Molecular Structure and Organic Synthesis Laboratory Syllabus
CHEM 3220 LB - 2 Semester Hours
Spring 2010

NCF Annex, Room 360

Section 01: MW	**2:00 – 4:50 PM**	**Dr. Jason Abrams**
Section 02: TR	**7:50 – 10:40 AM**	**Dr. Hua Mei**
Section 03: WF	**11:00-1:50 PM**	**Dr. Galina Goloverda**

Dr. Mei Office: NCF 301C Phone: 520-5408 e-mail: hmei@xula.edu
Office hours: T 1:30-4:30 PM, W 9:00-12:00 noon
Dr. Abrams Office: NCF 301-B Phone: 520-5075 e-mail: jabrams@xula.edu
Office hours: T 1:00-3:00 PM, R 2:00-4:00 PM, F 11:00-1:00 PM
Dr. Goloverda Office: NCF 321 Phone: 520-5417 e-mail: gzgolove@xula.edu
Office hours: MT 9:00-11:00 AM, W 2:00-3:00PM

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):
1. Zubrick, J. W. *The Organic Chem Lab Survival Manual*, 7th ed., John Wiley & Sons, Inc., New York, 2008. (previous editions are acceptable)
2. Williamson, K. L. *Macroscale and Microscale Organic Experiments*, 4th ed., Houghton Mifflin Company, New York, 2003. (3rd edition is OK too)
3. **Bound** notebook for the laboratory (Your lab notebook from Quant can be used **but only if there are enough consecutive pages left for this class**, ~20 pages.)
4. Lab coat, Goggles and Calculator

Additional books suggested for reference:
(References are available in the library. Several reference texts are available in the lab as well)
- Shriner, R. L.; Hermann, C.K.F.; Morrill, T.C.; Curtin, D.Y. ; Fuson, R. C. *The Systematic Identification of Organic Compounds*, 7th Ed.; J. Wiley & Sons, Inc. New York, 2004. (on reserve)
- Pasto and C.R. Johnson, *Organic Structure Determination*, Prentice-Hall, Inc., Englewood Cliffs, N.J., 1989. (on reserve)
- Silverstein, R. M.; Webster, F. X. *Spectrometric Identification of Organic Compounds*, 6th ed., John Wiley and Sons, New York, 1998. (Copies are in lab and on reserve)
- *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.

<u>In the event that classes are cancelled due to a hurricane evacuation, assignments and other course materials will be posted on Blackboard. Students should access the Blackboard site as soon as possible following the evacuation. Also check xula emergency web site for information.</u>

Course Objectives:

- To develop qualitative thinking skills and problem solving techniques through the identification of unknowns and through 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. 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 75 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)
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 3 syntheses = 150 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	(75 points)
Total Possible =	785 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:
"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."

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, purify and identify the product. There are three written parts for this unknown: spectroscopic characterization and identification (35 pts; word processed), reaction pre-lab write-up (25 pts), final report (40 pts; word processed).

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 <u>all</u> 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 3220LB Schedule for Spring 2010

Jan 11-13	Orientation & discussion of unknowns Safety and spectroscopy lectures Work on 1st spectroscopy PS in class Check in	Jan13-15	**Due: PS #1** IR demo Start Unknown #1
Jan 18 Jan 19-20	Martin Luther King, Jr. Holiday Continue Unknown # 1	Jan 20-22	**Due: PS #2** Continue Unknown #1
Jan 25-27	**Due end of class: Prelim report** Complete Unknown #1	Jan 27-29	**Due: Unknown #1 report** Discuss reaction prelab NMR demo Start Unknown #2
Feb 1-3	**Due: Esterification reaction prelab** GC/MS lecture and demo Continue Unknown #2	Feb 3-5,	**Due: PS #3** Discuss synthesis projects Continue Unknown #2
Feb 8-10	**Due: Synthetic preferences** Complete Unknown #2 Start esterification reaction	Feb 10-12	**Due: SQUALOR** **Due: Unknown #2 report** Continue reaction
Feb 15-16 Feb 17	Mardi Gras Holidays Continue reaction Complete esterification reaction	Feb 17-19	**Due: First synthetic prelab** Complete esterification reaction Prepare for 1st synthetic reaction
Feb 22-24	Begin or continue 1st reaction	Feb 24-26	**MIDTERM EXAM**
Mar 1-3	Continue reaction	Mar 3-5	**Due: report on esterification** Continue reaction
Mar 8-10	**Due: Second synthetic prelab** First synthetic step completed	Mar 10-12	Start 2nd synthetic step
Mar 15-17	**Due: Report on first rxn** Continue 2nd synthesis	Mar 17-19	Continue 2nd synthesis
Mar 22-24	Finish 2nd synthesis	Mar 24-26	Start 3rd synthetic step
Mar 29- April 2	Spring Break	April 5-7	**Due: Report on 2nd rxn** Continue synthesis
April 7-9	Continue synthesis	April 12-13 April 14	Continue synthesis <u>Due: Notebook</u> Continue synthesis
April 14-15 April 16	**Due: Notebook** Continue synthesis <u>Due: 3rd rxn report</u> Clean-up; checkout	April 19-21 April 21	**Due: 3rd rxn report** Clean-up; checkout <u>Due: final synthesis report</u> Oral reports
April 21-22 April 23	**Due: final synthesis report** Oral reports Oral reports	April 26-27	Oral reports