

## SPRING 2020, Chemistry 3220: Organic Chemistry Laboratory II

### COURSE & INSTRUCTOR DETAILS:

#### LAB LECTURE:

3220.001	Monday	1.00 – 1:50 pm. (SAGE 116)
3220.002	Tuesday	12.30 – 1:20 pm. (CHEM 109)
3220.003	Wednesday	3.00 – 3:50 pm. (GAB 104)

**INSTRUCTOR:** Dr. Sreekar Marpu

**OFFICE:** Chemistry Building, Room 371

**E-MAIL:** sreekarbabu.marpu@unt.edu

**OFFICE HOURS:** Monday: 10.00 am to 12.30 pm; Tuesday: 10.00 am to 11.00 am or by appointment.

**PHONE:** 940-565-4850

#### UNT REQUIREMENTS:

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 apply some of the theoretical concepts learned during the CHEM 2380 course.

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

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

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

Realize the significance of working in groups.

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

## B. TEXTBOOK(S):

**Recommended Reading:** "Macroscale and Microscale Organic Experiments," 6<sup>th</sup> or 7<sup>th</sup> 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.*

## C. LAB LECTURES DESCRIPTION:

	TOPIC	DESCRIPTION
1	<b>Synthesis of Cyclohexanone from cyclohexanol</b> Tests: 2,4-DNP test and oxidation of alcohols.	The lab will allow for the synthesis of cyclohexanone from cyclohexanol. Conversion of secondary alcohol into ketone using a household oxidizing agent will be performed in the lab. The lab will facilitate the identification of organic compounds by using simple-common organic reagents.
2	<b>Grignard Synthesis</b>	The significance of 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).
3	<b>Diels Alder Reaction</b>	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.
4	<b>Electrophilic Aromatic Substitution</b>	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.

5	<b>Friedel-Crafts Acylation of Ferrocene</b>	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.
6	<b>Esterification and Hydrolysis (preparation of soap)</b>	The students will have an opportunity to synthesize soap starting from a fatty acid. The as-synthesized soap will be tested for its hardness.
7	<b>Dyes and Dyeing: The coupling of diazonium compounds</b>	The lab will provide an opportunity for the students to learn the synthesis of a pH 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.
8	<b>Aldol Condensation.</b>	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.
9	<b>Synthesis of Acetylsalicylic acid (Aspirin)</b>	A very famous pain killer, Aspirin (acetylsalicylic acid) will be synthesized starting from salicylic acid.
10*	<b>*Identification of Unknown</b>	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.

\*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**

**NOTE: The number of experiments is not affected due to COVID 19.**

#### D. SAFETY

Texas State Law and common sense require eye protection in the form of goggles for all persons in academic chemical laboratories. Along with eye protection, all other laboratory safety guidelines that are listed in the item "R"- "**New Safety Rules Fall 2019**" will be strictly enforced. TA's and Laboratory Supervisors have the right to ask the students to leave the lab with a "0" grade for disobeying or not following any of these rules or guidelines. **PERSISTENT OFFENDERS WILL BE DROPPED WITH "WF". Please see the list of guidelines at the end of this document.** The instructor will discuss these new rules during the first recitation/lecture.

**NOTE: Students will have to sign the “New Safety Rules Fall 2019” form before they begin their labs. Please do not hesitate to ask the lab TA or the instructor any questions related to safety and good lab practices.**

#### **E. 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. Note that students must obtain a new letter of accommodation for every semester and must meet with each faculty member before implementation in each class. 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.

#### **F. ATTENDANCE POLICY:**

##### **Lab-Lectures (also called lab recitation) and Labs: Required!**

The lab-lectures are designed to help you understand not only the procedural details of the scheduled experiments but also the theoretical principles underlying the experimental design. **You are required to attend all scheduled lab-lectures.** It is your responsibility to contact the instructor for your absence(s).

**One excused absence is allowed without penalty for both labs and lectures. Please contact the instructor and the TA immediately for more than one excused absence(s).**

***There are no makeup labs or makeup quizzes.*** If you miss a lab or a lab lecture due to the acceptable reason (listed below), it will be excused. Otherwise, a missed laboratory will result in a zero grade. A partial grade can be earned for an unexcused absence, depends on TA’s discretion! upon submission of the lab report for the absent lab.

##### **Acceptable reasons for missing an experiment or lab lectures include:**

1. Active military service, including travel for that purpose
2. Illness or other extenuating circumstances- please submit the necessary documentation as requested by the TA.
3. Religious holy day – please reach out and inform the instructor and Lab TA in advance
4. Participating in an official University function

5. Pregnancy and parenting under Title IX
6. When the University of officially closed by the President

Please check the [https://policy.unt.edu/sites/default/files/06.049\\_CourseSyllabiRequirements\\_2017\\_0.pdf](https://policy.unt.edu/sites/default/files/06.049_CourseSyllabiRequirements_2017_0.pdf) for more details.

