

**TENNESSEE STATE UNIVERSITY
COLLEGE OF LIFE AND PHYSICAL SCIENCES
DEPARTMENT OF CHEMISTRY**

**CHEM 2021-01, Chem2021-02: Organic Chemistry II Laboratory
Fall 2019**

Instructor's Name: Dr. Al-Masum
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Office Hours: M 10:00-12:00, T 10:00-12:00, W 11:00-12:00

Required Textbook: Organic Chemistry Lab, Tennessee State University

OR

"Macroscale and Microscale Organic Experiments", 7th edition, Kenneth L.
Williamson; Brooks Cole, 2016 [ISBN-13: 978-1305577190]

Prerequisites: First Term Organic Chemistry – Lecture and Lab

Class days and location: M 2:20-5:20 PM, Room: CHM 224 / 227

Number of Credit Hours: 1

Objective: This course is designed for chemistry majors, pre-professionals, biology and other scientific areas. Its aims are to expose students to some of the important experimental techniques, equipment and reactions in organic chemistry, and to illustrate the chemical reactions and properties of molecules discussed in lecture.

Course Description: A continuation of CHEM 2011: Organic Chemistry I Lab. This course exposes students to some of the important experimental techniques, equipment and reactions in organic chemistry. In addition, the course illustrates the chemical reactions and properties of molecules discussed in lecture. Students will receive hands-on experience with nucleophilic substitution reactions, addition/elimination reactions, nucleophilic aromatic substitution reactions, Friedel-Craft's reaction, reduction reactions, Diels-Alder reaction, esterification/hydrolysis reactions and aldol condensation reactions. Students will extensively be involved with the extraction, purification and identification of products by spectroscopic techniques.

Course Competencies: Upon completion of this course, the student should have obtained the following competencies:

- A thorough understanding of the common isolation and purification techniques used in organic chemistry
- A working knowledge of the important spectroscopic techniques used for characterization and

- identification of organic molecules
- A working knowledge of the equipment and techniques used in performing simple organic synthesis
- A thorough understanding of the scientific writing process
- A working knowledge with major instruments- such as IR, UV, Raman, GC-MS and NMR
- Analysis of spectral data

GRADING:

Notebook grade	50%
Formal report	20%
Workstation Organization/Tidiness and Teamwork	10%
Final Exam	20%
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Total	100%

Grading Scale

<u>Grade</u>	<u>Score Range</u>	<u>Significance</u>
A	90 - 100%	Excellent, work of exceptional quality
B	80 - 89%	Good, work above average
C	70 - 79%	Work of average quality
D	60 - 69%	Poor, representing passing work
F	0 - 59%	Failure, representing unacceptable performance

LAB PERFORMANCE INCLUDES:

- Preparation
- Participation in actual experiment
- Results

LABORATORY REPORTS AND DUE DATES:

LATE LABORATORY REPORT: Any laboratory report not turned in on or before the due date will be assessed a 10-point penalty per day. Reports will not be accepted if they are past due more than two days.

OUTLINES: A detail outline of the experimental procedure for each experiment must be completed before coming to class. The outline will help you complete the lab faster and also help you better understand the experiment.

ATTENDANCE AND EXPECTATIONS: Students are expected to regularly attend all classes in this course for credit and to complete all required work in this course. Students are expected to read the *Undergraduate Catalog* for further instructions on attendance policy.

DEPARTMENT POLICY ON STUDENT MAKE-UP LABS WITH A *VALID* EXCUSE:

- 1st Absence: Student must provide a proper University excuse or an excuse that has been validated by the "One-Stop Shop" in 133 Kean Hall; no penalty or make-up lab required. Student must still complete the pre-lab and post-lab quizzes.

- 2nd Absence: Student must provide a proper University excuse or an excuse that has been validated by the “One-Stop Shop” in 133 Kean Hall and the student must complete the designated make-up lab exercise. If the make-up lab is completed successfully, the student will receive full credit for the absence.
- More than 2 Absences: A 3rd absence will be evaluated on a case-by-case basis by the lab instructor, but absences in excess of 3 are not eligible for make-up or earned credit.

