

CHEM 320L – Experimental Organic Chemistry, Summer 2019

Course Description: Elementary organic chemistry experiments to teach basic laboratory operations. Course

Attributes: None. Restrictions/Exclusions: None.

Section 001L (30019), Lab (P), 1 unit; Meets: MW 8:00 - 10:50 am, BSB 385

Instructor: Dr. Todor Gounev (gounevt@umkc.edu – preferred method of communication); Dept. of Chemistry, Flarsheim Hall 410B, 816-235-2294, Office hours, MTWR 10:00-11:00 AM

Course Website: <http://d.web.umkc.edu/drewa/Chem321L/index321L.html>

Teaching assistants: will manage the operation of the laboratories.

Section	Day	Time	GTA 1	GTA 2
1	Mondays and Wednesdays	8:00 – 10:50 AM	Michael Just majyr8@mail.umkc.edu	Dr. Robert Clevenger rgcdcb@mail.umkc.edu

Office Hours: GTA Office hours will be: [Schedule to be distributed in Week 1].

Please see teaching assistants first with a problem, then come and see the professor in charge if it cannot be resolved with the GTAs.

Pre-lab will meet in BSB 385

CATALOG DESCRIPTION: Elementary organic chemistry experiments to teach basic laboratory operations. Three hours of laboratory a week. Prerequisite: CHEM 212R and CHEM 212LR or their equivalents (each with a C-or better); Co-requisite: CHEM 320; Offered: Spring and Summer.

STUDENT LEARNING OUTCOMES

Upon completion of Chemistry 320L, students should be able to:

- Apply knowledge obtained in Chemistry 320 lecture to problem solving and critical thinking in the laboratory.
- Utilize mathematical knowledge gained from general chemistry to perform common calculations, including mass balance, limiting reagent, and percent yield.
- Engage in safe laboratory practices by handling laboratory glassware, equipment, and chemical reagents appropriately, using general guidelines and basic knowledge about the common hazards associated with them in an organic chemistry laboratory.
- Maintain an appropriate scientific notebook using notational and descriptive content containing MSDS information on relevant chemical reagents, experimental procedure followed, data collected, and observations made during the experimental process.
- Assemble glassware and perform the following techniques as a part of synthetic procedures: aqueous workup, distillation, reflux, separation, isolation, and crystallization.
- Predict the outcome of several common organic reaction types through a basic understanding of starting materials, functional groups, mechanism, and typical reaction conditions.
- Characterize prepared substances by physical and spectroscopic means.

Optional text: Mohrig, et al. Laboratory Techniques for Organic Chemistry, 4rd ed.; 2014, W.H. Freeman (ISBN- 9781464134227). 3rd edition is ok too. You can order online, or in the bookstore under CHEM 320L.

There will be other reference materials on BlackBoard.

Experimental handouts: Handouts will be provided on the course website (see URL above)

Required materials:

- A lab notebook capable of making carbon copies. (a 100-page version, should get you through Organic Lab 1 and 2...)
- A pen with black or blue ink.
- A small padlock.
- Safety glasses.
- Black felt-tip pen with permanent ink (e.g. Sharpie).
- Appropriate clothing.

Class Attendance: Attendance is required for all of the laboratory classes. The class starts on time and finishes on time. No makeup labs are allowed. Students must attend their assigned laboratory section.

Glassware drawers: You must provide a padlock for your glassware drawer. You are responsible for maintaining the glassware clean and in good condition and for keeping your equipment drawer clean. Even though you are expected to take appropriate care with equipment and glassware, it is reasonable to expect a small amount of breakage. Notify the GTA as soon as possible if something is broken.

Pre-Lab Videos:

Before each lab period, you are expected to watch videos that demonstrate each experiment you will be performing in the lab and the techniques associated with it. These videos were created in an effort to better prepare you for organic lab and to enhance your learning experience there. The videos can be found on the course website (see URL above). When you enter the lab, it is expected that you have already watched these videos!

