

Organic Chemistry Laboratory II (CH234) Spring 2011-2012

Time/Place:

Teaching Assistant (TA):

Office: Phone:
Office hours: e-mail:

Laboratory Director: Prof. Lynne O'Connell
Office: Merkert Chemistry Center, Room 125D
Phone: 552-3626
e-mail: oconnell@bc.edu

Textbook and other required materials:

Techniques in Organic Chemistry, 3rd edition, J. R. Mohrig, C. N. Hammond and P. F. Schatz; W. H. Freeman and Co.
Organic Chemistry Laboratory Notebook, Chemical Education Resources, Inc.
Safety goggles

Final Grade: The final grade will be calculated based on the following distribution:

55% Laboratory Assignments
30% Laboratory Performance
15% Quizzes

The final grades in the course are normalized such that the average letter grade for each Teaching Assistant's section(s) is approximately a B.

Laboratory Assignments

Each student will be given a grade of (from lowest to highest) 0, 1, 2, 3, 4 or 5 for each of the individual sections of the lab assignments. Each section of the assignment will be weighted, and a final numerical grade out of 5 for the lab assignment will be calculated based on the section scores and their weighting factors. The weighting factors will differ for each lab assignment (see attached).

A note regarding plagiarism As with any written assignment, the answers and information which you present in your laboratory assignments must be expressed *in your own words*. It is unacceptable to copy material from the lab textbook, your class textbook or another student's lab assignment. If you feel a direct quote from another source is warranted, you must reference the source in your report. Taking credit for someone else's writing is not only unethical, it is illegal.

Quizzes

Four unannounced quizzes will be given to each lab section over the course of the semester which will consist of 3-5 short answer questions. The questions on the quizzes will pertain to principles and techniques that will be used in the experiment being performed that day. The quiz will be administered by the TA at the beginning of the lab period, before the pre-lab presentation. The quizzes will be graded on a scale of 0-5.

Note: No quizzes will be given during the weeks of Qualitative Analysis.

Laboratory Performance

Each student will be given a grade of 0, 1, 2, 3, 4 or 5 for each of four categories comprising the lab performance grade. Each category will be weighted according to its importance, and a final numerical grade out of 5 for lab performance will be calculated based on the category scores and their weighting factors. The TA will assign the performance grade every fourth week of the semester for a total of three performance grades. The following are the categories and their respective weightings:

Experimental technique	30%	Preparation/Understanding	30%
Dexterity		Being prepared	
Use of chemicals/reagents		Efficient use of time	
Apparatus set-up		Ability to follow instructions	
Use of instruments		Understanding concepts	
Proper recording in notebook			
Safety attitude	20%	General attitude/Independence	20%
Observance of all safety rules		Attendance	
Cleaning up spills promptly		Being on time	
Removing gloves before leaving lab		Lack of excessive dependence on others	
Lack of clutter		Respect for others	
Cleaning up at end of lab period		Patience	
		Returning equipment properly	

Laboratory Make-up Policy

Students making up a missed experiment present a hazard in the laboratory. Overcrowding in the fume hoods and the presence of incompatible chemicals in a laboratory are just two of the potential hazards. For this reason, when a student is absent for **one** laboratory period for *any* reason, s/he will not be allowed to make up the missed experiment. The weighting factors for the assignment associated with the missed experiment will be adjusted accordingly to account for the missing grades. Any quiz which might have been taken during the missed period will be considered to be a "dropped" grade. A "dropped" grade is one that is not included in the calculation of the final grade. A student who does not miss any laboratory periods will have his/her lowest quiz grade dropped before the final grade is calculated.

If a student has excused absences for **more than one** laboratory period, the necessary make-up experiment(s) and quiz(ze)s (all *except* the first missed experiment and quiz) must be arranged through the Laboratory Director. Excuses for missed laboratory periods, such as an infirmary admission slip, letter from a doctor, notice from a coach, etc., should be submitted to the Director. Arrangements for make-up experiments must be made **within one week** following the missed lab period(s).

Exceptions: All students *must* complete **Experiments 11, 13, 15, 18 and the Qualitative Analysis**. Contact the Lab Director *as soon as possible* if you miss one of these lab periods (no later than one week following the missed period).

