Chemistry 106a. Laboratory in Organic Chemistry I …… Spring 2007

> ***"A good observer is one who sees what he is not looking for if it is there and who does not see what he is looking for if it is not there:"*** Frank Galbraith
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> "***In science the credit goes to the man who convinces the world, not the man to whom the idea first occurs."*** Sir Francis Darwin

Course Description: Laboratory work in organic chemistry. Experimental work may include measurement of physical properties, study of reactions, and synthesis. Techniques employed include chromatography, distillation, extraction, infrared spectroscopy, measurement of optical rotation, and recrystallization. This course must be taken concurrently with CHEM-106. Prerequisite: CHEM-105a. Three hours per week. *One semester hour.*

Class meetings: Monday or Tuesday or Wednesday or Thursday or Friday;
1:30 – 4:20 PM in the J.R. Lovett Organic Chemistry Laboratory (Pfahler 315).
Pre-lab discussion/lecture in Pfahler 210 at 1:30 PM.

Textbook: John W. Lehman, Multiscale Operational Organic Chemistry;
Prentice Hall: Upper Saddle River, NJ, 2002 [ISBN: 0-13-015495-4].

Notebook: Student Lab Notebook with Permanent Binding (100 pages),
Hayden-McNeil Publishing Company [ISBN: 1-930882-00-9].

Instructors:

Mon: Dr. Tom Ruttledge	Pfahler 313A	Ext. 2383	e-mail: truttledge
Tues: Dr. Tom Ruttledge	Pfahler 313A	Ext. 2383	e-mail: truttledge
Wed: Dr. Ronald Hess	Pfahler 313C	Ext. 2345	e-mail: rhess
Thu: Dr. Ronald Hess	Pfahler 313C	Ext. 2345	e-mail: rhess
Fri: Dr. Marian Knechel	Pfahler 205B	Ext. 23612	e-mail: mknechel

Safety: Suitable safety GOGGLES must be worn at all times in the laboratories by all students. Appropriate clothing must be worn to ensure your personal safety. Read carefully the material contained in your textbook on pages 9 – 18; these topics are entitled "Laboratory Safety" and "Reacting to Accidents."

Grading: Your grade in this course will be based on the following components, the quoted number of points being approximate and subject to change:

	Points
Laboratory reports ……	200
Laboratory notebook ..……..	100
Laboratory quizzes ……..…….	50
Laboratory practical exams (2) ……	200
Total	550

Examinations: Two laboratory practical exams as noted on the schedule. There are no written exams.

Missed Labs: We expect you to attend your regularly scheduled lab section each week. All labs are full and we cannot generally accommodate moving students from section to section. In ***extraordinary*** circumstances, you may ask for prior approval to move sections for a week. This request must be made by Thursday 12 noon of the week **preceding** the week you request to change sections. This request must be done by e-mail to Dr. Ruttledge (for ALL sections). You must clearly state your reasons for requesting a temporary change and alternative sections you can attend. You must also list a relevant contact person for this change (i.e. a coach if your team is traveling, a parent or legal guardian in the case of family emergency, a doctor's note in the case of illness or medical procedures). You must also include a summary of your weekly course schedule in this e-mail. Dr. Ruttledge will evaluate these requests on an individual basis. If insufficient information is provided (as requested above), it will be rejected. Any and all requests *may* be rejected, depending on circumstances and lab loads. Any approval granted will be printed by you to give to the lab instructor of the lab you will be attending. For obvious reasons, superfluous requests and requests with inaccuracies using deception will be rejected. Requests based upon deception/dishonesty in any way **will be considered cases of academic dishonesty and handled as outlined in this syllabus**.

Quizzes: Unannounced quizzes will be given to different sections during different weeks, at the discretion of the individual instructor. There will be at least one quiz for each section per semester and as many as 11 quizzes. This is at the discretion of your instructor. **The total points of all quizzes given to your section will be 50 points, whether you are given a single quiz or 11 quizzes. Thus, it is imperative that you arrive on time for every pre-lab!!**. To prepare for a quiz, simply have your notebook fully completed as per the instructions contained in this syllabus. The quizzes begin promptly at 1:30 PM and will typically last only a few minutes. ***You may use only your laboratory notebook***, which may not contain any photocopied or other electronically-reproduced sections. The notebook may contain only that which is hand-written in pen by you. You may not use the text or any other source. There will be no make-up quizzes given or extensions of time for lateness granted. **Arrive on time!!!!**

Lab Reports: You will write, individually or as part of a group as assigned by us, a total of three formal written lab reports (see description of expectations elsewhere in this packet). These reports will be due in your section (at 1:30 p.m., a loss of ten percentage points credit per hour or portion thereof will occur starting at 1:30:01!!) one week after the experiment is completed (see schedule of labs elsewhere in this packet for due dates). These will be graded based upon the criteria set out in the description for these reports, *as well as things like grammar, punctuation, spelling, and composition*. In addition, you may be asked to fill out occasional report sheets, which may or may not be graded. These will always be due one week after they are handed out to you, unless otherwise stated. Again, the same policy on late report sheets applies as stated for the formal reports above (i.e. loss of ten percentage points per hour). All of these reports and any occasional sheets will be worth 200 points total.

