

FS/2007
ORGANIC CHEMISTRY I LAB
CHEM 226

Instructor: Terry Bone
234 Schrenk Hall
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341-4820
<http://www.umr.edu/~tbone>
9:00-10:00, Tu-F, or by appt.

TA:	Vishal Patil	Robert Hull
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Grader: Minghang Chen
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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. Safe handling of chemicals and proper disposal of waste products are priority goals in this course. To help in attaining such goals, the first week of the semester is devoted to SAFETY in the chemistry laboratory. For the remainder of the semester, most of the experiments are conducted on a micro scale.

BOOKS:

1. CER-Chemical Education Resources, Inc., Packet of experiments
2. 100 pg. set Organic Chemistry Laboratory Notebook
3. "Microscale Techniques for the Organic Laboratory, 2nd ed", (MTOL), Mayo, Pike, Butcher and Trumper, John Wiley & Sons, Inc., 2001
4. "Right to Know pocket guide for School & University Employees", Genium Publishing Corp., 1990.

(All can be purchased from the book store)

LAB SYLLABUS:

Originally prepared by Prof. S. B. Hanna, modified by D. E. Hoiness

GENERAL GUIDELINES

SAFETY

Goggles must be worn at all times in the lab.

Unless you need them for another lab, you should keep goggles in your desk in a ziplock bag or the original box. Nitrile gloves and lab aprons are recommended but not required. Shorts or short skirts and open toed shoes are not permitted.

MSDS SIGNOFF SHEETS

These are due on entering the lab before you begin work on each lab. Turn them in to the TA. All chemicals, solvents, drying agents, etc. used in the experiment should be listed in alphabetical order. Additional blank sheets are available online through my web page, <http://web.umr.edu/~tbone>, in the 226 web link.

MSDS info is available online via web browser at <http://www.umr.edu/~msdshtml>.

Chemistry CLC's are located in rms. 120 & 305.

NOTEBOOK

1. A 100 pg. Computation Notebook, with white and yellow pages, available at the bookstore, should 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. Be sure to press firmly so that the yellow copy is readable. If you have bad 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, to be updated weekly.
4. Print at the top of every page. Your name, Course and Section number, Date, **Title** of the Experiment
5. 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.
Objective: Brief summary of the objective of the experiment.
Chemical Equations: (if applicable)
Physical Properties: For all chemicals used in the experiment, list as a table,

Compound name	Structure	CAS#	BP or MP lit. °C	RI (liquids only)	Hazards

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 234.

Reference(s) should follow the table as to the source of the property data.

Prelab Questions: answer any assigned questions and include the prelab questions and answers in the lab book.

6. **Procedure:** This section must be written as you do the lab. 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.
7. **Observations:** Report what you witnessed concerning the reaction, such as color changes, ppt, gas, etc. Observations should be written on the left page of the notebook. (back of facing yellow sheet)
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 or unknown #	Yield (gm) exp.	Yield (gm) theor.	% Yield	MP, BP, RI exp.	MP, BP, RI lit. (ref)	% Error 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 initialled by the TA before leaving the lab.**
14. To make corrections: 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 yellow pages of your notebook are to be turned in to the grader 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)
16. Samples are to be turned in upon completion of each lab. There will be a labelled box with the course name on it on top of the ice machine in the lab. Samples should be placed in 2 ml snap cap vials with a label listing:

Your Name, 226 A/B/C
 Expt. no., Date
 Compound name (or sample id. no.)
 % Yield, MP or BP

5 points will be assigned to %yield and 5 points to appearance of the sample

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.

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 instructors 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.

GRADES

Weekly quizzes will be given prior to the beginning of 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 for CER prelab questions (due at beginning of lecture)
20 pts 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 (Table(s))

Total pts per experiment: 110 pts

Penalties (-5 each, -25 pt maximum)

Improper method of calculation
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 before the end of lab the week following the completion of the experiment..

