CHEM 321L  Organic Chemistry Laboratory Syllabus  Fall 2016
Linfield College, McMinnville, Oregon (Also See Course Syllabus)

Required Text and Materials:
- Course Website: [http://www.linfield.edu/chem/current-courses/chem-321.html](http://www.linfield.edu/chem/current-courses/chem-321.html)
- **New** bound lab notebook with duplicate pages (purchased from bookstore or LSUC in the chemistry department)
- Appropriate laboratory attire: safety glasses, long pants, closed toe shoes

**NOTE: Disability Statement:** Students with disabilities are protected by the Americans with Disabilities Act and Section 504 of the Rehabilitation Act. If you are a student with a disability and feel you may require academic accommodations please contact Jennifer Gossett, Learning Support Services Coordinator (LSS), as early as possible to request accommodation for your disability. The timeliness of your request will allow LSS to promptly arrange the details of your support. LSS is located in Walker Hall, Room 126 (503-883-2562). We also encourage students to communicate with faculty about their accommodations.

**Academic Integrity:** Please read the section “Academic Integrity” in the college catalog (or at [http://www.linfield.edu/academic-affairs/curriculum.html](http://www.linfield.edu/academic-affairs/curriculum.html)). This class will adhere to the policy as published in the Linfield College Course Catalog. Work presented with your name on it is assumed to be your own, unless designated otherwise. Students engaging in cheating, plagiarism, fabrication, or aiding in such activities are subject to disciplinary action.

**COURSE OBJECTIVE AND PURPOSE:**
Experiments are chosen to offer practical experience with, and enhance the understanding of, topics covered in the lecture portion of the course. They also introduce students to common methods and procedures used in the organic chemistry laboratory.

On Mondays in class or Tuesdays in discussion there may be a brief outline of the experiment to be performed that week, depending on its difficulty. Bring your lab notebook with the protocol for the experiment written in your own words and be prepared to discuss the material for that week’s experiment (see below for laboratory notebook requirements).

An integral component of this course is learning how to write a laboratory report, and to properly prepare a laboratory notebook, complete with purpose, procedure summary, material toxicity, data tabulation, observations, and conclusions. Equally important is a focus on conducting experiments in a safe and efficient manner, and keeping a clean and organized workspace.

### Laboratory Schedule

<table>
<thead>
<tr>
<th>Dates</th>
<th>Report</th>
<th>Laboratory Experiment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Week 1</td>
<td>Aug 29 - Sept 02</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Check In</strong> - All students must participate</td>
</tr>
<tr>
<td>Week 2</td>
<td>Sept 05 – 09</td>
<td>Introduction Focus</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Choose one of the options for Experiment #1 on the course website</td>
</tr>
<tr>
<td>Week 3</td>
<td>Sept 12 – 16</td>
<td>Optional Draft</td>
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<tr>
<td></td>
<td></td>
<td><strong>Isolation</strong> of Caffeine Experiment Part One</td>
</tr>
<tr>
<td>Week 4</td>
<td>Sept 19 – 23</td>
<td>Experimental Focus</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Isolation</strong> of Caffeine Experiment Part Two</td>
</tr>
<tr>
<td>Week 5</td>
<td>Sept 26 – Sept 30</td>
<td>Workshop (No Report)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Organic Chemistry Nomenclature and Chair Modeling</td>
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<tr>
<td>Week 6</td>
<td>Oct 03 – 07</td>
<td>Experimental Focus</td>
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<tr>
<td></td>
<td></td>
<td><strong>Resolution</strong> of Enantiomers</td>
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<tr>
<td>Week 7</td>
<td>Oct 10 – 14</td>
<td>Experimental Focus</td>
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<tr>
<td></td>
<td></td>
<td><strong>Sx2:</strong> Synthesis of 2-ethoxynaphthalene</td>
</tr>
<tr>
<td>Week 8</td>
<td>Oct 17 – 21</td>
<td>Results and Discussion Focus</td>
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<tr>
<td></td>
<td></td>
<td><strong>E2:</strong> <strong>Dehydrohalogenation</strong> of cyclohexyl bromide</td>
</tr>
<tr>
<td>Week 9</td>
<td>Oct 24 – 28</td>
<td>Results and Discussion Focus</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>E1:</strong> <strong>Dehydration</strong> of cyclohexanol</td>
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<tr>
<td>Week 10</td>
<td>Oct 31 – Nov 04</td>
<td><strong>Major Report</strong></td>
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<tr>
<td></td>
<td></td>
<td><strong>Oxidation</strong> of cyclohexene to adipic acid</td>
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<tr>
<td>Week 11</td>
<td>Nov 07 – 11</td>
<td>Worksheet (No Report)</td>
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<tr>
<td></td>
<td></td>
<td><strong>Sx1:</strong> <strong>Solvolysis</strong> of t-butyl chloride</td>
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<tr>
<td>Week 12</td>
<td>Nov 14 – 18</td>
<td>Worksheet (No Report)</td>
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<tr>
<td></td>
<td></td>
<td>NMR and IR: Deduce the Structure</td>
</tr>
<tr>
<td>Week 13</td>
<td>Nov 21 – 25</td>
<td>Thanksgiving Break – No Lab</td>
</tr>
<tr>
<td>Week 14</td>
<td>Nov 28 – Dec 02</td>
<td><strong>Major Report</strong></td>
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<tr>
<td></td>
<td></td>
<td><strong>Reduction</strong> of a ketone to an alcohol</td>
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<tr>
<td>Week 15</td>
<td>Dec 05– 09</td>
<td>None</td>
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<tr>
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<td></td>
<td><strong>Check Out</strong> – All students must participate</td>
</tr>
<tr>
<td>Week 16</td>
<td>Dec 12 - 16</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Finals Week</strong></td>
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</tbody>
</table>
Grading System:
The total of all lab credit will be normalized to 150 pts of 800 total course pts. *If you fail the lab, you fail the entire course*

