SUMMER II 2020, Chemistry 3220: Organic Chemistry Laboratory II

COURSE & INSTRUCTOR DETAILS:

LAB LECTURE: 3220.001 ONLINE

LABS - ALL SECTIONS (301, 302, 303, 304, 305, 306) ONLINE

INSTRUCTOR: Dr. Sreekar Marpu

OFFICE: Chemistry Building, Room 371
E-MAIL: sreekarbabu.marpu@unt.edu

OFFICE & LECTURE HOURS: Monday: 9.30 am to 10.50 am (zoom meeting) or by email.

PHONE: 940-565-4850

LABS:

Section 301: TWR 8.00 am to 10.50 am - Farvid Majid - seyedmajidfarvid@my.unt.edu

Section 302: TWR 8.00 am to 10.50 am - Roshani Asuramana - roshaniariyagnana@my.unt.edu

Section 303: TWR 1.30 pm to 04.20 pm - Lu Zhou - ZhouLu@my.unt.edu

Section 304: TWR 1.30 pm to 04.20 pm - Matt Tiemann - MatthewTiemann@my.unt.edu Section 305: TWR 4.30 pm to 07.20 pm - Kazemi Shahrzad - ShahrzadKazemi@my.unt.edu

Section 306: TWR 4.30 pm to 07.20 pm - Rajitha Perera - rajithaperera@my.unt.edu

The TA's will schedule the online Zoom meeting for the labs. During these meetings, TA's will explain and go over the video demonstrations useful for lab reports. Attendance at these meetings is "MANDATORY".

The instructor will go over the PowerPoint presentations of the experiment during the office hours. Attendance at these meetings (Monday's 9.30 to 10.50 am) is not mandatory but highly recommended!!!

UNT REQUIREMENTS:

PLEASE CHECK THE COURSE REQUIREMENTS at https://registrar.unt.edu/ or reach out to the chem department (Heather Vidaurri or Miriam Freeman) for additional details. MUST HAVE HAD OR BE CONCURRENTLY REGISTERED IN CHEM 2380. MUST ALSO BE ENROLLED IN CHEM 3220 LAB. MUST HAVE ALREADY COMPLETED CHEM 3210.

A. LEARNING OBJECTIVES:

Objectives of this course include:

Providing a foundation of organic chemistry skills and techniques for students considering advanced studies in chemistry or medical fields or other life sciences.

After completing the course, the students are expected to:

Correlate and gain a deeper experimental (hand-on) understanding of many theoretical concepts learned during the CHEM 2380 course.

Able to perform at least five different types of organic reactions in the lab and understand the underlying mechanism of these reactions.

Perform medium to complex organic synthesis reactions following a written experimental procedure.

Understand the methodology and rules of writing a good and scientific lab report, able to write the lab report on their own.

Understand guidelines for working safely in a chemical/organic lab.

B. TEXTBOOK(S):

Recommended Reading: "Macroscale and Microscale Organic Experiments," 6th or 7th Edition,

Kenneth L. Williamson and Katherine M. Masters

Any format of the textbook is acceptable for the course.

All the information required for performing the experiments in the lab will be provided by the instructor in the form of handouts and lab lectures. Any missing information can be obtained from openly available online resources.

No special lab notebooks are needed, students can use regular plain white paper notebooks for writing the reports. Online Reports needs to be legible and clear for TA's to grade – Reports that are not legible will be returned to the students

Word or PDF documents are preferred formats for online submission. Canvas will accept doc, docx. TA's will provide more information regarding format and uploading of the reports.

"Class recordings are reserved for use only by students in this class for educational purposes. The recordings should not be shared outside the class in any form. Failing to follow this restriction is a violation of the UNT Code of Student Conduct and could lead to disciplinary action."

C. LAB LECTURES DESCRIPTION:

	TOPIC	DESCRIPTION
1	Synthesis of Cyclohexanone from	The lab will allow for the synthesis of cyclohexanone from cyclohexanol. Conversion of
	cyclohexanol	secondary alcohol into ketone using a household oxidizing agent will be performed in the
	Tests: 2,4-DNP test and	lab. The lab will facilitate the identification of organic compounds by using simple-
	oxidation of alcohols.	common organic reagents.

