



CHEM2480 Organic Separations and Elementary Organic Synthesis

The University of Toledo
Department of Chemistry & Biochemistry
College of Natural Sciences

CRN: 41987 (Sect. 1) or 41989 (Sect. 91), 45513 (Sect. 2) or 45514 (Sect. 92), 41988 (Sect. 3) or 41990 (Sect. 93),
55303 (Sect. 4) or 55305 (Sect. 94)

Name:	Prof. Steve Sucheck	Class Location:	Bowman-Oddy Labs 1045
Email:	steve.sucheck@utoledo.edu	Class Day/Time:	T (001 091) 11:30-12:25
Office Hours:	M 1-2PM, TR 9-10:30AM, By Appt.	Lab Location:	BO 3097
Office Location:	WO 3276B	Lab Day/Time:	TR (002) 1:00-3:50PM, TR (003) 5:30-8:20PM, or WF (004 094) 1:00-3:50
Instructor Phone:	(419)530-1504	Credit Hours:	2
Offered:	Fall 2021		
Course Website:	Blackboard Learn		

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 / TEACHING METHODOLOGY

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 2480 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_f 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), ^1H - 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 (degrees of unsaturation) 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 labeled 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

Students must have completed the General Chemistry sequence before enrolling in this course (CHEM 1230/1240, 1280/1290) with a grade of C- or better, as well as having declared chemistry or biochemistry as a major. Students also are required to be concurrently enrolled in (or have successfully passed) Organic Chemistry I lecture (CHEM 2410). Please note that withdrawal from CHEM 2410 during the semester will necessitate withdrawal from CHEM 2480. A student, registered for both CHEM 2410 and 2480 concurrently, who is intending to drop/withdrawal from the lecture course by mid-semester (in first 8 weeks) must also drop the associated lab course. A student withdrawing from the lecture during the last weeks of allowed withdrawal (weeks 9-10) may be allowed to finish the lab course if they have a grade of C or better in the lab and permission of the lab instructor.



REQUIRED INSTRUCTIONAL MATERIALS (TEXTS AND ANCILLARY MATERIALS)

A. Required Materials:

- A laboratory notebook (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

TECHNOLOGY REQUIREMENTS

A. Required Technology:

- Computer and accesses to the internet to download and view instructional materials, cell phone or access to scanner to upload assignments to blackboard.

ACADEMIC POLICIES

Please read: [Undergraduate Policies: http://www.utoledo.edu/policies/academic/undergraduate/](http://www.utoledo.edu/policies/academic/undergraduate/)

UNIVERSITY POLICIES*

Federal law requires the university to have an Institutional Attendance Policy that requires faculty to track student participation by the census date, which varies for each POT, these dates can be found here: <https://www.utoledo.edu/offices/provost/mandatory-attendance-tracking.html>, for federal financial eligibility and disbursement.

Institutional Classroom Attendance Policy (Fall and Spring Statement)

Please be aware that the university has implemented an attendance policy, which requires faculty to verify student participation in every class a student is registered at the start of each new semester/course. For this course, if you have not attended/participated in class (completed any course activities or assignments) within the first 14 days, I am required by federal law to report you as not attended. Unfortunately, not attending/participating in class impacts your eligibility to receive financial aid, so it is VERY important that you attend class and complete course work in these first two weeks. Please contact me as soon as possible to discuss options and/or possible accommodations if you have any difficulty completing assignments within the first two weeks.

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](#). Students can find this policy along with other university policies listed by audience on the [University Policy webpage](#) (<http://www.utoledo.edu/policies/audience.html/#students>).

Academic Accommodations* The University of Toledo embraces the inclusion of students with disabilities. We are committed to ensuring equal opportunity and seamless access for full participation in all courses. For students who have an Accommodations Memo from the Office of Accessibility and Disability Resources, I invite you to correspond with me as soon as possible so that we can communicate confidentially about implementing accommodations in this course.



For students who have not established accommodations with the Office of Accessibility and Disability Resources and are experiencing disability access barriers or are interested in a referral to health care resources for a potential disability, please connect with the office by calling 419.530.4981 or sending an email to StudentDisability@utoledo.edu.

ACADEMIC AND SUPPORT SERVICES*

Please follow this link to view a comprehensive list of [Student Academic and Support Services](http://www.utoledo.edu/studentaffairs/departments.html) (<http://www.utoledo.edu/studentaffairs/departments.html>) available to you as a student

SAFETY AND HEALTH SERVICES FOR UT STUDENTS*

Please use the following link to view a comprehensive list [Campus Health and Safety Services](#) available to you as a student.