DEPARTMENT POLICY ON MAKE-UP LABS DUE TO UNIVERSITY CLOSINGS:

(10-experiment semester)

- Missed Day 1: Since there is an extra day in the syllabus for review, this day will be used for make-up of that experiment. There will be 10 experiments for evaluation purposes.
- Missed Day 2: This lab will be made-up on Fridays. The specific time for each lab class will be forwarded to the students by the lab professor via the department chair. There will be 10 experiments for evaluation purposes.
- Missed Day 3: This lab will be made-up on Fridays. The specific time for each lab class will be forwarded to the students by the lab professor via the department chair. There will be 10 experiments for evaluation purposes.
- Missed Day 4: There will be no make-up. There will be 9 experiments for evaluation.
- Additional Missed Days: There will be no make-up. The number of experiments for evaluation purposes will be reduced by one for each additional missed day.

SAFETY GOGGLES: Safety Goggles are required in this course. No student will be allowed to work in the laboratory without a pair of Safety Goggles. Safety Goggles must be worn at all times while in the laboratory.

ACADEMIC MISCONDUCT, CHEATING & PLAGIARISM: In accordance with the university’s policy on academic and classroom misconduct found in the catalog, cheating will not be tolerated in this course and a zero-tolerance policy regarding cheating will be followed throughout the course. A student who is caught cheating or attempting to cheat will be given a zero “(F)” for that particular assignment/test/quiz for the first offense. If a student is caught cheating a second time, that student will be given an overall grade of “(F)” for the course. To this end, the following classroom policies will be in effect and enforced.

1. Cell phones and any other electronic devices (including smart watches) that connect to wireless networks will not be permitted during any exam or quiz. These devices may not be on your desk during an exam or quiz and must be stored in your bag or purse and/or turned off. Calculators may be used, only if the questions on the exam or quiz warrant their use.
2. Once an exam or quiz period has started, you will not be permitted to leave to go to the restroom during the exam period. Please be sure to use the restroom before coming to class. Exceptions will only be made for those with documented medical needs.
3. No outside materials may be used during an exam or quiz. Any necessary materials (*i.e.* periodic table, equations & constants, scratch paper, *etc.*) will be provided for you.
4. Sunglasses and hats may not be worn during an exam or quiz period.
5. The use of headphones and/or earbuds during an exam or quiz is strictly prohibited.
6. Duplication or copying of homework assignments will result in a score of zero (F) for each student submitting a copied homework assignment.

DISABILITY ACCOMODATIONSTATEMENT

TSU is committed to creating inclusive learning environments and providing all students with opportunities to learn and excel in their course of study. Any student with a disability or condition which might interfere with his/her class performance or attendance may arrange for reasonable accommodations by visiting the Office of Disability Services (ODS). ODS is located in Kean Hall, room 131 and can be reached at 963-7400 or www.tnstate.edu/disabilityservices . You will be required to speak with ODS staff and provide documentation of the need for an accommodation. If you qualify for an accommodation you will be provided with a document stating what type of classroom accommodations are to be made by the instructor. It is your responsibility to give a copy of this document to the instructor **as soon as you receive it**. Accommodations will only be provided **AFTER** the instructor receives the accommodation instructions from ODS; accommodations are not retroactive. You must follow this process for each semester that you require accommodations.

SEXUAL MISCONDUCT, DOMESTIC/DATING VIOLENCE, STALKING

TSU recognizes the importance of providing an environment free of all forms of discrimination and sexual harassment, including sexual assault, domestic violence, dating violence, and stalking. If you (or someone you know) has experienced or is experiencing any of these incidents, there are resources to assist you in the areas of accessing health and counseling services, providing academic and housing accommodations, and making referrals for assistance with legal protective orders and more.

Please be aware that most TSU employees, including faculty and instructors, are “responsible employees”, meaning that they are required to report incidents of sexual violence, domestic/dating violence or stalking. **This means that if you tell me about a situation involving sexual harassment, sexual assault, dating violence, domestic violence, or stalking, I must report the information to the Title IX Coordinator.** Although I have to report the situation, you will still have options about how your situation will be handled, including whether or not you wish to pursue a formal complaint. Our goal is to make sure you are aware of the range of options available to you and have access to the resources you need.

You are encouraged to contact TSU’s Title IX Coordinator to report any incidents of sexual harassment, sexual violence, domestic/dating violence or stalking. The Title IX coordinator is located in the Office of Equity and Inclusion, General Services Building, Room 210 and can be reached at 963-7494 or 963-7438. For more information about Title IX and TSU’s SART or policies and procedures regarding sexual, domestic/dating violence and stalking please visit: www.tnstate.edu/equity.

