CHEM2500 Instrumental Methods in Organic Synthesis
The University of Toledo
Department of Chemistry & Biochemistry
College of Natural Sciences
CRN: 41991 (Sect. 1) and 45515 (Sect. 2)

Name: Dr. Samantha Schachermeyer
Email: samantha.schachermeyer@utoledo.edu
Office Hours: TR 9:00-11:30am
or By Appointment
Office Location: BO 2036
Instructor Phone: (419)530-4592
Offered: Fall 2018
Class Location: TBA
Class Day/Time: R 11:30-12:25
Lab Location: BO 3097
Lab Day/Time: TR 1:00-3:50PM
Credit Hours: 2

CATALOG/COURSE DESCRIPTION

Practice of organic laboratory techniques. Three hours of laboratory per lab session, twice a week. Approved chemical safety goggles meeting the American National Standard Z87.1-1968 must be worn by every student during every laboratory class meeting.

COURSE OVERVIEW

Welcome to Organic Chemistry! The purpose of this laboratory course is to introduce students to the techniques that organic chemists (as well as biochemists, physical chemists, etc.) use in their daily routines. After learning and understanding those techniques, students will apply their knowledge to new situations to understand synthesis reactions, molecular structure determination, and analysis of (un)known compounds.

Organic chemistry laboratory is important for several reasons. It introduces students to many different laboratory practices and concepts that will be used in subsequent chemistry laboratory classes (CHEM 2500 and beyond) and in other laboratory situations in biology, pharmacy, and chemical engineering (just to name a few!). It is anticipated that by the completion of this course, students will be familiar with all of the following topics and techniques:

- Safety in the laboratory
- Interpreting and following scientific directions
- Keeping a proper lab notebook
- Names and proper usage of lab instruments
- Understanding of general properties of compounds (including solubility, miscibility, acid/base chemistry, etc.)
- Proper usage of glassware
- Isolation and purification techniques (including filtration, solvent removal, drying solutions, distillations, chromatography (thin-layer, column, and gas) and crystallization/recrystallization)
- Characterization techniques including spectroscopy and melting point determination
- Interpretation of scientific results including percent yield and recovery, melting point, boiling point, IR and NMR spectra, and R<sub>f</sub> values
STUDENT LEARNING OUTCOMES

Upon completion of this course, the student will be able to:

- Demonstrate their knowledge of departmental safety rules through their laboratory practice, including the ability to dispose of waste properly.
- Apply basic stoichiometric algorithms (calculating limiting reagents, theoretical yield, mole ratios) in the context of organic chemistry.
- Demonstrate a command of the rules for assigning significant figures in their work, specifically in calculations and laboratory measurements and calculations.
- Understand and be able to use the basic operations of an organic chemistry laboratory including gravity and vacuum filtration, liquid-liquid extraction, simple and fractional distillation, reflux, recrystallization, thin-layer chromatography, gas chromatography, column chromatography, drying of solids and solutions, and the theories behind these techniques.
- Know the significance of $pK_a$ values in experimental steps.
- Identify and assess the purity of organic compounds using analytical techniques, including melting point, thin-layer chromatography, IR, and gas chromatography.
- Deduce organic structures using spectroscopic methods, including infrared (IR), $^1$H- and $^{13}$C-nuclear magnetic resonance spectroscopy, and mass spectrometry.
- Determine molecular formulas from a mass spectrum by using the Rule of 13 and other techniques.
- Deduce hydrogen deficiency from a molecular formula and use this information to help deduce a structure.
- Be able to follow a detailed experimental procedure and construct a flow diagram to illustrate it.
- Depict and explain detailed chemical mechanisms for all laboratory reactions (and their related reactions).
- Demonstrate the ability to maintain a properly laboratory notebook.
- Construct a lab report that includes an analysis of the data collected, and discussion of the outcomes and answers to open questions associated with the experiment.

PREREQUISITES AND COREQUISITES

This course is for students who have the equivalent of CHEM 2420 and CHEM 2470 with a minimum of D-in both.

REQUIRED INSTRUCTIONAL MATERIALS (TEXTS AND ANCILLARY MATERIALS)

A. Required Materials:

- A laboratory notebook with carbon(less) pages (can be purchased from the UT bookstore)
- Approved safety goggles (can be purchased from the UT bookstore or from the UT-ACS group)
- Lab manual will be posted through Blackboard
UNIVERSITY POLICIES

Policy Statement on Non-Discrimination on the basis of Disability (ADA): The University is an equal opportunity educational institution. Please read The University’s Policy Statement on Nondiscrimination on the Basis of Disability Americans with Disability Act Compliance.

