CM 244 Organic Chemistry Laboratories

Spring 2017

Instructors:  
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123 SC  
Office hours: by appointment (send an e-mail)

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Office Hours: Tue 3-6 pm

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Office Hours: MonWed 3:30-5 pm, 136SC

Laboratory hours:  
Mon, Wed 8:00 am – 10:45 am; 236 Science Center; section 10  
Mon, Wed 2:00 pm – 4:45 pm; 236 Science Center; section 20  
Tue, Thu  8:00 am – 10:45 am; 236 Science Center; section 30  
Tue, Thu  11:00 am –1:45 pm; 236 Science Center; section 40  
Tue, Thu  2:00 pm – 4:45 pm; 236 Science Center; section 50

Lab notebook: Everyone will need to purchase a LABORATORY notebook (Continuation of Spectroscopy Lab notebook is acceptable) with the following features:

1. Table of Contents;
2. Contains duplicate pages;
3. Contains 100 pages;
4. Spiral bound laboratory notebook is suitable.

Safety glasses with side shields: Everyone will need to purchase safety glasses. Students will not be allowed to perform an experiment without glasses; A ZERO GRADE WILL BE RECORDED FOR THE MISSED LAB AND FOR THE FORMAL REPORT FOR SUCH MISSED LAB.

Lab coats: Everyone will need to purchase a lab coat. They can be kept in the lab lockers between lab sessions. Students will not be allowed to perform an experiment without lab coat; A ZERO GRADE WILL BE RECORDED FOR THE MISSED LAB AND FOR THE FORMAL REPORT FOR SUCH MISSED LAB.

Course description: The course will provide an introduction to the synthesis of organic compounds as well as methods of purification, and identification of organic compounds. Each section of OrgChem lab meets twice a week. Each session lasts for 2 hours and 45 minutes. Quizzes on comprehensive understanding of the particular lab will be taken prior each lab in electronic form through the Moodle. Introductory lecture given at the beginning of every lab will focus on theoretical and practical aspects of the particular laboratory. The prelab writing in the required format will be checked every lab. Recorded data and observations will be checked upon every lab completion. Laboratories related to the same topic will be accomplished with a write-up of the lab experiment, data and observation, and discussion of the results including spectroscopic data in a form of formal report. The formal report checklists will be provided by instructors. (See instructions for formal report writing on page 6 of the Syllabus).

All labs will be performed in mini scale.

Main goals of the laboratory: (a) to learn how to handle and work with chemicals safely; (b) to gain familiarity with a variety of laboratory equipment and techniques; (c) to learn proper laboratory record keeping; (d) to be introduced to organic synthesis, including the setting up of reactions, purification and identification of organic compounds.

Topics covered will include: recrystallization, melting points; distillations; extractions; chromatography; spectroscopic techniques; SN1 and SN2 reactions; elimination and addition reactions; radical chain reactions, Diels-Alder reaction; electrophilic substitution; oxidation and reduction reaction; Grignard reaction; reactions of carbonyl compounds; and multi-step synthesis as a final project.