INCLUSIVE CLASSROOM STATEMENT

In this class, we will work together to develop a learning community that is inclusive and respectful. Our diversity may be reflected by differences in race, culture, age, religion, sexual orientation, gender identity/expression, socioeconomic background, and a myriad of other social identities and life experiences. We will encourage and appreciate expressions of different ideas, opinions, and beliefs so that conversations and interactions that could potentially be divisive turn, instead, into opportunities for intellectual and personal development.

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. Absolutely no late work will be accepted without prior approval of your teaching assistant!***

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. As many experiments require multiple days, you should expect to stay for the full duration on the first day of the lab – failure to complete an experiment because of poor time management on your part will not be overlooked.*
- *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, with occasional assistance from myself (Prof. Sucheck), as well as Dr. Yong-Wah Kim and the NMR TA.*

- You will be required to have appropriate clothing before being allowed to enter the lab.
- Prelabs are due at the beginning of the lab, and results and postlabs are due at the beginning of the lab 1 week after completion of the experiment! Assignments should be uploaded to Blackboard
- You will be expected to adhere to all of the lab safety rules.
- You are all expected to do your part to maintain a clean lab environment as part of GLP (Good Lab Practices):
 - All reagent and solvent bottles should be completely closed immediately after use;
 - All spills and dribbles should be cleaned immediately;
 - All glassware should be put away at the end of the lab, and walkways should be kept free of debris.
 - Failure to adhere to these rules will lead to punitive measures in the form of lost points as decided by your TA!
- Your TA will regularly ensure that you know what you're doing – Failure to adequately comprehend the lab may also lead to punitive measures in the form of lost points, so pay attention during pre-lab lectures!

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** at the next scheduled lab meeting. We will adjust the experiments to account 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. Sucheck or the Teaching Assistant (Uzoamaka “Clara” Bokolo (TR (002) 1:00-3:50PM), Leart Sejdarsi (TR (003) 5:30-8:20PM), Prem Gurung WF (004 094) 1:00-3:50) 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 except:	
25 Pts/training, 150 for Unknown Lab	725 pts 64.4%
200 Pts for Reaction Optimization	200 pts 17.8%
100 Pts for Lab Cleanliness (pts deducted when necessary)	100 pts 8.9%
Final Written Exam (December 7th, 11:30 am, BO 1045)	<u>100 pts 8.9%</u>
	Total: 1125 pts

Honors Credit

If you are an honors student, you will need to discuss with Prof. Sucheck the honors project immediately.

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 are not allowed in the laboratory.** The complete outline of procedures must be written in your laboratory notebook prior to performing the experiment.
- Scanned or photographed images of your lab notebook pages are required for grading. These scans and images should be uploaded to blackboard for grading.
- Your TA may periodically inspect your notebook.

YOUR LAB REPORT CONSISTS OF THREE (3) PARTS

Part I - Prelab Report. A scan/image of your lab notebook pages, uploaded to Blackboard, containing the lab write-up and answers to any prelab questions. This is due at the **start** of each experiment. Uploads are time stamped in Blackboard.

Part II - Results. A scan/image of your notebook pages, uploaded to Blackboard, 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 or directly in the notebook and scanned and uploaded to Blackboard. 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. You will be required to upload to blackboard, A scan or image of the original pages of this report prior to the experiment as a single file such as a PDF. 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 your actual notebook for must be inspected 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. You will be required to upload to blackboard, A scan or image of the combined copy of the Results/Postlab report after the experiment is complete as a single file such as a PDF.

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, in your notebook or using a word processor. You will be required to upload to blackboard, A scan or image of the post lab report as a single file such as a PDF combined with the notebook scans of results. 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)

Parts II, and III should be uploaded no later than the beginning of the next week's lab session. You should keep the notebook for yourself. Note you are required to provide legible scans of all parts in a file format like compatible with Blackboard.

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 1000 points.

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

A	90%	A-	87%	B+	84%	B	81%
B-	78%	C+	75%	C	72%	C-	69%
D+	66%	D	63%	D-	60%		

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 13th**.*
- *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 the end of the 10th week, **November 5th**.*
- *If you drop or withdraw from this CHEM 2480, you must also drop/withdraw from the lecture course CHEM 2410.*
- *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.