Note: Students who elect to miss a lab period without providing an adequate reason for their absence to their Teaching Assistant will receive a significant deduction on the associated performance grade.

Notice: If at some point during the semester you decide to drop or withdraw from the lab course, you *must* return your locker key to the stockroom.

Assignments for Organic Chemistry Lab II

Weighting factor

Assignment #1

Data and observations for Expt. 11 (notebook pages)	0.2
Results from Expt. 11	
Melting point and percent yield <i>cis</i> -stilbene product	0.15
Melting point and percent yield <i>trans</i> -stilbene product	0.15
Discussion (1-2 pages)	0.5

Assignment #2

Pre-lab outlines for Expts. 12, 14 & 16	0.15
Data and observations for Expts. 12, 14 & 16 (notebook pages)	0.3
Results from Expt. 12	
Melting point, yield triphenylmethanol, explanation	0.15
Results from Expt. 14	
Melting point and percent yield acetylferrocene	0.1
TLC R_f 's and interpretation	0.15
Results from Expt. 16	
Yields of polymers and nylon comment	0.15

Assignment #3

Pre-lab outline for Expt. 13 (includes calculation)	0.2
Data and observations for Expt. 13 (notebook pages)	0.1
Results from Expt. 13	
Melting point and percent yield diene product	0.1
IR spectrum	0.1
Identification of diene	0.05
Discussion (1-2 pages)	0.45

Assignment #4

Pre-lab outline for Expt. 15	0.1
Data and observations for Expt. 15 (notebook pages)	0.15
Results from Expt. 15	
Melting point and percent yield hydrobenzoin	0.1
Melting point, percent yield and sublimation recovery camphor	0.15
IR spectrum	0.1
Discussion (1-2 pages)	0.4

Assignment #5

Pre-lab outlines and calculations for Expts. 17 & 19	0.1
Data and observations for Expts. 17 & 19 (notebook pages)	0.2
Results from Expt. 17	
Melting point and percent yield aldol product	0.1
Melting point and percent yield phenylhydrazone product	0.1
Melting point and percent yield 2,4-DNP product	0.1
Identification of aldehyde and ketone with explanation	0.1
Equations for Expt. 17	0.05
Results from Expt. 19	
Discussion of colors	0.1
Discussion of fabrics	0.1
Structure of dye	0.05

Assignment #6

Pre-lab outline for Expt. 18	0.1
Data and observations for Expt. 18 (notebook pages)	0.2
Results from Expt. 18	
Melting point and percent yield cholesteryl benzoate	0.1
Melting point and percent yield aspirin	0.1
Discussion (1-2 pages)	0.4
Expt. 20 Green chemistry	0.1

Assignment #7/8 (worth twice other assignments)

Pre-lab assignment for Qualitative Analysis.....	0.1
Physical properties liquid.....	0.1
Physical properties solid	0.1
Beilstein test liquid	0.05
Beilstein test solid	0.05
Solubility tests liquid	0.15
Solubility tests solid.....	0.15
Includes interpretation of solubility results and equations in Part 7)	
IR spectrum liquid.....	0.1
NMR spectrum solid	0.1
Includes interpretation of spectra	
Classification tests liquid	0.2
Classification tests solid.....	0.2
Includes choice of tests, interpretation of results and equations in Part 7)	
Derivative(s) liquid	0.2
Derivative(s) solid.....	0.2
Includes choice of derivatives, interpretation of results, accuracy of m.p. and equations in Part 7)	
Correct identification of liquid.....	0.15
Correct identification of solid	0.15