Most experiments are worth **20 points** apportioned as follows:

**Preparation and Conduction of Experiments**

Your laboratory instructor and teaching assistant will evaluate the following before, during, and after lab (**10 points**):

- **Laboratory notebook and pre-lab questions** before you start the experiment. Pre-lab questions for a given experiment can be found at the end of that week’s experimental handout. A sample pre-lab is given in the ‘Notebook Requirements’ section of the syllabus. The initial notebook check is worth **5 points**.

- **Work habits & competence at common lab skills** - timeliness, safety, overall neatness (including care of glassware, common areas, and bench top), ability to efficiently assemble each laboratory apparatus, effectively use laboratory equipment, control reaction conditions, and obtain desired results, *etc*. Your competence at common lab skills is worth **3 points**.

- **Data and observations** have been recorded clearly in the laboratory notebook at the conclusion of your experiment. Your data and observations are worth **2 points**.

**Demonstrating Your Knowledge after Conducting Experiments**

Your laboratory instructor and teaching assistant will evaluate what you’ve learned with several types of assignments:

1. **Laboratory Reports:**

   Reports are **due at the start of lab ONE week** after the conclusion of an experiment as a hard copy with all notebook pages (pre-lab and in-lab) attached AND an electronic copy sent to vsiller@linfield.edu. Name the file with last name, year, and experiment (EX: “Siller2016E1”). Laboratory reports will be assessed for proper English grammar, clarity, appropriateness of prose, conciseness, scientific accuracy, and proper formatting.

   Laboratory reports will focus on 6 main areas:
   
   1. **Introduction** - complete with the reaction and/or mechanism
   2. **Experimental methods** - protocol
   3. **Results** – Supporting data and observations
   4. **Discussion** - Interpret results
   5. **Appendix** – Supporting calculations etc.
   6. **References** – All necessary citations

   There are two report formats:

   - **Minor Lab Report** – **(10 points)**. Minor lab reports will focus on a single section of the report. Focus sections are meant to allow mastery of each piece individually thus most of the score is based on the focus section. Templates may be provided to aid with formatting and content.

   - **Major Lab Report** – **(20 points)**. Major lab reports are full reports for which no template will be provided. See the ‘Lab report format’ section of this document for an outline and use the guidelines provided on the course website for details. Two major lab reports will be required for this course.

   Each section of the lab reports should be written to answer the following five questions:

   1. What were the objectives that this experiment was designed to address?
   2. What was the rationale for the objectives?
   3. Exactly how did you do the experiment?
   4. What did you discover? (i.e. what results did you get?)
   5. How did the results serve the objectives?
2. Quizzes (5 points):
Quizzes over an experiment will be given the following Tuesday during Discussion. The purpose of the quiz is to evaluate what you have learned about the experiment’s equipment, techniques, and objectives.