***Turn in the documentation for the missed laboratory to the TA or the instructor as soon as possible (within a week of the missed lab).***

For individuals with a single excused absence, the total possible points in the laboratory will be adjusted appropriately to reflect the one excused absence. For students with more than one excused absence, please discuss this with your TA.

**Shifting lab sections or Make-up Labs:** Only 20 students are allowed in each laboratory, students are not recommended to switch lab sections to cover for missing labs. Only under extreme situations, switching lab sections is allowed with permission from the current TA and the TA of the new lab section. **There are no makeup labs!!!**

## G. BEHAVIOR POLICY

- Students cannot come to the lab more than **10 minutes late**, students coming late more than once will be asked to leave with “0”.
- You are not allowed to work alone (in the absence of TA) in the lab.
- Disruptive students will be asked to leave and will receive a 0 for that week’s lab
- Disruptive behaviors include:
  - Cell phone use (games..)
  - “Horseplay” or running in the lab
  - Not following TA instructions
- Please check the “Safety Rules Agreement” and “Safety Tool Kit” documents for additional details on safety and behavior in the lab.

**Be punctual:** If you arrive late, you will miss the instructions given by your TA at the beginning of the lab period, which may lead to unnecessary confusion as well as unsafe work. Reagents will only be available for the indicated week. If you must miss a lab, contact your TA as soon as possible. Reports for missed labs will not be accepted - your TA will explain how you might earn partial credit for completing pre-lab component and post-lab questions for the missed experiment.

The students will receive a “0” if late to the lab by more than 15 minutes. Can turn on the prelab for partial credit during the first incident. Students showing up late for more than one occasion will receive a “0”.

Under special situation cases, TA’s can allow students with partial credit. “TA’s discretion”.

## H. 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:

<b>9 Experiments + 1 Unknown</b>	<b>Possible Points = 10 x 20 = 200</b>
<b>3 Quizzes (one safety, two-take home)</b>	<b>Points Possible = 20 x 3 = 60</b>
<b>TA Assessment of Safe Laboratory Practices</b>	<b>Points Possible = 10</b>
<b>Total:</b>	<b>= 270</b>
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

### **COVID 19 Effect – Students are given the option to select letter grade or Pass/No Pass**

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

**QUIZZES:** There will be three 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. You may not begin your experimental work for the course unless you have satisfactorily completed the safety quiz. Quiz 2 and 3 will be handed over to the students in the lab or by the instructor during the lab lecture.

- I. **LAB REPORTS: MANDATORY!** DETAILS FOR WRITING A LAB REPORT ARE ENCLOSED IN A SEPARATE DOCUMENT – “Spring\_2020\_CHEM Organic Labs\_Report\_SM 1.1”

**COVID 19 Effect** – The students write their lab reports watching the online video demos of the experiments. Handouts contained brief procedure vs detailed procedure.

**J. TENTATIVE SCHEDULE FOR LAB LECTURES AND LABORATORY EXPERIMENTS**

<i>EXPERIMENT (Page numbers on both editions of the manual)</i>	<i>Lab Lecture –M, T, W</i>	<i>Experiment Dates for All Sections</i>
<b>Week of Jan 13<sup>th</sup>, No Lab Lecture, No Labs</b>		
<p><b>*Check-In, Safety</b>  <i>Passing a safety quiz with 70% points is mandatory before starting any experiments in the lab. Any additional information required for completing the safety quiz will be provided by the TA in the labs.</i></p>	<p><b>Week of Jan 20<sup>th</sup></b>  <b>(section 001 will not meet)</b>  <i>Sections 002 and 003 will receive a safety quiz during the lab lecture. 001 section should reach out to TA's for safety quiz or obtain quiz during the first lab (check-in lab)</i></p>	<p>Week of Jan 27<sup>th</sup>  <i>Quiz #1 (safety quiz)- Return to TA on the same day during the lab.</i></p>
<p><b>EXP 1: Cyclohexanone from cyclohexanol</b>            Williamson, Ch. 22            22.3: Cyclohexanone  <b>Tests:</b> 2,4-DNP test and oxidation of alcohols  <a href="#">p.361-363 / p.363-365.</a></p>	<p><b>Week of Jan 27<sup>th</sup></b></p>	<p>Week of Feb 3<sup>rd</sup></p>
<p><b>EXP 2: Grignard Synthesis</b>            Williamson, Ch. 38            38.1: Phenylmagnesium bromide            38.3: Benzoic acid  <a href="#">p.495-497 / p. 498 -500 and p. 500 / p. 503</a></p>	<p><b>Week of Feb 3<sup>rd</sup></b></p>	<p>Week of Feb 10<sup>th</sup></p>