If you wish to speak to someone confidentially, who is not required to report, you can contact the TSU Counseling Center, located in the basement of Wilson Hall, at 963-5611 or TSU Student Health Services, located in the Floyd Payne Campus Center room 304, at 963-5084. You may also contact the following off campus resources: Sexual Assault Center of Nashville at 1-800-879-1999 or www.sacenter.org or the Tennessee Coalition to End Domestic & Sexual Violence at 615-386-9406 or www.tncoalition.org.

HARRASSMENT & DISCRIMINATION

Tennessee State University is firmly committed to compliance with all federal, state and local laws that prohibit harassment and discrimination based on race, color, national origin, gender, age, disability, religion, retaliation, veteran status and other protected categories. TSU will not subject any student to discrimination or harassment and no student shall be excluded from participation in nor denied the benefits of any educational program based on their protected class. If a student believes they have been discriminated against or harassed because of a protected class, they are encouraged to contact the Office of Equity and Inclusion at General Services Building, Room 210, 615-963-7494 or 963-7438, www.tnstate.edu/equity.

CHEM 2021– EXPERIMENT SCHEDULE

Session	Reading Assignments	Experiments
Week 1	Check-In Procedures. Chapter 1: Introduction, Laboratory notebook writing and related calculation. Chapter 2: Laboratory Safety and waste disposal. Safety Quiz	
Week 2	Chapter 17: Nucleophilic Substitution Reactions of Alkyl Halides.	Experiment: Microscale Procedure (pp 320-327)
Week 3	Chapter 20: Bromination and debromination: Purification of Cholesterol	Experiments: 1. Microscale Bromination of Cholesterol (p 344) 2. Microscale Zinc Dust Debromination (p 345)
Week 4	Chapter 28: Nitration of Methyl Benzoate	Expt 1: Microscale Nitration of Methyl Benzoate (p 405)
Week 5	Chapter 32: Friedel-Crafts Acylation of Ferrocene	Expt: Acetylferrocene – Microscale procedure. p 443
Week 6	Chapter 55: Borohydride Reduction of a Ketone: Hydrobenzoin from Benzil.	Expt: Sodium Borohydride Reduction of Benzil. Microscale Procedure (p 671)
Week 7	Chapter 48: Diels -Alder Reaction,	Experiment 1: Cracking of Dicyclopentadiene, p. 622 Experiment 2: cis-Norbornene-5,6,-endo- dicarboxylic Anhydride, p. 625
Week 8	IR and NMR Spectroscopy of cis-Norbornene-5,6,-endo-dicarboxylic Anhydride	Chapter 48 (con't.)
Week 9	Chapter 40: Esterification and Hydrolysis.	Experiment 2: Isobutyl propionate 92- Methylpropyl propanoate) by Fischer Esterification. P 523
Week 10	Chapter 40: Esterification and Hydrolysis.	Experiment 5: Hydrolysis (Saponification): The preparation of Soap. Microscale procedure: P.527
Week 11	Chapter 37: Dibenzalacetone by the Aldol Condensation	Expt: Synthesis of Dibenzalacetone: Microscale Procedure p 488
Week 12	Review for final examination and lab reports	
Week 13	Make up for missed experiments / Check out	
Week 14	Final Examination, Lab reports and Lab notebooks will be collected for grading	

GENERAL LAB DIRECTIONS

A. Planning and Efficiency

Plan your lab work ahead of time. This is essential if you expect to complete an experiment within the allotted time. Furthermore, you will have some understanding of what you are doing.

1. Read the experiment and all reference material before coming to lab. (Prepare a complete outline of the experiment).
2. Make good use of your time. When you are waiting for something to dry, or waiting for something to cool, etc., start on or prepare for the next step or experiment. Learn to do more than one thing at a time.
3. Listen for announcements from your T.A. and/or instructor. Periodically check the lab blackboard for notices.
4. Do not be afraid to ask the instructor questions about the experiment. They (we) are here to help you. At the same time, you should be prepared for them to ask you questions about the experiment or technique.
5. Every student in this course is required to do his or her own work: this includes all experimental work, calculations, and reports.
6. Be sure to label all solutions or chemicals in your desk so you do not mix them up. Do not rely on your memory. Use pencil to label. Do not use tape or ink.
7. Upon entering the lab, you will be assigned to a specific locker and work area. It's a good idea to keep a towel in your locker. It is your responsibility to keep your work area, adjacent sink, and floor clean. Failure to do so will affect your grade. You are responsible for, together with your fellow students, for the cleanliness of community equipment - balances, hoods, and reagent tables. Again failure to assist in keeping the laboratory clean will lower your grade.

