Molecular Structure and Organic Synthesis Laboratory Syllabus CHEM 4320 LB
2 Semester Hours
Spring 2014
NCF Annex, Room 360

Section 01:  MW  2:00 – 4:50 PM  Dr. Jayalakshmi Sridhar
Section 02:  TR  9:25 – 12:15 AM  Dr. Florastina Payton-Stewart

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Office hours: MTR 10am - 12pm

Dr. Payton-Stewart Office: NCF 305 Phone: 520- 7381 e-mail: flpayton@xula.edu
Office hours: M 3-5PM; R 1-4PM; F 11AM-noon

We are frequently available for help beyond the office hours by appointment, so please use this opportunity as well.

Course Description:
Students perform multi-step synthesis and identify unknown and synthesized compounds. A variety of chemical and spectroscopic characterization methods are used. This course seeks to bridge the gap between the elementary organic lab and the advanced organic research lab. It allows the student to develop critical reasoning skills, computational skills and oral and written presentation skills necessary for a professional career in science.

Prerequisites: C or better in Chem 2220 Lecture/Drill, Chem 2230-2240 Labs (Organic Chemistry)

Required Text (and other required material):
2. Bound notebook for the laboratory (Your lab notebook from Quant can be used but only if there is enough consecutive pages left for this class, ~20 pages.)
4. Lab coat, goggles, closed-toe shoes and calculator

Additional books suggested for reference:
(References are available in the library. Several reference texts are available in the lab as well)
• The Aldrich Library of FT-IR Spectra, Charles J. Pouchert, Aldrich Chemical Company
• The Aldrich Library of NMR Spectra, Charles J. Pouchert, Aldrich Chemical Company
• The Aldrich Library of C-13 and H-1 NMR Spectra, Charles J. Pouchert and Jacqlynn Behnke, Aldrich Chemical Company
• CRC Handbook of Chemistry and Physics
• Merck Index
• Aldrich chemical catalog and other chemical catalogs (copies are in lab and on reserve) There is also a Blackboard webpage with extra handouts.

In the event of evacuation, assignments and other course materials will be posted on Blackboard. Students will be informed of class expectations by e-mail, the Xavier Chemistry website and/or the Blackboard website. It is the student’s responsibility to check the websites and his/her Xavier e-mail account for information.

Course Objectives and Student Learning Outcomes:
• To develop qualitative thinking skills and problem solving techniques through the identification of unknowns and data analysis. These skills are needed in any scientific or technical career.
• To develop the ability to organize and carry out a scientific investigation independently.
• To develop the laboratory skills and techniques (both instrumental and chemical) used to identify and characterize organic compounds, both unknown and products of reactions.
• To develop the skills and technique to carry out a multi-step synthesis.
• To develop and strengthen the skills to present laboratory results both in writing and orally.
• To develop and strengthen library research skills.
• To develop computer skills for chemical research.

Course Requirements: Students are expected to attend every class meeting as scheduled; there are no make-up lab sessions. Students who miss 3 or more classes cannot pass the course. Students are also expected to check the Blackboard and Xavier e-mail account for information regularly. The midterm exam is closed book and will cover information related to the identification of unknown compounds, including spectroscopy. The lab performance grade includes factors such as: attitude, effort, following directions, being on time for class, wearing safety goggles, cleaning up work space. Of the 50 points, one point per day will be awarded for daily check-out. It is the student’s responsibility to find the instructor for check-out.

Course Assignments and Evaluation:
Unknown Reports (100 points x 2 reports = 200 points)
Column Chromatography (25 points)
Squalor Exercise (20 points)
Spectroscopy problem sets (20 + 25 + 20 points = 65 points)
Midterm Examination (100 points)
Synthesis pre-labs (25 points x 2 steps = 50 points)
Report for each synthetic step (50 points x 2 syntheses = 100 points)
Final Report on synthesis (50 points)
Oral Report (25 points)
Daily objectives list (5 points x 6 unannounced checks; count the best 5 = 25 points)
Notebook (25 points)
Lab Performance (50 points)
Total Possible = 710 points
The method of assigning letter grades will not be more stringent than the following:
A, 90% and above; B, 80-89%; C, 70-79%; D, 60-69%; and F, below 60%.