Schedule for Organic Chemistry Experiments for Spring 2011-2012

Week	Monday	Tuesday/Wednesday	Thursday	Friday
1/16	*Holiday*	Check-in <i>and</i> Expt. 11 Bromination	Check-in <i>and</i> Expt. 11 Bromination	Check-in <i>and</i> Expt. 11 Bromination
1/23	Check-in <i>and</i> Expt. 11 Bromination	Expt. 12 Grignard Reaction	Expt. 12 Grignard Reaction	Expt. 12 Grignard Reaction
1/30	Expt. 12 Grignard Reaction	Expt. 13 Diels-Alder	Expt. 13 Diels-Alder	Expt. 13 Diels-Alder
2/6	Expt. 13 Diels-Alder	Expt. 14 Friedel-Crafts	Expt. 14 Friedel-Crafts	Expt. 14 Friedel-Crafts
2/13	Expt. 14 Friedel-Crafts	Expt. 15 Reduction/Oxidation	Expt. 15 Reduction/Oxidation	Expt. 15 Reduction/Oxidation
2/20	Expt. 15 Reduction/Oxidation	Expt. 16 Polymers	Expt. 16 Polymers	Expt. 16 Polymers
2/27	Expt. 16 Polymers	Expt. 17 Aldehydes/Ketones	Expt. 17 Aldehydes/Ketones	Expt. 17 Aldehydes/Ketones
3/5	*Break*	*Break*	*Break*	*Break*
3/12	Expt. 17 Aldehydes/Ketones	Expt. 18 Esters	Expt. 18 Esters	Expt. 18 Esters
3/19	Expt. 18 Esters	Expt. 19 Azo Dyes	Expt. 19 Azo Dyes	Expt. 19 Azo Dyes
3/26	Expt. 19 Azo Dyes	Expt. 20 Green Chemistry	Expt. 20 Green Chemistry	Expt. 20 Green Chemistry
4/2	Qualitative Analysis (start)	No lab	*Holiday*	*Holiday*
4/9	*Holiday*	Qualitative Analysis (start)	Qualitative Analysis (start)	Qualitative Analysis (start)
4/16	*Holiday*	Qualitative Analysis (finish)	Qualitative Analysis (finish)	Qualitative Analysis (finish)
4/23	Qualitative Analysis (finish)	Luminol <i>and</i> Check-out	Luminol <i>and</i> Check-out	Luminol <i>and</i> Check-out
4/30	Expt. 20 Green Chemistry <i>and</i> Check-out	No lab	No lab	*study day*

In the organic chemistry laboratory course, you will learn how to synthesize and purify a wide variety of organic compounds. You will perform many of the reactions that you will study in class. The semester will culminate with a Qualitative Analysis project for which you will collect physical, chemical and spectroscopic data in order to identify two unknown compounds.

Source Material

Two sources of material will be used throughout the course. The instructions for the experiments that you will perform, along with background information for each and guidelines for writing the lab assignments, are found at the following web site:

www.bc.edu/schools/cas/chemistry/undergrad/labcourses.html

The experiments are available in PDF format.

You will also use the book *Techniques in Organic Chemistry* by J. R. Mohrig et al., published by W. H. Freeman and Co. Chapters 1-19 describe in detail the lab techniques necessary for carrying out organic experiments. A companion website for the book contains movies that demonstrate each lab technique. To view them, go to:

bcs.whfreeman.com/mohrig2e/
and click on the link for "Techniques in Organic Chemistry Movies and Spectra".

The instructions for the experiments found at the lab course web site will often refer you to a section of *Techniques in Organic Chemistry* using the acronym "TOC". You must read these sections carefully and view the corresponding movie before coming to lab. You should also bring this book to lab to use as a reference source during the lab period. Before you begin any work in the laboratory, you should read pages 1-48 in *Techniques in Organic Chemistry*.

Pre-Lab Exercise

For each experiment, you must outline the steps of the **Procedure**, found in the instructions for the experiment at the web site, *in your own words*. You will carry out the procedure following the outline that you have prepared. You will **not** be allowed to bring a printed copy of the page(s) of the **Procedure** into the laboratory with you. Diagrams of apparatus set-ups have been included at the web site, and you *will* be allowed to print out and bring these pages with you. You can bring *Techniques in Organic Chemistry* into the laboratory, also.

In addition to the outline, you must:

- List all the safety hazards associated with the chemicals and procedures of the experiment.
- Calculate the number of moles of each reactant used. What is the limiting reagent (if applicable)?
- Calculate the theoretical yield (if applicable).

