## CHEM 2219 ORGANIC CHEMISTRY I LAB FS/2023

#### **Instructor:**

Name:\_\_\_\_\_

Section:\_\_\_\_\_

Desk No.:

Combination:\_\_\_\_\_

### **LEARNING OBJECTIVES:**

Course objectives are to develop facility with performing laboratory techniques involving the handling of organic chemicals safely and the keeping of proper records of experiments conducted in the laboratory. Crystallization, distillation, extraction and chromatography are emphasized as separation and purification techniques. Melting points, boiling points, and refractive indices are used routinely as measures of purity and identity. Safe handling of chemicals and proper disposal of waste products are also goals in this course. FTIR and HNMR will be used briefly as analytical and identification techniques. Most of the experiments will be conducted on a micro scale.

Lab lectures will be provided live in rm 126. A lecture recording and powerpoint slides will be stored online on Canvas. Assignments and lab writeups will be turned in via Canvas. All lab work will be done in person in rm 129 unless we are required to go remote due to covid, when only videos of the experiments will be used, however lab books will continue to be written up and graded via Canvas. Weekly quizzes, a midterm and comprehensive final exam will be given.

### **BOOKS:**

- 1. 100 pg. set Organic Chemistry Laboratory Notebook or 110 pg bound composition book. (#1 above can be purchased from the book stores or Amazon)
- 2. "Organic Chemistry Laboratory Laboratory Techniques, 2<sup>nd</sup> ed". Available as a free pdf download at https://open.umn.edu/opentextbooks/textbooks/369.
- 3. OPTIONAL "Microscale Techniques for the Organic Laboratory, 2nd ed", (MTOL), Mayo, Pike, Butcher and Trumper, John Wiley & Sons, Inc.,2001 (on reserve at library circulation desk-currently out of print but available on Amazon)

### LAB SYLLABUS:

Originally prepared by Prof. S. B. Hanna, modified by D. E. Hoiness, T.Bone, C. Bolon.

## GENERAL GUIDELINES

### SAFETY

Currently, no covid masking or temperature checks are required for lab.

### Goggles or faceshields must be worn at all times in the lab. Shorts or short skirts and open toed shoes are not permitted for safety reasons.

Nitrile gloves (blue or purple) are required. You may want to purchase nitrile gloves (blue or purple) from the bookstore or Walmart. Do NOT use vinyl or latex gloves as these provide inadequate protection. Lab aprons or lab coats are optional.

MSDS info is available online via the class web page or at <u>https://ehs.mst.edu/sds/</u>

### **NOTEBOOK**

1. A bound composition book with the pages numbered is acceptable as a lab book since electronic scans of the pages are to be submitted. Three ring and spiral bound notebooks are NOT acceptable. A 100 pg. Computation Notebook, with white and yellow pages, available at the bookstore, may be used. These are designed so that a carbon copy is made on the yellow pages which can then be torn out and turned in to the grader on completion of the experiment. In all cases be sure to press firmly so that the yellow copy is readable. If you have bad handwriting, you may want to print.

You should double space your writeup in either case for ease of reading and grading.

- 2. ALL entries in your notebook should be recorded in permanent blue or black ink. NO pencil.
- Table of Contents: Two pages should be retained at the front of the notebook or composition 3. book for the table of contents, to be updated weekly.
- Print at the top of every page: Your name, Course and Section number, Date, Title of the 4. Experiment
- Prelab: This portion must be completed before you come to lab lecture and submitted online 5. on Canvas two days before lab. Prelabs will be considered late upon start of the lab lecture.

**Objective:** Brief summary of the objective of the experiment. **Chemical Equations:** (if applicable for reactions) **Physical Properties:** For all chemicals used in the experiment, list as a table,

| Compound<br>name | Structure | CAS# | MWt. | BP or MP<br>lit. ∘C | State (s, 1)<br>color | RI<br>(liquids only) | Hazards |
|------------------|-----------|------|------|---------------------|-----------------------|----------------------|---------|
|                  |           |      |      | _                   |                       | ( <b>1</b>           |         |
|                  |           |      |      |                     |                       |                      |         |

Property data may be found in the Merck index, CRC, Aldrich catalogs or online. These are available at the library circulation desk or my office, rm 120B. **Reference(s)** should follow the table as to the source of the property data.

