ORGANIC CHEMISTRY LAB I Spring 2017 Syllabus

Course & Instructor Information:

Lab Instructor/Section:

Office: Email:

Office Hours:

Lab Coordinator: Prof. Guillermo Gerona-Navarro

Office: 2146 Ingersoll

Email: ggerona@brooklyn.cuny.edu

Office Hours: Wed/Th 11:30am-12:30pm & by Appt

Statement of Course Goals:

The goal of the laboratory component of Organic Chemistry I is to introduce students to fundamental concepts used by organic chemists in the lab. By the end of the semester, a successful student will have acquired a basic fundamental understanding of important laboratory techniques critical to organic chemists, and will be able to carry out reactions from set-up to purification, to data analysis.

Required Purchases:

- 1. Organic Chemistry I Lab Manual, 2016 edition (available at Brooklyn College bookstore)
- 2. Composition Notebook
- 3. Lock for Lab Drawer
- 4. Paper Towels
- 5. Soap

Laboratory Safety:

The primary goal for the instructors of this laboratory is for each and every person to safely make it through the semester. Never endanger the safety of yourself or others for any reason. Your instructor will discuss detailed safety policies before every lab session. However, the items presented bellow are so important that they will be stressed from the outset:

- Safety goggles (provided) and lab coat (provided) must be worn in the lab at all times. They must be worn from the second you walk in until the second you leave, even if you are not doing chemistry. If you are caught not wearing either of them in the lab, you will be asked to leave and you won't be allowed back for that session.
- No shorts or open-toed shoes are permitted in the laboratory. You will be immediately sent

home if you enter the lab wearing shorts, sandals, flip-flops, open-toed shoes, etc.

- No food or drinks are allowed in the laboratory. No cell phone use is allowed in the laboratory.
- You should wear gloves anytime you are working in the lab. However, students are not allowed to wear gloves outside of the laboratory. The moment you are leaving the lab, even if it is to go to the stock room, you should take your gloves off.

Fume Hood Safety and Energy Conservation

Fume hoods protect lab workers from inhaling chemicals by constantly pulling air into the hood and exhausting it out of the building. However, they will only protect you if operated properly. A lowered sash provides much higher protection against contamination or injury. Because they constantly pull air from the room when their sashes are open, fume hoods use an enormous amount of energy. A single open fume hood can use as much energy in a year as 3.5 houses! By simply closing the sash when you're not using the hood, you can reduce energy consumption by 60%, or about \$3000 a year. Here are the basic instructions:

- Use the fume hood for all chemical manipulations.
- Keep everything at least 6 inches back from the hood face.
- Don't lean into the hood or put your head into it when contaminants are present.
- Try to keep the sash lower than the level of the arrow sticker. The lower the sash, the greater the energy savings.
- Close the sash entirely when you're finished with your work.

Chemicals

If you spill anything on your skin, immediately wash the area with lots of water, and notify your instructor asap. If you spill a chemical all over your hood or the floor, tell your instructor, who will help you clean it up. If you spill a large amount of chemicals or a particularly dangerous chemical on your body and/or your clothes, you may need to use a safety shower. Get your instructor attention by yelling and proceed directly to a safety shower and pull the chain. Remove the affected clothing (trust us, modesty should be the last thing on your mind if you need the safety shower). The instructor will direct other student's attention away from you and provide a blanket and a lab jacket for you when the rinsing is done.

Wash your hands thoroughly before you leave the lab. There is soap at each sink.

Keep the lab clean. If everyone cleans up after themselves, the lab will be safer and easier to work in. The TFs are authorized to assign students to clean up certain areas of the lab. Put waste in the appropriate waste containers. Material Safety Data Sheets (MSDS's) are available for every chemical that you will be using in the lab. MSDS's are intended to give toxicity information, safe handling, and information on what to do in case of exposure. In general, it is often difficult to ascertain how harmful chemicals actually are. Err on the side of caution. Your limited exposure is well under the suggested dosage limits if you use safety procedures. If you have any questions about the chemicals you're working with, ask your instructor. The lab was designed, and the procedures written, so that your contact with hazardous chemicals is minimized. You simply can't do chemistry without chemicals. You can, however, think about what you are doing and stay safe.