3. Extra Credit (10 points):
There will be one ongoing opportunity for extra credit during the fall semester – a journal reflecting on topics you’re learning about in lab. This is an all-or-nothing opportunity: All entries must be complete to receive credit. Journal entry prompts can be found on the ‘Current Course Offerings’ page of the department website.

Grading and Feedback

The lab instructor puts thought and effort into feedback on lab reports. Please keep in mind that there are a lot of students in this course. Please feel free to submit any questions about grades and feedback via email (vsiller@linfield.edu) and/or set up a time to discuss it in person.

If your score on a report falls at or below 60% please make an appointment as soon as possible with the instructor. They will discuss with you some ways to improve your writing. You may revise and resubmit up to two lab reports during the semester. If you choose to revise a report, the revision is due no later than ONE week after the original report was returned to you. No revision will be accepted after that time. The submission must include the original report and the revision.

Proper writing is an important component of this course, please ask someone to proofread your laboratory report prior to submitting it. Check that reports are grammatically correct and make sense. Remember that what you turn in must be your own work. If you to work with a partner or a group on a lab assignment, give credit where credit is due. The Linfield Writing Center is a great place to go for basic writing help. Also, it is often nice to receive help from someone who doesn’t know the chemistry concepts – they can’t fill in all the conceptual gaps on their own.

Writing Center Locations and Hours: T.J. Day 321 SUN-THURS 3:30-5:30 PM; 6:30-8:30 PM
Nicholson Library SUN-THURS 8:00-10:00 PM

Drawing structures

The computer program ‘ChemDraw’ will be used to draw molecular structures and reactions electronically. It can be downloaded to personal computers (PC or Mac), or used on campus computers. The download information can be found on the course webpage.

Reference citations

Cite all sources in American Chemical Society (ACS) format. The direct link on the course webpage can help with proper citation format.

There is an online reference generator to help create accurate references for lab reports (it’s also an app). Sign up for free at: https://www.refme.com

Also, references need to be cited for the physical properties given in pre-lab materials, along with any additional external sources utilized to complete the lab assignment.
LABORATORY NOTEBOOK REQUIREMENTS

The objective of the lab notebook is to encourage you to develop a habit of keeping complete and useful records of your lab work. All writing must be legible.

PRE-LAB: When you enter the lab your notebooks are checked for a complete pre-lab. Pre-labs must be complete BEFORE you start that day’s experiment. Pre-labs should contain the following eight items:

1. **TITLE OF EXPERIMENT** (entire title given on handout)
2. **SOURCE REFERENCE** (where did you find the protocol? – COMPLETE URL AFTER DOCUMENT DISPLAYS, OR PROPER BOOK CITATION INCLUDING PAGE NUMBERS – USE AMERICAN CHEMICAL SOCIETY FORMAT)
3. **PURPOSE STATEMENT**: (Objective of experiment: chemical concepts to be illustrated, type of reaction, techniques not previously used, etc…)
4. **DEFINITIONS**: (Define all NEW concepts and techniques to the laboratory – required every week; suggested terms may be underlined in the experimental handout)
5. **BALANCED CHEMICAL REACTION AND REACTION MECHANISM IF APPROPRIATE** (omit spectator ions)
6. **MATERIAL CHARTS** (reactants + products in Table 1, and solvents + potential byproducts to follow – See PRE-LAB SAMPLE for how to set this up; Include citations to footnote references provided at end of pre-lab pages; Include calculations for finding limiting reagent – see below, and pre-lab sample

**BASIC SETUP FOR DETERMINING LIMITING REAGENT AND CALCULATING THEORETICAL YIELD:**

\[
\text{2X + Y} \rightarrow \text{YX}_2
\]

\[
\begin{array}{ccc}
\text{Grams} & 10.00** & 8.65** \\
\text{MW} & 132.0 & 173.0 \\
\text{Moles} & 0.07576 & 0.050 \\
\text{MOLES OF REACTION THAT CAN BE RUN} & 0.07576/2 & 0.050/1 \\
\text{= MOLES USED/STOICH.MOLES*} & \text{LR} & \text{LR} \\
\text{=0.0379} & =0.050 & =0.050 \\
\text{THEORETICAL YIELD} & 11.56 & 305.0 \\
\text{0.0379*1=0.0379} & & \\
\end{array}
\]

