SYLLABUS

CHEM 226 – Organic Chemistry LABORATORY B
Summer 2008 - LOYOLA UNIVERSITY CHICAGO (LUC)

Laboratory: #1590      Section 003    Mon + Wed, 12:30 – 3:20 p.m.    Quinlan Life Science Bldg 115

Instructor: Dr. C. Szpunar    Contact: 773-508-3128, cszpuna@luc.edu
Student Office Hours: by prior appt

Required: Text, Catalyst: CHEM 226, Thomas, Tim
(with duplicate-page, numbered-page, lab notebook included in the text)
Safety glasses (LUC Bookstore, Beck’s Bookstore, Chemistry Department)
Sponge *and* rubber gloves (Dominick’s, Walgreen’s)
Closed-toe shoes
Tie or ribbon or scrunchy for long hair
Lab coat or apron or expendable buttoned-up, long-sleeve shirt **and** full-length pants or skirt
T-shirt or handkerchief – cotton, white, no writing or embroidery, pre-washed
(for last experiment)

Course Objective: To illustrate some of the topics covered in Organic Chemistry lecture; to learn and use basic
Organic Chemistry laboratory techniques in synthesis, purification, separation, analysis, and/or characterization;
to learn to analyze and to interpret data from various instruments used in separating and identifying organic
compounds: IR, NMR, and UV-vis spectrophotometers and mass spectrograph, as applicable.

Safety: Safety is our top priority. Safety is YOUR priority. The Organic Chemistry lab poses far more potential
dangers than a General Chemistry lab. Safety is everyone’s job. Safety is the student’s job. Safety is
YOUR job. Students are expected to follow all safety instructions and guidelines, and to exercise common
sense. Students must wear goggles at all times during an experiment, until the last person has cleaned
and stored his/her equipment and has cleaned his/her work area(s).

*** STUDENTS WHO DO NOT COOPERATE WITH SAFETY GUIDELINES WILL BE ASKED TO
LEAVE THE LAB AND ARE ASSIGNED A ZERO SCORE FOR THAT EXPERIMENT. ***

Grading Guidelines: > 92%, A; 84-92%, B; 76-83.9%, C; grading may be curved.
- An unexcused absence merits a zero score.
- An excused absence is handled on a case-by-case basis; grade weighting may be adjusted,
depending on the circumstance, but an excused absence must be corroborated and documented, e.g.,
ampanied by a note from the doctor, dentist, hospital rep, or funeral director, or by a court summons.

Grading: 8-10 experiments, 25 points each (200-250 points anticipated total) to be adjusted as applicable.

Pre-lab preparation (recorded IN THE STUDENT’S NOTEBOOK, 7 pts, may be adjusted as applicable):
  5 pts a) title, date, refs, reaction(s); b) procedure summary and pre-lab exercises, if any; and
c) substances / reactants table (see notebook format below)
  2 pts MSDS data (see instructions below)

Lab report (15 pts, may be adjusted as applicable):
  5 pts experimental data (make notes alongside procedure steps / activities – “to do list”)
  5 pts analysis / calculations / results
  3 pts discussion (qualifications, observations)
  2 pts conclusion (what occurred overall) and afterthoughts (if experiment were to be done
again)

Technique (3 pts, subject to adjustment at the instructor’s discretion), includes: 1) attention to safety,
housekeeping, and level of preparation; 2) following directions, effectively interacting with others, and
independent work; 3) effort expended.
Pre-Lab Preparation: Success in the lab depends on sufficient advance planning. Reading, understanding (to the extent possible), and preparing must be undertaken BEFORE an individual student may proceed with an experiment. The pre-lab write-up must be witnessed by instructor or teaching assistant (and signed-off), before proceeding to the experiment of the period. An unannounced quiz may be administered before any experimental work begins; for that quiz, students may access their pre-lab write-ups in their notebooks, but not their texts. For safety reasons, if any student does not arrive ON TIME, that student will not be permitted to perform that experiment and that student will be ASSIGNED A ZERO SCORE FOR THAT EXPERIMENT; and the student’s pre-lab preparation will NOT be graded.

Notebook Format and Organization:
- Numbered pages: every page must be numbered, sequentially (1, 2, 3, 4, etc.).
- Title of experiment
- Date
- References: cite any original procedure, as applicable (publication, author[s], date of publication, and page numbers, etc.).
- Reaction(s): write out pertinent reaction(s).
- Procedure summary: write one paragraph briefly summarizing a) the purpose of the experiment and b) how that purpose will be achieved.
- Chemicals: as applicable, for each substance used in the reaction, record, in a table format, its:
  - name
  - quantity used (include density for liquids)
  - formula weight
  - number of moles
  - number of equivalents
  - other, as needed
- MSDS information: mention safety qualifier(s) and proper safety disposal(s).
- Procedure: write detailed steps / activities – “to do list.”
- Analysis, any calculations, and results
- Discussion and observations; record any pertinent thoughts and consider what one might do differently if given another opportunity.
- Conclusion: state the obvious and how you might better perform your experiment, given an opportunity to do so

