ORGANIC CHEMISTRY LAB I  
Spring 2013 Syllabus

General Information

Lab Instructor/Section:  YOUR NAME/Section  
Office: your preferred place  
Email: your email@brooklyn.cuny.edu  
Office Hours:  Your preferred time

Lab Coordinator:  Dr. Maria Contel  
Office: 355 New Ingersoll  
Email: mariacontel@brooklyn.cuny.edu  
Office Hours:  Tues 11‐12:00, Thurs 11‐12:00 (355 NE)  
Website:  http://userhome.brooklyn.cuny.edu/mariacontel/teaching.html

Statement of Course Goals:

The goal of the lab 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 several important laboratory techniques critical to organic chemists, and will be able to carry out reactions from set-up to purification, and analyze their results. In addition, the course will strive to encourage critical thinking.

Required Purchases For Lab:
1. Organic Chemistry I Laboratory Manual  
2. Composition Notebook  
3. Lock for Lab Drawer  
4. Rubber Gloves  
5. Paper Towels  
6. Soap  

Highly Recommended  
Lab coat (ask in stock room for names of some vendors)

Course Grade Breakdown:

Safety Quiz - 10%*  
Pre-Lab/Post-Lab (with question) – 35%  
Instructor Evaluation/ Laboratory Technique – 10%  
Notebook – 10%  
Final Exam – 35%**

*The safety quiz will be taken immediately following the safety video during lab, and a score of 80% must be achieved to continue in the lab. In the event that a student fails the safety quiz, they must rewatch the safety video and take a second safety quiz, held Tuesday Feb. 5 during common hours.

** The final exam will be cumulative, and will test a student's knowledge of the concepts learned in lab (similar questions to those in the post-lab questions), their knowledge of apparatus set-up, and their ability to process data.

All grades will be normalized according to laboratory sections with the exception of the Final Exam.
Administrative Dates

**Last day to add a course** – Sunday February 3 (ON LINE with department permission)
**Last day for Late add period** – Friday February 8 (instructor and department approval in the late-add form; late add period Feb4-Feb8)
**Last day to file pass/fail application** – Monday January 28
**Last day to drop a course without a grade** – Friday February 15
**Last day to apply to take fall 2012 and intersession 2013 Absentee Final examinations** – Friday, February 8
**Absentee Final Examinations**
**Last day to file for spring graduation** – Friday March 15
**Last day to resolve fall 2012 incomplete grades for fall 2012 and intersession 2013** – Thursday April 25
**Last day to resolve fall 2012 ABS grades** - Last day to take absentee final exams for Fall 2012 and Intersession 2013 (students must have applied in February) Thursday April 25
**Last day to withdraw from a course with a W (non-penalty) grade** – Friday April 12

COURSE POLICIES AND PROCEDURES

**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). Then please provide me with a copy of your course accommodation form and if necessary please schedule an appointment with me to discuss your specific accommodation needs.

**Absence from Examinations:**
No make up examinations will be given to students who are absent from the final lab exam. In the event of absence from the final exam, students must apply to the Academic Advisement Center for permission to take a make up final examination given during following semester. No make-up final will be given to any student who is failing the course heading into the final.
Laboratory Instructions and Regulations:

Safety is the number 1 priority in lab. You will be provided with an approved pair of safety goggles. **Wearing goggles at all times in the laboratory is mandatory.** If you are caught not wearing goggles in the lab, you will be asked to leave and you won’t be allowed back for that session. You will receive a score of 0 for that day’s prelab and notebook check.

During the first laboratory session, 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 to return to your lab instructor. You must read, understand and agree to abide by these rules if you want to take the course.

Please follow the instructions regarding check-in and check-out 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.** Report any missing or broken items to Ms. Belyayeva.

If you miss a lab, please follow the following procedure in order to make up the experiment. Pick up a makeup form from the stock room and ask your instructor to sign it. Check with the makeup instructor to make sure there is room for you to work in his or her laboratory. Have the makeup instructor sign the form so that credit can be given to you for having completed the experiment.

You **should not run heating devices** (hotplates, heating mantles and melting point devices) **more than the midpoint on the controller.** When the controllers are run and left in the maximum position the units overheat. This causes thermometers to crack and mercury spills on the bench and floors. Individuals can suffer bad burns by running heating mantles with controllers set on high or using a heating mantle without a controller. You do need to learn from your instructor the **proper way lubricate your glass fittings** to avoid breakage.