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

Course

9 experiments	765 pts	59%
10 CER prelabs	100 pts	8%
9 highest quizzes	135 pts	10%
Mid term exam	100 pts	8%
Final exam	<u>200 pts</u>	<u>15%</u>

Total 1300 pts 100%

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

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

GENERAL LAB SUPPLIES

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

Safety Items A telephone is in rm. 240, by the ice machine. Dial 4300, for campus police.

You are responsible for knowing the location of the following safety items in the lab: Mark these on your lab map, included in this handout.

Safety shower, eyewashes, safety blanket (orange), fire extinguishers

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 solid waste container in waste hood.

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

Supply Cart -by ice machine

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, Toothpicks
Filter paper (dia.(cm), grade (porosity))
Phase separation paper
pH paper
Aluminum foil, Parafilm
Cotton batting (insulation)
KimWipes
Scissors
Stopcock grease
Magnetic stir bar retriever (micro stir bars are \$8.00 ea!)

Glass Waste -box in the waste hood

Pasteur pipets -(acetone rinse before disposal)
Snap cap vials -(empty)
Broken glass
Used MP cover glasses

Waste Hood -please replace the lid on 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-(yellow dishpan, for contaminated filter paper, etc. -NO glass items)
Mercury Waste (broken thermometers)
Sharps Waste (syringe needles)
Wash bottles of acetone, CH_2Cl_2 , toluene, hexane,(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. 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 an orange flow shutoff valve on each that must be opened before use and closed after use.

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

Breakage Payment-Contact the TA to fill out a green slip and obtain a replacement item. Payment may be by cash or a check to "UMR Chemistry Dept."

Be sure to obtain a cash receipt from the TA when paying.

Bills not paid after lab checkout will incur an additional \$5.00 billing charge.

Broken glass should be disposed of in the glass waste box, located in the waste hood.

GROUP STATION CABINET CONTENTS

Shelf

- 1 Filter Flask, 250 ml
- 1 Filter Paper, 3.0 cm dia (for Hirsch Funnel)
- 1 Hirsch Funnel, 20 mm
- 1 Cover Glass for MP (Container)
- 1 Cotton (glass container)
- 1 Glass Wool, (glass container)
- 1 Litmus Paper, Blue (plastic container)
- 1 Litmus Paper, Red (plastic container)
- 2 Glass Stirring Rods
- 1 Mortar and Pestle
- 1 Test Tube Rack with 6 Test Tubes
- 1 Tweezers

Tray

- 2 Apparatus Clamps, large
- 2 Apparatus Clamps, small
- 4 Clamp Brackets
- 1 Buret Clamp
- 1 Beaker Tongs
- 1 Crucible Tongs
- 1 Cu Wire, heavy gauge
- 1 Needle-Nose Pliers (may be missing)
- 1 Ring Clamp, Small
- 3 Test Tube Clamp/Holder

Bottom

- 1 Aluminum Heating Block
- 1 Hair Dryer
- 2 Ring Stands
- 2 Steam Bath, Cu
- 2 Vacuum Hoses (1/2" O.D.-heavy wall)
- 1 Vacuum Trap
- 3 Water/Gas Hoses (3/8" O.D.-thin wall)

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

DESK CONTENTS: (checkin sheet items)

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/cap
- 1 Plastic 1 ml syringe

Extra Components

- 1 Watch glass, 3" dia
- 1 Vacuum filtering flask, 25 ml
- 1 Hirsch funnel with adapter
- 1 Casserole
- 2 Erlenmeyer flask, 50 ml/125 ml
- 3 Beakers, 50 ml, 100 ml, 150 ml
- 1 Micro spatula
- 1 Short stem glass funnel
- 1 Micro filter paper in glass vial, 0.5 cm dia
- 1 Thermometer
- 1 Glass stirring rod with policeman
- 1 Graduated Cylinder, 10 ml

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.

You must check out even if you drop the course.

Failure to check out will result in a \$25.00 fine.

Breakage bills not paid after lab checkout will incur an additional \$5.00 billing charge. See above for information on payment of breakage.

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.