Pre-lab Assignments: You are also required to prepare a pre-lab before each experiment. Your pre-lab work must be completed before you enter the laboratory. Writing a pre-lab ensures that you have an understanding of your tasks during an experiment before you begin. Ideally, the pre-lab should be thorough enough so that you can perform the experiment from what you have written without needing to refer to the handouts or to your book. The GTAs collect a copy of the pre-lab along with your other notebook entries for the day. You will not be allowed to perform the lab unless the pre-lab is complete. The pre-lab should consist of the following entries in your notebook (carbons collected as you leave lab):

- *Title:* Begin each experiment with a title, your name, the date, your GTA's name and your lab section.
- *Purpose:* A brief statement of the experimental objectives
- *Net equation and/or mechanism of reaction:* Include for all important reactions.
- *Table of quantities and physical constants:* Collect in tabular form the name, structure, molecular weight, density, melting or boiling point, solubility, mass or volume used, moles used, of any substance whose mass or volume you must measure. Some of this information can be found in the handouts. That which is not in the handouts can be looked up in *The Aldrich Chemical Company Catalog*, *The Merck Index*, *The CRC Handbook of Chemistry and Physics*, and on-line at <http://www.sigmaaldrich.com/united-states.html> (after completing a search, click on the SDS icon to see the data) or webbook.nist.gov/chemistry.
- *Procedure:* Provide an outline of the experimental procedure to be carried out. Do not simply copy what is written in the handouts; use your own words and diagrams. It is often useful to construct a flow chart of the procedure.

Lab notebooks:

- Read relevant reference sections or chapter in Mohrig, et al. or Zubrick, et al.
- Start a “Table of Contents” page near the beginning of your notebook.
- Each experiment should begin on a new page.
- Make every entry in your notebook in ink, never in pencil. Don't erase or black out entries. Instead, ~~draw a line through the error~~ and add the new information.
- Record all laboratory observations and data directly in the lab notebook at the time they are observed. Do not use scratch paper. Do not expect to transcribe any information into the notebook at a later time. Carbon copies of your notebook entries are handed in at the end of every laboratory session.
- Organize your lab notebook using headings such as *Title, Purpose, Procedure, Apparatus* or *Glassware set-up, Balanced reaction* or *Mechanism, Observations, Purification*, etc.

Lab reports: The due dates for lab reports are listed in the class schedule accompanying the syllabus. Lab reports must be turned in at the beginning of the pre-lab lecture prior to entering the laboratory. **Late lab reports will never be accepted.** No exceptions will be made.

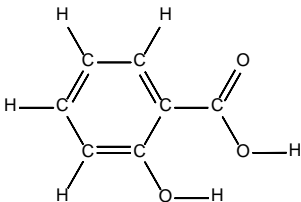
- For each experiment you will hand in (1) carbon copies of your lab notebook before leaving the lab, and (2) a completed post-lab report form on the scheduled due date. These two items will be combined and graded.
- The individual lab handouts will be available for download at the course website. Post-lab report forms appear after each experimental lab handouts on the course website.
- Your post-lab report must be typed using the Microsoft Word files provided on the website.
- **Chemical structures and mechanisms should be carefully depicted (hand drawing in pen or software drawings), showing all the relevant atoms and bonds, particularly at the reactions sites.**
- Also please note that in post-lab reports, numbers should never begin with a decimal and units must be given (even if they are implied)! Additionally, melting points that are measured are given as a range, not as a single temperature, and are reported in °C. An example is:

Initial weight of salicylic acid	0.198 g
Volume of water used to recrystallize salicylic acid	2.50 mL
melting point of compound above (°C)	158-160 °C

Example of how to set up your lab notebook (and see examples online)

Before the Experiment Begins

- **Experiment Title:**
- **Date:**
- **Name:**
- **Course: Chemistry 320L**
- **Section:**
- **GTA Name:**
- **Purpose:** (Purpose of the experiment; write a brief (1-3 sentences) statement of purpose for the synthesis or analysis, or state the question you are addressing)
- **Balanced Chemical Reaction:** (Write balanced chemical equations that show the overall process, not a mechanism)
- **Table of Physical Quantities:** Include all reactants, products and solvents: name of compound, molecular formula, molecular structure, molecular weight, melting point (solids) or boiling point (liquids), density, hazards.

Name (Other names)	Molecular formula	Molecular structure	Molecular Weight (g/mol)	Melting or Boiling Point	Density
Salicylic acid (2-hydroxybenzoic acid, o-hydroxybenzoic acid)	C ₇ H ₆ O ₃	 <p>(Note that stereochemistry and all atoms are shown!)</p>	138.12	mp = 158 - 159°C	1.4
Benzoic Acid					
Phthalic Acid					
Naphthalene					
Biphenyl					
Ethanol					
Water					

Name	Hazards (According to MSDS)
Salicylic acid	HARMFUL IF SWALLOWED, INHALED OR ABSORBED THROUGH SKIN. AFFECTS CENTRAL NERVOUS SYSTEM, KIDNEYS, AND PANCREAS. CAUSES IRRITATION TO SKIN, EYES AND RESPIRATORY TRACT.

