LECTURER:  
Dr. Steve Kinsley  
Office: LS 101D  
Phone: 314-935-9401  
e-mail: kinsley@wustl.edu

LABORATORY TEACHING ASSISTANTS:  
TBA

OFFICE HOURS:  
M T W Th F 11-12  
TA Office Hours  
TBA

If you are not free during the hours listed above, you may arrange an appointment directly with me by e-mail or phone. I am also available for walk-ins anytime my door is open.

TEXTBOOKS:

Chem 261-262 Laboratory Manual and carbonless-copy laboratory notebook.

OPTIONAL TEXT:

"Techniques in Organic Chemistry" (3rd Edition), Jerry R. Mohrig, Christina Noring Hammond and Paul F. Schatz, W.H.Freeman & Co, N.Y. This reference book will be available in the laboratory.

Sections, E,F,G,H Friday 3-4 p.m. Louderman 458, First Lecture Aug. 29.  
Lecture Slides will be posted on Blackboard

LABORATORY SECTIONS: (Note: Lab schedule does not have lab every week)

<table>
<thead>
<tr>
<th>Section</th>
<th>Day</th>
<th>Time</th>
<th>Dates</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Wednesday</td>
<td>1:00-5:00 pm</td>
<td>9/3, 9/10, 9/24, 10/8, 10/22, 10/29, 11/12, 11/19</td>
</tr>
<tr>
<td>B</td>
<td>Thursday</td>
<td>8:00 a.m.-Noon</td>
<td>9/4, 9/11, 9/25, 10/9, 10/23, 10/30, 11/13, 11/20</td>
</tr>
<tr>
<td>C</td>
<td>Thursday</td>
<td>1:00-5:00 pm.</td>
<td>9/4, 9/11, 9/25, 10/9, 10/23, 10/30, 11/13, 11/20</td>
</tr>
<tr>
<td>D</td>
<td>Friday</td>
<td>1:00-5:00 pm.</td>
<td>9/5, 9/12, 9/26, 10/10, 10/24, 10/31, 11/14, 11/21</td>
</tr>
<tr>
<td>E</td>
<td>Saturday</td>
<td>9:00 am-1:00 pm</td>
<td>9/6, 9/13, 9/27, 10/11, 10/25, 11/1, 11/15, 11/22</td>
</tr>
<tr>
<td>F</td>
<td>Monday</td>
<td>1:00-5:00 pm</td>
<td>9/8, 9/15, 9/29, 10/13, 10/27, 11/3, 11/17, 11/24</td>
</tr>
<tr>
<td>G</td>
<td>Tuesday</td>
<td>8:00 am-Noon</td>
<td>9/9, 9/16, 9/30, 10/14, 10/28, 11/4, 11/18, 11/25</td>
</tr>
<tr>
<td>H</td>
<td>Tuesday</td>
<td>1:00-5:00 p.m.</td>
<td>9/9, 9/16, 9/30, 10/14, 10/28, 11/4, 11/18, 11/25</td>
</tr>
</tbody>
</table>
LABORATORY EXPERIMENTS:

Experiment 1. Modeling Organic Molecules
Discovering the 3-dimensional structure is essential in understanding an organic molecules’s physical properties, reactions, or biological activity. There are many methods available to help visualize organic molecules. In this experiment, you will use various methods to determine or represent the 3-dimensional structure of organic molecules including computer modeling with Sparten®.

Experiment 2. Purification of Solids: Recrystallization
This experiment demonstrates recrystallization, which is an important method for the purification of crystalline materials and uses melting point to determine the purity of the crystalline solid.

Experiment 3. Paper Chromatography of AminoAcids and Thin Layer Chromatography of Herbal Drugs
In this experiment, paper chromatography will be used to separate mixtures of amino acids; thin layer chromatography will be used to study the components of a common herbal drug.

Experiment 4. Natural Products Isolation: Caffeine
There are four parts to this experiment: (1) Converting the caffeine salt in tea to the neutral form (neutralization), (2) extraction of the neutral caffeine by partitioning between two immiscible liquids (two phase liquid-liquid extraction), (3) isolation of the caffeine from the organic extraction solvent by distillation, and (4) purification of the caffeine by sublimation. The pure caffeine will be analyzed by $^1$H-NMR.

Experiment 5. The Synthesis and Recrystallization of 4'-Isopropoxyacetanilide
In this experiment, 4'-hydroxyacetanilide (the active ingredient in Tylenol®) will be transformed to 4’isopropoxyacetanilide. Many of the techniques used earlier in the course, specifically chromatography, recrystallization and NMR, will be used to monitor the reaction, isolate and identify the product.

Experiment 6. Addition Reactions of Alkenes: Isomerization of Dimethyl Maleate
In this experiment, possible mechanisms for the addition first step of the addition to a double bond will be studied by examining the reaction of various compounds in the catalyzed isomerization of dimethyl maleate to dimethyl fumarate.

Experiment 7. Physical Organic Chemistry: Elimination Reactions
The regiochemistry of a double bond created from the elimination of a hydrogen halide from either 2-chloro-2-methylbutane or 2-bromo-2-methylbutane will be determined. The reaction will be carried out at reflux; the product(s) will be isolated by fractional distillation and analyzed by $^1$H-NMR to help determine the mechanism of the reaction.

Experiment 8. Pericyclic Reactions: The Diels-Alder Reaction
In this experiment, the stereochemistry of the Diels-Alder reaction of cyclopentadiene and maleic acid will be studied using $^1$H-NMR spectroscopy.

