

Chemistry 2312H

Honors Organic Chemistry Laboratory

Fall 2023, 5 credits

<http://www1.chem.umn.edu/groups/hoye/teaching/Teaching2312HFall2023>

- Instructor:** Thomas R. Hoye, 419 Smith Hall, 612-625-1891, hoye@umn.edu
- Office Hours:** Tuesdays 11:00-noon (334 Smith)
(starting Sept. 8th) Fridays 1:15-2:15 (334 Smith)
- Teaching Assistants:**
- | | | | |
|-----------------------|-----------|--------------|------------------|
| Katharine Toll | 413 Smith | 508-596-6805 | toll0166@umn.edu |
| Jingyang Shi | 417 Smith | 612-876-7038 | shi00243@umn.edu |
| Rong Tang | 415 Smith | 612-446-9792 | tang0658@umn.edu |
- Lectures:** Tu, Th 8:00 - 8:50 AM, Smith 111 (plus 2-3 *ad hoc* sessions in the first two weeks)
- Prerequisite:** Chem 2301/2331 (or equivalent or concurrent) and permission of instructor
- Laboratory (491 K) Hours:** See “LabHoursF23” handout. A teaching assistant **will and must** be present at all times.
- Text:** Jerry R. Mohrig, David Alberg, Gretchen Hofmeister, Paul F. Schatz, Christina Noring Hammond *Laboratory Techniques in Organic Chemistry*, 4th ed., Freeman, New York, 2014 (earlier editions would also be fine).
- First Reading Assignment:** **Read chapters** 1. *safety* in the laboratory; 2. green chemistry; 3. laboratory notebook; 4. laboratory glassware; 5. measurements and transferring reagents; 6. heating and cooling methods; 10. extraction; and 18. thin layer chromatography **during the first week.**
- Second Reading Assignment:** **Read chapters** 19. liquid chromatography; 20. gas chromatography; 21. Infrared spectroscopy **during the second week of class.**
- Third Reading Assignment:** **Read chapters** 22. ¹H NMR spectroscopy; 24. mass spectrometry **during the third week of class.**
- Fourth Reading Assignment:** **Read chapters** 8. computational chemistry and 12. boiling points and distillation **during the fourth week of class.**
- Supplemental Reading:** You may also want to augment your reading and learning of the principal spectroscopic techniques we will use (MS, IR, and NMR) by (re)reading the relevant chapters in an introductory organic chemistry textbook such as L. G. Wade (Chaps. 12/13) or Carey et al. (Chap. 14). A very useful tutorial on infrared spectroscopy called “IR Tutor” can be found on the PC's in the lab computer room.
- Required Supplies:** Laboratory **safety goggles**, available for purchase in the stockroom, **must** be worn in the laboratory at all times. Wearing of **masks** is optional, per University regulation. A **permanent** (not loose-leaf) **notebook** of your choosing, dedicated to record keeping of your experimental work. Policy on wearing a lab coat is discussed below in the Safety section.
- Grades:** A written Lab Report will be required for each of five experiments (see separate handouts that I will provide for style/content guidelines, due dates, etc.).
- 80% of your course grade will be based on these reports and exercise(s).
 - 20% of your course grade will be based upon teaching staff judgement of the development and growth of your laboratory technique and prowess over the course of the semester.

Course Outline:	~Week #	Experiment #
	1-2	1
	3-5	2
	7-9	3 or 4 or 5
	10-12	4 or 5 or 3
	13-14	5 or 3 or 4

Exp. 1. Ketone Reduction by Sodium Borohydride

Exp. 2. Terpene Chemistry: a) Preparation of Nopinone by Ozonolysis of β -Pinene and b) Preparation (and Equilibration) of Menthone/Isomenthone by Hydrogenation of Pulegone

Exp. 3. Enolate Alkylation, Ester Saponification, and DCC Coupling with (*R*)-(+)-1-(1-naphthyl)ethylamine

Exp. 4. Catalysis: Palladium(0) Alkyne/Arene Coupling, Enzymatic Resolution, and Mosher Ester Analysis

Exp. 5. Diels-Alder Cycloaddition, Reduction, and Photocycloaddition

Instrumentation:

- gas chromatography-mass spectrometry (GC-MS) with autosampler
- Fourier-transform infrared spectroscopy (FT-IR)
- nuclear magnetic resonance spectroscopy (NMR, 400 MHz, via TA submission/autosampler)
- medium pressure liquid chromatography (MPLC)
- PC Workstations for processing nmr data with MNova software
- software for word processing, chemical structure drawing, searching the chemical literature (Reaxys and SciFinder), and

*You are expected to purify **the major product from each reaction** and to characterize **each purified sample** of each product by the battery of IR and NMR spectroscopy and mass spectrometry and to interpret these data in your lab reports.*

Tutorials Short (30 minute) **tutorial sessions** will be given by the TA's to introduce the use of:

Round 1/week 1:

- liquid/liquid extraction, pouring of solvents, syringe handling, rotary evaporators
- Fourier transform infrared (FTIR) spectroscopy; Laboratory safety features
- PC workstation: NMR data retrieval (and sample prep) and Reaxys/SciFinder connect.
- gas chromatography/mass spectrometry (GC/MS): sample preparation and queue submission
- hazardous waste handling; glass vs. paper waste; reagent weighing (solids vs. liquids; tare weights), closed chemical bottles, spills, etc.