B. General Comments Regarding Notebook

1. Your notebook is the only record you have of your work in the lab.
2. You must use a bound composition notebook. Loose pages, three-ring notebooks, or spiral notebooks are not accepted.
3. The pages in the notebook should be numbered. The first 6 pages should be an up-to-date "Table of Contents" so that any experiment can be located quickly.
4. All entries in the notebook must be in ink. A mistake is not erased, but crossed out with a single line and replaced with the correction written above.
5. All data obtained in the laboratory are recorded in the notebook at the time the data are obtained.
Recording data on loose scraps of paper is not acceptable.
6. Your notebook must be sufficiently neat that someone else (especially the instructor) can read and understand it. Use plenty of space. Remember, you will have to extract data from your notebook to calculate results and more importantly write reports.

C. Format of Notebook:

1. Reserve pages 1-6 for Table of Contents. Keep Table of Contents current.
2. Start Experiment 1 at the top of page 7.
3. For each new experiment, begin at the top of a new right-hand page.
4. For each experiment, begin with the following items:
 - I. Title of experiment, date experiment began, and date experiment was completed. List name(s) of lab partner(s) if applicable.
 - II. A **brief** (3-4 sentences) statement of introduction, purpose, and principles on which the experiment(s) is based. Relevant balanced chemical equations should appear here when applicable.
 - III. An outline of the procedure to be followed.
 - IV. A complete and neat lists of data taken during the experiment. Data must be properly identified and labeled and presented in a logical order. Use appropriate structural drawings when necessary. Use tables where appropriate.
 - V. All other observations that you make during the course of the experiment should also be recorded. Examples include: color changes, changes you make in the general procedure, observed errors such as adding too much solvent or solute or too much of a certain reagent. If you are in doubt as to if the observation is relevant, make a note of it in your notebook. Do not trust your memory!

******* Sections I, II and III above must be completed before coming to the lab. This will save significant time during the lab period. *******

The Chemistry Laboratory Formal Report

There is not one absolutely correct format or method for writing up a laboratory report. The format given below is for a published experimental study and is the format that is expected in general. The goal is to "include everything necessary in the shortest possible way." Standard English is required, and everything that is written must be scientifically sound and logical.

I. Title Page:

This should as specific as possible. State the actual substances used and the method: Determination of the Molar Mass of Acetone by the Dumas Method.

1. Name:
2. Date Experiment Started and Date Finished
3. Date Submitted

II. Abstract

Put on the next page. In general, summarize the results of the experiment and state any significant conclusions. Specifically:

1. State the method used, and actual experimental values that you obtained with uncertainties
2. If there are literature values, put these here as well; (this is not necessary when actually publishing, but it is good practice for in-house reports, unless there are many literature values).
3. Put statement comparing your results with the literature values. If there is disagreement exceeding experimental error, state a plausible reason why there is a difference.

III. Introduction

1. State the purpose of the experiment.
2. Give a brief outline of the theory behind the experiment as well as any pertinent definitions. This includes the "working equation(s)" that will be used in treating your experimental data and the actual experiment quantities to be measured.
3. Make sure that all symbols are defined in any equations that are used.

IV. Experimental Method

1. Cite the lab manual where the original procedure was obtained. This would be a good "first sentence" for this section. Here is a sample sentence along with two ways of citing a reference:
 - a). The method as given by Crockford et al. Was followed, except that heating mantle was used in place of a micro-burner (1).
 - b). The method as given by Crockford et al. Was followed, except that heating mantle was used in place of a micro-burner.¹
2. As shown above any modifications to the procedure in the reference should be noted.
3. Give a brief description of the procedure
4. This section is always written in the past tense (was, were, was used, etc.)
5. State the number of runs made and the conditions (concentrations, temperatures, etc.) at the end of this section.