Glassware

Be aware of the dangers of handling glassware. The most common laboratory injury is cuts received from a piece of broken glass. Be especially careful when connecting pieces of glassware together and taking them apart. *Don't force anything*. If you can't get two pieces of glassware together or apart, ask your instructor for help.

Injuries

If you are injured, no matter how small the injury, tell your instructor. In the case of minor injuries (small cuts, minor burns, splinters, etc.) first-aid can be given on-site with laboratory first-aid kits. In the case of anything worse than a minor injury, notify your instructor immediately so he/she can look for professional help. Never leave for treatment without notifying your instructor. After treatment of any injury, you will be asked if you wish to continue that day. You will not be penalized if you are unable to finish that day, even if it's because you simply feel uncomfortable returning to the bench.

Fires and Serious Chemical Spills

The dangers from fire and spills can be minimized and effectively handled by observing the following procedures.

Think! Common sense should prevent most fires and spills. Is it a good idea to heat flammable solvents on a hot plate? (NO!) If a fire does occur, immediately step away from it and notify your instructor. Most fires occur inside of a fume hood and will burn themselves out on their own. You are not responsible for putting out a fire.

Notify your instructor of all spills. Small spills are easily contained, and your instructor can tell you how to deal with them. If the spill looks large or is of something particularly nasty, notify your instructor and get out of the area.

Evacuation

If the fire alarm goes off, immediately walk out of the lab and exit the building. If an instructor tells you to get out of the lab or a certain area, do it!. Dedication is a valuable asset to have in the laboratory, but we would much prefer to have you safe than finished ten minutes early.

During the first laboratory period, you will receive 2 copies of a hand-out of safety rules. One is for you to keep and the other one is for you to sign and return to your lab instructor. You must read, understand and agree to abide by these rules if you want to take the course.

You must not run heating devices (e.g. hotplates, heating mantles and melting point devices) more than the halfway up their temperature setting unless instructed otherwise by your instructor. Doing otherwise can cause thermometers and other glassware to crack and break and put you at risk of injury.

Exposure to Chemicals:

There is scientific evidence that exposure to laboratory chemicals (especially volatile solvents) during pregnancy increases the risk of birth defects. Any student who has sensitivity toward chemicals or who may be pregnant is strongly advised to check with his or her doctor to determine if taking this course may pose a hazard to his or her health. A list of chemicals to be used in the laboratory experiments will be made available upon request.

Pre-lab reports (written before the lab)

Before you come to lab, carefully read through the assigned experiment and complete your Pre-Lab report. These Pre-Lab reports are designed to ask you to think about the lab procedure to be performed, understand how it relates to other aspects of chemistry, and guide you in your preparation for the experiment. You may need to refer to your lecture text to help you answer some of the questions. Don't wait until just before lab to get prepared – instead, work on your Pre-Lab ahead of time so you can ask your instructor for help if you are confused about anything.

You must turn your pre-lab assignment in to your instructor at the beginning of the lab period. No one will be permitted to do the lab without a completed pre-lab report. Your instructor will ask you to leave the lab if you don't turn you pre-lab assignment or if the pre-lab is graded as "Failed". Additionally, you will find that the labs will go much smoother if you have read through everything ahead of time, so be sure to do a good job in getting organized.

Please don't sit in the hallway outside the lab and copy the pre-lab from your classmates. It just makes you look extremely unprepared and not very serious about your coursework. Copying a pre-lab assignment from a classmate will be considered cheating, therefore, both parties involved in the plagiarism will receive a grade of "zero" for that laboratory session.