Grignard Synthesis	The significance of the Grignard reagent and Grignard reaction will be demonstrated.
	Starting from phenyl magnesium bromide, the Grignard reagent will be in situ
	synthesized in step one. In step two the as-synthesized Grignard reagent will be utilized
	to synthesize an aromatic compound (benzoic acid).
Diels Alder Reaction	The objective is to synthesize a six-membered ring starting from a combination of a
	diene and dienophile. The lab lecture will emphasize on the mechanism of formation of
	the six-membered rings and the stereochemistry associated with the reaction.
Electrophilic Aromatic Substitution	Synthesis of an electrophilic substitution product is taught in the lab. Mechanism and
	chemistry associated with electrophilic substitution reactions are emphasized in the lab
	lecture. The students will perform nitration of methyl benzoate in the lab.
Friedel-Crafts Acylation of Ferrocene	Synthesis of acetylferrocene from ferrocene, followed by purification of acetylferrocene.
	The labs will demonstrate the utility of the HPLC technique for the purification of organic
	compounds from a mixture during an organic reaction.
Esterification and Hydrolysis	The students will have an opportunity to synthesize soap starting from a fatty acid. The
(preparation of soap)	as-synthesized soap will be tested for its hardness.
Dyes and Dyeing: The coupling of	The lab will provide an opportunity for the students to learn the synthesis of a pH
diazonium compounds	sensitive dye/indicator by following a multi-step organic synthesis procedure. Methyl
	Orange (MO) will be synthesized starting from sulfanilic acid. The MO will be used for
	dyeing the fabric.
Aldol Condensation.	An Aldol product (Dibenzalacetone) will be synthesized starting from Benzaldehyde. The
	reaction will explain the significance of molar ratios of starting materials in the organic
	synthesis reactions.
Synthesis of Acetylsalicylic acid	A very famous pain killer, Aspirin (acetylsalicylic acid) will be synthesized starting from
(Aspirin)	salicylic acid.
*Identification of Unknown	The handout contains a molecular formula, Infrared Spectrum, and Nuclear Magnetic
	Spectrum of an unknown organic molecule. The students are required to identify the
	molecule and draw the structure from the given information.
	Diels Alder Reaction Electrophilic Aromatic Substitution Friedel-Crafts Acylation of Ferrocene Esterification and Hydrolysis (preparation of soap) Dyes and Dyeing: The coupling of diazonium compounds Aldol Condensation. Synthesis of Acetylsalicylic acid (Aspirin)

^{*}Unknown identification can also involve experimental lab (instrumentation) in addition to spectral identification. The details will be posted within a few weeks of the start of the labs.

NOTE: Any minor changes in the syllabus will be informed immediately. Check announcements on Canvas

SAFETY

Even for online labs, understanding safety guidelines for working in an organic lab is very important.

D. ADA STATEMENT

DISABILITY: The University of North Texas makes reasonable academic accommodations for students with disabilities. **Students seeking accommodation must first register with the Office of Disability Accommodation (ODA) to verify their eligibility.** If a disability is verified, the ODA will provide an accommodation letter that is delivered to the instructor on record directly. Students are welcome to discuss with the faculty regarding specific needs in a course. You may request accommodations at any time, however, **ODA notices of accommodation should be provided as early as possible in the semester** to avoid any delay in implementation. For additional information see the Office of Disability Accommodation website at http://www.unt.edu/oda. You may also contact them by phone at 940.565.4323. Please talk to the instructor or the TA for any further questions.

E. ATTENDANCE POLICY:

All course materials are available on Canvas for the students to review and use them appropriately for generating the lab reports. Online (zoom) meetings with the instructor and with the TA's are not mandatory but are highly encouraged to get the best of the course material and also to get the required help with the lab reports. Students should submit all the REPORT(S) & QUIZ(S) ON/BEFORE THE DUE DATE. Be prepared to lose points for LATE SUBMISSIONS!!! Unless arranged with the TA in advance. TA-Zoom meetings are mandatory! Please arrange with your TA for absences.

Lab-Lectures: Monday 9.30 am to 10.50 am; not mandatory but highly recommended.

The lab-lectures are designed to help students understand the procedural details of the scheduled experiments and also the theoretical principles underlying the experimental design.

Lab Meetings with TA's: Mandatory, please contact the TA immediately for an excused absence(s).

There are no makeup quizzes. A missed lab report will result in a zero grade. A partial grade can be earned for late submission of the lab report due to an emergency or extraordinary situation. Reach out to your TA and prearrange for a late submission. Students are required to submit all the reports and quizzes on/before the due date. Be prepared to lose points for late submission, unless arranged with the TA. Be prepared to lose points for not attending the meeting with the TA.

F. BEHAVIOR POLICY/RULES OF ENGAGEMENT

Treat your instructor and TA with respect in email or any other communication. Use clear and concise language.

G. GRADING:

Teaching Assistants will enter grades on Canvas under the course page for labs. The TA's are expected to update the grades on the Canvas for every 2 weeks. It is the responsibility of the student to regularly check for consistency of grades and report to the TA for any inconsistencies. Students are advised to report to the instructor regarding any delays in the grading of their lab reports. Please wait for at least 2 weeks before complaining to the instructor or talk to your TA first.