Notebooks: You are required to keep an up-to-date scientific notebook (see description of expectations elsewhere in this packet). These notebooks *may be collected for grading at anytime throughout the semester with no advance notice provided*, but will be collected at the end of the course for final grading. **Your notebook must be complete, meaning it must contain all experimental procedures, all observations (colors, smells, temperatures during distillation, etc.), all results (i.e. melting points, copies of spectra or gc traces affixed into the notebook, etc.), and your analysis of results (i.e. major assignments of absorptions in your IR spectra, percent yields of all products isolated, etc.).** It is items such as these that will be the basis of the notebook grade. The more complete and organized your notebook is, the higher the number of points you should receive.

Check-Out: You must, upon completion or withdrawal from the lab course, check out of your assigned drawer. *For students completing the course, this check out must occur during the time prescribed for your section (which will be during the final exam period assigned to your lab section by the registrar)*. We will give you more details on the precise time within your exam period that check out will occur for each section. *For those students withdrawing from the lab course, you must check out of your drawer within one week of your withdrawal from the course or within one week of the day you stop attending the lab (whichever is earlier), during your prescribed lab section*. Failure to check out of your drawer as stated above will mean your student account will be charged a $50.00 fee, in addition to fees for broken/missing glassware and equipment.

Classroom Etiquette:

Our expectations: Each student is expected to do every experiment, using the proper amounts of materials specified in the Lehman text, the syllabus, the *Blackboard*® site, and the course materials provided on the course CD, whichever pertains. Each student is also expected to submit the results that he or she actually obtained. It is a serious breach of laboratory ethics (i.e., cheating) to do otherwise, except when properly credited in your report. Your laboratory notebook should reflect your work, should be accurate, well-organized, and kept up-to-date at all times. Your notebook may be collected for grading by your lab assistant or your instructor at any time with no advance notice.

Lab Etiquette: A clean lab is a safer lab (not to mention a happy lab). This means you are expected to clean up your own messes. Do not spill solids all over the balance area and not clean them up. Do not dispense liquids and spill them on the benches without cleaning them up. Make sure when you use an instrument, return it to a proper and clean state for the next person. If you use the last of a reagent, inform your instructor or a TA.

ACADEMIC HONESTY. Your work must be written in your own words and comprise your own ideas. We hold you responsible for knowing ***ALL*** the rules on academic honesty, including plagiarism, spelled out in the Student Handbook, some of which follow:

"… ***You are cheating if you***:

1. Copy answers or use information from a fellow student's paper during a quiz, test, or examination.

2. Divulge answers or information, or otherwise give improper aid to another student during a quiz, test, examination or accept such aid.
3. Relay or receive any improperly obtained or confidential information concerning a quiz, test, or examination. (Example: if one sees the test before it is given and transmits information concerning its contents or whereabouts to other students.)
4. Use or refer to any unauthorized notes, books, calculators, problem solving aids such as "cheat sheets" during a quiz, test, or examination.
5. Collaborate improperly with another student on an open-book or take-home quiz, test, or examination; or obtain information from an unsuspecting fellow student during such an exercise.
7. Commit an act of plagiarism in any form.
8. Borrow under false pretenses, steal or otherwise improperly obtain lecture or research notes, laboratory data, or any information gathered by another student and present it as your own work (examples … laboratory reports or experimental yields …), or knowingly collaborate with another student by making such material available to him/her.
9. Falsify laboratory data, notes, results, or research data of any type in any course and present it as your own work.
10. Steal or intentionally damage or destroy notes, research data, laboratory projects, library projects, library materials, computer software (including the intentional passing of a computer virus), or any other work of another student (or faculty member), out of malice, or for the purpose of sabotaging that person's work and thereby gaining an unfair advantage to yourself.
11. Knowingly and willingly violate any special rules concerning research procedures, group assignments, or inter-student collaboration, which may be established by any instructor in any course.
13. Misrepresent yourself to an instructor or an administrator for the purpose of gaining special favors or extensions for academic work missed. Examples include but are not limited to lying about your health or the health of a relative, forging doctor's notes.
14. Forge signatures on forms, documents, or letters pertinent to College business. This may include but is not limited to course of study sheets, drop/add forms, or doctors' notes.
15. Request an accommodation of any kind (extra time, temporarily moving to another lab section) using deception/dishonesty.