Laboratory Notebook

You will be keeping a detailed record of your laboratory work in a laboratory notebook. Use a notebook that contains perforated graph paper (*Organic Chemistry Laboratory Notebook*, available in the bookstore, is a good choice). If the pages of the notebook which you purchase are not made of carbonless copying paper, you must purchase a packet of carbon paper. When you write in your notebook, you will be making a carbon copy of your data and observations as you record them. At the end of the lab period, your TA will initial your notebook and tear out and collect the carbon copies of the pages you used that day. You will then be able to take your notebook with you after the lab period is over to refer to as you write your lab assignment.

The general procedure for using the notebook is as follows:

- Write your name, lab period, lab instructor's name and locker number on the cover.
- Keep an up-to-date Table of Contents at the front of the notebook by entering each experiment when it is completed.
- Put the title of the experiment and the date at the top of **each** page.
- Use **ink** and enter all data and observations **directly** into the notebook. **Never** write data and observations on a scrap piece of paper to copy it neatly into the notebook at a later date. The notebook must be the **original** record of your work. Do not trust data to memory. Write it down immediately.
- Observations of physical and chemical changes should be recorded immediately. Describe what happens, how things look, any changes that take place, etc. A seemingly insignificant observation could be of crucial importance later. In addition, record any procedural changes, mistakes or difficulties that might affect the results of the experiment. Be alert for sources of error and limitations in the experiment.
- The assignments will be written by referring to your lab notebook while you are away from the lab; therefore, it is vital that you record **all** essential information in your notebook. Within reasonable limits, both it and the carbon copies should be legible.
- Mistakes should not be erased or covered with "white-out". If you make an error, draw a single line through the incorrect number, word or statement and write the correct entry beside it. A brief explanation for the change should be written in parentheses. The original value must remain legible, for you may later decide to use it.
- Use tabular form to record data whenever appropriate. The table should be planned before coming to lab. Always record data with the correct number of significant figures and the appropriate units. A numerical value is incomplete without the units.
- Begin each experiment on a new page. Never skip pages to be filled in later.
- All experimental data and results bearing your name must be your work and yours alone.
- At the end of each laboratory period, the laboratory TA **must** sign your notebook entries for the day and collect the carbon copies.

Laboratory Assignments

The laboratory assignments will consist of several different parts, some of which are handed in at the end of the lab period and some of which are handed in a week or more after the experiment is completed. Include your name, the name of your laboratory TA, the experiment number and title of the experiment, and the date in the heading of any document that you hand in. The parts of the assignments are as follows:

Pre-lab procedure: This is handed in to your TA at the end of the laboratory period, after the experiment is completed.

Data and observations: This section consists of the carbon copies from your laboratory notebook which are handed in to your TA at the end of the laboratory period. Observations should be detailed and data should include correct units and significant figures.

Results and Calculations: This section is handed in a week or more after the experiment is completed. Perform any calculations asked for on the last page of the experiment instructions. Show one sample for each calculation performed. Construct necessary tables, graphs, figures, etc. Be sure to use correct units and significant figures.

Discussion: Discuss the objectives of the experiment, what happened, what principles were illustrated and what conclusions can be drawn. Relate the observations from your notebook to what happened during the experiment at the molecular level. This will include writing out balanced chemical equations for any reactions (including side-reactions) that were carried out. Discuss what errors may have occurred in the experiment and their effect on your results. More explicit instructions for the discussions can be found at the website under the **Assignment** links. ***Be sure you read these links!*** Some assignments will include a discussion and others will not.

Interpretation: Assignments that do not include a discussion may have an additional section which asks you to interpret your data and draw conclusions based on your experimental results.