ACADEMIC ACCOMMODATIONS

The University of Toledo is committed to providing equal access to education for all students. If you have a documented disability or you believe you have a disability and would like information regarding academic accommodations/adjustments in this course please contact the Student Disability Services Office.

COURSE EXPECTATIONS

You are expected to come to both the lectures and labs on time. Failure to attend the prelab lecture will lead to your being excluded from the lab for that particular day. You are expected to come to each lab, and there will be pre-lab assignments which must be complete before you will be allowed to participate in the lab.

COURSE STRUCTURE

Lecture

- Lecture sessions are designed to clarify the concepts covered in the lab, as well as give an overview of techniques that will be used in the lab.
- Attendance is expected: The labs are only 3 hours in duration, so these lectures will be where you learn everything that you’ll need.
- Lab exercises will be available on Blackboard for each week.
- Please be considerate of your fellow students during the lecture period. Disruptions of any kind will not be tolerated and may result in expulsion from the classroom.

Laboratory

- Labs will be principally conducted by your TA (Dr. Samantha Schachermeyer), with assistance from myself (Prof. Young), as well as Dr. Yong-Wah Kim.
- You will be required to have appropriate clothing before being allowed to enter the lab.
- You will be expected to adhere to all of the lab safety rules.

Blackboard

- Blackboard is a course management system provided by the University of Toledo and can be accessed at https://blackboard.utdl.edu/ . Your access code is your UTAD user name and password.
- You should consult the site regularly for news and announcements. Handouts, lecture notes, and lab experiments will be posted. The system also permits you to check your grades at any time and to email your instructor or other students in the class.

Inclement Weather Policy
If classes are cancelled on a lab day, lab WILL PROCEED as normally scheduled. We will adjust the experiments to adjust for the reduced availability of lab time. If both portions of a lab are cancelled, your grade will be determined based on the labs we have been able to complete.

Lab Absence Policies
Refer to UT Missed Class Policy (https://www.utoledo.edu/policies/academic/undergraduate/pdfs/3364-71-14%20Missed%20class%20policy.pdf).

Communication
You are urged to communicate with Prof. Young or the Teaching Assistant (Dr. Samantha Schachermeyer) about any aspect of the course which concerns you or which might limit your success. We want you to be successful in this course, so let’s work together!

Chemistry Help Center
The Chemistry Help Center, Room BO 2043, is where the teaching assistants hold their office hours so it is a great place to receive assistance. It is generally open all day Monday through Friday and evenings Monday through Thursday. A schedule will be posted early in the term. No appointment is necessary.

OVERVIEW OF COURSE GRADE ASSIGNMENT

Course Points:
The following is the distribution of possible points in the course:

Lab Notebook and Reports (50 pts/lab, 25 Pts/instrumentation training) 400 pts
Library Training and Report 50 pts
Multi-Step Reaction and Report 150 pts
Unknown Identification and Report 150 pts
Final Written Exam and Practicum 100 pts

Total: 850 pts

FORMAT FOR LABORATORY NOTEBOOK REPORTS
Keeping an accurate laboratory notebook is essential to your success in this class. Some guidelines are given below:

• The laboratory notebook must not be loose leaf or spiral bound. Lab notebooks are available from the campus bookstore and are designed so that they permanently contain the original pages of your Prelab and Postlab reports.
• Use permanent blue or black ink only (ballpoint pen, NO red ink!).
• Other textbooks, lab manuals, loose sheets of paper, iPads or cellphones are not allowed in the laboratory. The complete outline of procedures must be written in your laboratory notebook prior to performing the experiment.
• Copies of your lab notebook pages are required for grading. The assigned notebooks are designed so that the carbon copies can be removed and handed in to your TA.
• Your TA may periodically inspect your notebook.

YOUR LAB REPORT CONSISTS OF THREE (3) PARTS
Part I - Prelab Report. A copy of your lab notebook pages containing the lab write-up and answers to any prelab questions. This is due at the start of each experiment.
Part II - Results. A copy of your notebook pages containing observations noted during the lab experiment. Is due with Part III one week from the conclusion of the experiment.
Part III - Postlab Report. A summary of results and answers to postlab questions. This can be written on separate loose-leaf paper. Is due with Part II one week from the conclusion of the experiment.