Assignments are due at the beginning of the class unless otherwise noted. No late problem sets will be accepted. If one of the other assignments is submitted late, 10% of the points for that exercise, per day (not per class) will be deducted. Assignments turned in too late to receive credit must be turned in to pass the course, no exceptions. Assignments will not be accepted after Quiet Day. The following is quoted from the Xavier University Faculty Handbook:
“...”

Academic Integrity: The following is quoted from the Xavier University Faculty Handbook:
“If a student's test, examination paper, laboratory report, term paper, or other written assignment gives evidence of not being completely his/her own work, he/she may be given an F for the course. A student who communicates with anyone during the course of an examination or test, unless with the permission of the instructor, may be immediately dismissed from the room and given an F. Such communication includes attempt to read from another's paper. If a student is found to have brought study materials into the examination room without the instructor's permission, it may be assumed that he/she intended to use such materials unlawfully, and he/she may be penalized accordingly.”

Students should also consult the Academic Integrity Policy:
http://www.xula.edu/cas/documents/cas_academicIntegrity.pdf

Laboratory Notebooks:
Lab notebooks are used by scientists to record data as they are collected and not later. Another person reading the notebook should be able to tell what experiments were done, what results were obtained, and when. Important questions involving priority, patent rights, and scientific fraud are often settled by referring to original data in lab notebooks. Therefore, it is important that they be kept correctly.

You must use a bound notebook and write in pen. You may use the notebook you have from Quant Lab, if enough consecutive pages are remaining. Loose-leaf and spiral notebooks and (especially) scraps of paper are not permitted. Cross out mistakes once; white-out may not be used. Pages may not be torn out. Write only the results of your lab work; do not include pre-lab lecture notes. Clarity and sound organization are prime virtues in a lab notebook. Neatness is nice, but it is often impossible. However you must write legibly so that others can read it. The notebook should be dated and signed each day you work. Leave the first page of your notebook blank for a Table of Contents. All pages should be numbered. Start each new unknown or new synthetic procedure on a new page. For unknowns, your data should include the measurements obtained (such as BP/MP) and not just the tests performed but also your observations, ie, green ppt formed. Include other information such as what spectra you obtained. Be sure to give the identity of the unknown at the end. For the reactions, you need to include the pre-lab write-up and any changes made to this
procedure when you ran the reaction. Observations and spectra obtained are also included. Notebooks will be evaluated at the end of the semester. Your instructor may inspect your notebook at any time during the semester, without warning.

**Unknown Reports:**
The first unknown has a preliminary report (30 pts) and final report (70 pts; word processed). For the preliminary report, the results of a specific battery of tests are tabulated on a form, and you will learn if you are on the right track in determining the unknown. The final report is a discussion of how a student determined the identity of the unknown and why other compounds are not the answer. This discussion must describe your thought process thoroughly, for maximum credit. The second unknown will be identified using at least three spectroscopic methods (IR, $^1$H NMR, $^{13}$C NMR, GC/MS). You will then carry out a reaction of the unknown, and isolate and identify the product. You will then purify the reaction product by column chromatography. There are four written parts for the second unknown: spectroscopic characterization and identification (35 pts; word processed), reaction pre-lab write-up (25 pts), final report (40 pts; word processed) and report on column chromatography (25 pts).

**Reports on Synthetic Project:**
Each of the synthetic steps has two written assignments, and in addition there is a final report and oral report. For each synthetic step, a detailed pre-lab write-up must be turned in and approved before a student can begin that procedure; see handout for details. After each synthetic reaction is completed, you will write out a complete experimental procedure, including characterization data, in *Journal of Organic Chemistry* format (www.chemistry.org; choose publications tab; find JOC). You will need to discuss whether the reaction worked and how you knew. The final, summary report is modeled on a *JOC* Note. Consult recent issues of *JOC* in the pharmacy library or online for examples, and handouts for more specific information. The oral report is a 15-minute presentation summarizing the final written report on your synthetic project.