Make-up labs will be at the discretion of the instructor. If you know you are going to miss specific days please consult in advance.
<table>
<thead>
<tr>
<th>Date of laboratory</th>
<th>Topic of the laboratory</th>
<th>Chapter in the textbook 5-th edition</th>
<th>Formal reports due dates</th>
</tr>
</thead>
<tbody>
<tr>
<td>1/16 1/18 1/23 1/25 1/30 2/1 2/6 2/8 2/13 2/15 2/20 2/22 2/27 3/1</td>
<td>1/12 1/17 1/19 1/24 1/26 1/31 2/2 2/7 2/9 2/14 2/16 2/21 2/28 3/2</td>
<td><strong>Lab 1:</strong> Check In; Safety; General Overview <strong>Lab 2:</strong> Solvent selection experiment Recrystallization and melting point of acetonilide <strong>Lab 3:</strong> Recrystallization and melting point of benzoic and cinnamic acids <strong>Lab 4:</strong> Simple distillation <strong>Lab 5:</strong> Steam distillation <strong>Lab 6:</strong> One base extraction <strong>Lab 7:</strong> Two base extraction <strong>Lab 8:</strong> Acid-Base extraction <strong>Lab 9:</strong> Separation of spinach pigments by TLC <strong>Lab 10:</strong> Spectroscopic characterization of products obtained starting from recrystallization lab <strong>Lab 11:</strong> Free-radical chain chlorination of 1-chlorobutane <strong>Lab 12:</strong> Elimination with alcoholic potassium hydroxide <strong>Lab 13:</strong> Bromination of (E)-stilbene <strong>Lab 14:</strong> Hydration of norbornene</td>
<td>Ch. 1 and 2 Ch. 3.2A and B2, 3.3 and 3.3 B Exp.1 pp.11 of the syllabus, Ch. 3.2A and B1, 3.3 and 3.3B Ch. 4.1-4.3 Ch. 4.5 and 4.6 Ch. 5.1-5.3, 5.3A Ch. 5.3B Ch. 5.3C Ch. 6.1-6.2 Ch. 8 Chapter 9 Ch. 10.1-10.2; 10.2A Ch. 10.4-10.6; Ch. 10.7</td>
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<td>Date(s)</td>
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<td>3/6</td>
<td>3/7</td>
<td><strong>Lab 15:</strong> Diels-Alder reaction</td>
<td>Ch. 12.1-12.3 12.3A</td>
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<td>3/8</td>
<td>3/9</td>
<td><strong>Lab 16:</strong> Preparation of 1-bromobutane, an S$_{N}$2 reaction</td>
<td>Chapter 14.4</td>
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<td>3/13</td>
<td>3/14</td>
<td><strong>Lab 17:</strong> Preparation of 2-chloro-2-methylbutane, an S$_{N}$1 reaction</td>
<td>Chapter 14.5</td>
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<td>3/15</td>
<td>3/16</td>
<td><strong>Lab 18:</strong> Nitration of bromobenzene</td>
<td>Chapter 15.4</td>
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<td>3/20, 3/22</td>
<td>3/21, 3/23</td>
<td><em>Spring break</em></td>
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<td>3/27</td>
<td>3/28</td>
<td><strong>Lab 19:</strong> Oxidation of cyclododecanol</td>
<td>Chapter 16.2A</td>
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<td>3/29</td>
<td>3/30</td>
<td><strong>Lab 20:</strong> Formation and reduction of N-Cinnamylidene-m-nitroaniline</td>
<td>Chapter 17.3</td>
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<td>4/3</td>
<td>4/4</td>
<td><strong>Lab 21:</strong> Synthesis of <em>trans</em>-p-anisalacetophenone</td>
<td>Chapter 18</td>
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<tr>
<td>4/5</td>
<td>4/6</td>
<td><strong>Lab 22:</strong> Preparation of Grignard reagent from bromobenzene</td>
<td>Chapter 19</td>
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<td>4/10</td>
<td>4/11</td>
<td><strong>Lab 22 continuation:</strong> Preparation of triphenylmethanol</td>
<td>Chapter 19</td>
</tr>
<tr>
<td>4/12</td>
<td>4/13</td>
<td><strong>Lab 23:</strong> Preparation of luminol and its chemiluminescence</td>
<td>Chapter 20.4 A and B</td>
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<td>4/17</td>
<td>4/18</td>
<td><strong>Lab 24:</strong> Multi-step lidocaine synthesis</td>
<td>Chapter 21</td>
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<td>4/19</td>
<td>4/20</td>
<td>Multi-step lidocaine synthesis</td>
<td>Chapter 21</td>
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<td>4/24</td>
<td>4/25</td>
<td>Multi-step lidocaine synthesis</td>
<td>Chapter 21</td>
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<tr>
<td>4/26</td>
<td>4/27</td>
<td>Check out and evaluation</td>
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</table>

*Note: FR stands for Friday Remains.*
The course will be graded according to the following:

<table>
<thead>
<tr>
<th>Requirement</th>
<th>Weighting</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lab reports</td>
<td>50%</td>
<td>Formal report will be due in one week after completion of the topic, see due dates in the tentative schedule.</td>
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<td></td>
<td></td>
<td><strong>Formal report will be submitted in hard copy to a lab TA. The text of the introduction, discussion, and conclusion sections of every formal report has to be uploaded to TURNITIN on Moodle. This text will be routinely checked for plagiarism and cheating. Reports that will appear too similar will have their grade reduced by 50% for the first infraction. All instances thereafter will result in a zero score.</strong> General checklists for writing formal report for purification lab and for preparative (synthetic) lab will be posted on Moodle for several first labs. The lab reports will be accepted only for the experiments performed by the student. In a case the lab was missed 0 (zero) grade will be recorded for both the performance and the lab report. The only exception is medical excuse.</td>
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<tr>
<td>Notebooks</td>
<td>10%</td>
<td>Pre-lab and lab writing in required format will be checked at the beginning of the lab and signed at the end of the lab by TA. The pre-lab and data and observation copy will be submitted with formal report for grading.</td>
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<tr>
<td>Lab performance</td>
<td>10%</td>
<td>Every student will submit the final product of every lab to a TA. The grade will be given for every lab according to lab completion on time, purity and yield of the obtained compound that will be analyzed by the instructor.</td>
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<tr>
<td>Quizzes</td>
<td>30%</td>
<td>Prior every lab in electronic form through the Moodle. 0 grade will be recorded for students submitting the quiz after the lab starts.</td>
</tr>
</tbody>
</table>

