University of Rhode Island; Department of Chemistry
CHM 425: Qualitative Analysis Laboratory
Fall 2014; TR 2:00-5:00pm, Pastore Annex

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Office Hours  RF 12:30-1:30 pm and by appointment

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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.

Grading  Student grades will be based on 3 in-class group presentations of their experimental results (100 pts each), laboratory notebook and record keeping (100 pts), lab technique and citizenship (100 pts), and 5 brief reports (100 pts each). Brief lab reports will be due at the beginning of the lab 1 week after the experiment in 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 1 week from when the evaluations are returned. 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

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<tr>
<th>Meeting</th>
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<th>Topic</th>
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<td>1</td>
<td>9/4</td>
<td>Course introduction; check-in</td>
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<tr>
<td>2</td>
<td>9/9</td>
<td>Unknown separation and identification</td>
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<tr>
<td>3</td>
<td>9/11</td>
<td>Unknown separation and identification</td>
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<tr>
<td>4</td>
<td>9/16</td>
<td>Diels-Alder Cycloaddition</td>
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<td>5</td>
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<td>Diels-Alder Cycloaddition</td>
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<td>6</td>
<td>9/23</td>
<td>Synthesis and COSY Analysis of an Unknown Ester</td>
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<td>7</td>
<td>9/25</td>
<td>Synthesis and COSY Analysis of an Unknown Ester</td>
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<td>Multi-step Synthesis of a Sulfa Drug</td>
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<td>10/7</td>
<td>Multi-step Synthesis of a Sulfa Drug</td>
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<td>Polystyrene</td>
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<td>15</td>
<td>10/23</td>
<td>Diastereoselective Reduction of a Ketone</td>
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<td>Diastereoselective Reduction of a Ketone</td>
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<td>Stilbene Synthesis by Wittig Reaction</td>
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<td>Veterans Day – no class</td>
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<td>Stilbene Synthesis by Metathesis Reaction</td>
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<td>22</td>
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<td>Stilbene Synthesis by Metathesis Reaction</td>
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<td>23</td>
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<td>24</td>
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<tr>
<td>26</td>
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<td>Experiment Section 3 Presentations</td>
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<td>27</td>
<td>12/4</td>
<td>Check-out</td>
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### Experiment Sections

#### Section 1
1. Separation and Identification of Unknowns
2. Diels-Alder Cycloaddition
3. Synthesis and COSY Analysis of an Unknown Ester

#### Section 2
4. Multi-step Synthesis of a Sulfa Drug
5. Polystyrene
6. Diastereoselective Reduction of a Ketone

#### Section 3
7. Stilbene Synthesis by Wittig Reaction
8. Stilbene Synthesis by Metathesis Reaction
Evaluation Rubrics

Presentations (3 @ 100 pts each)

Oral presentations are designed to familiarize students with speaking publically 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.
7. Clarity of presentation.

Visuals for the presentations should be prepared in Powerpoint.

Laboratory notebook and record keeping (100 pts)

All students are expected to maintain a laboratory notebook which will be evaluated in spot checks during lab (50 pts) and handed in after each experiment (50 pts).

Spot Check 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 or reagents are noted.
   c. A table of contents entry is made.

Post-lab Evaluation Criteria (all, half or no credit):

1. Amounts of reagents are given in mass/volume, moles and molarity.
2. A clear description of their actions during the experiment are given.
3. Existence and identity of spectroscopic/characterization are correctly noted.
4. All relevant information is recorded.
5. Usefulness of entry.

Lab Technique and Citizenship Evaluation Criteria (100 pts)

Each ‘demerit’ will result in the loss of 5 pts, so behave.

Demerit-worthy violations:
1. Students engage in unsafe practices.
2. Students maintain an untidy work space and does not return the lab to its starting condition.
3. Students fail to follow the lab safety policies.
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**: a purpose of the experiment is given (5 pts)
2. **Reaction scheme**: a reaction scheme is given, if applicable (10 pts)
3. **Data**: relevant data is given in table or list form (25 pts)
4. **Spectra**: relevant spectra are provided and assigned (25 pts)
5. **Conclusion**: briefly discuss the meaning of your results, answer all questions posed in the lab experiment, give suggestions for improving the experiment/yield/etc (30 pts)
6. **Give one technique that you learned or learned how to do better** (5 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. Students will be required to hand in a copy (a carbon copy or photocopy) of their lab book entries for each experiment; hand in all pages. These copies are due to the TA/prof. by noon the day after a lab experiment concludes.

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.