Notebook Content: all data must be included in the notebook and all writing must be done in ink. Mistakes are to be crossed out with a single line (you may need to refer to the mistake at a later time and even reconsider its deletion) and the revised data or observation noted. If data are missing, a lab report may be downgraded. If applicable, any spectra collected must be labeled with the experiment name and/or compound name, then taped or stapled into the notebook. As applicable, each student must have the spectrum or spectra in his/her own notebook; if only one original is acquired, the partner may insert a photocopy. Make experimental notes as you perform the procedure. Write down what you do, as you do it. Write down what you observe, as you observe it. This is important because in some cases we may not be following the written procedure exactly as written. Be ready and be attentive to respond accordingly if the lab instructor announces any changes or tips or extra cautions at any time during the lab period.

Lab Reports: Lab reports will vary in scope depending on the experiment. Lab reports may consist of:

a) a detailed multi-page report (which is incorporated in the lab notebook to be collected subsequently) or
b) a separate stand-alone written report.

The student will be informed as to what is required in each instance, at the beginning of the lab. If you are not sure what is required, ASK!!! It is YOUR responsibility to do so!
Safety and Material Disposal (using MSDS):

**Safety:** Summarize any safety concerns and material disposal concerns for the experiment. Safety information can be obtained from a Material Safety Data Sheet (MSDS). The student experimenter is required to look up the MSDS for any and all organic compounds used or made in the experiment. Also, look up the MSDS for any inorganic compounds noted as hazardous in the lab manual (e.g., strong acids or bases). For each substance, write key words from Section 3, Hazards Identification (MSDS) for each compound. Some examples of key words: poisonous, toxic, sensitizer, cancer hazard, fatal if swallowed.

The student may obtain the MSDS for a compound via the Internet. There are free websites where the MSDS can be searched and printed. One website index is: [www.ilpi.com/msds/index.html](http://www.ilpi.com/msds/index.html). Although there are many websites for this purpose, a few previously used websites are described below. (Let us know which work the best for you.)

- **Sigma-Aldrich** – a supplier of organic chemicals, at [www.sigmaaldrich.com](http://www.sigmaaldrich.com). This site requires you to register and create a password. There is no charge for registering; it is for tracking purposes.

- **J. T. Baker** – an organic chemical supplier, at [www.jtbaker.com](http://www.jtbaker.com); you may need to type in the URL directly. These MSDSs are very informative and sometimes easier to read.

- **Alfa Aesar** – at [www.alfa.com](http://www.alfa.com). This site may be easier to use than the Sigma site and may offer more compounds than the Baker site.

The LUC Chemistry Department Organic Chemistry Laboratory Coordinator offers a suggestion: link: [www.hz.genium.com](http://www.hz.genium.com); username: Loyola; password: msdslibrary.

**Disposal:** Most substances used in the organic laboratory CANNOT be flushed down the drain. Therefore, waste containers are located in the hoods for your disposals. As a part of your pre-lab preparation, classify into which container each substance should be placed for disposal or whether it should go down the drain. Generally, there is a container for organic liquids and a separate container for organic solids. Most inorganic compounds (salts, acids, and bases) can be washed down the drain with copious amounts (LOTS) of water. But some inorganics containing certain metals (Ag, Pb, Hg, Cr, Mn, Cd) CANNOT go down the drain. These compounds have their own special containers. **If you are not sure where to dispose of a particular substance, ask the instructor for advice before disposal.** All substances used in an experiment should be listed under one of the four headings, as shown in the sample table below:

<table>
<thead>
<tr>
<th>Organic-Liquid Waste</th>
<th>Organic-Solid Waste</th>
<th>Water-Soluble Waste (down the drain with large amounts of water)</th>
<th>Special Waste (heavy metals, halogens, etc.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ethyl acetate*</td>
<td>benzoic acid*</td>
<td>sodium sulfate*</td>
<td>KMnO$_4$ *</td>
</tr>
<tr>
<td>acetanilide*</td>
<td>sulfuric acid*</td>
<td></td>
<td>Br$_2$ *</td>
</tr>
</tbody>
</table>

* The sample compounds above are listed as examples only.

**Pre-lab safety and disposal must be included in your pre-lab preparation.** If you come to lab without the pre-lab preparation, we must assume that you are not yet prepared for the experiment. In that you will be working in groups or with partners, it is not fair that one student should shoulder most of the work in an experiment because his/her partner chooses to come to the lab unprepared. More importantly, unprepared students are a safety hazard to themselves and to their classmates. Therefore, **unprepared students will not be allowed to perform the experiment** and those students will be **ASSIGNED A ZERO SCORE FOR THAT EXPERIMENT**, as an unexcused absence; and the student’s pre-lab preparation will NOT be graded.
**Lab Clean-Up:** Laboratory hygiene is important and essential to lab safety. You must clean up your hood and any community equipment you have used when you are finished using it. **Students leaving messes in their hoods or in community areas will have EXTRA points deducted.** At the end of a lab, the student is responsible that:

- community equipment has been returned to its proper place.
- community chemicals have been returned to their proper hood.
- the hood spaces are free of chemical spills and debris.
- bench tops are clean and free of chemicals and debris.
- balances are clean and free of chemicals and debris.