V. Results

1. Tabulate the data that were measured. Tables should have a title with units, and explanatory captions. Do not exclude data if it seems out of line. The cause of this discordant data should be pointed out in the Discussion. Your final calculated results with error estimates should be listed in this section. Sometimes, data can be presented in form of graphs, in which case it is not necessary to tabulate it. A common example would be in a freezing point determination, a plot of time versus temperature would suffice for this data. Then report the actual freezing point from the graph in the results section.
2. Give a typical calculation (avoid the details). If the calculation is excessively long, it may be placed in the end in an Appendix.
3. Any graphs of data should be included in this section. Here are rules for graphs, especially if they are hand drawn.
 - A). Title the graph
 - B). Label the X and Y axis with units

- C). Use "x's" to make the data points, not individual dots. Dots with circles, triangles, squares, etc. are fine.
- D). Label any lines drawn in
- E). The data points should cover at least half of the paper, especially for hand-drawn graphs.

VI. Discussion

This is the most flexible section. It should always include:

1. A comparison of your results with literature values
2. Plausible reasons for discrepancies with the accepted values
3. Possible systematic errors and ways to reduce them
4. Improvements for the experimental technique
5. Special circumstances or difficulties that may have influenced the results
6. Suggestions of experiments to confirm suspicions of discrepancies of your results with accepted values
7. Suggestions of new/different chemical systems which may be interesting to try in the future

VII. References

1. The **book** title can be either in italics or in quotations.
Garland, D. P.; Garland, C. W.; Nibler, J. W. *Experiments in Physical Chemistry*, 6th ed. McGraw-Hill: New York, **1996**.
2. A book with a plethora of information and an editor (like CRC Handbook of Chemistry and Physics):
Handbook of Chemistry and Physics, 72nd edition., R. C. Weast, editor, p F-10, CRC Press, Boca Raton, Florida, **1991**
3. A book with many authors (say 10 or more) with some editors:
Justice, J. C. In *Comprehensive Treatise of Electrochemistry*, Vol. 5, *Thermodynamic and Transport Properties of Aqueous and Molten Electrolytes*; Conway, B.E.; Bokris, J. O'M; Yeager, E. Eds.; Plenum: New York, **1983**; pp. 223-337.
4. A journal Article:
Gunning, H. E.; Gordon, A. R. *J. Chem. Phys.* **1942**, *10*, 126.
Here the year is in bold, the volume italicized, and the last number is the first page number. An option here is to underline the volume, and use regular type for both the year and the page number.
5. A journal Article with Title of the paper:
"Novel methods for the synthesis of functionalized indoles from arylhydroxylamines and activated acetylenes." Hwu, J.R.; Patel, H.V.; Lin, R.J.; Gray, M.O. *J. Org. Chem.* **1994**, *59*, 1577-1582 and references cited therein.

FALL SEMESTER 2019

- Aug 9 Faculty contracts begin
- Aug 9 Faculty report for fall semester activities
- Aug 12-13 Faculty/ Staff Institute
- Aug 13-14 Residence Halls Open (New Students)
- Aug 14-16 Freshmen Orientation
- Aug 16 Residence Halls Open (Returning Students)
- Aug 19 Classes begin
- Aug 19- Aug 23 Late registration/Schedule Adjustment
- Aug 30 Campus Wide Assembly – First Year Students 9:00 am – Kean Hall
- Sept 2 Holiday-Labor Day
- Sept 17 Recognition of Constitution Day
- Sept 22-28 Student Study Week – No Activities Scheduled
- Sept 30 – Oct 4 Mid-term Examination Week-all classes meet as scheduled
- Oct 11 Last day to withdraw from a course and/or the University
- Oct 15 Founders Day (9:00 a.m. – Kean Hall)
- Oct 19 Homecoming
- Oct 28 – Jan 17 Registration for Spring 2020
- Nov 11 Veterans' Day
- Nov 25-29 Fall Break/Thanksgiving Holiday- No Classes

Nov 30 Last Day of Class
Dec 2-6 Final examinations
Dec 7 Commencement – Gentry Complex
Dec 9 Faculty must have posted all grades via “MyTSU”
Dec. 13 Records Office releases all posted grades via “MyTSU”
Dec 24 – Jan 1 Holiday Break – University Closed