

CHEMISTRY 213
Laboratory in Organic Chemistry
Fall 2009

Important Information:

Lab Section	001 T (11:30 AM – 2:15 PM) 002 W (2:30 AM – 5:15 PM) 004 R (11:30 AM – 2:15 PM)	003 R (8:00 – 10:45 AM)	005 R (2:30 – 5:15 PM)
Instructor	Mrs. Tracy Halmi	Dr. Michael Justik	Dr. Martin Kociolek
Office	35 Hammermill	32 Hammermill	37 Hammermill
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Web Page	chemistry.bd.psu.edu/halmi/	chemistry.bd.psu.edu/justik/	chemistry.bd.psu.edu/kociolek/
Office Hrs	M 10:10 AM – 12:05 PM W 11:15 am – 12:05 pm & by appointment	T 9:00 AM – 12:00 PM & by appointment	M 2:00 – 3:30 PM R 9:30 – 11:00 AM & by appointment

Required Materials:

Handouts: Must be downloaded from instructor's webpage prior to each new experiment.

Text: Techniques in Organic Chemistry, 2nd edition
Jerry R. Mohrig et al, W. H. Freeman and Company, 2006

Notebook: A 100 page carbon-copy spiral notebook is **required** and must be purchased through the bookstore.

Goggles: Any goggles or safety glasses with side-shields are acceptable.

Grading: The end of the semester grades will be determined from the following graded material:

Lab Reports (7 x 50 pts)	350 pts
Laboratory Procedures & Safety	50 pts
Final Exam	50 pts
Total	450 pts

The minimum grade you will receive in this course will be based on a standard scale as follows: A (100-93%), A- (92-90 %), B+ (89-87%), B (86-83%), B- (82-80%), C+(79-75%), C (74-70), D (69-60%), F (<60%).

Graded Assignments:

Lab Reports (350 pts): Lab reports will contain three parts; a Pre-lab, In-lab and Post-lab. All three should be neatly handwritten into the carbon copy notebook.

The notebook will follow the general guidelines as detailed in the text, Technique 3, pages 31 – 33. On the front cover or inside front cover, you should neatly write your name, desk number, locker combination, section number, contact information and your instructor's name. In addition, an up-to-date table of contents should be recorded on the appropriate pages in the front of the notebook.

Pre-lab: At the start of each new experiment, you must complete a short Pre-lab. The Pre-lab should follow the format given in your text with the sections listed below. Your instructor will collect them during the beginning of the lab period. Students who do not complete the Pre-lab will not be allowed to start the lab and will lose 5 points from the report grade. The Pre-lab should include the following sections:

Title of Experiment & Date
Purpose
Balanced Equation*
Data Table
Pre-lab Questions (found at the end of each experiment handout)

Each page should be clearly labeled with the title and date. The original (top) copy of this part of the notebook will be submitted at the **beginning** of each lab period. Please note, a detailed procedure is **NOT** required.

* This section will be dependent on the type of experiment. The lab handout will indicate when a balanced equation is required.

In-lab: The In-lab portion contains the specific details of the experiment performed during class. These pages should be detailed enough to have a classmate repeat the experiment using only your notebook. During the lab, describe what you do (especially any deviations from the procedure in the handout), make observations and record data in a well organized manner.

Post-lab: The Post-lab summarizes the results and provides a conclusion for the experiment. It should include:

Raw Data & Data Treatment including but not limited to raw numerical or physical data, calculations that accompany data, percent recovery or yield calculation and data tables (RI, IR, GC, MP, etc.)
Discussion of Data
Conclusion
Post-lab Questions

Post-labs will be due as indicated on the attached schedule. Lab reports will be graded on neatness, organization of material, proper calculations and quality of results. Late reports will be penalized two points per day until they are one week late, after which they will not longer be accepted.

Laboratory Procedures & Safety (50 pts): Failure to collaborate, cooperate and abide by the safety rules will result in a deduction of points, at the discretion of the instructor. Point deduction can also result from being late, disrupting the lab, inconsiderate behavior, coming to lab unprepared, incorrect or unsafe techniques, not following directions and/or improper lab procedures. Persistent absence or chronic tardiness will be heavily penalized. Students who do not checkout their desk equipment at the end of the semester will have their final grade lowered by one letter grade and be charged a \$25 fee.

Final Exam (50 pts): A comprehensive written final exam will be given. It will include theoretical and practical problems relating to the experiments and may include problems, short answer, multiple choice, true/false and some essay. The final exam will be given during finals week.

