# Chemistry 2312H Honors Organic Chemistry Laboratory

Fall 2024, 5 credits

# http://www1.chem.umn.edu/groups/hoye/teaching/Teaching2312HFall2024

**Instructor:** Thomas R. Hoye, 422 Smith Hall, 612-625-1891 (hard to reach there), hoye@umn.edu

**Office Hours:** Thursdays 11:00-noon (334 Smith) (starting Sept. 5<sup>th</sup>) Fridays 3:00-4:00 (334 Smith)

**Teaching Assistants:** Jack Orr 431 Smith 952-412-4871 orrxx168@umn.edu

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Lectures: Tu, Th 8:00 - 8:50 AM, Smith 111 (plus 2-3 ad hoc sessions in the first two weeks)

**Prerequisite:** Chem 2301/2331H (or equivalent or concurrent)

**Laboratory (491 K)** See "LabHoursF24" handout (to be generated soon after our first class meeting).

**Hours:** 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., Macmillan, New York, 2014

(earlier editions would also be fine).

**First Reading** Read chapters 1. safety in the laboratory; 2. green chemistry; 3. laboratory

Assignment: 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 Read chapters 19. liquid chromatography; 20. gas chromatography; 21. Infrared

Assignment: spectroscopy during the second week of class.

Third Reading Assignment: Read chapters 22. <sup>1</sup>H NMR spectroscopy; 24. mass spectrometry during the third week of class.

Fourth Reading Read chapters 8. computational chemistry and 12. boiling points and distillation

**Fourth Reading Assignment:**Read chapters 8. computational chemistry and 12. boiling points and distillation during the fourth week of class.

Supplemental 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 Wade & Simek (Chaps. 12/13) or Carey et al. (Chap. 14). A very useful tutorial (within a piece of ancient, but pleasingly effective, software) 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. 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.

<b>Course Outline:</b>	~Week#	Experiment #
	1-2½	1
	$2\frac{1}{2}-5$	2
	6-8	3 or 4 or 5
	9-11	4 or 5 or 3
	12-14	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:

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

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; lab safety features; Reaxys/SciFinder
- PC workstation: NMR data retrieval (and sample prep)
- 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.
- thin-layer chromatography (tlc), flash chromatography, tlc staining/visualization

# Round 2/week 3:

- 7. 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 all of you, 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 (especially so, solvents), irritating, 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 2312H 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 <u>may</u> 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 textbook section of the Coffman bookstore (style 3138, ~\$30). Try on the <u>sample</u> coats

hanging on the racks to guide your choice of size.

Goggles: Again, laboratory safety goggles, available for purchase from the stockroom (or the Bookstore,

\$13 there) 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 need 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. (more details in WasteHandlingGuidelinesF'24)

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 my occasionally need something from the stockroom (e.g., replacement of a broken glassware item).

#### **Modality**

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

#### Legitimate (i.e., Excused) Absences

While <u>makeup work for legitimate absences</u> 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.
- 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.

# **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: <a href="https://cse.umn.edu/chem/diversity-equity-inclusion">https://cse.umn.edu/chem/diversity-equity-inclusion</a>.

# **Ally Statement**

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.

#### **Mutual Respect**

I will endeavor to treat each of you with the respect that you deserve and expect that you will do the same in your interactions with your fellow students in the class as well as with the teaching staff.

# Additional Syllabus Requirements - Policy Statements (UMN)

The following is a set of UMN Policy resources that are relevant to the U's mission and/or to the courses taught here and that instructors are asked to provide in their course syllabus.

#### **Education & Student Life Policies**

The University's Education & Student life policies are available in the online Policy Library.

#### **Student Conduct Code**

The University seeks an environment that promotes academic achievement and integrity, that is protective of free inquiry, and that serves the educational mission of the University. To support this environment, the University seeks a community that is free from violence, threats, and intimidation; that is respectful of the rights, opportunities, and welfare of students, faculty, staff, and guests of the University; and that does not threaten the physical or mental health or safety of members of the University community.