Safety Rules of Boston College

- 1) Wear approved eye protection in the laboratory **continuously**, even when not performing an experiment. This means eye covering which will protect both against impact and splashes. This is a state law. Do **NOT** wear contact lenses in the laboratory. If you wear prescription glasses in the lab, you must wear goggles. If you should get a chemical in your eye, wash it with flowing water from the eye wash for 15 minutes.
- 2) Perform no unauthorized experiments. Horseplay, pranks and other acts of mischief will not be tolerated. No visitors are allowed in the laboratory.
- 3) To prevent fire, keep flammable substances away from open flames, heat (this includes hot plates) and sparks. Do not place gas burners near wooden surfaces or compressed gas cylinders. In case of fire or accident, call the instructor at once. Note location of safety shower and fire blanket now so that you can use them if needed. If the fire alarm sounds, leave the building **immediately**. *Do not use the elevator.*
- 4) You must go to the Infirmary, accompanied by someone, for treatment of cuts, burns or inhalation of fumes. Your instructor will arrange for transportation if needed. Any personal injury, regardless of how minor, must be reported.
- 5) Eating *and* drinking in the laboratory are absolutely prohibited! All food and drink should be stored in the lockers before entering the lab. Smoking and the application of cosmetics are also not allowed. Do not touch your mouth with your hands while you are in the laboratory, and wash your hands well before leaving. Never taste a chemical.
- 6) Exercise great care in noting the odor of fumes, and avoid breathing fumes of any kind. Keep chemicals in the fume hood whenever indicated.
- 7) **NEVER** use mouth suction to fill pipets with chemical reagents. Use a suction bulb or other pipeting device.
- 8) Do not force glass tubing into rubber stoppers. Lubricate with glycerin or water and protect your hands with a towel.
- 9) Confine long hair and loose clothing when in the laboratory. Do **NOT** wear: nylon stockings or high heels. It is recommended that you avoid wearing short sleeve shirts, shorts and skirts. Shoes must be close-toed and must cover the majority of the foot. A laboratory apron is essential. It is fire resistant and will protect you and your clothing from corrosive chemicals. Wear gloves when working with corrosive or toxic chemicals. Gloves *must* be removed if you need to leave the laboratory for *any* reason (e.g., to go to the stockroom). The apron must be removed if you go to the restroom.
- 10) **Never** work in the laboratory alone or without the presence of a supervising instructor.
- 11) Never leave heated laboratory reactions unattended. Beware of hot glass. It cools slowly and is identical in appearance to cold glass. Always use the proper equipment when handling hot glass (tongs, insulated gloves, etc.).
- 12) Clean-up broken glass immediately with a dust pan and brush, and dispose of it in the proper waste box. Handle broken glassware with a towel or thick gloves. Glass tubing and rods must be fire polished. Do not subject a flask to sudden changes in temperature.
- 13) Keep your work space uncluttered. Do not place coats, purses, or backpacks on the laboratory bench or in the aisles— use the lockers located on the loading dock to store these

items. Keep drawers and cabinet doors closed while working. Do not place chemicals or equipment on the floor. Do not leave pipets sticking out of bottles, flasks or beakers.

- 14) Assemble all required materials before beginning a task. Place glassware and equipment well back from the front edge of the lab bench.
- 15) Mercury is toxic. Call your instructor whenever a mercury thermometer is broken so that he/she can take charge of the clean-up.
- 16) Dispose of chemical wastes only as instructed. **Never** mix acid waste with organic waste.
- 17) Clean up all chemical spills at once. Acid spills should be neutralized with sodium bicarbonate. Plenty of running water is the best first-aid treatment for all chemical spills to the skin, clothing and lab bench. If a large amount of corrosive material is spilled on your person, rinse it off under the safety shower, and notify your instructor. Do not "dry sweep" spilled solid material. Cover the solid with wet paper towels, and pick up the material. If the material is hazardous, dispose of the towels as a hazardous waste. Clean the area with soap and water.
- 18) Always add acid to water; **never** water to acid. Mix slowly. Use care when performing acid-base neutralization reactions. They can generate heat and bubbling.
- 19) Avoid slipping hazards by picking up ice, stoppers, glass rods and other small objects from the floor.
- 20) Radios are a distraction and therefore are not allowed in the undergraduate labs.
- 21) Do not handle electrical equipment with wet hands, while standing in water or leaning against a metal object or pipeline. Keep cords away from cabinets or drawers.
- 22) Never remove chemicals from the laboratory. If you see someone on the elevator in Merkert who is transporting chemicals, do not get on the elevator with them. Take the stairs instead.
- 23) Be alert to unsafe conditions, and report them to the supervisor so that corrective action will be taken.
- 24) Clean up your lab bench area at the end of the lab period. Do not leave contaminated or dirty glassware or tools in the work area.

Prevent accidents by using **common sense** and being **well-informed**. Read each experiment thoroughly before coming to the laboratory and follow directions carefully. Your instructor will inform you weekly of specific safety hazards in each experiment. Pay strict attention to these warnings in addition to the above rules.