Academic Integrity: It is expected that each student will do his/her own work on all assignments including quizzes and laboratory reports. This includes but is not limited to stealing, purchasing or copying laboratory reports from present or previous students. Proven cases of academic dishonesty will result in an XF for the course. For more information see the Academic Integrity & Academic Dishonesty (Senate Policy 49-20) at <http://www.psu.edu/ufs/policies/> or Behrend's Academic Integrity policy at <http://www.pserie.psu.edu/faculty/academics/integrity.htm>.

Note to students with disabilities: Penn State welcomes students with disabilities into the University's educational programs. If you have a disability-related need for modifications or reasonable accommodations in this course, contact the Disability Specialist in the Office of Student Affairs, Room 115 Reed Union Building, 898-6111. Information can be found at <http://www.behrend.psu.edu/student/services-index.htm>.

CHEMISTRY 213
Laboratory Schedule
Fall 2009

Week of	Readings	EXPERIMENT	ASSIGNMENT DUE DATES
8/24	Techniques 1, 2, & 3	Check-in	Safety Quiz
8/31	Techniques 6, 9, & 10	Experiment #1: Recrystallization	Exp #1 Pre-lab
9/7	Techniques 11, 13, 16, & 18	Experiment #2: Distillation	Exp #2 Pre-lab
9/14	-	Experiment #2: continued	Exp #1 Post-lab
9/21	Technique 8 & 11.7	Experiment #3: Steam Distillation	Exp #3 Pre-lab
9/28	-	Experiment #3: continued	Exp #2 Post-lab
10/5	Handout	Experiment #4: Acid/Base Extraction	Exp #4 Pre-lab
10/12	-	Experiment #4: continued	Exp #3 Post-lab
10/19	Technique 15	Experiment #5: Thin-Layer Chromatography	Exp #5 Pre-lab
10/26	-	Experiment #5: continued	Exp #4 Post-lab
11/2	Technique 17	Experiment #6: Column Chromatography	Exp #6 Pre-lab
11/9	-	Experiment #6: continued	Exp #5 Post-lab
11/16	Handout	Experiment #7: Puzzle	Exp #7 Pre-lab
11/23	-	Thanksgiving Week – No Lab	-
11/30	-	Experiment #7: continued	Exp #6 Post-lab
12/7	-	Check-out	Exp #7 Post-lab
12/14	-	Final exam (Date & Time TBA)	-

Notebook Keeping Tips

Immediately following the Pre-lab section, will be the in class observations and raw data. Good note taking skills are critical to success in CHEM 213 and any laboratory situation. Listed below are some tips for keeping a good notebook.

1. Number each page and leave several pages at the front of your notebook for a title page and a table of contents.
2. Never remove pages from your notebook. If you make a mistake, correct it by crossing it out with a single line. Do not use whiteout or erasable pens.
3. The first entry in your journal for each particular experiment must be a title, date and any applicable reaction equations, followed by the remainder of the Pre-lab assignment.
4. Include the date each time you continue the same experiment on a different day.
5. Always write in blue or black permanent ink.
6. Data should be recorded directly into your notebook. Do not write data on scraps of notebook paper, weighing paper or any scrap paper in the lab.
7. It is helpful to have an appendix on the back pages of your notebook with common information. This may include boiling points, melting points, molecular weights, abbreviations, spectroscopic data, etc.
8. Always record weights, measurements, colors, smells, unusual events, spills, and any other observations. Some examples include:
 - a) ...beaker fell over on steam bath, 50% of solution was lost, yield will be low
 - b) 5 mL 3M HCl was added to the solution and pH checked. Paper turned blue (should be red) more HCl added until pH paper turned red (approx. 10 mL)
9. Use as many standard abbreviations and chemical formulas as possible. It will help you practice for CHEM 210/212 and save you time in the lab.
10. Record your data as it occurs. If you wait until the end of the class period you will not remember that actual steps you took. Very often it will deviate from the text, so the text is no longer a reliable source. You will need this detailed information for the procedure section of your final report. You do not have to write in complete sentences in your notebook.

Safety Rules

Below are some essential laboratory safety rules. They should be read immediately. Safety is no accident. All students are responsible for safety in the laboratory and must understand the following rules.

