



CHEM2500 Instrumental Methods in Organic Synthesis

The University of Toledo
Department of Chemistry & Biochemistry
College of Natural Sciences
CRN: 11161 (Sect. 1) and 15895 (Sect. 3)

Name:	Dr. Zin-Min Tun	Offered:	Spring 2021
Email:	zinmin.tun@utoledo.edu	Class Location:	MFH 1110
Office Hours:	By appointment.	Class Day/Time:	R 11:30 AM -12:20 PM
Office Location:	BO 2034	Lab Location:	BO 3097
Instructor Phone:	(419) 530-4591	Lab Day/Time:	TR 5:30-8:20PM
		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_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 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 C- in both.

REQUIRED INSTRUCTIONAL MATERIALS (TEXTS AND ANCILLARY 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

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 completed 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 the instructor, 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.*



IMPORTANT COVID-19 SPECIFIC RULES

Due to the current additional requirements implemented to prevent the spread of Covid-19, the following summary outlines some important changes to this semester's lab course.

- Due to COVID-19, the maximum number of students that can safely work in each lab at a given time is 12.
- There will be **no loaner goggles** supplied by the Chemistry Stockroom. You must own your own goggles. You must arrive appropriately dressed (no exposed skin except for arms shoulders down, closed toe shoes that will not be penetrated by a simple spill) and with approved goggles.
- **NO students will be permitted in the lab without a face covering. We recommend that you bring a spare mask to lab in case the mask you are wearing gets exposed to chemicals by accidental touching etc.** If you have a medical reason that prevents you from wearing a face covering due to a health condition deemed high-risk for COVID-19 by the Centers for Disease Control and Prevention (CDC), please contact the Student Disability Services Office (SDS).
- The lab doors will be a "one-way", where one door serves as entrance and the other serves as exit.
- Backpack storage cubicles and hoods are numbered, and you will be assigned a specific set of hood and cubicle that you must use every time you are in the lab.
- When you arrive at the lab, wash your hands with water and soap, and put on gloves. Wipe your cubicle and your work area with paper towels sprayed with ethanol.
- When you are finished for the day, you should wipe your work area(s) and cubicle again. Whenever you use a shared instrument or piece of equipment, wipe it off with a paper towel/ethanol as soon as you are finished.
- Be conscientious of the distance between yourself and others. If you need somebody to briefly step aside so that you can pass through while **maintaining a 6-foot distance**, please ask them to do so.
- If you show any kinds of symptoms – do not attend lecture or lab, and inform Dr. Tun and your TA before your lab time. Virtual materials will be made available to you, and you will be able to finish your work and earn your grade this way.

Inclement Weather Policy

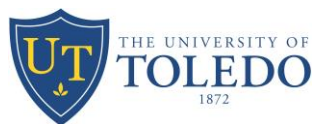
If classes are cancelled on a lab day, lab **WILL PROCEED** as normally scheduled. We will adjust the experiments to adjust for the reduced availability of lab time. If both portions of a lab are cancelled, your grade will be determined based on the labs we have been able to complete.

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 me about any aspect of the course with concerns you or which might limit your success. Please allow for 24-48 hours, not including weekends or holidays, for a reply from Dr. Tun. Please also make sure that you include your course number and section number (CHEM2500-001) either in the subject line or in the body of the email to facilitate the communication. We want you to be successful in this course so let's work together!



Chemistry Help Center

The Chemistry Help Center is where the teaching assistants hold their office hours so it is a great place to receive assistance. The spring semester virtual help center link is <https://us.bbcollab.com/quest/80670d8c3ff9469dbb520091a0612503>. The Help Center hours are MW 9-12, 1-4, 5-8; TR 9-8 and F 9-4, from January 25 until the end of classes. 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)	250pts
Informatics Training and Worksheet	50pts
NMR Training Worksheet	50pts
Reaction Optimization (Technique and Participation)	50pts
Reaction Optimization Final Report	100pts
Unknown Identification Report	150pts
Cleanliness Points (Starts at 100, points deducted as necessary)	<u>100pts</u>

Total: 750 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 Pre-lab and Post-lab 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.
- Your instructor may periodically inspect your notebook.

YOUR LAB REPORT CONSISTS OF THREE (3) PARTS

Part I - Prelab Report. The lab write-up and answers to any prelab questions. This is due at 11:59 p.m. the day before each experiment. Pre-lab must be submitted electronically on Blackboard.

Part II - Results. Scanned in 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. Typed up summary of results and answers to postlab questions 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 scanned in copy of these pages must be submitted electronically on Blackboard by 11:59 p.m. the day before each experiment.