LABORATORY GRADING:

(1) Laboratory Report and Notebook (320 points out of 350 points): Experiments Two thru Eight will be worth a maximum of 50 points and will consist of notebook and the experiment report sheet. Experiment One is worth 20 pts and consists only of the report sheet. The reports are to be turned in on the forms provided at the end of the experiments. Any additional laboratory notebook pages should be attached. These are pages used for calculations as well as any other required supplementary material such as graphs, spectra, or relevant discussion of your results. A schedule of when laboratory reports are due is on Blackboard. Each report is to be turned in on the day of the student's laboratory period in the week the report is due. Late reports will be fined 5 points for up to 24 hours late and 10 points if more than 24 hours late. (If the student does not attend a lab period when a report is due, the report will be due during the first succeeding lab period.) Note: For determination of the Laboratory Report Score, the score for the lowest scoring report from experiment 2-8 will be dropped. If you miss any experiment between 2 thru 8, this lab will automatically be the dropped score. Experiment 1 cannot be dropped.

(a) Notebook: Each student's notebook will be graded by awarding points for items that should be included in a notebook. An example of a notebook is given in Appendix C of the laboratory manual. The point distribution will be as follows:

<table>
<thead>
<tr>
<th>Pre-lab</th>
<th>10 pts</th>
<th>Must be completed before lab and includes Purpose, Table of Chemicals, Structures and Reactions.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Procedure and Observations</td>
<td>10 pts</td>
<td>Must be written during lab</td>
</tr>
<tr>
<td>Experimental Performance</td>
<td>20 pts</td>
<td>Includes yield, purity, calculations, labeling, workup of data and laboratory conduct.</td>
</tr>
<tr>
<td>Conclusion</td>
<td>10 pts</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>50 pts*</td>
<td></td>
</tr>
</tbody>
</table>

* Experiment One includes only the Experimental Performance (no pre-lab, notebook or conclusion) and is worth 20 pts.

(b) Experimental Performance:

(i) Preparations: Each preparation will be judged on the basis of effectiveness of synthesis, general appearance, such as color and crystal form, and proper labeling of the sample. Samples will also be spot checked for accuracy in reporting melting point ranges and yields in the student's report.

(ii) Other experiments: Other experiments involve identifying unknowns, obtaining or observing kinetic or physical data. The results of these experiments will be judged on the basis of the accuracy of the results and/or the quality of data.

(iii) Laboratory Conduct: All students will be evaluated on the basis of (a) the student’s understanding of the experiments, (b) the student’s laboratory technique, (c) the neatness of
the student’s bench, apparatus set-up, etc., (d) efficiency in carrying out experiments, and (e) lab citizenship (helpful to others, listening and following TA suggestions, etc.) The TA will keep a record of each student’s laboratory proficiency, and will also note if the student obeys the rules concerning laboratory safety, personal protective equipment, waste disposal, etc.

(2) Lab Quizzes (30 points out of 350 points): At the start of lab, a 5 point quiz will be given. Note: For determination of the Laboratory Quiz Score, the score for the lowest scoring quiz will be dropped. If you miss a lab, this quiz will automatically be the dropped score.

LABORATORY GRADE ASSIGNMENT: Laboratory grades will be assigned as follows:

<table>
<thead>
<tr>
<th>Grade</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-</td>
<td>301 – 350</td>
</tr>
<tr>
<td>A/A+</td>
<td></td>
</tr>
<tr>
<td>B-/B/B+</td>
<td>251 – 300</td>
</tr>
<tr>
<td>C-/C/C+</td>
<td>201 – 250</td>
</tr>
<tr>
<td>D-/D/D+</td>
<td>151 – 200</td>
</tr>
<tr>
<td>NCR</td>
<td>&lt; 151</td>
</tr>
</tbody>
</table>

LABORATORY REGRADES REQUESTS: Occasionally, mistakes are made during grading of laboratory reports or quizzes. In such cases, all regrade requests for laboratory experiments should be first directed to your TA. In the event that there is some disagreement or that the TA does not have the authority to make the requested change, the regrade request will be forwarded to the Laboratory Director. A regrade request will include a complete regrade of the report.

FINAL LABORATORY GRADE CALCULATION: Final lab scores will be converted to a 100 pt scale and treated like one 100 pt exam to be combined with the exam scores to determine the final course grade (see section on Course Letter Grades Above).

SAFETY GOGGLES: Students are required to provide an approved pair of safety goggles in order to be admitted to the laboratory. Approved safety goggles can be purchased from the School Book Store). These goggles meet specifications of the U.S. Occupational Safety and Health Act (OSHA-ANSI287.1-1979) and are approved by the Chemistry Department Safety Committee. Other goggles may be acceptable, but must be approved by the course instructor.

DISPOSABLE GLOVES: A box of disposable gloves must be purchased from the School Book Store). Students can share boxes of gloves as long as the gloves are always available to each student during the appropriate lab period.

LAB COATS: Students are required to provide and wear a lab coat for the laboratory. A multi-use disposable Tyvek ® lab coat can be purchased from the School Book Store. A cloth lab coat is also acceptable.
LABORATORY EVACUATION PLAN: In the rare event requiring evacuation of lab, such as fire or major chemical spill, the following procedure will be followed.

1) The evacuation will be signaled by the fire alarm. This alarm consists of a purposefully obnoxious, repetitive honking sound and flashing lights.

2) Upon hearing the alarm, students should immediately exit the laboratory by the door shown on the diagram below. The student does not have to worry about shutting off electrical equipment; this task is performed by the TA.

3) The TA will designate a student (usually the one closest to the exit) to lead the students to the assembly site. The assembly site for the 2nd floor laboratories is across Thorpe Drive to the street level of the parking garage (see diagram below).

4) The TA will turn off the main power switch for the laboratory and follow their students out of lab to the assembly site.

5) At the assembly site, remain calm and quiet; the TA will take attendance.

6) Wait at the assembly site for further instructions or an “all clear” announcement.