1. **ALWAYS WEAR EYE PROTECTION WHILE INSIDE A CHEMISTRY LAB.**
The requirement that you wear approved safety goggles in the laboratory is mandatory and is most effective in preventing the largest number of potential accidents of serious consequence. For their own protection, those unwilling to comply with this rule will be excluded from the laboratory. Contact lenses absorb many organic vapors and this may lead to severe eye irritation. Therefore, please try not to wear contact lenses in the lab.
2. The use of rubber gloves for all chemical manipulations is recommended. If you spill any chemical on yourself, wash the effected part immediately and thoroughly with soap and water. Washing your hands regularly is an excellent practice.
3. Chloroform, carbon tetrachloride, benzene, and p-dioxane are known or anticipated carcinogens. You may not use them in the Chemistry 213 laboratory without permission and then only in a hood. These solvents have been replaced with methylene chloride or toluene in most experiments.
4. **DO NOT USE OPEN FLAMES IN THE LAB!** If you need to light a Bunsen burner, check with the instructor first. Use heating mantles, sand baths, or steam baths!
5. Be sure you know the location of the fire extinguishers, eyewashes and safety showers.
6. Report any accident immediately to your instructor. If fire extinguishers are used, make sure you report this fact to the instructor immediately so that it can be recharged.
7. **ALL REACTIONS SHOULD BE RUN IN THE HOODS.** Many toxic and noxious chemicals will be used in the course. Remember that such solvents as ether, toluene, methanol and ethanol are highly flammable. Ether, in particular, is extremely volatile and flammable. Methanol is toxic and should never be inhaled or brought close to your eyes.
8. Noxious or highly toxic chemicals must be weighed in a hood. Transfer the weighed material to your own hood in a stoppered or corked container.
9. You may work in the laboratory only when an instructor is in the same lab supervising.
10. Nobody is allowed in the prep room in B108. Nobody may take chemicals from the prep room without permission.
11. Dress properly for work in the lab. Shorts are not permitted unless a long lab apron is worn. Open-toe sandals can not be worn in the lab. Wear old clothing and if possible use a lab coat for protection. Students will be sent home if proper clothing is not worn.

The laboratory text (Techniques 1 & 2) thoroughly covers organic lab safety, chemical disposal methods and glassware. These sections of the text should be read prior to the first experiment.

Courtesy in the Laboratory

The following guidelines will make for a safer and more pleasant laboratory atmosphere in which to work:

1. Show up on time having made the necessary preparations for the day's work. Clean up and leave promptly at the end of the three-hour period. If you anticipate a problem with timing, talk to the instructor.
2. Always replace the cap on a reagent bottle after use and **return all reagent and solvent bottles to their proper place on the side shelves or in the refrigerator immediately!** If you empty a bottle, take it to the instructor or a student assistant and inform them that it needs refilling.
3. Clean your desktop and sink before you leave the laboratory so that they will be ready for the next person who works at your bench. Be certain that no items such as litmus papers, used matches, and filter papers collect in the sink. Dispose of all trash properly.
4. When you use other parts of the laboratory, such as the reagent shelves or balances, leave them clean. If you should spill some chemical, it is your responsibility to clean it up!
5. Keep the instrument rooms clean and neat. Remove what you bring in, and properly store the instrument accessories in their proper containers or in drawers. Throw away unwanted spectra and scraps of paper. Never take stock chemicals into the instrument room!!
6. Wire the rubber hoses to an unattended running condenser so as to avoid flooding. Make sure the sink drains properly!
7. **Keep balances clean!!!!** The electronic balances are worth almost \$2000 each!! Brushes, tissues, and methanol dispensing bottles are provided to allow you to clean up spills.
8. Dispose of chemicals in the proper containers provided and properly record on waste disposal list. Do not leave unlabelled flasks and beakers lying around in the lab. There is no legal way we can dispose of unknown waste unless we pay a large penalty fee (\$1,000 per item) or identify the item ourselves.
9. If you wish to leave an experiment overnight of unattended at any time, obtain the permission of the instructor, and leave a note on the apparatus stating your name, what it is, and your home phone number. Any unlabelled reactions left out will be discarded.
10. Return all equipment that you are no longer using. We have limited supplies of these items. Someone else may be waiting to use it.
11. When you use books available in the lab, replace them in the correct place when you are finished with them. Do not remove them from the lab without signing for them.

Waste Disposal

"The entire procedure of waste disposal starts with the laboratory worker.....The laboratory worker must first decide if a material is no longer needed and is thus a candidate for disposal. Unneeded material does not become a waste until a decision is made to discard it. All reasonable possibilities for use, recovery, recycling, or reuse of the material should be considered.....Once material is declared a waste, the first responsibility for guiding its proper disposal rests with the laboratory worker. He or she is in the best position to know the degree of hazard posed by the material they have used or synthesized and must provide sufficient information to fit it into the correct channel for disposal."