A complete pre-lab consists of:

A. *Title (1 pt)*: Preface each experiment with a descriptive title, your name, the date, your instructor's name, and the day and time of your lab section.

B. *Purpose* (2 pt): Write a brief statement outlining your experimental objective(s). If you are using a technique, i.e distillation, crystallization, etc, describe with your own words the principle of the technique to be used (5 sentences maximum)

C+D+E (9 pts)

- C. Balanced chemical equation and Theoretical Yield (if applicable).

 Present the balanced chemical equation and calculate the theoretical yield for all reactions, showing the appropriate math.
- D. Reagent Table:

List all chemicals to be used, including solvents. List any hazards associated with each chemical. When a reaction is run, give the quantity of each reagent in grams or mL, along with MW, density and moles.

E. Mechanism (if applicable):

Write clear stepwise mechanisms for all synthetic transformations, showing important intermediates where appropriate.

F. Flowchart of Procedure (8 pts)

Provide a diagram or scheme of the procedural steps you will be conducting (see page p. 23 of your lab manual for an example of a flowchart). Be sure to include specific information such as chemical names, quantities, temperatures, time frames, etc. You should not transcribe the manual word for word; sketch it out, make notes to yourself, and use pictures and abbreviations. You should also write a list of glassware and chemicals you'll need for the experiment, and draw your glassware setup(s).

Post-lab reports

Post-labs are due one week after completion of an experiment and are due promptly at the start of lab, unless stated otherwise in the handouts or by your instructor. Late reports will not be accepted (late lab reports equal a "0" for the report).

A complete post-lab report consists of:

A. *Title*: Preface each experiment with a descriptive title, your name, the date, your instructor's

name, and the day and time of your lab section.

- B. Abstract (3 pts): Write a brief abstract about the experiment and your findings (150 words maximum)
- C. Observations (5 pts).

List 3 important physical observations you made. Briefly state why each is significant. Physical observations are things you see or feel. They are not conclusions or interpretations. A stepwise sequence of short phrases is often sufficient for this purpose.

D. Results (5 pts):

D.1 Product data. List any data that you have obtained (e.g. melting point, boiling point, yield) and report % recovery or % yield when applicable. Show graphs of data where applicable.

D.2 Interpretation and explanation of results obtained in your experiment, including spectral data (if relevant). Include an analysis of the likely sources of error in your experiment (human and instrumental).

E. Conclusion (3 pts):

Brief concluding remarks about the experiment.

F. Post-lab Questions (4 pts)

Answer the designated postlab question (see below) concisely, but completely. Your answer should typically be about one to two paragraphs long

Laboratory Notebook

Your notebook is a tool to help you prepare for the experiment and to record your observations during the lab period. Ever experiment should start in a new page. You should prepare the pre-lab repot in your notebook and hand a copy of it to your instructor as you enter every lab session. Any observations you make and any numerical data you collect should be recorded directly into your notebook in pen. (Data should never be written on scrap paper and later transferred to a notebook.). Notebooks should be legible so that another person can follow what you have done; however they need not look perfect. Errors should not be erased or covered over with whiteout, but rather should be crossed out neatly so that the original error is still visible.

At the end of the semester you will turn your notebook in to your instructor so it can be graded.

Grade Breakdown:

Laboratory Reports (Prelab +Postlab) - 40%

- Pre-lab 20%
- Post-Lab 20%

Safety Assignment - 5%

Laboratory Notebook - 5%

Instructor Evaluation - 20%*

- Coming to class on time 5%
- Performance in the laboratory (preparation, execution, discipline) 10%
- Maintaining/Leaving the work space tidy (including the front bench and hoods) 5%

Final Exam - 30% **

One common final exam will be administered to all laboratory sections on **Thursday May 18 at 9:30AM. The final exam will be cumulative. It will test students' knowledge of the techniques covered (both theoretical and practical aspects) as well as their ability to analyze and interpret data. A copy of previous semesters' lab finals can be found on Horowitz's website.