Your grade will be determined entirely by your performance in the laboratory and quizzes. This semester 10 laboratory experiments and three quizzes. The 270 total points in the laboratory are broken down as follows:

9 Experiments + 1 Unknown	Possible Points = 10 x 20 = 200	
2 Quizzes (one safety, two-take home)	Points Possible = 20 x 2 = 40	
TA Assessment of Safe Laboratory Practices	Points Possible = 10	
Total:		= 250
90 – 100 % of the total points	Grade = A	
80 – 89 % of the total points	Grade = B	
70 – 79 % of the total points	Grade = C	
60 – 69 % of the total points	Grade = D	
Below 60 %	Grade = F	

The additional bonus points for recitation, labs, and quizzes are at the instructor's discretion.

QUIZZES: There will be two quizzes. Quiz 1 is a safety quiz that deals with aspects of general lab safety and the location of various safety items in the organic laboratory. Quiz 3 will be related to experimental modules and will be available on Canvas following the schedule.

H. LAB REPORTS: MANDATORY! DETAILS FOR WRITING A LAB REPORT ARE ENCLOSED IN A SEPARATE DOCUMENT – Course grade depends on lab reports. Students will write lab reports watching the online video demos of the experiments. Handouts can be used only for reference purposes. Do not copy the handouts, the experimental procedure for the report must match the video demo. The students are required to answer the extra/additional questions in some of these demo videos. These questions do not carry extra points, they are part of the report. Be prepared to lose points for not answering these questions. A simple and straightforward answer is required, please discuss it with the TA.

I. TENTATIVE SCHEDULE: PLEASE FOLLOW THE DUE DATES. The exact date/time for the meeting(s) with the TA's will be announced by your TA. Please look for announcements on Canvas.

5 week 2: First class – July 6th; Census – July 9th; Final exam – August 7th; Grades due – August 9th

EXPERIMENT (Page numbers on both editions of the organic manual)	Lab Lecture – Web- based (online)	Experiments- Web- based (online)	Due Dates for Reports/Quiz(s)
EXP 0: Lab Safety Experiment (online simulation presentation)-	July 6 th (Monday)	Week of July 6 th	July 7 th – Quiz#1 due;
EXP 1: Cyclohexanone from cyclohexanol			June 13 th – Lab
Williamson, Ch. 22			Report 1 Due
22.3: Cyclohexanone			
Tests: 2,4-DNP test and oxidation of alcohols			
p.361-363 / p.363-365.			
EXP 2: Grignard Synthesis	July 13 th (Monday)	Week of July 13 th	July 20 th – Lab
Williamson, Ch. 38		_	Reports for Exp 2, 3,
38.1: Phenylmagnesium bromide			and 4 due.
38.3: Benzoic acid			
p.495-497 / p. 498 -500 and p. 500 / p. 503			
EXP 3: Diels Alder Reaction			
Williamson, Ch. 48			
48.2: Reaction with Maleic anhydride			
p.623-624 /p.625-626			
EXP 4: Electrophilic Aromatic Substitution			
Williamson, Ch. 28			
28.1: Nitration of Methyl Benzoate			
p.402-403 / p.405-406			

EXP 5: Friedel-Crafts acylation of ferrocene	July 20th (Monday)	Week of July 20th	July 27 th – Lab
Williamson, Ch. 32	No quiz this week		Reports for Exp 5, 6,
32.1: Acetylferrocene			and 7 due.
Column chromatography			
p. 440 -441 /p. 443 - 444			
EXP 6: Esterification and Hydrolysis (preparation of soap)			
Williamson, Ch. 40.5			
The preparation of soap			
p. 525 – 526 /p. 527 - 528			
EXP 7: Dyes and Dyeing, the coupling of diazonium compounds			
Williamson, Ch. 46			
46.1: Diazotization of sulfanilic acid p. 592/ p. 594			
46.4: Methyl Orange p. 596/ p. 598			
46 Direct dyes, with methyl orange p. 599/ p. 601			
EXP 8: Aldol Condensation	July 27 th (Monday)	Week of July 27 th	Quiz #2 due on July
Williamson, Ch. 37	Quiz #2 will be		30 th
37.1: Synthesis of dibenzalacetone.	accessible on Canvas		August 3 rd – Lab
p.485 -486 / p.488 -489			Reports 8, 9 and
EXP 9: Acetylsalicylic acid (Aspirin)			Unknown
Williamson, Ch. 41			Assignment due.
41.1: Synthesis of Acetylsalicylic acid (Aspirin)			
p. 531-532 / p. 533-534.			August 7 th - all grades
			returned to the
EXP 10: Unknown Handout			instructor.