**Final grade** will be determined on the following scale and there will be no bumping.

- **A+** (96-100);
- **B** (80-83.95);
- **C-** (64-67.95);
- **A** (92-95.95);
- **B-** (76-79.95);
- **D** (60-63.95);
- **A-** (88-91.95);
- **C+** (72-75.95);
- **F** (<60);
- **B+** (84-87.95);
- **C** (68-71.95);

**Your safety** in the laboratory is the top priority. Chemicals can easily turn into a threat if you are unaware of their reactivity. Therefore it is your responsibility to learn about the reagents before using them in the lab. This data can be found in Material Safety Data Sheet (MSDS) which is a summary of information on a given chemical, identifying material, health, and physical hazards, exposure limits, and precautions. MSDS of all commercially available compounds can be found on [www.sigmaaldrich.com/Area_of_Interest/The_Americas/United_States.html](http://www.sigmaaldrich.com/Area_of_Interest/The_Americas/United_States.html) or are available from Rebecca Hourihan in the chemistry stockroom.

**The use of laboratory fume hoods is required. No experiment will be performed outside the hood. Safety glasses or goggles as well as a lab coat must be worn at all times. Students wearing prescription glasses must wear safety glasses or goggles over their glasses. Secure long hair and loose clothing away from equipment and chemical contamination. Closed shoes are required at all times. Eating, drinking and smoking are prohibited in the lab any time.**
**Preparation for the laboratory** is crucial for your safety and successful experiment.

You must read all assigned material.

You must have a pre-lab write-up in your notebook when you arrive. A TA will check the pre-lab write-up at the beginning of every lab. A student who failed to complete pre-lab write-up will not be allowed to start the experiment.

A quiz that must be completed prior every lab will cover the theoretical and experimental part of the particular laboratory.

**Lab Notebook**

Everyone will need to purchase a lab notebook (Continuation of Spectroscopy Lab notebook is acceptable) from the bookstore with the following features:

1. Table of Contents;
2. Contains duplicate pages.

Documentation must be clearly and neatly written and nothing can be added or removed from the notebook once it is taken out of the lab. **No pencil can be used for record keeping purposes.**

A carbon copy of all lab notes will be turned in with Formal lab report. These page(s) along with the notebook check for pre-lab and during the lab write-ups will be the basis of your notebook grade.

**Pre-lab writing (will be checked and signed prior every lab by TA):**

1. Page number and Heading (use a new page of the notebook to start the entries for the experiment);
2. Table of Reactants (*an MSDS must always be consulted before handling hazardous substances*);

<table>
<thead>
<tr>
<th>Compound Name and structure</th>
<th>Molecular weight</th>
<th>Weight in grams</th>
<th>Volume in milliliters (if applicable)</th>
<th>Moles used (for the reagents)</th>
<th>Density</th>
<th>Boiling point</th>
<th>Melting point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naphthalene</td>
<td>128 g/mol</td>
<td>1 g</td>
<td>-</td>
<td>7.8 mmol or 0.0078 mol</td>
<td>solid</td>
<td>solid</td>
<td>77°C</td>
</tr>
<tr>
<td>Ethanol</td>
<td>46 g/mol</td>
<td>4 g</td>
<td>5 ml</td>
<td>solvent</td>
<td>0.79 g/ml</td>
<td>78 °C</td>
<td>liquid</td>
</tr>
</tbody>
</table>

3. For synthetic labs the overall balance equation of the particular reaction with all products has to be written, the mechanism of the reaction has to be drawn.
4. In reaction equation limiting reagent has to be assigned.
5. Experiment outline explaining all operations in the stepwise manner as exemplified below:
**For example in your textbook you have the following procedure for recrystallization of naphthalene 3.2.B3:**

Naphthalene may be conveniently recrystallized from methanol, 95% ethanol, or 2-propanol. Because these solvents are somewhat toxic and/or flammable, proper precautions should be taken. The sequence of steps up through the hot filtration should be performed in a hood if possible. Alternatively, if instructed to do so, position an inverted funnel connected to a vacuum source above the mouth of the flask being used for recrystallization.