6. thin-layer chromatography (tlc), flash chromatography, tlc staining/visualization

Round 2/week 3:

- medium pressure liquid chromatography (MPLC)
- ozonolysis apparatus and vacuum distillation equipment
- molecular modeling software (MacroModel via Maestro)

Tutorial Schedule: Round 1 Tutorial sign-up sheets (6) will be posted in 491K by the end of Labor Day. Sign-up for each one and note times on your calendar; spread yourself out to a less busy session if your schedule is flexible. You will be able to begin using these techniques immediately in your work. **Plan to start experimental work (Experiment #1) before the end of this, the first week of classes.**

Electronic Mail: Feel free to share useful information by e-mail and/or ask questions of the entire class and/or teaching team via this route. I have e-mailed you all, so you should have everyone's address. You might want to store this as a group list in the address book of your e-mail client.

Safety: **Safety in the lab:** Standard safety practices are an essential part of all laboratory operations. Some of the chemicals used in this course are flammable, irritating or corrosive, or possess toxic characteristics. The chances of accidents in any laboratory are reduced when researchers come prepared for the experiment and if they follow the basic safety rules outlined below. The risk of any given operation escalates significantly as the scale of the procedure increases (more flammable solvent, larger apparatus, etc.). The experiments in Chem 2312H3 have been designed with this reality in mind. Nearly every reaction will be performed on a scale of less than one gram

of limiting reactant and less than 50 mL of organic solvent.

No food or drink is permitted to be in room 491 K (or the adjoining computer/IR room). These should be consumed outside the laboratory (e.g., in the hallway). You may store beverage and food inside closed containers in a backpack inside 491K.

Lab apparel: You may NOT wear shorts or sandals/flip-flops/etc. in the laboratory. Minimization of the amount of exposed bare skin is excellent protection against both a flash fire and a chemical spill or splash.

Lab coat: Although not mandatory, it is recommended that you acquire and wear a laboratory coat during experimental work. This not only protects your clothing but, more importantly, serves as an effective shield toward burns to the skin if there should be a flash fire in the laboratory. Many of the solvents used in organic chemistry experimental work are flammable. It is advisable that the coat be sized so that sleeves do not extend beyond your wrists. Coats are available for purchase in the medical section of the Coffman bookstore ("for Chem 2311" style 415, ca. \$30). Try on the sample coats hanging on the racks to determine your correct size.

Goggles: Again, laboratory **safety goggles**, available for purchase from the stockroom if you do not already own a pair, **must** be always worn in the working area of the laboratory.

Never work alone in **any** laboratory. Students in 2312H will only be permitted to work in the lab during the scheduled lab times and when a TA or instructor is present.

You should take time to **locate** the following **safety features** in the lab: *fire extinguishers, eye wash stations, safety shower, the two exits*, and the first aid kit (by the stockroom window and by the main entrance door to the lab). Hopefully you will never have need for any of these, but in an emergency, you should know exactly where to go if needed.

You should not listen to music while working, not even with headphones. It is prudent to be aware of what other people around you are doing in addition to your own manipulations. Full sensory awareness can often give advanced warning of a potentially hazardous event where your response, whether cognitive or instinctive, can minimize or prevent undesired outcomes.

If you need to receive or make a phone call, please conduct your conversation in the hallway outside the laboratory.

Keep your work area and lab bench neat and uncluttered. Wash and put back into your drawer glassware that is not being used. Discard used thin-layer chromatography (tlc) plates once their comparative information is recorded in your notebook and/or no longer needed.

Cleanup chemical (liquid or solid) and water spills at once. If a spill occurs and you are unfamiliar with the safe cleanup procedure for that chemical, immediately contact a TA or the instructor for guidance. Avoid skin contact with chemicals. If you spill a chemical on your skin, immediately wash the affected area. Wash your hands after coming into direct contact with chemicals. It is always advisable to wash your hands before you leave the lab.

Any accident that results in an injury, no matter how minor, needs to be reported to a TA or to me, the instructor, ASAP.

All chemical and hazardous wastes must be disposed by being placed into proper waste containers. Never place organic chemicals or solvents in the sink drains. If you are unsure of the correct disposal of something that you use or generate in your experiments, please ask. Incorrect disposal of chemicals is both a safety as well as an environmental concern.

Thank you for being attentive to the above rules and guidelines.