As a student at the University, you are expected to adhere to Board of Regents Policy: <u>Student Conduct Code</u>. Note that the conduct code specifically addresses disruptive classroom conduct, which means "engaging in behavior that substantially or repeatedly interrupts either the instructor's ability to teach and/or a student's ability to learn." The classroom extends to any setting where a student is engaged in work toward academic credit or satisfaction of program-based requirements or related activities.

#### Use of Personal Electronic Devices in the Classroom

Using personal electronic devices in the classroom setting can hinder instruction and learning, not only for the student using the device but also for other students in the class. To this end, the University establishes the right of each instructor to determine if and how personal electronic devices are allowed to be used in the classroom. For complete information, please review the Administrative Policy: <u>Teaching and Learning: Student Responsibilities</u>.

# **Scholastic Dishonesty**

As students in a university community, you are expected to do your own academic work, and to cite sources of knowledge that you draw on in completing your assignments and other academic work. Failing to do so is scholastic dishonesty, which includes: plagiarism; cheating on assignments or examinations, including the unauthorized use of online learning support and testing platforms; engaging in unauthorized collaboration on academic work, including the posting of student-generated coursework on online learning support and testing platforms not approved for the specific course in question; taking, acquiring, or using course materials without faculty permission, including the posting of faculty-provided course materials on online learning support and testing platforms; submitting false or incomplete records of academic achievement; acting alone or in cooperation with another to falsify records or to obtain dishonestly grades, honors, awards, or professional endorsement; altering, forging, misrepresenting, or misusing a University academic record; or fabricating or falsifying data, research procedures, or data analysis. When it is determined that a student has cheated, the student may be given an "F" or an "N" for the course and may face additional sanctions from the University. For additional information, please see: *Teaching and Learning: Instructor and Unit Responsibilities*.

Additional resources pertaining to academic integrity can be found through the <u>Office for Community Standards</u> and the <u>Center for Educational Innovation</u>.

Beware of websites that advertise themselves as being "tutoring websites." It is not permissible to upload any instructor materials to these sites without their permission or copy material for your own homework assignments from these various sites. When you have additional questions and concerns, please speak with or email your instructor to clarify practices expected for the course. Your instructor can respond to your specific questions regarding what would constitute scholastic dishonesty in the context of a particular class, for example, when and whether collaboration on assignments is permitted, when citing sources is required and what citation method to use, or when and which electronic aids are permitted or prohibited during an exam.

# **Excused Absences and Makeup Work**

Students will not be penalized for absence during the semester due to unavoidable or legitimate circumstances. Such circumstances include verified illness, participation in intercollegiate athletic events, subpoenas, jury duty, military service, bereavement, and religious observances. Such circumstances do not include voting in local, state, or national elections. For complete information, please see Administrative Policy: *Excused Absences and Makeup Work*.

# **Appropriate Student Use of Class Notes and Course Materials**

Taking notes is a means of recording information and more importantly of personally recording, integrating, and connecting information as part of your educational experience. However, broadly disseminating class notes beyond the current classroom community or accepting compensation for taking and distributing classroom notes undermines instructor interests in their intellectual work product while not substantially furthering instructor and student interests in effective learning. Such actions violate shared norms and standards of the academic community. For additional information, please see: Administrative Policy: *Teaching and Learning: Student Responsibilities*.

### **University Grading Scales**

The University has two distinct grading scales: A-F and S-N.

A-F grading scale. The A-F grading scale allows the following grades and corresponding GPA points:

Grade	<b>GPA Poin</b>	ts Definitions for undergraduate credit
A	4.000 Re	epresents achievement that significantly exceeds expectations in the course.
A-	3.667	
$\mathbf{B}$ +	3.333	
В	3.000 Re	epresents achievement that is above the minimum expectations in the course.
B-	2.667	
C+	2.333	
C	2.000 Re	epresents achievement that meets the minimum expectations in the course.
C-	1.667	
D+	1.333	
D	1.000 Re	epresents achievement that partially meets the minimum expectations in the course.
	Cı	redit is earned but it may not fulfill major or program requirements.
F	0.000 Re	epresents failure in the course and no credit is earned.

S-N grading scale. The S-N grading scale allows for the following grades and corresponding GPA points:

S	0.00	Satisfactory (equivalent to a C- or better)
N	0.00	Not Satisfactory

For additional information, please refer to: Administrative Policy: *Grading and Transcripts*.