I. PRELAB REPORT (30% of the report grade)
The initial part of your lab report must be written in your laboratory notebook. A copy of the original pages of this report will be collected prior to the experiment and will be returned to you after the whole lab is graded. It will consist of:
• Your name, lab section and the name of your TA (on each page) (1 pt).
• The title and number of the experiment (1 pt).
• Objectives. This should include hypotheses about the outcome of the lab, which you will test by experiment. It is your responsibility to propose what you expect to determine from each experiment. (2 pts).
• Prelab question answers. These will always require an analysis of the hazards and risks associated with the experiment. It will also include the list of chemicals: masses or volumes, structures, and amounts. Look up molecular masses and calculate the material amount in moles (if appropriate), boiling/melting points (bp/mp, if appropriate) and density (if appropriate). Your prelab will suggest what is needed in the Reagent Table. (9 pts)
• List of equipment (sketch complex apparatus). (1 pt)
• Outline of procedure. This must be sufficiently detailed to allow you to perform the experiment. Make sure you note any necessary safety precautions. (1 pt).

The copy pages of this report must be handed in BEFORE you begin the experiment.

II. RESULTS (10% of the report grade)
This section should be started on a fresh page of your notebook, after the prelab report. A combined copy of the Results/Postlab report will be stapled and turned in to your TA after the experiment is complete.
This section should be completed during the lab session and consists of:
a) Your name, lab section and the name of your TA (on each page). (1 pt)
b) The title and number of the experiment. (1 pt)
c) Results: Date, times, measured masses and volumes used in the experiment (if you use different amounts from the procedure, note this), measured mp/bp of your products and any other observations (color changes, etc) recorded during the lab session. (1-2 pts, as appropriate)
d) Characterization materials: include copies of spectra, etc., recorded during the lab session. (0-2 pts, as appropriate)

III. POSTLAB REPORT (60% of the report grade)
This section does not need to be written in your lab notebook - it can be written on separate loose leaf sheets and stapled to your results copy pages. It is to be completed after the lab period at home, and consists of:

- Your name, lab section and the name of your TA (on each page). (1 pt)
- The title and number of the experiment. (1 pt)
- Analysis of results: In 5-10 sentences, comment on the outcome of your experiment, notably the quality of your results. Describe problems that may have occurred and possible solutions. If there was any deviation from what you expected, explain how and why did the outcome differ from that predicted in your prelab report? What was learned from the experiment? (10-20 pts, as appropriate)
- Answers to postlab questions, including labelling of spectral characterization. (20-40 pts, as appropriate)

Staple Parts II and III together and turn into your TA at the beginning of the next week's lab session. You should keep a copy of Part III for yourself.

Reaction Optimization
Details about the reaction optimization experiments will be posted to Blackboard with the Experiment Information.

Midterm Grading
Midterm grading serves as a point in the term where the instructor of record may provide a midterm grade assessment and may identify any student who has never attended, has stopped attending, or who is not actively participating in the course. In addition, students may use midterm grade to help make a decision in regards to withdrawing from the course.

The U.S. Department of Education requires the University to document both active participation and satisfactory academic progress as part of the compliance with federal financial aid regulations. Students receiving Title IV Federal Aid funds are required to have regular attendance and satisfactory academic progress in their courses to receive federal aid.

Final Grading
Your final grades will be calculated based on a total of 850 points.

Grade Scale  These are the minimum percentages (points) needed to receive the indicated grade:

<table>
<thead>
<tr>
<th>Grade</th>
<th>Minimum Percentage</th>
<th>Minimum Points</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>90% (765)</td>
<td></td>
</tr>
<tr>
<td>A−</td>
<td>87% (739)</td>
<td></td>
</tr>
<tr>
<td>B+</td>
<td>84% (714)</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>81% (688)</td>
<td></td>
</tr>
<tr>
<td>B−</td>
<td>78% (663)</td>
<td></td>
</tr>
<tr>
<td>C+</td>
<td>75% (637)</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>72% (612)</td>
<td></td>
</tr>
<tr>
<td>C−</td>
<td>69% (586)</td>
<td></td>
</tr>
<tr>
<td>D+</td>
<td>66% (561)</td>
<td></td>
</tr>
<tr>
<td>D</td>
<td>63% (535)</td>
<td></td>
</tr>
<tr>
<td>D−</td>
<td>60% (510)</td>
<td></td>
</tr>
</tbody>
</table>

Drop, Withdrawal and Incomplete Grades
- Course drop and withdrawal procedures have been set by the University faculty. Dropped courses do not appear on your transcript. If you are in a course after that date, there will be a
grade on your transcript (A-F, W, or Incomplete). The deadline for dropping is September 10th.