COURSE RULES & REGULATIONS

Academic Integrity:

Academic dishonesty of any type, including cheating and plagiarism, is unacceptable at Brooklyn College. Cheating is any misrepresentation in academic work. Plagiarism is the representation of another person's work, words, or ideas as your own. Students should consult the Brooklyn College Student Handbook for a fuller, more specific discussion of related academic integrity standards. Academic dishonesty is punishable by failure of the "test, examination, term paper, or other assignment on which cheating occurred" (Faculty Council, May 18, 1954). In addition, disciplinary proceedings in cases of academic dishonesty may result in penalties of admonition, warning, censure, disciplinary probation, restitution, suspension, expulsion, complaint to civil authorities, or ejection. (Adopted by Policy Council, May 8, 1991.)

Students with Disabilities:

If you have a disability, it is the responsibility of the university to provide you with reasonable accommodations. You should first register with Ms. Stewart-Lovell, the Director of the Student Disability Services Center (718-951-5538). Afterward, please provide the laboratory coordinator, Dr. Gerona-Navarro, with a copy of your course accommodation form and if necessary schedule an appointment with him to discuss your specific accommodation needs.

Laboratory Drawers:

Please follow the check-in and check-out instructions given by the senior college laboratory technician, Ms. Anna Belyayeva. Make sure that you clean your glassware and bench space everyday (with solvent if necessary) and that you return all your glassware and equipment to your laboratory drawer before you leave. **Make sure you lock your drawer at the end of every lab period.** At the end of the semester, you will be charged for the replacement cost of any missing or broken items.

Note that once you check in to the course, you **must check out** even if you only attend class for one day. Students who fail to check out will be charged a fee of **\$50** plus the cost missing or broken equipment. Students who drop should arrange a checkout day with the stockroom. Students who withdraw will checkout on the day designated for checkout for their lab section.

Makeups:

Students who must miss lab due to extreme circumstances beyond their control may submit a Request for Excused Absence. Please understand that personal issues with scheduling conflicts, such as work, non-emergency dentist or doctor appointments, extracurricular activities, family vacations, etc., do not justify an excused absence. The lab must be made up before the last date of the experiment listed under "Experiment Schedule". Under no circumstances will a make-up lab be permitted once the last date for that experiment has past.

Excused absences will be approved by your lab instructor. To make-up a lab, please, pickup a makeup form from the stockroom and ask your instructor to sign it. Then, schedule and arrange your makeup date by contacting the instructor teaching the makeup section (see contact information below). After you complete the makeup experiment, please have the makeup instructor sign your makeup form and return it to the stock room. A request for excused absence due to religious holidays and/or valid school activities (sports meet etc) must be submitted at least two weeks in advance, otherwise it will be rejected.

If you don't manage to make up your lab after you were granted an excused absence, your instructor will not count that lab for the final grade after approval from the Organic Chemistry coordinator (Prof Guillermo Gerona-Navarro, ggerona@brooklyn,cuny.edu). In order to get the approval you must justify your absence and clearly explain the reasons why you did not manage to make up your missing session.

Prof. Abeykoon <u>nwalalawela@brooklyn.cuny.edu</u>

Prof. Berkowitz Alex.Berkowitz@brooklyn.cuny.edu

Prof. Khajo Khajo@brooklyn.cuny.edu

Prof. LeRoi gdewaeleleroi@gradcenter.cuny.edu Prof. Zhang gan.zhang89@brooklyn.cuny.edu Prof Cordone pcordone@gradcenter.cuny.edu

Administrative Dates

Sunday 2/5 - Last day to add a course

Monday 2/13 - Lincoln's Birthday, College Closed

Wednesday 2/15 - Conversion Day, Classes Follow a Monday Schedule

Sunday 2/19 - Last day to drop a course without the grade of W

Monday 2/20 - President's Day, College is Closed

Monday 2/20 - First day to apply for a W grade (Course withdrawal period begins)