**Other Comments:**
1. You are more like an independent researcher in this class with all the responsibilities of making decisions where appropriate. It is assumed that you know all previously learned techniques from Organic I and II labs.
2. If you encounter a problem or are not sure what you're doing, ask.
3. My answer to the question “Is this a positive test” is “I don't know”. If you want to see what a positive (or negative) test looks like, find a compound you know will give the appropriate result, and test it. Compare these results for known compounds with the result from your unknown.
4. Cell phones, beepers, and other electronic communication devices must be turned off during class.
5. As in all laboratory classes, no eating, drinking or horseplay in lab. Students who do not follow these rules will be asked to leave lab for the day. Safety goggles must be worn over the eyes at all times in lab, and sensible clothing (including closed-toe shoes and no Crocs) must be worn. Not wearing goggles, coming late for class, and using cell phones during class will cost you performance points, at your instructor’s discretion.
6. In lab, spend time on things that can only be done in lab; plan and prepare before coming to class!
## CHEM 4320LB Schedule for Spring 2014

<table>
<thead>
<tr>
<th>Date</th>
<th>Task</th>
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<th>Task</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jan 13, 14</td>
<td>Orientation &amp; discussion of unknowns Safety and spectroscopy lectures Work on 1st spectroscopy PS in class Check in</td>
<td>Jan 15, 16</td>
<td><strong>Due:</strong> PS #1 IR demo Start Unknown #1 PS # 2 given</td>
</tr>
<tr>
<td>Jan 20, 21</td>
<td>MLK Holiday Continue Unknown 1</td>
<td>Jan 22, 23</td>
<td>Discuss reaction prelab Continue Unknown #1</td>
</tr>
<tr>
<td>Jan 27, 28</td>
<td><strong>Due:</strong> Prelim report Complete Unknown #1</td>
<td>Jan 29, 30</td>
<td><strong>Due:</strong> PS #2 GC/MS lecture and demo Start Unknown #2 PS # 3 given</td>
</tr>
<tr>
<td>Feb 3, 4</td>
<td><strong>Due:</strong> Unknown #1 report NMR demo Continue Unknown #2</td>
<td>Feb 5, 6</td>
<td>Continue Unknown #2</td>
</tr>
<tr>
<td>Feb 10, 11</td>
<td><strong>Due:</strong> Esterification reaction prelab Continue Unknown #2 Discuss synthetic projects</td>
<td>Feb 12, 13</td>
<td>Complete Unknown #2 <strong>Due:</strong> Synthetic preferences Start esterification reaction</td>
</tr>
<tr>
<td>Feb 17, 18</td>
<td><strong>Due:</strong> PS #3 Continue reaction</td>
<td>Feb 19, 20</td>
<td><strong>Due:</strong> Unknown #2 report Continue reaction</td>
</tr>
<tr>
<td>Feb 24, 25</td>
<td><strong>Due:</strong> SQUALOR Complete esterification reaction</td>
<td>Feb 26, 27</td>
<td><strong>Due:</strong> First synthetic prelab Column Chromatography</td>
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<tr>
<td>Mar 3-7</td>
<td>Mardi Gras</td>
<td>Mar 10, 11</td>
<td><strong>MIDTERM EXAM</strong></td>
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<tr>
<td>Mar 12, 13</td>
<td><strong>Due:</strong> report on esterification Column Chromatography</td>
<td>Mar 17, 18</td>
<td>Start 1st reaction</td>
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<tr>
<td>Mar 19, 20</td>
<td>Continue 1st reaction</td>
<td>Mar 24, 25</td>
<td><strong>Due:</strong> Report on Column Chromatography Continue 1st reaction</td>
</tr>
<tr>
<td>Mar 26, 27</td>
<td><strong>Due:</strong> Second synthetic prelab Complete 1st step</td>
<td>Mar 31, Apr 1</td>
<td><strong>Due:</strong> Report on first rxn Start 2nd synthesis</td>
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<tr>
<td>Apr 2, 3</td>
<td>Continue 2nd step synthesis</td>
<td>Apr 7, 8</td>
<td>Continue 2nd step</td>
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<tr>
<td>Apr 9, 10</td>
<td>Complete 2nd synthetic step</td>
<td>Apr 14, 15</td>
<td><strong>Due:</strong> Report on 2nd rxn <strong>Due:</strong> Notebook Clean-up, checkout Clean-up.</td>
</tr>
<tr>
<td>Apr 21, 22</td>
<td><strong>Due:</strong> final synthesis report Oral Reports</td>
<td>Apr 23, 24</td>
<td>Oral Reports</td>
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