Lab Services Coord Kylie Adams and her Chemistry Stockroom staff work out of the teaching stockroom located in the back of the General Chemistry laboratory space in room 249 Smith Hall (adjoining our lab in room 491 Kolthoff Hall and accessible by the back door/exit within 491K). That facility typically closes at 4:10 pm M-F. The TAs will be attentive to stocking the routine supplies (e.g., solvents) we will be needing, but you may occasionally need something from the stockroom (e.g., replacement of a broken glassware item).

Modality transparency:

This course is scheduled as an in-person course. I intend to hold all class sessions in-person except if situational factors arise, such as personal illness of the instructor, when the class may be held synchronously via Zoom or recorded for later viewing.

Legitimate (i.e., Excused) Absences:

While makeup work for legitimate absences is part of University policy, faculty and instructors choose how to accommodate absences based on their course. In this course, excused absences will be handled as follows:

- Students are expected to obtain notes from a classmate of class material missed.
- If you inform me of a legitimate absence in advance, I will attempt to record the class session on Zoom and make it available only to those who have a legitimate absence.
- Please note that I do not intend to record class sessions at the request of individual students.

Illnesses and Face Coverings:

Illness: You should stay at home if you experience significant signs of illness and, especially, if you have a positive COVID-19 test result. If this occurs, please consult with your healthcare provider about an appropriate course of action. I will follow these same protocols and will let you know if the delivery of this course has to be temporarily changed as the result of my own circumstances. Absences related to illness, including COVID-19 symptoms, for yourself or your dependents, are legitimate “excused” absences

Face coverings: I do not plan to wear a mask in the lecture room (although that could change as things evolve here in the Fall), but I fully support your individual choices around masking.

Additional Syllabus Requirements – Policy Statements (UMN)

Instructors must have as part of the syllabus copies of, references to, or statements on the following and are encouraged to discuss elements of the policies particularly applicable to their course (see Appendix - *Recommended Policy Statements for Syllabi*):

1. Grade definitions from the Administrative Policy: *Grading and Transcripts: Twin Cities, Crookston, Morris, Rochester*.
2. Scholastic Dishonesty (see Board of Regents Policy: *Student Conduct Code* and the Administrative Policy: *Teaching and Learning: Instructor and Unit Responsibilities: Twin Cities, Morris, Rochester*).
3. Administrative Policy: *Makeup Work for Legitimate Absences: Twin Cities, Crookston, Morris, Rochester*
4. Board of Regents Policy: *Student Conduct Code*; Administrative Policy: *Teaching and Learning: Student Responsibilities (Twin Cities, Crookston, Morris, Rochester)*
5. Board of Regents Policy: *Sexual Harassment, Sexual Assault, Stalking and Relationship Violence*
6. Board of Regents Policy: *Equity, Diversity, Equal Employment Opportunity, and Affirmative Action*
7. Board of Regents Policy: *Disability Services*
8. Statement about the availability of mental health and stress management services.
9. Board of Regents Policy: *Academic Freedom and Responsibility*

Department of Chemistry Diversity and Inclusion Committee

Collaboration among people of all cultures and backgrounds enhances our experiences and contributes to excellence in teaching, learning, and research. We strive for a climate that celebrates our differences and strengthens our department by embracing and working to increase diversity, equity, and inclusion. For more information about our departmental efforts and upcoming activities: <http://z.umn.edu/ChemDiversity>. For a list of diversity related resources: <http://z.umn.edu/DiversityandInclusionResources>.

Ally Statement

I strive to serve as an effective Ally for students who hold marginalized identities. I am available to listen and support you in a safe and confidential manner. I can help connect you with resources to help address barriers that may interfere with your academic and social success on campus as related to diversity, access, or safety. My goal is to help students be successful and to maintain a safe, accessible, and equitable campus.

University Stances/Positions

Equity, Diversity, Equal Opportunity, and Affirmative Action

We welcome individuals of all ages, backgrounds, beliefs, ethnicities, genders, gender identities, gender expressions, national origins, religious affiliations, sexual orientations, ability – and other visible and nonvisible differences to this course. Instructors, teaching assistants, and peer students are expected to contribute to a respectful, welcoming, and inclusive environment for every other member of the class. This is in agreement with university policy: http://regents.umn.edu/sites/regents.umn.edu/files/policies/Equity_Diversity_EO_AA.pdf

Access and Disability Accommodations

In this course, we support anyone requiring accommodations for access to class activities and materials. Please contact the instructor or the Disability Resource Center <https://diversity.umn.edu/disability/>, which will provide a letter to share with the instructor on how to facilitate an inclusive learning environment.

Sexual Harassment and related topics

In this course, we strive to provide a safe and positive environment for everyone. Please review policy regarding sexual harassment and related topics:

Sexual Harassment, Sexual Assault, Stalking and Relationship Violence

For support and help please contact the Aurora Center: <http://aurora.umn.edu>