<u>Prelab Questions:</u> answer any assigned questions in Canvas online.

- 6. **Procedure:** This section must be written <u>as you do the lab</u>. It should include experimental data and an apparatus sketch. It should be complete enough to allow someone to repeat the experiment and should note any procedural modifications used. (**Use 3**<sup>rd</sup> **person past tense**)
- 7. **Observations:** Report what you witnessed concerning the reaction, such as color changes, ppt, gas, etc.
- 8. **Results:** Results should be reported in the form of a table. The yield and physical properties may be listed as two separate tables if more space is required. Show theoretical yield, % yield and % error calculations below the table.

| Product name<br>or unknown # | .0. | Yield (gm)<br>theor. | %<br>Yield | MP, BP, RI<br>exp. | MP, BP, RI<br>lit. (ref) | % Error<br>MP, BP, RI |
|------------------------------|-----|----------------------|------------|--------------------|--------------------------|-----------------------|
|                              |     |                      |            |                    |                          |                       |
|                              |     |                      |            |                    |                          |                       |

- 9. **References:** Cite source(s) used for product physical property data in table.
- 10. Each section (5-9) must be clearly designated.
- 11. Each new experiment should begin on a new page.
- 12. Void all unused space. Sign and date each page.
- 13. You must have your lab book initialed by the TA before leaving the lab. Points will be deducted if the procedure is not completed before leaving lab.
- 14. <u>To make corrections</u>: simply draw a line through the old data or conclusions, add the new information and initial it. Do NOT erase or use white out.
- 15. Upon completion of each experiment, the <u>pages of your notebook are to be turned in as a</u> <u>single pdf via Canvas</u> before the end of the next lab session. This will allow time to complete MP or yield measurements if not finished the previous period. Late reports will receive only partial credit. (-5 pts/wk late, -25 pts max)

*Notebooks will NOT be graded if:* pencil is used, white-out is used, or a TA did not sign your notebook at the end of the day or the submitted scan is not legible.

### **CELL PHONES:**

Cell phones are to be turned off and put away during lecture. Any use of cell phones during lecture will result in confiscation of the phone for the remainder of the lecture.

### MAKE-UP OF LABS:

Make up labs must be completed within one week of the original scheduled date, as the chemicals for each lab are put away after each experiment. Exceptions may be made for a written medical excuse or with the instructor's permission. If you know in advance that you will have to miss a lab, please notify your TA and instructor. With advance warning, we may be able to fit someone into another lab section that week. Missed labs remaining unexplained after their due date will receive no credit and cannot be made up.

The lab schedule is online at: <u>https://web.mst.edu/~tbone/Subjects/TBone/scheduleWS2023.html</u>

## GRADES

A rubric for each experiment is on Canvas to assist in determining if you have everything required.

### Lab Notebook

10 pts for Canvas prelab questions (due at beginning of lecture) 20 pts Prelab Table (due at beginning of lecture, see general guidelines for details) 40 pts Procedure and Observations (to be recorded while in lab) 25 pts Results (Table(s))

Total pts per experiment: ~110 pts

Penalties (-5 each, -25 pts maximum)

Procedure not in 3<sup>d</sup> person past tense Improper method of calculation Missing or incorrect data or results Failure to void unused space No references for properties Results not in table form Failure to sign and date each page Procedure not written before leaving lab Procedure not double spaced Turned in late (-5 pt/wk)

<u>Weekly Experiments</u> are **due before the end of lab the week following the completion of the experiment.** These should be graded by the following week. If they are not, please email your TA. If this fails to resolve the problem, then contact me at tbone@mst.edu.

