CHEM 228 -ORGANIC CHEMISTRY LAB II- WS/2009

Instructor:

Terry Bone
Rm 234 ChE
tbone@mst.edu
341-4820
Office Hours: 9:00-10:00 M-F, or by appt.
http://web.mst.edu/~tbone

Teaching Assistants

Robert Hull
Rm 206 BOM #2
rvhyr8@mst.edu
341-4419

Nanditha Govindan Nair
Rm 340 ChE
nngmb2@mst.edu
341-7208

Formal Paper Grader

Kathryn Sandefur
Rm 206 BOM #2
kds@mst.edu
341-4419

Grader

Minghang Chen
Rm 206 BOM #2
mc6xb@mst.edu
341-4419

COURSE OVERVIEW:

The Winter Semester 228 Laboratory is made up of 2 parts. The first half of the semester involves a series of weekly single step syntheses and two short papers. 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 multi-step synthesis, introduction to chemical literature retrieval, and a typewritten report in the style of manuscripts submitted to the Journal of Organic Chemistry for publication. 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 228.

LAB NOTES:

Originally prepared by Prof. S. B. Hanna

BOOKS:

1) 100 pg. set Organic Chemistry Laboratory Notebook (continue from 226)
2) "Microscale Techniques for the Organic Laboratory, 2nd ed", (MTOL), Mayo, Pike, Butcher and Trumper, John Wiley & Sons, Inc.,2001
3) "Right to Know pocket guide for School & University Employees", Genium Publishing Corp., 1990. (Optional, you may still have one from chem 4)

(All can be purchased from the book store)

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 234.

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 SIGNOFF SHEETS

These are due before you begin work on each lab. All chemicals, solvents, drying agents, etc. used in the experiment should be listed in alphabetical order. Turn them in to the TA when you enter lab. Additional blank sheets may be printed from the Chem 228 web page. Please print some in advance.

MSDS info is available online in the CLC (rm. 120 ChE and via any web browser elswhere on campus or from home) at http://web.mst.edu/~msdshtml

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#	BP or MP lit. °C	RI (liquids only)	Hazards

Property data can be found in the Merck index, CRC, Aldrich catalogs or online.
These references 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 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 each lab. There will be a labeled box on top of the ice machine with the course and section name on it. See the TA for assistance.

Samples should be placed in 2 ml snap cap vials with a label listing:

Your Name,
Course(section),
Expt. no., WS/2008
Compound name (or sample id. no.)
% Yield, MP or BP

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)

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

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

Total pts per experiment: 100 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 at the beginning of the lecture the week following the completion of the experiment.

Course

4 experiments	400 pts	32%
2 reports	100 pts	8%
Mid term exam	100 pts	8%
IR/NMR hwk	50 pts	4%
Synthesis project	400 pts	32%
Final exam	200 pts	16%
Total	1250 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

TYPED SHORT REPORTS 2 required reports, 50 pts/ea.

First report, will be due one week after the oxidation of 9-Fluorenol to 9-Fluorenone experiment. The topic will be announced at a later date.

Second report, will be due one week after the Aldol condensation experiment. This short report will be defined in more detail later.

Second Half of the Semester

After mid-semester, the students enrolled in Chem 228 will carry out a special project which entails a multi-step synthesis, introduction to chemical literature retrieval, and a type written report in the style of manuscripts submitted to the Journal of Organic Chemistry as if you were publishing this work as original research. The exact nature 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, perform the synthesis and write a report.

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). At a later date, we will discuss the writing of the manuscript.

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."

GENERAL LAB SUPPLIES

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.

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

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 -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 (stir bars are $8.00 ea!)

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, 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 the 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. 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 may be by cash or a check to "MS&T Chemistry Dept.". Be sure to obtain a cash receipt from the TA when paying.

<u>You must check out even if you drop the course</u>
Failure to check out will result in a $25.00 charge in addition to any breakage.
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
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 Graduated Cylinder, 10 ml
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
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.)
1 Vacuum Trap
3 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

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 Microspatula
1 Short stem glass funnel
1 Micro filter paper, 0.5 cm dia.
1 Thermometer
1 Glass stirring rod with policeman

<u>Be sure to record your desk number and combination on something that you will be bringing to lab after check in.</u>

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, suppls. **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 compund 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.