Chemistry 2312H
Honors Organic Chemistry Laboratory

Fall 2019, 5 credits

http://www1.chem.umn.edu/groups/hoye/teaching/Teaching2312HFall2019

Instructor: **Thomas R. Hoye**, 419 Smith Hall, 612-625-1891, hoye@umn.edu

Office Hours: Tuesdays 1:30-2:30 in 122 Smith [**not** 11/19]
Fridays 2:30-3:30 in 193 Kolthoff [**not** 9/6 (122 Smith instead), 9/29, 11/15, 11/29]

Teaching Assistants:

Qian Xu	413 Smith	651-343-7684	xu000243@umn.edu
Casey Ritts	413 Smith	952-818-4948	ritts009@umn.edu
Kate Rynders		612-323-4056	rynde015@umn.edu

Lectures: Tu, Th 8:00 - 8:50 AM, 111 Smith Hall (plus several *ad hoc* sessions in first ca. 10 days)

Prerequisite: Chem 2301/2331 (or equivalent or concurrent) and permission of instructor

Laboratory (491 K) Hours: Open 33-36 hours each week. A teaching assistant will **and must** be present at all times. Finalized lab open times will be set and provided by the end of Tuesday, Sept. 3, 2019.

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. ^{1}H 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 want to augment your reading and learning of the common spectroscopic techniques (MS, IR, and NMR) by (re)reading the relevant chapters in the course textbook by L. G. Wade (Chaps 12/13) or J. G. Smith (Chaps 13/14) or J. Karty (Chaps 15/16). A very useful tutorial on infrared spectroscopy ("IR Tutor") can be found on the PC's in the lab computer room.

Required Supplies: Laboratory **safety goggles**, available in the stockroom, **must** be worn in the laboratory at all times. A **permanent** (not loose-leaf) **notebook** of your choosing for record keeping.

Grades: A written report will be required for each of five experiments (see separate handouts I will provide for style guidelines, due dates, etc.).

80% of your course grade will be based on these reports.

15% of your course grade will be based upon teaching staff evaluation of the development and growth of your laboratory technique and prowess over the course of the semester.

5% of your course grade will be given in a "Final" Exam (50 minute) administered in class on Tuesday, December 10, 2019.

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, Saponification, and EDCI Amide Coupling with α-Methylbenzylamine
Exp. 4. Catalysis: Palladium(0) Alkyne/Arene Coupling and 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, 300 MHz, via TA submission/autosampler)
d. medium pressure liquid chromatography (MPLC)
e. PC Workstations for processing nmr data
f. software for word processing, chemical structure drawing, searching the chemical literature (Reaxys and SciFinder), NMR data processing, and internet and e-mail access

*You are expected to purify **each and every** reaction product and to characterize **each and every** 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 present the use of:

Round 1/week 1:

1. liquid/liquid extraction, pouring of solvents, syringe handling, rotary evaporators
2. Fourier transform infrared (FTIR) spectroscopy; Laboratory safety features
3. PC workstation: NMR data retrieval (and sample prep) and Reaxys/SciFinder connect.
4. gas chromatography/mass spectrometry (GC/MS): sample preparation and queue submission
5. thin-layer chromatography (tlc), flash chromatography, tlc staining/visualization
6. hazardous waste handling; reagent weighing (solids *vs*. liquids), closed bottles, glass *vs*. paper waste

Round 2/week 3:

7. medium pressure liquid chromatography (MPLC)
8. ozonolysis apparatus and vacuum distillation equipment
9. molecular modeling software (MacroModel via Maestro)

Sign-up sheets:

Sign-up sheets for Round 1 tutorials have been posted in the lab (491 Kolthoff). You should sign-up and attend each of the Round 1 tutorials by the end of Friday, September 6 at the latest. 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 will e-mail you a complete list of everyone's address. You might want to store this 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 2312 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 room). These should be consumed (e.g., in the hallway) outside the laboratory.

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 deterrent toward burns to the skin if there should be a solvent fire in the laboratory. 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. $25). 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 worn in the working area of the laboratory at all times.

Never work in alone in **any** laboratory. Students in 2312H will only be permitted to work in the lab during the scheduled lab times 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/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 tlc plates once their comparative information is recorded and/or no longer needed.

Cleanup chemical 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 assistance. Avoid skin contact with chemicals. If you spill a chemical on your skin, immediately wash the affected area. Wash your hands after coming into contact with chemicals or chemical containers. 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 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 team work out of the teaching stockroom located in the back of the general chemistry laboratory space in room 249 Smith Hall (and adjoining our lab in room 491 Kolthoff Hall). That facility generally closes at 4:10 pm M-F.

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 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