Exams, (Midterm and Final) are both required and will have -5% / day deducted from the curved score if taken after the scheduled section time. They will be administered online in Canvas.

### **Course**

| 9 experiments     | 765 pts        | 59% |
|-------------------|----------------|-----|
| 10 Ĉanvas prelabs | 100 pts        | 8%  |
| Misc. hwk         | 135 pts        | 10% |
| Mid term exam     | 100 pts        | 8%  |
| Final exam        | <u>200 pts</u> | 15% |
|                   | -              |     |

Total 1300 pts 100%

(The exact course point total may vary slightly from 1300 but this should be close.)

Course grades will be based on the following % scale. (Some curving of raw scores may be applied.)

| 90-100 | Α |
|--------|---|
| 80-89  | В |
| 70-79  | С |
| 60-69  | D |
| <60    | F |

### **GENERAL LAB SUPPLIES**

Required Items: Students are required to supply their own masks, goggles, gloves and paper towels.

Safety Items Dial 911 for emergencies.

You are responsible for knowing the location of the following safety items in the lab: Mark these on your lab map, handed out during checkin and submitted on Canvas.

Safety shower, eyewashes(3), fire extinguisher, fire blanket

**Balances** are to be kept clean. A pan and broom for spill cleanup are available. Dispose of used weighing paper, gloves, etc. in the used solids waste bucket in the waste hood.

Chemicals & Samples for each lab are located under the hoods or by the balances.

Supply Cart -- in lab

1.5 ml latex bulbs (reuse these)Pasteur pipets, (9" glass, dispose in glass waste)Sample Vials, (Snap Cap , 2 ml), Corks/Stoppers, Labels (S, M, L), Q tips pH paper, litmus paperAluminum foil, Parafilm, Cotton batting (insulation), Scissors, stapler

## Waste Hood PLEASE REPLACE THE LID ON ALL WASTE CONTAINERS AFTER USE

Non Halogenated Solvent Waste Halogenated Solvent Waste (compounds containing F, Cl, Br, I eg. CH2Cl2) Mineral Acids Waste-(aqueous strong acids and bases) Glass Waste (box for Pasteur pipets, snap cap vials, broken glass, MP cover glasses) Used Solids Waste-(white bucket, for contaminated filter paper, etc. -NO glass items) Mercury Waste (broken thermometers) Sharps Waste (syringe needles) Wash bottles of acetone, water, (for cleaning glassware).

**Hoods** Large lab hoods will sound an alarm if the sash is raised too high. There is a reset button on the upper right front that will temporarily disable the alarm. The sash should be returned to a 4"-6" opening when finished.

**Mini hoods** at each lab bench location should be used for all experiments. There is a flow adjustment valve on each snorkel to control flow. They should be closed after use.

**Spills & Breakage Cleanup** Broken glass should be disposed of in the glass waste box, which is located in the waste hood. There is a pan and broom, kitty litter, baking soda etc. available in lab. Please see the TA for assistance.

**Students are responsible for all breakage, loss or other damage to equipment**. Damaged items must be repaired, replaced or paid for before check out is complete.

**Breakage Payment-**Contact the TA to fill out a green slip and obtain a replacement item. Payment is via charge to the student account. Be sure to obtain a receipt from the TA or instructor at the end of the semester.

Failure to check out will result in a \$25.00 fine. You must check out even if you drop the course.

## **GROUP STATION CABINET CONTENTS**

### **Top Shelf**

4 Filter Flask, 250 ml
4 Hirsch Funnel, 3.0 cm dia
1 Filter Paper, 3.0 cm dia (for Hirsch Funnel)
4 Aluminum Heating Block

### White Plastic Tray (top shelf)

4 Beaker Tongs

- 4 Crucible Tongs
- 4 Cu Wire, heavy gauge
- 4 Ring Clamp, Small

## **Bottom Shelf**

- 8 Steam Bath, Cu
- 4 Vacuum Hoses (1/2" O.D.-heavy wall)
- 8 Water/Gas Hoses (3/8" O.D.-thin wall)
- 2 Variacs (variable power supplies)

The above items are shared by all sections and must be returned to the common drawer after use.