Stockroom Procedures and Regulations

Keys

You will be issued a key for your laboratory locker by the stockroom personnel. It is important that you remember to bring your key with you to lab as a forgotten key will result in unnecessary delays in starting your experiment. If you forget your key more than two times during the semester, your performance grade will be affected.

Replacing broken or missing equipment

If you break or lose a piece of equipment, you must report it to the stockroom attendant. You will be given a replacement for the missing item from the stockroom after you have filled out a requisition form. **Note:** Remove your gloves before going to the stockroom window.

Equipment on loan

Frequently it is necessary that special equipment be loaned to students in order for them to do the scheduled experiment. This equipment **must** be returned clean and in good condition **immediately** after use, so that all the students in the next lab section will be able to perform the experiment. You will be checked off the sign-out sheet when you have returned the equipment and will be held responsible for anything that is not returned.

Broken glassware

Never use broken glassware. Dispose of it immediately into the "Broken Glass" box located in each lab. **Never** place glass into the waste baskets, even Pasteur pipets or capillary tubes. The following glassware is not to be disposed of but should be brought to the stockroom to get a replacement:

- chipped glassware that can be repaired
- pipets or volumetric flasks broken near the mouth
- ground glass joints
- buret barrels
- Teflon stopcocks
- glassware that is difficult to clean
- thermometers

Broken mercury thermometers

Mercury is toxic; consequently, a broken mercury thermometer should be reported to your instructor **immediately**. No replacement will be issued until the stockroom has been informed by your instructor that the spill has been cleaned up properly. Do **not** throw any part of the thermometer in the Broken Glass box!

Defective equipment

If you have any defective glassware or equipment, bring it to your TA or the stockroom, where it will be either repaired or replaced.

If you have any problems with your lock, drawer, sink, faucet, gas, steam or vacuum outlets, put these repair requests in writing. State specifically what the problem is, the room number and where it is located in the room.

Support rooms

Instrumentation, such as balances, gas chromatographs and the infrared spectrometer, can be found in the support room of the laboratory. Some lockers which are located in the support room contain equipment that is used by other students. After using this common equipment, please put it *neatly back exactly* where it belongs so that it will be readily available for use by the next student. **Do not place this equipment in your own drawer!** No one should have to waste time trying to find common equipment if everyone cooperates.

Safety glasses

Each student must purchase a pair of Uvex Stealth™ Safety Goggles which meet American National Standards Institute (ANSI) approval. These are available in the BC bookstore. Once you bring your goggles to lab, you will find it most convenient just to leave them in your drawer. Write your name on the strap. Remember, eye protection must be worn at all times.

To remove any dirt from the goggles, use a cold water rinse. Do not rub grit across the surface of the lens with your fingertips or nails. To remove grease or oil, use soap and water. Do not use acetone as it will damage the plastic.

If you wear prescription eye glasses, your safety goggles must be worn over your glasses. Ordinary glasses alone are not approved. If the Uvex goggles do not fit around your glasses, consult your TA. Contact lenses should not be worn in the laboratory even under approved safety goggles.

Aprons and gloves

Each student drawer is equipped with a vinyl laboratory apron which must be worn when working in the laboratory. You will also be issued a pair of nitrile gloves of the appropriate size during the first lab period.

Housekeeping

Many of the chemicals used in the labs are corrosive. This means that upon contact, they will destroy or damage not only your skin, but also the bench tops and some equipment. **Clean spills immediately!**

Every student is responsible for cleaning up their own work area before leaving lab. This includes:

- bench top
- fume hood
- sink and sink area

Show consideration for others and help keep the community areas clean, too. It is not part of the custodian's daily job to clean anything except the floors in these labs.

Vials or test tubes that contain chemicals (such as student products or unknowns) should **never** be discarded in the waste baskets or broken glass boxes. Ask your TA for a correctly labeled **Laboratory Byproducts** jar.

Before leaving the laboratory, **make sure the water and gas are completely turned off.** Allow the hot plate to cool as much as possible before returning it to the support room locker. Do not forget to lock your drawer and return any special equipment.