Lab Schedule – Fall 2021 (TR Section) (Red = NMR use)

<u>Week</u>	<u>Date</u>	<u>Lab Exercise</u>
1	8/31	Check-in/Safety Video/Exp 1A - Ethanol Biosynth.
1	9/2	Exp 2 - Solubilities of Organic Compounds
2	9/7	Exp 1B - Ethanol Distillation
2	9/9	Exp 3 - Crystallization Lab Due: Exp 2
3	9/14	Exp 4 – Extraction Due: Exp 1
3	9/16	Instrumentation (GC-MS/IR/UV-Vis) Due: Exp 3
4	9/21	Exp 5 - IR and UV/Vis- of Unknown Organic Compounds Due: Exp 4, GC-MS/IR/UV-Vis Instrumentation Worksheet
4	9/23	Instrumentation (NMR)
5	9/28	Exp 6 - NMR of Organic Compounds Due: Exp 5, NMR Instrumentation Worksheet
5	9/30	Exp 7 - Isolation of the Natural Product Caffeine
6	10/5	Exp 7 - Isolation of the Natural Product Caffeine Due: Exp 6
6	10/7	Exp 8 - Chiral Resolution
7	10/12	Exp 8 - Chiral Resolution Due: Exp 7
7	10/14	Fall Break

8	10/19	Literature Searching Exercise (<i>Carlson Library 1025</i>) Due: Exp 8
8	10/21	NMR/IR/GC MS Practice
9	10/26	Exp 9 - Identification of Unknowns Due: Literature Search Worksheet
9	10/28	Exp 9 - Identification of Unknowns
10	11/2	Exp 9 - Identification of Unknowns
10	11/4	No Lab (Work on Exp 9 Lab Report)
11	11/9	Exp 10 - Nucleophilic Substitution Reactions Due: Exp 9 Formal Lab Report
11	11/11	Veterans Day No Lab
12	11/16	Exp 11 - Elimination from <i>meso</i> -Stilbene Dibromide
12	11/18	Exp 12 - Optimization of a Heterocycle Synthesis Due: Exp 10
13	11/23	Exp 12 - Optimization of a Heterocycle Synthesis Due: Exp 11
13	11/25	Thanksgiving Break
14	11/30	Exp 12 - Optimization of a Heterocycle Synthesis
14	12/2	Exp 12 - Optimization of a Heterocycle Synthesis
15	12/7	Exp 12 - Optimization of a Heterocycle Synthesis
15	12/9	Exp 12 - Optimization of a Heterocycle Synthesis
16	12/14	Due: Exp 12 Formal Report



Lab Schedule – Fall 2021 (WF Section)

<u>Week</u>	<u>Date</u>	<u>Lab Exercise</u>
1	9/1	Check-in/Safety Video/Exp 1A - Ethanol Biosynth.
1	9/3	Exp 2 - Solubilities of Organic Compounds
2	9/8	Exp 1B - Ethanol Distillation
2	9/10	Exp 3 - Crystallization Lab Due: Exp 2
3	9/15	Exp 4 – Extraction Due: Exp 1
3	9/17	Instrumentation (GC-MS/IR/UV-Vis) Due: Exp 3
4	9/22	Exp 5 - IR and UV/Vis- of Unknown Organic Compounds Due: Exp 4, GC-MS/IR/UV-Vis Instrumentation Worksheet
4	9/24	Instrumentation (NMR)
5	9/29	Exp 6 - NMR of Organic Compounds Due: Exp 5, NMR Instrumentation Worksheet
5	10/1	Exp 7 - Isolation of the Natural Product Caffeine
6	10/6	Isolation of the Natural Product Caffeine Due: Exp 6
6	10/8	Exp 8 - Chiral Resolution
7	10/13	Chiral Resolution Due: Exp 7
7	10/15	Fall Break

8	10/20	Literature Searching Exercise (<i>Carlson Library 1027</i>) Due: Exp 8
8	10/22	NMR/IR/GC MS Practice
9	10/27	Exp 9 - Identification of Unknowns Due: Literature Search Worksheet
9	10/29	Exp 9 - Identification of Unknowns
10	11/3	Exp 9 - Identification of Unknowns
10	11/5	No Lab (Work on Exp 9 Lab Report)
11	11/10	Exp 10 - Nucleophilic Substitution Reactions Due: Exp 9 Formal Lab Report
11	11/12	Exp 11 - Elimination from <i>meso</i> -Stilbene Dibromide
12	11/17	Exp 12 - Optimization of a Heterocycle Synthesis Due: Exp 10
12	11/19	Exp 12 - Optimization of a Heterocycle Synthesis Due: Exp 11
13	11/24	Thanksgiving Break
13	11/26	Thanksgiving Break
14	12/1	Exp 12 - Optimization of a Heterocycle Synthesis
14	12/3	Exp 12 - Optimization of a Heterocycle Synthesis
15	12/8	Exp 12 - Optimization of a Heterocycle Synthesis
15	12/10	Exp 12 - Optimization of a Heterocycle Synthesis
16	12/14	Due: Exp 12 Formal Report