* limiting reagent = lowest quotient
** Once you have completed the experiment you will use the ACTUAL AMOUNTS USED to determine TRUE theoretical yield for your product

7. **NON-Routine EQUIPMENT SETUP; PROCEDURE** (condensed but clear protocol)

(left hand side, ¼ of pg width) (right hand side, ¼ of pg width)

**APPARATUS DRAWINGS (if needed)** **DATA**

**PROTOCOL (Number each step)** **OBSERVATIONS**

8. **PRE-LAB QUESTIONS** (answered completely, after procedure)

9. **REFERENCES** (for pre-lab sources such as properties of reagents and products, use ACS format)

IN LAB: Write data and observations as you see them. DO NOT wait until the end of lab to try to write everything down.

LAB CHECK-OUT: Before you leave the lab:

1. Areas you used must be clean. All equipment must be clean, complete, and in its proper place.
2. A staff member must sign your lab notebook.
   The staff member will check your notebook for the following:
   1. CONCISE OBSERVATIONS OF WHAT HAPPENED AND DATA
   2. ALL MISTAKES STILL LEGIBLE (cross out with only a single line)
   3. YOUR SIGNATURE AND DATE
3. Take a picture or write down information from the board to help you study for the quiz and write the report.
Sample PRE-LAB (written into your lab notebook)

FILL OUT TOP, PROVIDING AT LEAST NAME, PARTNER (IF APPLICABLE), DATE, AND LAB SECTION

**Title:** An E2 Reaction: Dehydrohalogenation of Cyclohexyl Bromide


**Purpose:** The purpose of the “Dehydrohalogenation of Cyclohexyl Bromide” experiment is to learn a typical E2 (elimination, bimolecular) reaction. A strong base removes a β-hydrogen atom as the halide leaving group departs from the α-carbon in an anti-periplanar orientation in the rate determining step. The most stable (most substituted) alkene product is the result. Other concepts to be practiced are reflux, extraction, distillation, and two chemical tests that can suggest the presence of unsaturated bonds.

**Definitions:**
- **Dehydrohalogenation:** an elimination reaction that results in the loss of H-X from adjacent carbons of the substrate and the formation of a π-bond between those carbons.
- **Reflux:** the boiling of a liquid in an apparatus that condenses the vapor and returns it to the boiling flask. The purpose is to avoid loss of liquid while maintaining a constant high temperature in the boiling flask.
- **β-hydrogen:** a hydrogen atom on the carbon adjacent to a carbon bearing a functional group.
- **Leaving Group:** an atom or group of atoms that departs from a substrate during a substitution or elimination reaction.
- **α-Carbon:** the carbon bearing a functional group of interest.

![Bimolecular elimination mechanism (E2)](image)

**Table 1: Reactant & Product Properties and Computed Theoretical Yield based on Actual Amounts Used**

<table>
<thead>
<tr>
<th>Reactant(s)/Product(s)</th>
<th>1 cyclohexyl bromide</th>
<th>+ 1 potassium hydroxide</th>
<th>→ 1 cyclohexene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amount used (g or mL)</td>
<td>13.29 g</td>
<td>19.5 g</td>
<td>6.724 g (theoretical)</td>
</tr>
<tr>
<td>MW</td>
<td>163.06 g/mol</td>
<td>56.11 g/mol</td>
<td>82.15 g/mol</td>
</tr>
<tr>
<td>Moles used (“moles of reaction” that can be run)</td>
<td>0.08185 / 1 = 0.08185 mol (LR)</td>
<td>0.3475 / 1 = 0.3475 mol</td>
<td>0.08185 (theoretical)</td>
</tr>
<tr>
<td>Equivalents</td>
<td>0.08185 / 0.08185 = 1</td>
<td>0.3475 / 0.08185 = 4.25</td>
<td>1</td>
</tr>
<tr>
<td>Physical properties (bp/mp, density)</td>
<td>d: 1.329 g/mL, bp: 163-165°C</td>
<td>salt*</td>
<td>d: 0.8098 g/mL, bp 83°C</td>
</tr>
<tr>
<td>Chemical hazards (or special notes)</td>
<td>Severe burns, corrosive</td>
<td>Flammable, irritant</td>
<td></td>
</tr>
</tbody>
</table>

