

CHEM 2229 -ORGANIC CHEMISTRY LAB II- WS/2020

Instructor:

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COURSE OVERVIEW:

The Winter Semester 2229 Laboratory is made up of 2 parts. The first half of the semester involves a series of weekly microscale single step syntheses. There will be a midterm exam. A review of IR and NMR for product characterization will be done in preparation for the second half.

The second half of the semester involves a special project which entails a larger scale multi-step synthesis, introduction to chemical literature retrieval. Each week prior to lecture the lab books will be collected for grading of the prelab section and a short quiz will be given.

There will also be a comprehensive final exam for Chem 2229.

LAB NOTES:

Originally prepared by Prof. S. B. Hanna

BOOKS:

1. 100 pg. set Organic Chemistry Laboratory Notebook (may continue from 2219)
2. "Organic Chemistry Laboratory Laboratory Techniques, 2nd ed". Available as free pdf download at <https://open.umn.edu/opentextbooks/BookDetail.aspx?bookId=369>
3. OPTIONAL "Microscale Techniques for the Organic Laboratory, 2nd ed", (MTOL), Mayo, Pike, Butcher and Trumper, John Wiley & Sons, Inc., 2001 (On course reserve at MST library)
4. OPTIONAL "Right to Know pocket guide for School & University Employees", Genium Publishing Corp., 1990. (You may still have one from chem 4)

(#1 can be purchased from the book store or Amazon)

There are reference books on reserve in the library at the circulation desk under the course number. See a later page in the handout for a list. Please consult them for the prelab preparation. There are also Aldrich chemical catalogs, useful for physical property information, in the lab and in my office, rm 120B.

GENERAL GUIDELINES

SAFETY

Goggles must be worn at all times in the lab.

Unless you need them for another lab, you should keep them in your desk in a ziplock bag or the original box. **Shorts, short skirts and open toed shoes are not permitted, for safety reasons.** You may want to purchase nitrile gloves from the bookstore. These are recommended but not required.

MSDS INFO

You should be aware of the hazards of all chemicals used in each lab.

MSDS info is available online via any web browser on campus or from home at:
<http://ehs.mst.edu/msds/MSDS.html>

LAB NOTEBOOK

1. A 100 pg. Computation Notebook, available at the UMR bookstore, should be used. These are designed so that a carbon copy is made on the yellow pages which can be torn out and turned in to the TA on completion of the experiment. Be sure and press firmly so that the yellow copy is readable. If you have poor handwriting, you may want to print.
2. ALL entries in your notebook should be recorded in permanent ink. NO pencil.
3. Table of Contents: Two pages should be retained at the front of the notebook for the table of contents.
4. Your name: Print at the top of every page.
5. Course and Section number: Print at the top of every page.
6. Date: Print at the top of every page.
7. Prelab: This portion must be completed before you come to lab lecture. Prelabs are due when you walk in to lecture and will be considered late upon start of the lab lecture.

Title: The title should go at the top of every page related to the experiment.

Objective: Brief summary of the objective of the experiment.

Chemical Equations: (if applicable)

Physical Properties: For all chemicals used in the experiment, list in the form of a table:

| Compound name | Structure | CAS# | MWt. | State (s,l) color | BP or MP lit. °C | RI (liquids only) | Hazards |
|---------------|-----------|------|------|-------------------|------------------|-------------------|---------|
| | | | | | | | |
| | | | | | | | |

Property data can be found in the Merck index, CRC, Aldrich catalogs or online.

These reference books 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.

Prelab Questions: answer any assigned questions and turn in the prelab questions on separate paper along with the lab book.

The above items (1-7) are due before lecture and are to be turned in to the TA to be graded during lecture. Your lab book will then be handed back so that you can complete the following sections.

8. Procedure: This section must be written as you do the lab. It should include data and diagrams. It should be complete enough to allow someone to repeat the experiment and should include any procedural modifications used. (**use 3rd person past tense**)
9. Observations: Report what you witnessed concerning the reaction. Observations should especially note any unexpected observations or changes from the standard procedure.
10. Results: Results should be reported in the form of a table. You must include %yield, BP or MP, RI, physical form (s or l), color, correct identification or name of the final product, and unknown number, if applicable.

Also include literature m.p. , b.p. or RI if not included in the prelab.