- You may withdraw from the course and receive a grade of W. W's do not affect your GPA. For both dropping the course or withdrawing you should go to the Registrar's Office in Rocket Hall. You do not need your instructor’s permission for either process. Please note that course registration changes might change your financial aid. The deadline for withdrawal is November 16th.

- A course grade of Incomplete is given only to those who have completed all but a small percentage of course requirements for an acceptable reason.

Attendance/Class Participation
On two occasions during the term, instructors are asked to report student attendance. However, you will remain enrolled in the class independent of these reports. That is, you remain registered for the class and will receive a final grade unless you take the action of dropping or withdrawing. Attendance reports will be based entirely on clicker responses.
# Lab Schedule – Fall 2018

<table>
<thead>
<tr>
<th>Week</th>
<th>Date</th>
<th>Lab Exercise</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>28-Aug</td>
<td>NMR Training + NMR Unknowns</td>
</tr>
<tr>
<td>1</td>
<td>30-Aug</td>
<td>Check-in/Safety Video/ Instrumentation training (GC/IR/UV-Vis)</td>
</tr>
<tr>
<td>2</td>
<td>4-Sep</td>
<td>Library Assignment</td>
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<tr>
<td>2</td>
<td>6-Sep</td>
<td>Suzuki Cross-Coupling Reaction</td>
</tr>
<tr>
<td>3</td>
<td>11-Sep</td>
<td>Suzuki Cross-Coupling Reaction</td>
</tr>
<tr>
<td>3</td>
<td>13-Sep</td>
<td>Chemo-selective Epoxidation</td>
</tr>
<tr>
<td>4</td>
<td>18-Sep</td>
<td>Chemo-selective Epoxidation</td>
</tr>
<tr>
<td>4</td>
<td>20-Sep</td>
<td>Chiral Resolution</td>
</tr>
<tr>
<td>5</td>
<td>25-Sep</td>
<td>Chiral Resolution</td>
</tr>
<tr>
<td>5</td>
<td>27-Sep</td>
<td>Synthesis of a Thiazoline Heterocycle</td>
</tr>
<tr>
<td>6</td>
<td>2-Oct</td>
<td>Synthesis of a Thiazoline Heterocycle</td>
</tr>
<tr>
<td>6</td>
<td>4-Oct</td>
<td>Guest Binding in a Self-Assembled Capsule</td>
</tr>
<tr>
<td>7</td>
<td>9-Oct</td>
<td>Guest Binding in a Self-Assembled Capsule</td>
</tr>
<tr>
<td>7</td>
<td>11-Oct</td>
<td>Fall Break - No Lab</td>
</tr>
<tr>
<td>8</td>
<td>16-Oct</td>
<td>Indigo Lab</td>
</tr>
<tr>
<td>8</td>
<td>18-Oct</td>
<td>Indigo Lab</td>
</tr>
<tr>
<td>9</td>
<td>23-Oct</td>
<td>Multi-step Synthesis of a Natural Product</td>
</tr>
<tr>
<td>9</td>
<td>25-Oct</td>
<td>Multi-step Synthesis of a Natural Product</td>
</tr>
<tr>
<td>10</td>
<td>30-Oct</td>
<td>Multi-step Synthesis of a Natural Product</td>
</tr>
<tr>
<td>10</td>
<td>1-Nov</td>
<td>Multi-step Synthesis of a Natural Product</td>
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<tr>
<td>11</td>
<td>6-Nov</td>
<td>Multi-step Synthesis of a Natural Product</td>
</tr>
<tr>
<td>11</td>
<td>8-Nov</td>
<td>Multi-step Synthesis of a Natural Product</td>
</tr>
<tr>
<td>12</td>
<td>13-Nov</td>
<td>Multi-step Synthesis of a Natural Product</td>
</tr>
<tr>
<td>12</td>
<td>15-Nov</td>
<td>ID of Unknowns</td>
</tr>
<tr>
<td>13</td>
<td>20-Nov</td>
<td>ID of Unknowns</td>
</tr>
<tr>
<td>13</td>
<td>22-Nov</td>
<td>Thanksgiving - No Lab</td>
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<tr>
<td>14</td>
<td>27-Nov</td>
<td>No Lab</td>
</tr>
<tr>
<td>14</td>
<td>29-Nov</td>
<td>No Lab</td>
</tr>
<tr>
<td>15</td>
<td>4-Dec</td>
<td>Lab Final/Check out</td>
</tr>
</tbody>
</table>