- **Yield Calculations:** Outline the formulas to be used in your experiment. Calculate the theoretical yield.
- **Procedure:** This is a procedural outline of what you are doing in the experiment. Remember, you will not be allowed to bring your lab report to class so it is important that you have the details of the experiment here. Leave space between lines here for any corrections that the GTA gives you to the procedures.

During the Laboratory Session

- **Observations:** Record all observations that take place while you are performing your experiment. This includes:
 - Actual quantities of all reagents used.
 - Amounts of crude and purified products obtained.
 - Mention measurements you took (temperature, time, melting point, and so on).
 - Odors you detect.
 - Color changes.

Assignments and Post Lab Reports are Due on MONDAYS or TUESDAYS (see schedule).

Grading: You will be graded on the basis of the following: nine (9) best out of ten lab reports and one (1) lab exam. Each lab report will be worth twenty (20) points, while the exam will be worth fifty (50) points. **Final grade:** (your points/total points) * 100 = % grade.

There are no lab make ups; if you have a valid, emergency excuse for missing, you must contact the TA and instructor ASAP for accommodation and verification. To deal with these situations note the following policy.

The lowest report score will be dropped. If you have an “excused” absence, it will become your dropped lab.

Grades will be assigned using the following scale: A, 100-90.0; B, 89.9-80.0; C, 79.9-70.0; D, 69.9-60.0; F, 59.9-0. The professor in charge reserves the right to make minor adjustments in the total number of points to the course and to make adjustments for inadvertent errors in the syllabus.

Grading Detail:

<u>Assignment</u>	<u>Total</u>
9 Lab Experiments (20 points each)	180
Lab Exam	50
TOTAL POINTS POSSIBLE	230

UMKC Resources & Policy Statements

Important UMKC Resources and Policies are applicable to every course and every student at UMKC. These are located in the Blackboard site for this course under the “UMKC Policies” tab. As a UMKC student, you are expected to review and abide by these policies. If you have any questions, please contact your instructor for clarification.

This course follows the “Faculty not allowing recording” option of the Academic Inquiry, Course Discussion and Privacy policy.

College of Arts & Sciences Course Policies & Resources

Please refer to the following web page and the linked resources for critical information regarding course policies and resources. You are expected to abide by all the rules and regulations regarding student conduct referenced in these pages. <http://cas.umkc.edu/CPR/>

Academic Calendar: Students are encouraged to review important add, drop or withdraw dates: <http://www.umkc.edu/registrar/acal.asp>

CHEM 320L-001 LABORATORY SCHEDULE

Day	Date	Lab	Assignment(s) DUE MONDAY
Monday	June 3	Check-in: Introduction; notebook requirements; lab safety and waste disposal; read pp 2-74 in Mohrig. [Z: Sections 1,2,3,4,5]	
Wednesday	June 5	1. Crystallization: Recrystallization [Z: Sections 9, 10]	
Monday	June 10	2. Distillation: Steam Distillation of Toluene - Benzil Mixture [Z: Sections 12, 13, 14, 15]	Exp.1 Post Lab
Wednesday	June 12	3. Extraction I: Separation of a Strong/Weak Acid	
Monday	June 17	3. Extraction I: Continued	Exp.2 Post Lab
Wednesday	June 19	4. Extraction II: Separation and Purification of the Components of an Analgesic Tablet	
Monday	June 24	5. Solid-Liquid Extraction: Trimyristin from Nutmeg	Exp.3 Post Lab Exp.4 Post Lab
Wednesday	June 26	6. Grignard Reaction	
Monday	July 1	7. Substitution Reactions S_N2 : Synthesis of <i>trans</i> -1,2-dibenzoylcyclopropane	Exp.5 Post Lab
Wednesday	July 3	NO LAB	
Monday	July 8	8. Substitution Reactions S_N1 : triphenylmethanol	Exp.6 Post Lab Exp.7 Post Lab
Wednesday	July 10	9. Elimination Reaction E1: Cyclohexene from cyclohexanol (fractional distillation)	
Monday	July 15	10. Elimination Reaction E2: Cyclohexene from bromocyclohexane.	Exp.8 Post Lab Exp.9 Post Lab
Wednesday	July 17	CLEAN LAB and Check-out	
Monday	July 22	LAB FINAL	Exp.10 Post Lab

CHEM 320L Laboratory Safety Regulations: Initial the items and sign the bottom.