Pre-lab will consist of:

- Your name, lab section and the name of your instructor (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).

II. RESULTS (10% of the report grade)

This section should be started on a fresh page of your notebook, after the prelab report.

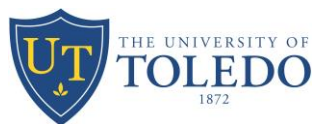
This section should be completed **during** the lab session and consists of:

- a) Your name, lab section and the name of your instructor (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 typed up. It is to be completed **after** the lab period at home, and consists of:

- Your name, lab section and the name of your instructor (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)

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 750 points.

Grade Scale

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

A	90% (765)	A-	87% (739)	B+	84% (714)	B	81% (688)
B-	78% (663)	C+	75% (637)	C	72% (612)	C-	69% (586)
D+	66% (561)	D	63% (535)	D-	60% (510)		

Drop, Withdrawal and Incomplete Grades: Course drop and withdrawal procedures have been set by the University faculty. Pay attention to those add/drop dates as they pass very quickly during the semester! For both dropping the course or withdrawing you should go to Rocket Solution Central 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.

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. If you have a serious problem near the end of the course, communicate with me as soon as possible. You will retain all of your previously determined grades.

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.

UNIVERSITY POLICIES

Your safety and well-being as a University of Toledo student is important to the faculty, staff, and administration; as such please take a minute to review the following university policies that apply to you as a student of the University:

- <https://www.utoledo.edu/title-ix/policies.html>
- The University of Toledo's Title IX (Sexual Misconduct) Policy - 3364-50-01
- Nondiscrimination 3364-50-02
- Nondiscrimination on the basis of disability - Americans with Disability Act compliance 3354-50- 03
- Consensual romantic and/or sexual relationships - Policy 3364-25-65
- Student Code of Conduct 3364-30-04

Please use this URL to view a more comprehensive list of student policies:

<https://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 Student Disability Services, 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 affiliation with Student Disability Services and are experiencing disability access barriers or are interested in a referral to healthcare resources for a potential disability or would like information regarding eligibility for academic accommodations, please contact the [Student Disability Services Office](http://www.utoledo.edu/offices/student-disability-services/) (<http://www.utoledo.edu/offices/student-disability-services/>) by phone: 419.530.4981 or email at 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.

OTHER SPECIAL NOTES

It is important to note that, based on the unpredictability of the COVID-19 virus, things can change at any time so please be patient and understanding as we move through the semester.



CHEM2500 Instrumental Methods in Organic Synthesis
Lab Schedule – Spring 2021

<u>Week</u>	<u>Date</u>	<u>Lab Exercise</u>
1	1/19	Check-in
1	1/21	Library Search Worksheet
2	1/26	Nuclear Magnetic Resonance Spectroscopy
2	1/28	Instrumentation
		Due: Library Search Worksheet
3	2/2	Exp 1 - NMR of Organic Compounds
3	2/4	Exp 1 - NMR of Organic Compounds
		Due: NMR Instrumentation Worksheet
4	2/9	Exp 2 - Suzuki Cross-Coupling Reaction
4	2/11	Exp 2 - Suzuki Cross-Coupling Reaction
		Due: Exp 1 Report
5	2/16	<i>No Lab – Instructional Break</i>
5	2/18	Exp 3 - Solid State Reactions
		Due: Exp 2 Report
6	2/23	Exp 3 - Solid State Reactions
6	2/25	Exp 4 - When Reactions Go Awry
7	3/2	Exp 4 - When Reactions Go Awry
7	3/4	Exp 5 - Guest Binding in a Self-Assembled Capsule
8	3/9	Exp 5 - Guest Binding in a Self-Assembled Capsule

8	3/11	Exp 3 - Solid State Reactions (Cont.) Due: Exp 4 Report
9	3/16	Reactions Catalyzed in a Supramolecular Capsule
9	3/18	Reactions Catalyzed in a Supramolecular Capsule Due: Exp 3 Report
10	3/23	Reactions Catalyzed in a Supramolecular Capsule
10	3/24	Reactions Catalyzed in a Supramolecular Capsule Due: Exp 5 Report
11	3/30	Reactions Catalyzed in a Supramolecular Capsule
11	4/1	Reactions Catalyzed in a Supramolecular Capsule
12	4/6	Reactions Catalyzed in a Supramolecular Capsule
12	4/8	Reactions Catalyzed in a Supramolecular Capsule
13	4/13	Identification of Unknowns
13	4/15	Identification of Unknowns
14	4/20	Identification of Unknowns
14	4/22	Identification of Unknowns Check-out Due: CURE Formal Report
15	4/27	Due: Identification of Unknowns Formal Report