Occasionally, we all forget to clean up after ourselves; but, a respectful, professional reminder from anyone should be all that is required. Chemical residues left uncleaned can be especially hazardous to the next experimenter who is not aware that the residue is present or of what they are composed. All glassware must be cleaned and stored in the assigned locker / space / drawer. **Poor chemical hygiene will not be tolerated.** If any remaining chemical spills or unstored glassware are discovered by the instructor or any designated personnel, **students will lose points for EACH violation in a given lab period.**

**Post-Lab Procedure:** As applicable, complete lab notebook entries that period and prepare stand-alone report to be handed in subsequently, if required. Finally, ask instructor to sign-off (witness completion of that period’s experiment).

**Laboratory Attendance, Punctuality, Attention, and Participation:**
- important and required.
- **NO MAKE-UPS.** A missed lab is a missed opportunity, earning ZERO for that experiment.

**Phones and Pagers:** Please be courteous and respectful of others. Silent mode during laboratory. No phone conversations in the laboratory, before, during, after the laboratory period.

**Academic Honesty:** Essential, expected, and enforced. Each student submits his/her own work, although data may be shared, activities during the experiment may be split, and collaborative discussion may ensue within a group during the experiment. Dishonesty dictates consequences which may include: (1) notification of Chemistry Department Chair, student’s Department Chair, and CAS Dean, (2) documentation in the student’s official record, and (3) dismissal from the university. Immediate consequences will include a *ZERO* on any item in question. Please refer to the LUC Undergraduate Handbook on policies or the CAS website: [http://ww.luc.edu/cas/pdfs/CAS_Academic_Integrity_Statement_December_07.pdf](http://ww.luc.edu/cas/pdfs/CAS_Academic_Integrity_Statement_December_07.pdf).

**Typical Day in the Lab:**

1. Arrive on time, having read the experiment(s) and procedure(s), having completed the pre-lab preparation, and having noted so in your notebook.
2. Listen to the pre-lab instructions, changes, cautions, and caveats. If unsure about the pre-lab readings or the pre-lab instructions, ask questions, get clarification.
3. Get pre-lab write-up witnessed, i.e., obtain instructor sign-off.
4. Take pre-lab quiz, if applicable, using pre-lab preparation in notebook only.
5. Set up experiment.
6. Perform experiment at standard scale, unless instructed otherwise.
7. Do analyses, calculations, and finish in-class notebook write-up.
8. Clean up bench and all common areas. Don’t be shy. Remind others to do their part. And be sure to do yours.
9. Hand in results and lab write-up and/or get instructor sign-off.
10. Submit lab notebook subsequently for grading, as applicable and as instructed.

**Check-Out Procedure:** At the end of the term, even if you choose to drop the course, the student must return at check-out to settle his/her University account. No grade will be issued for a student who has not formally checked out of lab. The University places a HOLD on any future registration and any documentation requests, if not formally checked out of lab. The student must account for all damaged or missing equipment, whether from the lab drawer, from community stock, or supplied extra for a specific experiment.
Schedule (tentative, subject to adjustment):

<table>
<thead>
<tr>
<th>Date</th>
<th>Activity / Experiment</th>
<th>Text pp.</th>
</tr>
</thead>
<tbody>
<tr>
<td>June 30</td>
<td>Safety, orientation, check-in</td>
<td></td>
</tr>
<tr>
<td>July 2</td>
<td>1. Reduction of benzophenone using sodium borohydride (→ alcohol)</td>
<td>1-4</td>
</tr>
<tr>
<td>July 7</td>
<td>2. Diels-Alder</td>
<td>5-8</td>
</tr>
<tr>
<td>July 9</td>
<td>3. Nitration</td>
<td>17-18</td>
</tr>
<tr>
<td>July 14</td>
<td>4. Ketone Derivatives</td>
<td>9-10</td>
</tr>
<tr>
<td>July 16</td>
<td>5. Acylation of an Aromatic Amine</td>
<td>15-16</td>
</tr>
<tr>
<td>July 21</td>
<td>6. Grignard Reaction (→ carboxylic acid)</td>
<td>Thomas handout + 23-26</td>
</tr>
<tr>
<td>July 23</td>
<td>Grignard Rxn (continued)</td>
<td></td>
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<td>July 28</td>
<td>7. Synthesis of Isopentyl Acetate (Ester) plus Combinatorial Ester Syntheses</td>
<td>27-30 + Szpunar handout</td>
</tr>
<tr>
<td>July 30</td>
<td>8. Aldol Condensation</td>
<td>35-36</td>
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<tr>
<td>Aug 4</td>
<td>9. Chemistry of Dyes</td>
<td>Thomas handout</td>
</tr>
<tr>
<td>Aug 6</td>
<td>Check-out</td>
<td></td>
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</tbody>
</table>