Semester Summer 2017 Section VO Room # _____
TA Name _____ Station # _____

1. _____ I will prepare for lab by studying the experiment before class and by trying to anticipate potential hazards from the chemicals or procedures to be used.
2. _____ I will wear approved safety goggles AT ALL TIMES in the laboratory unless the instructor gives specific approval to remove them.
3. _____ I will not work in the lab unless an instructor is present.
4. _____ I will not perform any unauthorized experiments.
5. _____ I will notify the instructor of any allergies or other health conditions (pregnancy, epilepsy, etc) that may affect my ability to work in a chemistry lab.
6. _____ I will not eat, drink, or smoke in the lab.
7. _____ I will not use cellular phones or radios in the lab.
8. _____ I will minimize my contact with chemicals by taking care to note odors, never tasting chemicals, using suction bulbs to fill pipettes, and washing any spilled chemicals off my person as soon as possible. I will wash my hands before leaving the lab.
9. _____ I will not wear shorts, sandals (or open-toed shoes), tank tops, or other clothing in the lab that allows unnecessary exposure to spilled chemicals. I am also aware that certain chemicals can ruin clothing and that wearing a lab coat or apron adds some degree of protection.
10. _____ I will secure long hair to keep it away from open flames and chemicals while I am working in the lab.
11. _____ I will immediately report all cuts, burns, personal injuries, fires, chemical spills, or other accidents to the instructor.
12. _____ I will keep my work area and the common areas of the lab clean.
13. _____ I will NOT return unused chemicals to their original bottles.
14. _____ I will consult with the instructor about the proper disposal of all waste chemicals.
15. _____ I know the location, operation, and appropriate uses of the eye-wash stations, safety showers, fire extinguishers, fire alarms, and fume hoods; and I know the locations of all lab exits.
16. _____ I agree to follow any specific or additional safety instructions that may be given for any experiments.
17. _____ I will conduct myself in a professional and respectful manner. I will leave the lab after I complete my work.

I understand all of these statements and agree to observe them at all times in the lab. I also understand that if I fail to observe them, I will be expelled from the laboratory.

CHEM 320L Academic Honesty Statement

I will perform the work by myself and will adhere to the UMKC Academic Conduct Standards for Students (http://www.umkc.edu/catalog/Student_Conduct.html and http://www.umkc.edu/catalog/Rules_of_Procedures_in_Student_Conduct_Matters.html). It is my responsibility to understand the facets of academic honesty and to uphold them. If I am not sure, I will consult with the instructor.

Sign: _____ **Date:** _____

Name: _____ TA: _____

Email _____@mail.umkc.edu

Section #: _____ Room #: 385 Hood #: _____ **L side or R side** (circle one)

Item (# Needed <u>per</u> Student)	# Received by Student	# Returned by Student
Beaker, 50 mL (2)	_____	_____
Beaker, 400 mL (1)	_____	_____
Beaker, 600 mL (2) plastic!	_____	_____
Erlenmeyer flask, 50 mL (3)	_____	_____
Erlenmeyer flask, 125 mL (1)	_____	_____
Erlenmeyer flask, 250 mL (1)	_____	_____
Graduated cylinder, 10 mL (1)	_____	_____
Spatula, (1)	_____	_____
Glass stir rod, large or small (2 total)	_____	_____
Powder funnel, large (1)	_____	_____
Small reaction tube, (4)	_____	_____
Large reaction tube, (1)	_____	_____

Student's Signature: _____ Date: _____

Checked IN by: _____ Date: _____

Checked OUT by: _____ Date: _____

TODAY'S TO-DO LIST!

- ✓ Sign the "post it" on your drawer so we know that drawer has been assigned.
- ✓ Put fresh paper towels to line your drawer.
- ✓ Get any glassware you are missing from your TA.
- ✓ Throw any used pipette bulbs into regular trash. Get 2 new pipette bulbs!
- ✓ Give any extra glassware to your TA.
- ✓ Wash any dirty glassware.
- ✓ Make sure there are pipettes and paper towels in the common hood space (underneath the hood).
- ✓ Put your safety goggles and Sharpie marker in your lab drawer.
- ✓ Put your lock on your drawer if you have it and give UMKC's lock to your TA.
 - If you don't have your own lock, use UMKC's lock to lock your drawer and bring your lock next week.
- ✓ Put this sheet in your drawer and give the Laboratory Safety Regulations sheet to your TA.

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