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

Calculations for % yield, % error should be shown below the table of results.

11. References: Any book, manual, handbook, etc., used for the lab write up.
12. Each section (7-11) must be clearly designated.
13. Void unused space. Sign and date each page.
14. To make corrections: simply draw a line through the old data or conclusions, add the new information and initial it.
15. Each new experiment should begin on a new page.
Lab books must be initialed by the TA before leaving lab.

Upon completion of each experiment

16. The yellow pages of your notebook are to be turned in to the grader before the end of the next lab period. This will allow time for MP, yield, etc. data that may carry over from the previous week.
17. Samples/products are to be turned in upon completion of some labs. See the TA or myself for assistance.

Notebooks will not be graded if:

pencil is used, white-out is used, TA did not sign your notebook at the end of the day.

Late reports will receive only partial credit. (-5 pts / wk late, -25 pts max)

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: <http://web.mst.edu/~tbone/Subjects/TBone/scheduleFS2019.html>

GRADES

Weekly quizzes will be given after each lab lecture covering general knowledge of the experiment you will be doing that week. Quiz topics may include compound names or structures, reactions, equipment, or techniques used.

Quiz

15 pts each

Lab Notebook

10 pts Canvas prelab questions (online)

20 pts Notebook Prelab (due at beginning of lecture, see general guidelines for details)

40 pts Procedure and Observations (to be recorded while in lab)

25 pts Results

Total pts per experiment: 100 pts

Penalties (-5 each, -25 pt maximum)

Procedure not in 3rd person past tense

Improper or missing method of calculation

Missing data or calculations

Failure to void unused space

No references for properties

Results not in table form

Failure to sign and date each page

Turned in late (-5 pt/wk)

Weekly Experiments are due at the beginning of the lecture the week following the completion of the experiment.

Course

| | | |
|-------------------|----------------|-----|
| 4 experiments | 400 pts | 35% |
| Mid term exam | 100 pts | 8% |
| IR/NMR hwk | 50 pts | 4% |
| Synthesis project | 400 pts | 35% |
| Final exam | <u>200 pts</u> | 17% |

Total 1150 pts

Course grades will be based on the following percentage scale
(*some curving of raw scores may be applied*).

| | |
|--------|---|
| 90-100 | A |
| 80-89 | B |
| 70-79 | C |
| 60-69 | D |
| <60 | F |

Second Half of the Semester

After mid-semester, Chem 2229 will carry out a special project which entails a multi-step synthesis on larger scale, introduction to chemical literature retrieval. Details of the synthesis will be given after mid semester.

The experimental details for this synthesis can be found in the chemical literature. Your task is to retrieve this information and perform the synthesis.

The quantities of materials used will need to be scaled to appropriate amounts from those reported in the literature, and you will be expected to determine the percent yield, physical constants and the IR and proton NMR spectra for all compounds synthesized.

You will keep a notebook with you during laboratory practice; it will be checked weekly to assess progress (or lack of it).

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."

Disability Support Services
203 Norwood Hall
573-341-6655
dss@mst.edu

TITLE IX INFO

Missouri University of Science and Technology is committed to the safety and well-being 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.

Missouri S&T's Title IX Coordinator is Neil Outar, J.D.. Contact him directly (naoutar@mst.edu; (573) 341-6038; 203 Centennial Hall) 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 <http://titleix.mst.edu>.

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.

GENERAL LAB SUPPLIES

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.

Safety shower, eyewashes(3), fire extinguisher

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

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

Supply Cart

1.5 ml latex bulbs (reuse)

Beral pipets, (9" glass, dispose in glass waste, after rinsing)

Sample Vials, (Snap Cap, 2 ml)

Corks/Stoppers, Labels (S, M, L)

Q tips

pH paper

Aluminum foil, Parafilm

Cotton batting (insulation)

Scissors

Waste Hood -please replace the lid on the waste containers after use.

Non Halogenated Solvent Waste

Halogenated Solvent Waste (compounds containing F, Cl, Br, I)

Mineral Acid Waste-(aqueous strong acids and bases)

Solid Waste-(white plastic bucket, for contaminated filter paper, etc. -NO glass items)

Glass Waste-(cardboard box for pipets, snap cap vials, used MP cover glasses, broken glass)

Mercury Waste (broken thermometers)

Sharps Waste (syringe needles)

Wash bottles of acetone, distilled water (for cleaning glassware).