J. EMERGENCY NOTIFICATION & PROCEDURES

UNT uses a system called Eagle Alert to quickly notify you with critical information in the event of an emergency (i.e., severe weather, campus closing, and health and public safety emergencies like chemical spills, fires, or violence). The system sends voice messages (and text messages upon permission) to the phones of all active faculty staff and students. Please make certain to update your phone numbers at

http://www.my.unt.edu. Some helpful emergency preparedness actions include: 1) know the evacuation routes and severe weather shelter areas in the buildings where your classes are held, 2) determine how you will contact family and friends if phones are temporarily unavailable, and 3) identify where you will go if you need to evacuate the Denton area suddenly. In the event of a university closure, labs scheduled during that time are excused absences in addition to the one dropped lab.

K. ACADEMIC INTEGRITY EXPECTATIONS AND CONSEQUENCES (UNT POLICY 06.003)

UNT promotes the integrity of learning processed and embraces the core values of trust and honesty. Academic integrity is based on educational principles and procedures that protect the rights of all participants in the educational process and validate the legitimacy of degrees awarded by the university. In the investigation and resolution of allegations of student academic dishonesty, the university's actions are intended to be corrective, educationally sound, fundamentally fair, and based on reliable evidence.

Please see the document for more details. https://policy.unt.edu/sites/default/files/06.003.pdf.

Reach out to your TA first and resolve the grade disputes. If the dispute is unresolved with your TA, make an appointment to set up the meeting. Give me a week to discuss with your TA, review the details, and to understand the quality of the work you turned in for resolving the grade dispute. Cheating is not tolerated and will result in a grade of "F" for the course. Plagiarism is not tolerated and will result in an "F" grade for the course. Both types of behavior will be reported in accordance with UNT policies regarding academic integrity. Identification of academic dishonesty in this class can result in penalties including additional work, a failing grade for the assignment or class, a grade being reduced or changed, and a referral to the Dean of Students. The APA publication manual and material on the UNT Center for Student Rights and Responsibilities webpage (www.unt.edu/csrr) can help you understand and avoid plagiarism. Please communicate in advance with your TA's if you have any questions or concerns regarding the lab reports to avoid plagiarism issues.

Turnaround Time and Late Work

Follow the due dates outlined in the syllabus (see the table above for actual deadlines for lab reports and quizzes). The lab reports are submitted online. The graded lab reports will be turned back to the students within one week after submission. When this is not possible, the TA's will send an announcement to the class.

Late reports will not be accepted unless the student encounters an emergency or extreme situations. All work turned in after the deadline will receive a grade of zero unless the student has a <u>university-excused absence</u> and provides documentation with 72 hours of the missed

deadline. Do not expect any partial credit for the late reports. Treat your instructor, TA, and classmates with respect in email or any other communication. Use clear and concise language. Profanity or use of "f" word in the lab reports is not acceptable. Will result in "ZERO"

L. STUDENT PERCEPTION OF TEACHING (SPOT)

Student feedback is important and an essential part of participation in this course. The Student Perception of Teaching (SPOT) is a requirement for all organized classes at UNT. This short survey will be made available at the end of the semester to provide you with an opportunity to evaluate how this course is taught.

Extra credit: TA may give up to 5 points extra credit for completing the SPOT online evaluation form.

M. SUCCEED AT UNT

UNT endeavors to offer you a high-quality education and to provide a supportive environment to help you learn and grow. And, as a faculty member, I am committed to helping you be successful as a student. Here's how to succeed at UNT: **Show up. Find Support. Get advised. Be prepared. Get involved. Stay focused.** To learn more about campus resources and information on how you can achieve success, go to http://success.unt.edu/

N. EXPERIMENTAL VIDEO LINKS

EXP 1: Cyclohexanone from cyclohexanol	https://youtu.be/_KUTswDIXGo
EXP 2: Grignard Synthesis	https://youtu.be/PcF8OjwfrEQ
EXP 3: Diels Alder Reaction (Part 1 and 2)	https://www.youtube.com/watch?v=qJv0nU1YqTk&t https://www.youtube.com/watch?v=lwy0ZogOpQM
EXP 4: Electrophilic Aromatic Substitution (Browning and Majd)	https://youtu.be/Fl2zbceRJMw
EXP 5: Friedel-Crafts acylation of ferrocene	Part 1: https://youtu.be/Ea57WXpRZUU Part 2: https://youtu.be/Ea57WXpRZUU

EXP 6: Esterification and Hydrolysis (preparation of soap)	https://www.youtube.com/watch?v=N37XM2OU7go&feature=youtu.be
EXP 7: Dyes and Dyeing, the coupling of diazonium compounds	https://youtu.be/_NP_OUfpZOE
EXP 8: Aldol Condensation	https://www.youtube.com/watch?v=pCNddIKpAQ4&feature=youtu.be
EXP 9: Acetylsalicylic acid (Aspirin)	https://youtu.be/G8xMPhfKKTo
EXP 10: Unknown Handout	https://www.youtube.com/watch?v=Hypk3bgUlJw&feature =youtu.be

 $\label{thm:missing} \mbox{Missing video links will be updated soon before the experiment day.}$