**Laboratory Breakage.** In some schools, a laboratory fee is charged to everyone. This department's practice is to charge you only for the replacement cost of any items you lose or break. After check out, a bill will be prepared which you may pay at the bursar's office the following semester.

**NOTE:** If you have checked in for any lab course you **must check out** even if you only attend class for one or two weeks before dropping the course. Students who fail to check out will be charged a fee of **$50** plus the cost missing or broken equipment. Students who drop a course must go to the stockroom to check out as soon as possible.
Lab Schedule:

<table>
<thead>
<tr>
<th>Lab</th>
<th>Domzalski (Monday Morning)</th>
<th>Hayes (Monday Morning)</th>
<th>Ghogare (Monday Afternoon)</th>
<th>Fraboni (Tuesday Afternoon)</th>
<th>Grigoryan (Wednesday Morning)</th>
<th>Ghogare (Wednesday Morning)</th>
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<tr>
<td>C-I</td>
<td>28-Jan</td>
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<td>7-May</td>
<td>8-May</td>
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* Lab 6 will be held in the computer lab of the learning center, which is located at 1300 Boylan

**Final Exam Laboratory:** May 14th Tuesday during lecture 9:30AM to 10:45AM, Room 2310N
### Lab and Reading:

<table>
<thead>
<tr>
<th>Lab</th>
<th>Experiment</th>
<th>Required Reading</th>
</tr>
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<tbody>
<tr>
<td>C-I</td>
<td>Check-in, Safety Video, Safety Video Quiz</td>
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</tr>
<tr>
<td>2</td>
<td>Solubility Experiment Experiments 1A, 1B &amp; 1C; Technique 10</td>
<td>1-4, 147-153</td>
</tr>
<tr>
<td>3</td>
<td>Extraction of Neutral Unknown Experiment 3D, Technique 12</td>
<td>28-31, 177-185, 191-198</td>
</tr>
<tr>
<td>5</td>
<td>TLC and Column Chromatography Experiment 4D; Techniques 19 &amp; 20</td>
<td>41-43, 249-287</td>
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<td>6</td>
<td>Electronic Structure of Molecules</td>
<td>Handout*</td>
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<tr>
<td>7</td>
<td>Simple &amp; Fractional Distillation: Unknown Mixture Experiment 5; Techniques 7.1A, 14 &amp; 15</td>
<td>45-48, 97-99, 215-221, 227-238</td>
</tr>
<tr>
<td>8</td>
<td>Nucleophilic Substitution Lab</td>
<td>Handout*</td>
</tr>
<tr>
<td>9</td>
<td>Elimination of Methylcyclohexanol Experiment 24</td>
<td>63-66</td>
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<tr>
<td>10 – 11</td>
<td>Oxidation of an Unknown Alcohol &amp; Preparation of Derivatives</td>
<td>Handout*</td>
</tr>
<tr>
<td>12</td>
<td>Grignard Reaction: Triphenylmethanol Experiment 36A; Techniques 7.5 &amp; 7.6</td>
<td>67-75, 104-107</td>
</tr>
<tr>
<td>13</td>
<td>Finish Grignard, Acetal Formation</td>
<td>Handout*</td>
</tr>
<tr>
<td>C-O</td>
<td>Check-out: No Experimental Work SUBMIT LABORATORY NOTEBOOK</td>
<td>NA</td>
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</table>

*Handouts can be found on Prof. Horowitz's website (http://userhome.brooklyn.cuny.edu/ghorowitz/index.htm)
Lab Assignments:

LABORATORY REPORTS AND NOTEBOOKS

You will be required to submit a prelab and postlab for each experiment or set of experiments. You will also be required to keep a laboratory notebook which you will turn in at the end of the semester. The instructions below describe what should be included in your prelab, postlab and notebook.

A good free online resource regarding organic laboratory techniques is Techniques in Organic Chemistry by Mohrig, Hammond, Shatz. It is available at googlebooks at the following address:
http://books.google.com/books?id=21acgWiaB5cC&lpg=PP1&ots=SmRvx3PEa4&dq=hammond%20mohrig%20techniques%20of%20organic%20chemistry&pg=PP1#v=onepage&q&f=false

Prelabs – Use the forms at the end of the syllabus. Pre-labs must be signed by your instructor and turned in with the post-lab.

Table of Chemicals Used (1 pt)
The table of chemicals should list all known chemicals that will be used, including solvents. List any hazards associated with each chemical. If a chemical reaction is conducted, you must also report the molecular weight, grams and moles for each chemical involved in the reaction.