#### Sexual Harassment, Sexual Assault, Stalking and Relationship Violence

The University prohibits sexual misconduct and encourages anyone experiencing sexual misconduct to access resources for personal support and reporting. If you want to speak confidentially with someone about an experience of sexual misconduct, please contact a confidential resource on your campus. If you want to report sexual misconduct or have questions about the University's policies and procedures related to sexual misconduct, please contact your campus <u>Title IX office or relevant policy contacts</u>.

Instructors are required to share information they learn about possible sexual misconduct with the campus Title IX office that addresses these concerns. This allows a Title IX staff member to reach out to those who have experienced sexual misconduct to provide information about personal support resources and options for investigation. You may talk to instructors about concerns related to sexual misconduct, and they will provide support and keep the information you share private to the extent possible given their University role.

For more information, please see Administrative Policy: <u>Sexual Harassment, Sexual Assault, Stalking and Relationship Violence</u>.

#### Equity, Diversity, Equal Opportunity, and Affirmative Action

The University provides equal access to and opportunity in its programs and facilities, without regard to race, color, creed, religion, national origin, gender, age, marital status, familial status, disability, public assistance status, membership or activity in a local commission created for the purpose of dealing with discrimination, veteran status, sexual orientation, gender identity, or gender expression. For more information, please consult Board of Regents Policy: *Diversity, Equal Opportunity, and Affirmative Action*.

# **Disability Accommodations**

The University of Minnesota is committed to creating learning environments that are inclusive and accessible to all students. If you are experiencing disability-related barriers to learning in your courses, the Disability Resource Center (DRC) is the office that collaborates with students to explore reasonable accommodations, tools, and resources.

- If you are registered with the DRC and have a current accommodation letter please share your letter with me as soon as possible so that we can discuss how your accommodations will be implemented in this course. The sooner I know about your disability access-needs, the more equipped I can be to facilitate accommodations. You may reach out to me or your (access consultant/disability specialist) if you have any questions or concerns about your accommodations.
- If you are **not** registered with the DRC and are experiencing or think you may be experiencing disability related to a mental health, attention, learning, chronic health, sensory, or physical condition, and would like to discuss accommodations and/or resources, please contact the DRC on our campus (e.g., Twin Cities campus 612.626.1333).
- If you have a short-term medical condition, such as a broken arm, I may be able to assist in minimizing class-room barriers. In situations where additional assistance is needed, you should contact the DRC as noted above.

# **Campus DRC information:**

• Twin Cities - <a href="https://disability.umn.edu/">https://disability.umn.edu/</a> drc@umn.edu)

# **Mental Health and Stress Management**

As a student you may experience a range of issues that can cause barriers to learning, such as strained relationships, increased anxiety, alcohol/drug problems, feeling down, difficulty concentrating and/or lack of motivation. These mental health concerns or stressful events may lead to diminished academic performance and may reduce your ability to participate in daily activities. University services are available to assist you. You can learn more about the broad range of confidential mental health services available on campus via the <a href="Student Mental Health Website">Student Mental Health Website</a>. As an instructor/University community member, we care about the wellbeing of students. If health, safety, or mental health concerns are conveyed, we may consult with campus support offices to provide support and resources to a student.

#### Academic Freedom and Responsibility: for courses that do not involve students in research

Academic freedom is a cornerstone of the University. Within the scope and content of the course as defined by the instructor, it includes the freedom to discuss relevant matters in the classroom. Along with this freedom comes responsibility. Students are encouraged to develop the capacity for critical judgment and to engage in a sustained and independent search for truth. Students are free to take reasoned exception to the views offered in any course of study and to reserve judgment about matters of opinion, but they are responsible for learning the content of any course of study for which they are enrolled.\*

Reports of concerns about academic freedom are taken seriously, and there are individuals and offices available for help. Contact the instructor, the Department Head, your adviser, the Associate Dean of the College, or the Vice Provost for Faculty and Academic Affairs in the Office of the Provost.

<sup>\*</sup> Language adapted from the American Association of University Professors "Joint Statement on Rights and Freedoms of Students."