

CHEM 224
ORGANIC CHEMISTRY LAB I
FS 2012

Instructor:

Terry Bone
333 Schrenk Hall
tbone@mst.edu
341-4820
Hrs: 9:00-10:00 Tu-F, or just stop by
<http://web.mst.edu/~tbone>

TA

Name: Rasika Nimkar
Office: Rm. 339 Schrenk
email: rnnmtf@mst.edu
Phone: 341-4924
341-6702

TA/Grader:

Malik Saeed
Rm. 137 Schrenk
ssnp7@mst.edu
341-6453

OBJECTIVES:

Course objectives are to develop facility with performing laboratory techniques involving the handling of organic chemicals safely and keeping proper records of experiments conducted in the laboratory. Crystallization, distillation, extraction and chromatography are emphasized as separation and purification techniques. Melting 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, Chem 224.
2. 50 pg. set Organic Chemistry Laboratory Notebook
3. OPTIONAL "Microscale Techniques for the Organic Laboratory, 2nd ed", (MTOL), Mayo, Pike, Butcher and Trumper, John Wiley & Sons, Inc., 2001
4. OPTIONAL: "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 them in your desk in a ziplock bag or the original box. Nitrile gloves and a lab apron are recommended, but not required. 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 SIGNOFF SHEETS

These are due on entering 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 may be printed from the Chem 224 lab web page. Please print some in advance. A single MSDS signoff sheet is provided at the end of this syllabus.

MSDS info is available online via web browser at <http://ehs.mst.edu/msds/MSDS.html>.

The chemistry CLC is located in room #G-34 in the basement of the new chemistry building.

NOTEBOOK

1. A 50 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 should be handed to the grader for grading during lecture. They will be considered late upon start of the lab lecture.

Objective: Brief summary of the objective of the experiment.

Chemical Equations: (if applicable, eg. a synthesis reaction)

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 333.

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

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

Graded prelabs will be handed back to the students after lab lecture.

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. (use 3rd person past tense)
7. **Observations:** Report what you witnessed concerning the reaction, such as color changes, ppt, gas, etc., especially if they differ from what you expected.
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.

Upon completion of each experiment

15. 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 in the previous period.
16. Samples may be turned in upon completion of some labs. There will be a labeled 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, 224 A or B
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.

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

MAKE-UP OF LABS:

Make up labs should be completed within one week of the original scheduled date, since 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 you 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 and structures, reactions, equipment, calculations or techniques used.

Quiz

15 pts each

Lab Notebook

10 pts for CER prelab questions

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

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

10 pts for CER postlab questions (some labs, see lab assignment schedule)

25 pts Results

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

Wrong tense in procedure (3rd person past tense reqd.)

Results not in table form

Failure to sign and date each page

Turned in late (-5 pt/wk)

Weekly Experiments: Yellow pages with completed writeups 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 56%

10 CER prelabs 100 pts 7%

7 CER postlabs 70 pts 5%

9 highest quizzes 135 pts 10%

Mid term exam 100 pts 7%

Final exam 200 pts 15%

Total 1370 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 with 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 the solid waste bucket in the 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 these)
Pasteur pipets, (9" glass, dispose in glass waste, after rinsing)
Sample Vials, (Snap Cap , 2 ml)
Corks/Stoppers
Labels (S, M, L)
Q tips
Filter paper, Phase separation paper
pH paper, litmus paper
Aluminum foil, Parafilm
Cotton batting (insulation)
KimWipes
Scissors, stapler
Stopcock grease
Magnetic stir bar retriever (micro stir bars are \$8.00 ea!)

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)
Mineral Acids Waste-(aqueous strong acids and bases)
Glass Waste (box for Pasteur pipets, snap cap vials, broken glass, MP cover glasses)
Solid Waste-(white bucket, 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 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 by the ice machine. 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 may be by cash or a check to "MS&T Chemistry Dept.". Be sure to obtain a cash receipt from the TA when paying.

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

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

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 dia
- 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 Vacuum Trap

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
- 1-Tweezers(forceps)

Bottom

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

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
- 1 Vacuum sublimation adapter
- 1 Thistle tube condenser

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 ea)
- 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 Plastic Ruler, 6"
- 1 Glass funnel, 14/10 joint
- 1 Glass syringe, 2 ml
- 1 Graduated cylinder, 10 ml

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 something 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."