Monday-Tuesday 4/10-4/18 - Spring Recess

Wednesday 4/19 - Course withdrawal period ends

Thursday 4/20 - Conversion Day, Classes Follow a Monday Schedule

Thursday 5/18 - Last Day of Undergraduate Weekday Courses

Friday 5/19 - Reading Day

As per our department policy students dropping the lecture must also drop the lab

LABORATORY SCHEDULE

Week	Berkowitz MQ9A (Mon AM)	Khajo MQ9B (Mon AM)	LeRoi MQ2 (Mon PM)	TQ2 (Tues PM)	Zhang WQ2 (Wed PM)	Abeykoon FQ9 (Fri AM)
1	1/30, Lab 1	1/30, Lab 1	1/30, Lab 1	1/31, Lab 1	2/1, Lab 1	2/3, Lab 1
2	2/6, Lab 2	2/6, Lab 2	2/6, Lab 2	2/7, Lab 2	2/8, Lab 2	2/10, Lab 2
3	Wed, 2/15 Lab 3	Wed 2/15 <i>Lab 3</i>	Wed 2/15 <i>Lab 3</i>	2/14, Lab 3	-	2/17, Lab 3
4	-	-	-	2/21 Lab 4	2/22, Lab 3	2/24, Lab 4
5	2/27, Lab 4	2/27, Lab 4	2/27, Lab 4	2/28, Lab 5	3/1, Lab 4	3/3, Lab 5
6*	3/6* , Lab 6	3/6, Lab 5	3/6* , Lab 6	3/7*, Lab 6	3/8* , Lab 6	3/10*, <i>Lab</i> 6
7	3/13, Lab 5	3/13* , Lab 6	3/13, Lab 5	3/14, Lab 7	3/15, Lab 5	3/17, Lab 7
8	3/20, Lab 7	3/20, Lab 7	3/20, Lab 7	3/21, Lab 8	3/22, Lab 7	3/24, Lab 8
9	3/27, Lab 8	3/27, Lab 8	3/27, Lab 8	3/28, Lab 9	3/29, Lab 8	3/31, Lab 9
10	4/3, Lab 9	4/3, Lab 9	4/3, Lab 9	4/4, Lab 10	4/5, Lab 9	4/7, Lab 10
11	-	-	-	-	-	-
12	Th, 4/20, <i>Lab</i> 10	Th, 4/20 <i>Lab</i> 10	Th, 4/20 Lab 10	-	4/19, Lab 10	4/21, Lab 11
13	4/24, Lab 11	4/24, Lab 11	4/24, Lab 11	4/25, Lab 11	4/26, Lab 11	4/28, Lab 12
14	5/1, Lab 12	5/1, Lab 12	5/1, Lab 12	5/2, Lab 12	5/3, Lab 12	5/5, Lab 13
15	5/8, Lab 13	5/8, Lab 13	5/8, Lab 13	5/9, Lab 13	5/10, Lab 13	5/12, Lab 14
16	5/15, Lab 14	5/15, Lab 14	5/15, Lab 14	5/16, Lab 14	5/17, Lab 14	-

*Lab 6 will take place in the computer lab of the learning center, located at 1300 Boylan. $\label{eq:computer} % \begin{subarray}{ll} \end{subarray} % \begin{subarray}{ll} \end{suba$