You are an accessory to cheating, and penalties may be applied to you, if you:

1. Witness or have direct knowledge of any of the aforementioned forms of cheating and fail to inform an authorized person (faculty member, administrator, proctor, or student assistant).
2. You bring unauthorized materials into a testing area and fail or refuse to remove them when instructed to do so.
3. You fail or refuse to comply with admonitions from a faculty member or authorized proctor to cease any activity, which might aid other students in cheating."

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Department of Chemistry

Policy on attire in chemistry teaching laboratories and chemistry research laboratories

1. Approved safety **<u>goggles</u>** must be worn at all times.

2. No open-toed shoes, sandals, or clogs. Feet must be completely covered, including heels. Socks are recommended.

3. Arms should be covered up to the elbow. No tank tops or muscle shirts.

4. Torso area should be completely covered.

5. Long hair should be tied back.

6. Long pants that come down to the ankles must be worn. No shorts, skirts, or kilts. ***Approved Fall 2005***

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The Nature of Organic Chemistry Laboratory 2007

The undergraduate organic chemistry laboratory experience generally has two goals: to teach the basic manipulative skills (such as recrystallization, distillation, reaction set-ups) and to make the organic chemistry lecture material come alive. In CHEM-106a and CHEM-205a, both of these goals are addressed.

Laboratory work can be placed in three broad categories:

Structural Analysis. In most organic reactions, the identity of the product must be determined. If the compound is ***known***, comparisons with properties reported in the chemical literature allow us to verify its identity. If ***new***, the compound is characterized by its physical and chemical properties (especially spectral techniques), and often its structure is proved by independent synthesis or by degredative methods. In the latter case, a molecule is broken down into simpler molecules by known chemical reactions; the smaller pieces are then identified and the original molecule is reassembled employing sound chemical logic, sometimes in the lab but most often in the minds of those who carried out the reaction.

In all cases, the reaction product must be purified; otherwise, the observed properties are those of the combination of the given compound and the impurities present in the sample, not just the compound of interest. Impure compounds are simply mixtures, whose properties are often intermediate to those of the components. Purification techniques that you will encounter in your organic chemistry courses include distillation, crystallization (recrystallization), sublimation, extraction, and chromatography. Compounds will be characterized by morphology (shape and color), melting and boiling points, solubility, refractive index, adsorption behavior, and spectroscopic parameters.

Purification can often be a very time-consuming and frustrating process (be warned!), and this, of course, must be reflected in the cost of a chemical. To illustrate this point, check out the prices of calcium carbonate ($CaCO_3$, limestone) in the 2005 – 2006 Aldrich catalog. The price you pay for 100 grams of ACS reagent grade $CaCO_3$, ≥ 99.0% purity is $26.20. However, the price of 100 grams (4 x 25 grams) of $CaCO_3$, 99.999+% purity is $806.00 (of course, plus shipping charges).

Synthesis. As the name implies, a desired compound is prepared from material(s) of known structure, through a sequence of chemical reactions that will optimize yield. In the early 19^{th} century, all organics were natural products. However, the exponential increase in empirical and theoretical knowledge over the past two centuries has allowed us to synthesize myriad compounds not found in nature, some for the good of humankind and some not. We create some molecules simply for the intellectual challenge; we create some molecules to make us well or keep us well, others to make our lives better or more comfortable, still others for less than noble reasons.

Many natural products were synthesized after their structures had been proven. The late Dr. Robert Burns Woodward—Professor of Chemistry at Harvard, 1965 Nobel Laureate in Chemistry, and arguably the greatest synthetic chemist who ever lived—carried out, with numerous co-workers, the total synthesis of such complex molecules as quinine, reserpine, cholesterol, vitamin B12, and chlorophyll, both as proof of the deduced structure and as a challenge to his intellect, imagination, and creativity. Not only a synthetic chemist, Woodward used theoretical predictions to arrive at the correct answers to many questions, which had arisen from his synthetic work.

Reaction Dynamics involves the study of the rates of chemical reactions (kinetics) and the energy changes (thermodynamics) that accompany these transformations. Experiments determine the effect of temperature, concentration, solvent, catalysis, molecular structure, light, and other variables on reaction rates and equilibrium constants.

Our physical observations in these experiments often enable us to draw scientifically sound conclusions concerning what is happening at the molecular level, a level we cannot directly observe. In other words, we can learn about reaction mechanisms.

In the year 2007, chemical theory has advanced to a position where we can often make accurate predictions—both qualitative and quantitative—about the course of chemical reactions—the nature of the products and their properties, the rate of the reaction, the effect of catalysis, solvent effects, and the magnitude of the thermodynamic parameters (enthalpy, entropy, and free energy).