Procedure (2 pts)
The procedure should be concise. Try to keep the procedure to less than half of a page if possible.

Postlabs- Use the forms at the end of the syllabus. Pre-lab/post-labs must be turned in on time. In the event that you miss lab, you must still turn in your lab report.

Observations (3 pts)
List 3 important physical observations that were made during the lab and briefly state why it is important. Physical observations are what you see or feel, not what you conclude or interpret.

(good example. Upon heating the reaction, the color of the solution turned from green to red. The color change is indicative of a chemical reaction taking place)

(bad example: I observed that my yield was better when I doubled the concentration. This means that higher concentrations are important for a more successful reaction)

Notice how the latter is not a physical observation, but an interpretation of data that you observed.

Data (3 pts)
List any data that you have obtained and report % recovery and/or % yield when applicable. Show graphs of data where applicable.

Post-lab Question (6 pts)
One post-laboratory question will help guide you to critically evaluate the laboratory you just performed. It should be concise and to the point, and should reflect upon data and observations that you have made whenever possible.
Post lab Questions

Lab 2 – Solubility (due week 3) - Compare the solubility results of biphenyl and benzophenone. Account for any differences observed. Explain the solubility results you observed for the alcohols in part B.

Lab 3 – Aqueous Extraction (due week 5) - Was aqueous extraction effective at purifying your neutral compound? Use your data and observations to support your claims.

Lab 4 – Recrystallization and Melting Point (due week 6) – Were you successful at purifying fluorene and sulfanilamide? Use your data and observations to support your claim.

Lab 5 – Column Chromatography (due week 7) – Were you successful at separating fluorene and fluorenone? Use your data and observations to support your claim.

Lab 6 – Computational Modeling No prelab is required for this lab. Turn in your completed packet week 7.

Lab 7 – Distillation (due week 8)- Analyzing your data, what differences do you notice between fractional and simple distillation? How do these help support the notion that fractional distillation is more efficient at separating liquids?

Lab 8 – Nucleophilic Substitution (due week 9) – Compare the results of the KI and KCl reactions. How do the results differ? How can you account for the difference? Predict the results you would have obtained if you had used 2-bromo, 2-methylheptane instead of 1-bromoctane. Explain your prediction.

Lab 9 – Elimination of Methylcyclohexanol (due week 10) – Do you think your synthesis was successful? Describe the alkene tests that you performed. Show structures to justify your answer.

Labs 10 and 11 – Alcohol Oxidation (due week 12) – Where you successful at oxidizing the alcohol to a ketone? What was your unknown, and how did you determine it?

Lab 12 – Grignard (due week 14) – Discuss your yield and purity. Do you think that the reaction was successful, and do you think there are ways you could have improved it?

Lab 13 – Acetal Formation (due week 14) – Discuss your yield and purity. Do you think that the reaction was successful, and do you think there are ways you could have improved it?

Notebook Guidelines

Keeping a good record of your work is as important as doing good work. The lab notebook is the only valid record of a scientific experiment. A good record means that all important information is recorded accurately in a timely fashion (i.e., IN LAB, not at home afterwards).

Your record should be clear enough for another chemist to understand what you did and to be able to reproduce it following your notes.
You must write your observations as you do your experiments (and include the date at the top of each page). Do not trust your memory. It is very difficult to remember a full lab session after you have finished the experiment; you will always forget details and they may be important.

Use only ink to write in your notebook. NEVER write in pencil.

Try to write as neatly as possible and make sure it is legible for anyone (it doesn’t have to look perfect though!). You will certainly make mistakes from time to time; that is normal. When that happens, just cross out the error and write the correct entry in the proper place (the crossed-out error must also be legible).

Some things you must NEVER do in your book: erase, use liquid corrector, and tear off pages.

Each entry should contain the following 7 sections:

1) **Heading**
2) **Reaction table**
3) **Hazards**
4) **Precautions**
5) **Procedure and Observations**: Summarize the sequence of important steps that were performed. This can be written either as a numerical list or in paragraph form. Record important observations as you go along (i.e. when a reactant was added, was there a color change? or did the solution bubble, indicative of gas evolution?).
6) **Data (or Results)**: Record any raw data. This can be something you measured (i.e. a weight) or the results of a test (i.e. the results of a litmus test).
7) **Conclusions**: Analyze or interpret your data in this section. (i.e. Answer the question: What was the % yield of the product? Or, what does your data suggest your unknown is?)