Place 1.0 g of impure naphthalene in an Erlenmeyer flask equipped for magnetic stirring or with boiling stones and dissolve it in the minimum amount of boiling alcohol. *Caution:* Because the sample may be contaminated with insoluble material, pay close attention to whether additional solid is dissolving as you add more solvent; if it is not, stop adding solvent. Then add 0.5 ml of additional solvent to ensure that premature crystallization will not occur during subsequent transfers. Record the total volume of solvent used.

Continue the procedure by following the directions for Decoloration, Hot Filtration and Crystallization, and Isolation and Drying given for benzoic acid in Part 1; however, rather than water use the solvent in which you dissolved the naphthalene.

**Analysis** Determine the melting points of the crude and recrystallized naphthalene, the weight of the latter material and calculate the percent recovery using equation 3.1.

**You convert it to the following experiment outline:**

a. Place 1.0 g of naphthalene in 50 mL Erlenmeyer flask
b. Dissolve it in the minimum amount of boiling alcohol with stirring on top of the hot plate
c. Add 0.5 ml of additional solvent to ensure that premature crystallization will not occur during subsequent transfers
d. Record the total volume of solvent used
e. If the solution is colored, proceed with decolorization:
e.1 cool the solution slightly, add a microspatula-tip full of carbon,
e.2 reheat to boiling for a few minutes,
f. If there are insoluble impurities or decolorizing carbon in the solution, perform a hot filtration:
f.1 rinse filtrate receiving 50 mL Erlenmeyer flask with about 1 mL of hot solvent,
f.2 place funnel with folded filter paper on the top of the rinsed Erlenmeyer flask,
f.3 wet the filter paper with the hot solvent,
f.4 filter the solution with insoluble impurities or decolorizing carbon.
g. Allow the filtrate to stand undisturbed until it has cooled to room temperature and no more crystals form
h. Place the flask in an ice-water bath for at least 15 min
i. Collect the crystals on a Buchner funnel by vacuum filtration
j. Wash the filter cake with two small portions of cold solvent (the one you have used for crystallization)
k. Dry the crystals under vacuum for 15-20 minutes
l. Transfer the crystals to the pretared watch glass
m. Record mass and melting point of the product
Data and observations (must be collected during every lab):

1. Your observations of the experiment as it progresses have to be recorded. Write these observations (continuation of the reaction in minutes, boiling temperatures, the pH of the reaction (if applicable), etc.) in your notebook as you do the experiment.

As you perform the experiment, some **physical changes** may or may not happen. For example, when you combine two substances, a change in color might occur, the solution might get hot, a precipitate may form, or bubbles might appear. The rule of thumb is to record any changes that can be detected with the senses. While most of them are visual (e.g. a change in color or formation of a precipitate), some are tactile (a change in temperature), or even auditory (an explosion, which hopefully will not happen in this course!). On occasion a smell might be detected, and this qualifies as a physical observation.

Example of observations of physical changes:

a) When the impure naphthalene was warmed in acetone, it dissolved after about 20 sec.

b) When the impure naphthalene was boiled in 95% ethanol, it dissolved after about 1 min.

c) When the filtrate was cooled to the room temperature the solution became milky-cloudy.

d) When the filtrate was cooled in ice-water bath, white, needle-shaped crystals formed.

Keep in mind that in this section all you do is record such observations as objectively as possible, but you do not try to infer a meaning from them. This is done in the results and discussion section, described in a formal report layout.

2. Record the weights of tare and products. Record the melting point of the final (purified or synthesized and purified compound in a case it is solid). Summarize the obtained data into a table:

<table>
<thead>
<tr>
<th>Product name and structural formula</th>
<th>Weight of a tare in grams</th>
<th>Weight of the tare with the product in grams</th>
<th>Weight of the product in grams</th>
<th>FW of the pure product in mol/g</th>
<th>Amount of moles of the product</th>
<th>Melting point of the final product (if applicable)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naphthalene</td>
<td>28 g</td>
<td>28.8 g</td>
<td>0.8 g</td>
<td>128 g/mol</td>
<td>6.25 mmol or 0.00625 mol</td>
<td>80°C</td>
</tr>
</tbody>
</table>

Do not re-write the Procedure section in observation section.

3. Calculation of the yield in the experiment:

   a. for purification: yield = moles of purified product / moles of used material * 100%

   *Example for the mentioned above naphthalene Yield = 6.25 mmol / 7.8 mmol * 100% = 80%*

   b. for synthesis: yield = moles of final product / moles of limiting reagent * 100%
**Formal Reports**: Upon completion of a topic a Formal lab write-up will be submitted to the instructor. Formal report due dates are listed in the tentative schedule of the course and reports submitted after due dates will incur penalties. Formal report should be written with 1.5 spacing with 12 font size. The general checklist for a report write-up can be found on the Moodle. The TA will grade your report according to the checklist as well. The length of the formal report is expected to be about 6-7 pages.

