6. Usefulness of entry.

Experimental (1 @ 200 pts)

For the last experiment, your report consists of writing an experimental that is ready for publication. This should include the following items:

1. A chemdraw figure of the product (10 pts).
2. A concise experimental procedure for preparing the compound in proper ACS format (*J. Am. Chem. Soc*. or *J. Org. Chem*.). (60 pts)
3. Reference (in proper ACS format) to prior synthesis or a statement: "This is a new compound." (20 pts)
4. Isolated yield. (10 pts)
5. Statement on the purity of your compound. If impure, state why it could not be purified and speculated with evidence as to the identity of the impurity. (40 pts)
6. 1H NMR, 13 NMR and mass spec data. Your spectra should be assigned to a structure. (60 pts)

CHM 425 Lab Safety Policies

Most laboratory accidents can be obviated by thinking ahead, communicating and planning for the worst. However, it is possible to do everything right and have something go wrong. Below are some guidelines to help prevent lab accidents.

1. Students should be familiar with and adhere to all URI and Chemistry Department safety guidelines.
2. It is the responsibility of all students to be familiar with the chemicals that they are using, the procedures they are executing, the hazards associated with them and the proper safety precautions.
    a. *Arrive* at lab familiar with the procedure that you will be conducting and with questions in mind. You will be allowed to refer to the lab handout during the lab session, but you should only need to do so for clarification.
    b. If you are unsure of a procedure, ask someone! It is better to wait to do an experiment until the prof/TA or a knowledgeable colleague is available than to rush and cause an accident.
    c. Be especially cautious and respectful of chemicals and procedures that can result in serious injury such as those involving flammable reagents, pressurized gasses, mutagens, carcinogens, especially when conducted on a large scale.
3. Safety glasses, closed-toe shoes, gloves and lab coats should be worn at all times in the laboratory area. Long pants are advised.
4. The use of flammable gases can result in the formation of gas pockets. Care should be used to minimize the accumulation of gases and avoid sparks.
    a. The spill of large amounts of flammable materials should be managed according to URI and department guidelines. REMEMBER, the spill remains dangerous after all liquid is cleaned up as flammable vapors remain.
5. Be defensive! Always plan for something to go wrong and decide what you can do to respond. Plan what you can do to avoid something going wrong in the first place.
6. Know the location and type of all fire extinguishers in the lab.
7. Create a pleasant work environment for all collaborators.
8. Maintain an orderly lab space.
    a. Put chemicals back to where they are inventoried.
    b. Clean your glassware!

Keeping a Laboratory Notebook

Students should acquire a laboratory notebook and maintain it according to the rules below.

An example lab page is included.

In short: A lab notebook should contain enough information that a person 'skilled in the art' could reproduce your experiments.

At length:

1. All students must keep a lab notebook with consecutively numbered pages and bring it to lab every session ready to go (i.e. do not *start* filling out your book in lab).
2. Each experiment should receive its own page (or more than one page).
3. Notebooks should be numbered consecutively.
4. Place a chemical drawing of the reaction being performed at the beginning of the entry, if applicable.
5. All spectra/data should be numbered (physically on the paper) and saved to the spectrometer/instrument so that they reference a specific notebook page. Use the format: 'your initials'-'notebook number'-'page number' (ex. MK-1-19). The type of data collected and what was analyzed should be clearly labeled in the notebook.

a. Multiple analyses should be given letters. Example: If MK-1-19 is the crude material, the purified material could be called MK-1-19b. Consider giving complicated purifications their own notebook page/experiment.

6. Cross reference your pages, if applicable.
7. For experiments that use more than one page, clearly label at the bottom of the first page where the second page starts (it may not always be on the next page).
8. For each reagent used, clearly give 1) the desired amount; 2) the amount used in grams/mL/etc; 3) (m)mol; and 4) (m)M; 5) formula weight and density (if applicable)

a. These data will help as you go to write up experiments and helps trace errors later on.

9. Backup your data and/or print everything out (and organize it!).
10. Date your entries.
11. Write down a purpose or hypothesis statement. It can be difficult to remember why you did something 6 months after you do it.
12. Beware of cross-contamination. A notebook that travels from desk to lab takes unknowns along for the ride.
13. Create and update a table of contents.