Prudent Practices for Disposal of Chemicals from Laboratories, prepared by the National Research Council's Committee on Hazardous Substances in the Laboratory (National Academy of Sciences)

To help insure that all chemical waste is properly treated, the following guidelines are provided. Instructions on how to dispose of most wastes are given in the "Cleaning Up" section at the end of each experiment. If you have questions about specific cases, ask your lab instructor.

Waste Containers: On the side shelves are special collection containers for the following categories of chemical wastes. These chemicals should **NEVER** go down the sink.

1. **Nonhalogenated Organics (NHO)** - nonwater-soluble organic liquids and solids.
2. **Halogenated Organics (HO)** - all halogenated organic liquids and solids. This does **not** include inorganics such as sodium chloride (NaCl), etc. (See **Inorganic salts**.)
3. **Hazardous Metals (HM)** - Hg, Cr, Ce, Mn, Zn, etc. in any form.

The **Nonhalogenated Organics** and **Halogenated Organics** collection containers are glass five-liter bottles or five-gallon blue containers that are placed on the side shelves or the hood. **Hazardous Metals** containers are also found on the side shelves of the labs normally in a smaller glass bottle.

Each container will have a **contents manifest form** near the bottle in lab notebook. Take time to properly enter the **Chemical Name, Amount, Desk #, Date and your initials** for the materials that you have placed in the container. Failure to make the entry correctly is just as bad as dumping it down the drain and could have very costly consequences. You need only estimate the quantity, but must define it as completely as possible. An example would be:

Waste Description	Waste	Amount	Desk #	Date	Initials
Cyclohexane	NHO	~ 5 mL	45 G	1/2/99	MGK
ethanol filtrate of Cl-phenoxyacetic acid	HO	15 mL	24 B	1/5/98	MC
toluene distillation residue	NHO	~ 4 mL	27 F	1/7/99	TAH

Prudent Practices for Disposal of Chemicals states that small quantities of certain chemicals used or produced in the academic laboratory can be disposed of by flushing them down the drain with water.

Down the Sink

Inorganic acids (such as hydrochloric, nitric or sulfuric) and **inorganic bases** (such as sodium or potassium hydroxide) which do not contain hazardous metals (see below). These should be neutralized with sodium bicarbonate or hydrochloric acid and washed down the drain.

Acetone used to rinse glassware. Acetone containing more than a few percent of organic residues should be placed in the appropriate Organics disposal bottle-- Halogenated or Nonhalogenated. If used to rinse off water, it can go down the drain.

Nonhalogenated Organics that are very water soluble such as methanol, ethanol, propanol, acetic acid, acetone, etc. Modern biological sewage treatment facilities can degrade these.

Solutions of inorganic salts such as NaCl, CaCl₂, Na₂SO₄, NaNO₃, or Na₂HPO₄ which do not contain hazardous metals. Solids of this type that are wet with organic solvents should be allowed to dry in a hood before disposal in the trash bin. Aqueous solutions of these same salts can be flushed down the drain.

NOT Down the Sink, but.....

Nonhazardous Solids such as sand, alumina, silica gel, sodium sulfate, filter paper, boiling stones or sticks, etc. should be dried by spreading them on a sheet of paper or paper towel in the hood and allowing all ad- or absorbed organic solvents to evaporate. When completely dry, they can then be thrown in the **trash bin**.

Glass disposal must be made into special containers, not the trash bins!! Used Pasteur pipets, syringe needles and all other broken glassware should be disposed of in the small bins labeled "GLASS ONLY" located throughout the lab.

Recycling is the best way to handle waste materials. For a number of the experiments, you will be directed to recycle your products in appropriate labeled containers. Please reuse the Pasteur pipets as much as possible. A simple water and/or acetone rinse is usually all that is necessary to clean them.

What happens to organic waste once you properly dispose of it?

For the most part, the organics are burned, including the halogenated organics, which are mixed with nonhalogenated materials to aid their combustion. The combustion gases produced by the halogenated materials must be thoroughly scrubbed to prevent hydrohalic acid release to the atmosphere. Organic solids are dissolved in organic solvents and burned. That is why we can combine both liquid and solid organics in one bottle. It also is the reason that inorganic salts can not be put into the organic container as they are non-combustible and would clog the combustion nozzle.