<b>EXP 3: Diels Alder Reaction</b> Williamson, Ch. 48 48.2: Reaction with Maleic anhydride <a href="#">p.623-624 /p.625-626</a>	<b>Week of Feb 10th</b>	Week of Feb 17 <sup>th</sup>
<b>EXP 4: Electrophilic Aromatic Substitution</b> Williamson, Ch. 28 28.1: Nitration of Methyl Benzoate <a href="#">p.402-403 / p.405-406</a>	<b>Week of Feb 17th</b>	Week of Feb 24 <sup>th</sup>
<b>EXP 5: Friedel-Crafts acylation of ferrocene</b> Williamson, Ch. 32 32.1: Acetylferrocene Column chromatography <a href="#">p. 440 -441 /p. 443 - 444</a>	<b>Week of Feb 24<sup>th</sup></b> <i>Quiz #2, will be given during the lecture</i>	Week of March 2 <sup>nd</sup> .
<b>EXP 6: Esterification and Hydrolysis (preparation of soap)</b> Williamson, Ch. 40.5 The preparation of soap <a href="#">p. 525 – 526 /p. 527 - 528</a>	<b>Week of Mar 2nd</b>	Week of March 16 <sup>th</sup> <i>Quiz #2, DUE: Hand over to your TA in the lab.</i> Late returns without prior permission will be penalized.
<b>UNT SPRING BREAK – WEEK OF MARCH 9<sup>th</sup> – NO LECTURE AND LABS</b> <b>COVID 19 Effect – Week of March 16<sup>th</sup> UNT CLOSED for In-person classes and labs – Resulted in one week delay.</b>		
<b>EXP 7: Dyes and Dyeing, the coupling of diazonium compounds</b> Williamson, Ch. 46 46.1: Diazotization of sulfanilic acid <a href="#">p. 592/</a> <a href="#">p. 594</a> 46.4: Methyl Orange <a href="#">p. 596/</a> <a href="#">p. 598</a> 46 Direct dyes, with methyl orange <a href="#">p. 599/</a> <a href="#">p. 601</a>	<b>Week of March 23<sup>rd</sup> – Online Lecture</b>	Week of March 30 <sup>th</sup> - <b>Online Lab</b>
<b>EXP 8: Aldol Condensation</b> Williamson, Ch. 37	<b>Week of March 30<sup>th</sup> - Online Lecture</b>	Week of April 6 <sup>th</sup> - <b>Online Lab</b>



37.1: Synthesis of dibenzalacetone. p.485 -486 / p.488 -489		
<b>EXP 9:</b> Acetylsalicylic acid (Aspirin) Williamson, Ch. 41 41.1: Synthesis of Acetylsalicylic acid (Aspirin) p. 531-532 / p. 533-534.	<b>Week of April 6<sup>th</sup> - Online Lecture</b> <i>Quiz #3, will be uploaded on Canvas – Due in one week</i>	Week of April 13 <sup>th</sup> - <b>Online Lab</b> (Tour of NMR facility – Cancelled due to online labs) The TA's will send the unknown handout to the students.
<b>EXP 10: Unknown Handout and Checkout</b>	<b>Week of April 13<sup>th</sup> - Online Lecture</b>	Week of April 20 <sup>th</sup> - <b>Online discussion with TA's</b>
Quiz # 3 and Unknown Handouts are due the week of April 27 <sup>th</sup> TA's will finalize the grades and submit the final grades to the instructor the week of May 4 <sup>th</sup> .		

\*Any changes in the syllabus and grading due to chemicals availability, reaction issues, and other unexpected situations will be immediately updated to the students by the instructor and the TA's.

\*Section 001 will not meet the week of Jan 20<sup>th</sup>.

**COVID 19 Effect – Number of experiments have not decreased or changed**

**The experiments and quiz remained the same.**

**The online labs and lectures are carried through the zoom platform.**

**Experiments 7, 8, and 9 are recorded in the labs and video links are posted to the students during the week of the experiment.**

**Zoom online meeting with TA's during the actual lab hours is made mandatory.**

## K. 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.

#### **L. 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>

#### **M. 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.

#### **N. 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/>

#### **O. STUDENT LABORATORY SAFETY AGREEMENT- SEE THE FALL 2019 AGREEMENT ON CANVAS**

#### **P. HELPFUL VIDEO DEMONSTRATION LINKS**

[https://chem.libretexts.org/Core/Inorganic\\_Chemistry/Chemical\\_Reactions/Limiting\\_Reagents](https://chem.libretexts.org/Core/Inorganic_Chemistry/Chemical_Reactions/Limiting_Reagents)

**For TLC:** <https://www.youtube.com/watch?v=CmHFVxTxkGs>  
<https://www.youtube.com/playlist?list=PL03C01E9EB4EF6B45>  
[https://www.youtube.com/watch?v=sq\\_LFsVlaq8](https://www.youtube.com/watch?v=sq_LFsVlaq8)  
<https://www.youtube.com/watch?v=VtkwBLTd0rA>

For Diels-Alder Reaction: <https://www.youtube.com/watch?v=HdvrTQpzfjc>

For Formation of Cyclohexanone from cyclohexanol: <https://www.youtube.com/watch?v=2adECwZdH80>