Tips:
- In 5) write your account of what you did (e.g., the solution was heated under reflux for 2h and a color change from blue to yellow was observed; it was then allowed to cool to room temperature and chloroform (10 mL) was added...). Do not write what you think or expect will happen (e.g., “The reaction mixture will be heated until the new product precipitates”).
- Write only facts, not opinions (e.g. “after heating for 10 min, the red mixture turned black and an intractable black solid was obtained”, but never write “the synthesis didn't work” or “crappy black decomposition product”. It might be a new compound in fact more interesting that the one you wanted to make in the first place!).
PRE-LAB REPORT

Your Name ____________________

Title of Experiment ______________

Today’s Date ____________________

Table of Chemicals:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Hazards</th>
<th>Mol. Wt.</th>
<th>Density</th>
<th>Grams</th>
<th>Moles</th>
</tr>
</thead>
<tbody>
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</tbody>
</table>

Procedure:
POST-LAB REPORT

Your Name ________________
Title of Experiment ____________
Today’s Date ________________

Observations:

Data:

Post-lab Question:
EXAMPLES OF FINAL EXAM QUESTIONS

1. Name the following pieces of equipment (5 points each)

   a. ______________________________

   b. ______________________________

   c. ______________________________

   d. ______________________________
2. Explain the following miscibility trend (10 points)

<table>
<thead>
<tr>
<th></th>
<th>Water</th>
<th>Hexanes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>Miscible</td>
<td>Immiscible</td>
</tr>
<tr>
<td>1-Butanol</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
<tr>
<td>1-Octanol</td>
<td>Immiscible</td>
<td>Miscible</td>
</tr>
</tbody>
</table>

3. Explain how you would go about separating the following molecules from one another using aqueous extraction. Use structures to support your answer (10 points).

\[
\text{\begin{center}}
\text{\begin{tabular}{c c c}
\hline
\text{Structure 1} & \text{Structure 2} \\
\hline
\text{\begin{center}}
\text{\begin{tabular}{c}
\text{N-}
\end{tabular}}
\text{\begin{center}}
\text{\begin{tabular}{c}
\text{O-}
\end{tabular}}
\end{center}}
\end{tabular}}
\end{center}}
\]
4. The following is a silica gel TLC plate of 3 compounds run in 50% EtOAc in Hexanes. Predict which spot corresponds to which compound. Explain your answer (10 points).

A

B

C

5. Use the data below to identify the appropriate solvent that should be used to re-crystallize a mystery compound X. Explain your answer. (10 points)

<table>
<thead>
<tr>
<th>Solvent (bp)</th>
<th>Solubility at rt</th>
<th>Solubility at reflux</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water (100oC)</td>
<td>Insoluble</td>
<td>Insoluble</td>
</tr>
<tr>
<td>Dimethyl Sulfoxide (190oC)</td>
<td>Insoluble</td>
<td>Soluble</td>
</tr>
<tr>
<td>Ethanol (70oC)</td>
<td>Insoluble</td>
<td>Insoluble</td>
</tr>
<tr>
<td>Ethyl Acetate (65oC)</td>
<td>Soluble</td>
<td>Soluble</td>
</tr>
<tr>
<td>Dichloromethane (35oC)</td>
<td>Insoluble</td>
<td>Soluble</td>
</tr>
<tr>
<td>Diisopropyl Ether (70oC)</td>
<td>Insoluble</td>
<td>Soluble</td>
</tr>
<tr>
<td>Hexane (70oC)</td>
<td>Insoluble</td>
<td>Insoluble</td>
</tr>
</tbody>
</table>

6. What is wrong with the following fractional distillation setup? What would go wrong if it were run as currently setup? (10 points)
7. Elias wanted to perform an oxidation of ethanol to generate acetic acid.

\[
\begin{align*}
\text{OH} & \quad \xrightarrow{\text{H}_2\text{CrO}_4} \quad \text{O} \\
\text{amu} = 46 & \quad \rightarrow \quad \text{amu} = 60
\end{align*}
\]

a. After the workup of the reaction, Elias had left over a liquid. Give a way in which Elias could determine if his compound was acetic acid, and whether it was pure? (10 points)

b. Elias soon discovered that his compound had only gone to \(~50\%\) conversion, and he had a mixture of acetic acid and ethanol. Give two ways in which Elias would be able to purify the acetic acid away from the ethanol? Be specific in your answer, and use structures if necessary. (10 points)

c. After the purification, Elias recovered 5 grams of pure acetic acid. He had begun with 20g of ethanol, which was the limiting reagent. What was Elias’ yield? (10 points)