**Non-reactant chemicals (solvents, potential byproducts, etc.):**

<table>
<thead>
<tr>
<th>Substance/Solution</th>
<th>Physical Properties</th>
<th>Toxicity/Hazards</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>MW:46.07g/mol, d:0.794g/ml, bp:78°C</td>
<td>flammable liquid, irritant, toxic</td>
</tr>
<tr>
<td>Calcium chloride</td>
<td>MW:110.98g/mol</td>
<td></td>
</tr>
<tr>
<td>Hexane</td>
<td>MW:86.18g/mol, d:0.660g/ml, bp:69°C</td>
<td>flammable liquid, irritant</td>
</tr>
<tr>
<td>Bromine</td>
<td>MW:159.82g/mol, d:3.102g/ml, bp:59.5°C</td>
<td>highly toxic, oxidizer</td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>MW:153.82g/mol, d:1.594g/ml, bp:76-77°C</td>
<td>cancer suspect agent, toxic</td>
</tr>
<tr>
<td>Potassium permanganate</td>
<td>MW:158.04g/mol</td>
<td>oxidizer, corrosive</td>
</tr>
</tbody>
</table>

*Note: the density and m.p. for salts are not shown (These data are not relevant to organic chemical experiments)*
Procedure:
(Outline)

1) Weigh 10 g cyclohexyl bromide into 100 ml rb flask
2) Weigh ~20 g KOH into rb flask & add stir bar
3) Add 25 ml 95% EtOH
4) Reflux gently for 45 minutes
5) Allow to cool to RT
6) Pour into sep. funnel containing 50 ml of H₂O
7) Wash rb flask 2X w/10 ml of H₂O & add to sep. funnel
8) Shake; discard lower layer
9) Extract upper layer again with 80 ml H₂O
10) Dry organic layer in erlenmeyer over anhydrous CaCl₂ (Swirl 10 min)
11) Distill product into pre-weighed receiving flask
12) Bromine absorption test:
    a) Mix 1 ml hexane & 1 drop 2M Br₂/CCl₄ in each of 3 test tubes
    b) Label test tubes +, -, & s (positive control, negative control and sample, respectively)
    c) Add 1 drop of an alkene, an alkane and the product to the respective test tubes and observe the result
13) Permanganate test:
    a) Mix 1 ml water & 1 drop KMnO₄ soln in each of 3 test tubes
    b) Label test tubes +, -, & s (positive control, negative control and sample, respectively)
    c) Add 1 drop of an alkene, an alkane and the product to the respective test tubes and observe the result
14) Dispose of chemicals properly and clean up

You may want to draw a special setup here.

Answer pre-lab questions at end of protocol, clearly labeled.

References
2 Ibid.
Lab Report Format

Sample Outline with key requirements.

- **Title**

- **Introduction** containing:
  - *Experiment objective(s)* (what was the purpose of performing the experiment?)
  - *Context* (how the experiment is meant to meet the objectives—pertinent concepts and techniques included)
  - *Rationale* (why is the objective worthwhile?)
  - *Figure 1* (see experimental handouts for reactions, mechanisms, etc.)

- **Experimental methods** (Refer to experiment handout for examples of writing a *concise* experimental protocol)

- **Results** present actual data/observations that support your conclusions (e.g., mp(bp range, IR spectra, chemical tests, appearance, percent yield, etc.)

- **Discussion** containing:
  - Reiteration of the objective.
  - Comments on how the results compare to published values and what conclusions can be drawn.
  - % yield or % recovery: plausible reasons for deviations from 100 percent,
  - Chemical concepts supporting results/interpretations (including figures when appropriate)
  - Summary sentence indicating whether or not the objectives were met.

- **References** Provide appropriate citations throughout the report as superscripts (include citation for protocol source)

- **Appendix** containing:
  - Calculations, protocols for chemical tests, and/or additional supporting figures.

**Required format:**

- Complete sentences.
- Times New Roman 11 or 12 point font.
- 1.5 or 2 line spacing for the main body of each section.
- Bolded section headings.

**Do not include:**

- Glassware
- Equipment
- Common laboratory techniques
- Solution making procedures
- Routine operational details

**Do include:**

- All reagents and solvents
- Solution concentrations
- Reaction times and temperatures
- Concise isolation and purification method
- Chemical amounts used

See the course website for guidelines to writing each section of a lab report.