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

Mini hoods at each lab bench location should be used for all experiments. There is a flow shutoff valve on each that may be adjusted.

Spills & Breakage Cleanup There is a pan and broom, kitty litter, baking soda etc. available in the lab. Please see the TA for assistance.

Breakage Payment-Contact the TA to fill out a green slip and obtain a replacement item. A pan and broom are located by the ice machine to clean up broken glass, which should be disposed of in the glass waste box. Payment is charged to student account. Be sure to obtain a cash receipt from the TA when paying.

You must check out even if you drop the course

Failure to check out will result in a \$25.00 charge in addition to any breakage.

GROUP STATION CABINET CONTENTS

Top Shelf

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

White Plastic Tray (top shelf)

4 Beaker Tongs
4 Crucible Tongs
2 Cu Wire, heavy gauge
4 Ring Clamp, Small

Bottom Shelf

4 Aluminum Heating Block
8 Steam Bath, Cu
4 Vacuum Hoses (1/2" O.D.)
8 Water/Gas Hoses (3/8" O.D.)

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

DESK CONTENTS

Microscale Kit Components

1 Air condenser
1 Jacketed condenser
1 Claisen adapter
1 Hickman still
1 Drying tube
1 5.0 ml conical vial
2 3.0 ml conical vial
1 1.0 ml conical vial
1 0.1 ml conical vial
1 Teflon spin vane-large
1 Teflon spin vane-small
2 2 ml GC sample vial/screw cap
1 Plastic 1 ml syringe
1 Micro filter paper, 0.5 cm dia.

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
4 Beakers, 50, 100, 150, 250 ml
1 Microspatula
1 Short stem glass funnel
1 Thermometer
1 Glass stirring rod with policeman

Record your desk number and combination on something that you will be bringing back to lab.

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.

LITERATURE SOURCES FOR ORGANIC COMPOUND INFORMATION

GUIDES TO THE LITERATURE

How to Find Chemical Information: A Guide for Practicing Chemists...and Students (3rd ed.)
REF QD8.5 M34 1998 by Robert E. Maizell

Information Sources in Chemistry (4th ed.) REF QD8.5 .I47 1993 eds, R.T. Bottle, J.F.B. Rowland.

HANDBOOKS

CRC Handbook of Chemistry and Physics (80th ed.) REF QD65 .H3 1999-00

Latest edition located at the Reference Desk.

Useful source of physical property data as well as an extensive section on mathematical tables, information on sources of critical data, and rules for nomenclature of organic chemistry.

Lange's Handbook of Chemistry (15th ed.) REF TP151.H25 1999

Latest edition located at the Reference Desk. A standard reference source for chemistry.

Merck Index: An Encyclopedia of Chemicals, Drugs, and Biologicals (12th ed.) REF RS51.M4 1996

Latest edition located at the Reference Desk.(also available on CD - UMR Glass Case (1st RS51 .M4) Descriptive information on over 10,000 chemicals, drugs, and biologicals. Arranged alphabetically by generic name. Includes organic name reactions, a comprehensive cross index of synonyms, and a formula index. Also available online through library.

HANDBOOKS: Organic Chemistry

Dictionary of Organic Compounds (6th ed) v.1-7, suppl. REF QD246 .D5 1996

A seven volume set plus supplements providing concise data on many common compounds. Much less comprehensive than Beilstein's but more up-to-date information. Alphabetical arrangement and indexes by chemical name (including systematic, trivial, and trade names), molecular formula, heteroatom, and CAS Registry Number.

Handbook of data on organic compounds REF QD257.7 .H36 v.1-v.7

A seven volume set with organic compound data including solubility in various solvents and IR, NMR peak locations.

Purification of laboratory chemicals REF TP156.P83 P47 1997

Purification methods for inorganic and organic compounds.

The Organic Chem Lab Survival Manual, 8th ed. (On course reserve at library circulation desk.)

Contains info on micro and macro techniques, IR, NMR, background theory.

The Student's Lab Companion (On course reserve at library circulation desk)

Contains info on micro and macro techniques, IR, NMR, background theory.