### **DESK CONTENTS**

### **Microscale Kit Components**

1 Jacketed condenser 1 Claisen adapter 1 Hickman still 1 5.0 ml conical vial 1 Teflon spin vane-large 1 Micro filter paper in glass vial, 0.5 cm dia 1 septum with hole 2 screw caps w/septa

### **Extra Components**

- 2 Watch glass, ~3" dia
- 1 Vacuum filtering flask, 25 ml
- 1 1 cm Hirsch funnel with adapter
- 1 Casserole
- 2 Erlenmeyer flask, 50 ml, 125 ml
- 8 Beakers, 50, 100, 150, 250 ml (2 ea)
- 1 Short stem glass funnel
- 1 Thermometer
- 1 Glass stirring rod with policeman
- 1 Plastic Ruler, 6"
- 1 Graduated cylinder, 10 ml
- 1 Mortar and Pestle
- 1 Test Tube Rack with 6 Test Tubes
- 2 Test Tube Clamp/Holder
- 1 Tweezers(forceps)
- 6 #1 rubber stoppers for test tubes
- 1 microspatula

**Opening combination locks:** Turn the dial 3 turns clockwise and stop on the first number of the combination. Next, turn counterclockwise, passing the middle number once and stop on the middle number of the combination the second time it comes up. Finally, turn clockwise and stop on the last number of the combination. The locks will not open if you miss any number by more than one digit.

Be sure to record your desk number and combination on this syllabus that you will be bringing to lab after check in.

# **Disability Support Services**

If you have a documented disability and anticipate needing accommodations in this course, you are strongly encouraged to meet with me early in the semester. You will need to request that the Disability Services staff send a letter to me verifying your disability and specifying the accommodation you will need before I can arrange your accommodation.

G-10 Norwood Hall, MST 573-341-7734 <u>dss@mst.edu</u>

# TITLE IX Info.

Missouri University of Science and Technology is committed to the safety and wellbeing of all members of its community. US Federal Law Title IX states that no member of the university community shall, on the basis of sex, be excluded from participation in, or be denied benefits of, or be subjected to discrimination under any education program or activity. Furthermore, in accordance with Title IX guidelines from the US Office of Civil Rights, Missouri S&T requires that all faculty and staff members report, to the Missouri S&T Title IX Coordinator, any notice of sexual harassment, abuse, and/or violence (including personal relational abuse, relational/domestic violence, and stalking) disclosed through communication including but not limited to direct conversation, email, social media, classroom papers and homework exercises.

Contact directly Dr. Paul Hirtz (<u>equity@mst.edu</u> (573) 341-7734; (900 Innovation Drive, Suite 500) to report Title IX violations. To learn more about Title IX resources and reporting options (confidential and non-confidential) available to Missouri S&T students, staff, and faculty, please visit <u>https://equity.mst.edu/title-ix/</u>.

# Student Honor Code and Academic Integrity

Academic integrity is important to Missouri S&T and to future employers. Missouri S&T's Honor Code was developed and endorsed by the Missouri S&T Student Council. The Honor Code can be found at <a href="http://stuco.mst.edu/honor-code">http://stuco.mst.edu/honor-code</a> . Please read the Honor code and its emphasis on HONESTY and RESPECT. The use of ChatGPT to write lab reports is against university policy and is subject to disciplinary action.

## COVID

Work through Student Health Services (<u>mstshs@mst.edu</u>), 573-341-4284, if you are quarantined, become ill, or are unable to attend class or take tests on campus. <u>The student is now responsible for forwarding a student health absence notice to instructors</u>.

## FILES

There are numerous files of prelab & postlab questions circulating that have incorrect answers, but due to previous careless grading, were marked as correct. Incorrect answers will not be given credit, so you should not rely on files as a source of easy answers.