EXPERIMENTS AND READINGS

Lab	Experiment	Required Reading
1	Check-in, Safety, Techniques 1, 3	548-565, 573- 580
2	Solubility Experiment Experiments 1A, 1B & 1C; Technique 10	2-5, 655-661
3	Extraction of Neutral Unknown Experiment 3D, Technique 9, 12	22-24, 645- 654, 683-690, 695-704
4	Recrystallization of Sulfanilamide & Fluorene Experiments 2A & 2B; Techniques 8 & 11	9-13, 632-641, 664-682
5	TLC and Column Chromatography Experiment 5D; Techniques 19 & 20	35-37, 780- 797, 804-814
6	Computer Modelling Lab	Handout*
7	Simple & Fractional Distillation: Unknown Mixture Experiment 6; Techniques 7.1, 14 & 15	38-42, 610- 613, 722-729, 733-743
8	Isolation of Eugenol Experiment 13; Technique 24	88-90, 848-852
9	Preparation of Dibromosuccinic Acid Handout	Handout*
10	Nucleophilic Substitution: Iodide versus Chloride Handout	Handout*,
11	Phenyl Alanine Handout; Technique 23	Handout*, 840- 846
12	Elimination of Methylcyclohexanol Experiment 22	171-174
13	Substitution versus Elimination Handout; Technique 7.6	Handout*, 619
14	Check-out: No Experimental Work SUBMIT LABORATORY NOTEBOOK	NA

^{*} Handouts can be found on Prof. Horowitz's website: http://userhome.brooklyn.cuny.edu/ghorowitz

Postlab Questions:

Lab 2 - Solubility (due week 3) -

- 1) Compare the solubility results of biphenyl and benzophenone Explain why you would expect these results to differ.
- 2) Explain the solubility trends you observed for the alcohols in part B.

Lab 3 - Aqueous Extraction (due week 5) -

- 1) Identify the neutral compound.
- 2) Was your extraction effective at purifying it? Use your data and observations to support your claims.

<u>Lab 4 - Recrystallization and Melting Point</u> (due week 6) -

- 1) Describe
- 2) Which solvent did you determine was best for the recrystallization of fluorene? Discuss, in terms of polarity, why this solvent should be optimal.

Lab 5 - TLC & Column Chromatography (due week 7) -

- 1) Why does fluorenone travel more slowly than fluorene?
- 2) Why does acetone speed up the rate at which fluorenone travels? (Make sure to include a picture of your TCL plate in your lab report)

<u>Lab 6 - Computational Modeling</u> (due week 7) - No prelab is required for this lab. Turn in your completed packet week 7._

Lab 7 - Distillation (due week 8)-

Graph your data.

- 1) Identify the two unknowns. What differences do you notice between the fractional and simple distillation graphs?
- 2) How do these differences help support the notion that fractional distillation is more efficient at separating liquids?

Lab 8 - Isolation of Eugenol (due week 9) -

1) Why is steam distillation used in this experiment? (Hint: Look up the boiling point of Eugenol and read about steam distillation at https://en.wikipedia.org/wiki/Steam_distillation.)

Lab 9 - Preparation of dibromosuccinic acid (due week 11) -

1) Which stereoisomer of dibromosuccinic acid (racemic melting point = 170°C, meso melting point = 290°C) did you obtain? Draw it and show a mechanism to justify its formation.

<u>Lab 10 - Nucleophilic Substitution</u> (due week 11) -

- 1) Compare the results of the KI and KCI reactions. How do the results differ?
- 2) How can you account for the difference in the results of the KI and KCI reactions?
- 3) Predict the results you would have obtained if you had used 2-bromo, 2-methylheptane instead of 1-bromooctane. Explain your prediction.

Lab 11 - Phenyl Alanine (due week 14) -

Given that the specific rotation of S-2-hydroxy-3-phenylpropanoic acid is -22°:

- 1) Determine the absolute configuration of the product you obtained (Is it R or S?)
- 2) Draw the 3D structure of the product you obtained.
- 3) Show a mechanism for the substitution step to justify the formation of this isomer.

Lab 12 - Elimination of Methylcyclohexanol (due week 13) -

1) Do you think your synthesis was successful? Use the data from your alkene tests to justify your answer.

Lab 13 - Substitution versus Elimination (due week 14) -

- 1) What product(s) did you obtain?
- 2) Predict what product or products you would have obtained if you had used the other base for this reaction (but still used the same alkyl halide).