Formal reports can be of two kinds, depending on whether they refer to experiments involving **physical operations** (purification and characterization) or **chemical preparations** (synthesis).

**Physical operation report** refers to lab work during which no change of the chemical nature of the substances is involved. They are typically conducted as part of a synthesis.

**Synthesis report** refers to experiments whose main goal is to prepare a pure substance from specific starting materials.

**Formal Reports consist of the following**

1. **Title**: e.g., *Separation of Spinach Pigments by TLC*.

2. **Introduction** (about 2-3 pages): The introduction should identify the particular problem or issue addressed by the lab and give background information about that problem. The instructor provided checklist will help you to write the introduction.

   E.g., *Thin layer chromatography (TLC) is a solid-liquid adsorption chromatography technique used to rapid analysis of small quantities of sample. In organic chemistry laboratory TLC is used for monitoring the progress of reactions and analyzing purity of reaction products.*

   *The common principle of chromatography involves unequal distribution of the mixture components between two immiscible phases, mobile and stationary. For TLC a stationary phase consists of a thin layer of adsorbent material, usually silica gel, aluminum oxide, or cellulose immobilized onto a flat, inert carrier sheet. An analyzed mixture is dissolved in an appropriate solvent in concentration ca. 10 mg/mL and spotted at the bottom of TLC plate. One end of TLC plate is immersed into a mobile, liquid phase (eluting solvent/s) used to “run” the TLC. An eluting solvent is drawn up the plate via capillary action. Components of the analyzing mixture go up with solvent. Due to the processes of sorption and desorption different compounds in the sample mixture travel different distances according to how strongly they interact with the adsorbent, mainly due to different polarity of the compounds.*

   *This allows the calculation of an \( R_f \) value which is characteristic for particular compound under particular conditions. \( R_f = \frac{\text{traveling distance of the compound}}{\text{traveling distance of the solvent}} \)

3. **Materials and Methods** (must be neatly written): Copy pages of pre-lab writing written in understandable manner.

4. **Data and Observations** (must be retyped from the notebook)
5. **Results and Discussion (about 3-4 pages):** This is the section in which you interpret the data obtained in the previous section.

5.1 You have to write the main reaction equation (write the mechanism of the reaction using curved arrows, draw intermediate products and transition state for each step of the reaction, all this must be **handwritten**) which was used for preparation of new material.

5.2 You have to **handwrite** all reactions that have been used in your purification techniques using curved arrows, most of the time these are acid base reactions. Mention the pKa values of the acid and conjugate acids.

5.3 You have to indicate the amount of purified compound that you obtained and how the purity and identity of the compound was assessed. In a preparative experiment, report the yield in both grams and percent.

5.4 Include neatly assigned instrument printouts, such as GC traces and IR, UV/Vis and NMR spectra. If the analyzed spectra contain some additional signals the source of them (possible impurities-starting materials, solvents) has to be identified.

5.5 In this section, you can state whether or not the procedure was a good method for making the desired compound; if not, try to make suggestions to improve the method for future experimenters.

5.6 Explain how any changes to or problems with the experimental procedure may have affected the results, or offer other suggestions as to why your results may have been different from or similar to described experiment.

6. **Conclusions:** A brief summary reiterating the finer points of the experiment should be given at the end of the report. It should provide the reader a sense of closer on the subject discussed in the previous sections of the report but it should also restate the major findings.

7. **Literature Cited** refers to a list of all materials used to get background knowledge on a subject and a list of papers actually mentioned within the report.
Experiment #1: Recrystallization and melting point of either benzoic or cinnamic acid

Protocol:

1. Obtain 1g of pre-prepared crude sample (save the number of the sample) of either cinnamic acid with 5-10% of benzoic acid as an impurity or benzoic acid with 5-10% of cinnamic acid as an impurity;

2. Measure the melting point of the crude sample as described in Chapter 3.3B;

3. Recrystallize the crude sample to obtain pure compound (either benzoic or cinnamic acid) as described in Chapter 3.2B (follow the procedure for benzoic acid);

4. Measure the melting point of purified compound as described in Chapter 3.3B;

5. Analyze results of purification:
   5.1 name the pure compound you obtained after recrystallization;
   5.2 calculate yield of recrystallization;
   5.3 calculate molar and weight percentage of impurities in the crude sample according to the binary phase diagram (use the upper curve of the graph, pay attention that the temperature is given in Kelvin and ratio is molar). The diagram is printed on the next page.
   5.4 calculate molar and weight percentage of impurities in the final compound, if you have some, according to the same binary phase diagram.