ORGANIC CHEMISTRY LABORATORY (CHEMISTRY 0123)

SYLLABUS — FALL 2005
Ambler, evening — TEMPLE UNIVERSITY

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COURSE DESCRIPTION:

This course provides an introduction to microscale laboratory techniques in organic chemistry. Offered in the Fall, Spring, and First Summer session it places an emphasis on each student's independent learning to manipulate equipment, to separate efficiently, purify and characterize organic compounds. It meets for one session of two hour and fifty minute each week during Fall and Spring semesters.

Pre- or Corequisite: Chemistry 121 - Organic Chemistry Lecture (Note: This course requires completion of General Chemistry 072 and 074. Knowledge of stoichiometry including determination of limiting reagents, solution preparation and the use of balances is assumed).

TEXTBOOK and SUPPLEMENTAL MATERIALS:

1). Mayo, D W, *et al*. "**Microscale Organic Laboratory** . . .", 4th edition, J. Wiley & Sons, New York, 2000.

2). Eye Protection that meets ANSI standards

3) "**General *Safety Guidelines for CST Labs***" including the release waiver******

4). "***Supplemental materials for Organic Chemistry 121 Lecture"*****

5). A hardbound composition book to serve as permanent lab notebook.

** These materials are available from the Copy Center in Room 601 Conwell Hall or the Copy Center in Bright Hall. Plan on purchasing them two days before class so that you may be prepared for lab.

SCHEDULING:

The **FIRST LABORATORY DAY IS TUESDAY, 30 August 2005.**

Your Organic Chemistry 123 lab is scheduled for only one meeting per week. Students are expected to arrive on time with a prelab write up that appears in their lab notebook. You should also be prepared for the quiz that begins every lab. By the **scheduled** end of class students will have cleaned their bench spaces and returned all equipment to the drawer used that period. The timely attention to these responsibilities will be rewarded.

There is no catch-up make-up session scheduled for the "wet or dry labs", and there is no provision for a "make up" of a missed quiz. As a result of an absence you will have lost the opportunity to accumulate points towards your final total and grade. *When you return from an absence, come prepared to undertake the laboratory work scheduled for the time noted...not for what you missed!*

The schedule below is divided into two portions. THREE CLASSES WILL BE DIFFERENT FROM ELEVEN. THREE classes will be “dry labs” consisting of workshops. The eleven other meetings of the term will be “wet labs” spent in the laboratory to which you are now assigned doing BENCH WORK - more traditional for this course. You should plan now on having your "Safety Guidelines for CST Labs" and EYE PROTECTION (SAFETY GLASSES OR GOGGLES) READY; YOU WILL NOT BE PERMITTED IN THE LABORATORY UNLESS YOU HAVE EYE PROTECTION!

Laboratory Schedule: [There is a new Lab each week.]

PART 2

Laboratory Schedule:

30 August Check in, explanation of lab procedures, Examine equipment. (examine sand heating baths (SEE, p. 19 and Fig 3.12) and pipettes (p. 26; 1a, b). [Work with automatic pipette optional; p.27 - 28; Fig.3.21.])

6 September Experiment 4A, p 123: Determination of a Partition Coefficient for the System Benzoic Acid, Methylene Chloride, and Water. **NOTICE**. After you complete the microscale experiment described in the text, **it will be repeated, as per the SUPPLEMENT on a 50 fold scale-up for purposes of comparison and in order to learn to use a separatory funnel.** ***Use your 10-mL graduated cylinder to collect CH_2Cl_2***

13 September GC of Gasoline. You must have read the material through p. 43, the GC DISCUSSION (pp. 44 - 49) and the material in the Supplement FOR THE EXPERIMENT TODAY!!

20 September Experiment 3B: Fractional Semi microscale Distillation. You will be separating hexane (bp 66°C) from toluene (bp 110°C). Procedure on p. 115. You will not measure refractive index. Use GC to check the purity. Each student of pair will work with her/his own Hickmann Still assembly. Each student will collect both first distillation components. Inject these fractions into GC, then reinject "re-distilled combined cuts" as directed by instructor to check purity.

27 September Experiment 11B: Isolation of caffeine from tea. Read the introduction on page 199, then the material beginning on p. 204. **SAVE THE SAMPLE OF CAFFEINE IN A LABELED VIAL*****

4 October Molecular Modeling Exercise #1, a "dry lab". Using molecular models you will be assigned a group of exercises building molecules and recording your observations in answer to posed questions

11 October Experiment 9, p 184: Dehydration of 2-Butanol: "the butenes". *GC needed.* ***WARNING: Concentrated sulfuric acid will be used.*** *Note: the gas delivery tube has been modified. Your instructor will provide you with the modification.****

18 October Experiments D2, p 478: Bromination of *trans*-Cinnamic Acid: *erthyro*-2,3-Dibromo-3-phenylpropanoic Acid, Use dichloromethane for recrystallization solvent. Procedures found on pages 481.

25 October Experiment 13, p 223: Oxidative Hydroboration of **1-Decene to 1-Decanol, substitute 1-decene for 1-octene.** You must have read the material pp. 223 ff before coming to class. Perform the Column chromatography and obtain an IR spectrum. What methods can determine if the reduction has succeeded. How will you know?

1 November Experiment 12: Reductive Hydrogenation of an Olefin: ***n-Decane***, p. 218 ff.**, substitute 1-decene for 1-octene.** What methods can determine if the reduction has succeeded. How will you know?

8 November Molecular Modeling Exercise #2, a "dry lab." **Stereochemistry.**

15 November "Spectroscopy Workshop" Dry lab #3 devoted to solving **spectroscopic** problems.

29 November **CHECK OUT. LAST LABORATORY QUIZ. Your notebook is due.**

GRADING:

You will be judged on your skills. Thus, the work in this laboratory is done on your own.

Course grading is as follows:

Laboratory quizzes/computer hand-in.....	30%
Laboratory Notebooks.....	30%
Last laboratory quiz.....	30%
Technique*	10%

Performance on the last quiz above minimum standards is required to pass the course.

>>EACH LABORATORY SESSION BEGINS WITH A QUIZ*<<

There is no make-up for missed quizzes or labs.

The requirements for your LABORATORY NOTEBOOK are noted below in the GENERAL INFORMATION SECTION. The notebook will be brought to every lab so that it can be kept current. It may be collected or reviewed at any time. A carbon copy of the pre-lab write up can be submitted in the event that the original is misplaced or lost. The format and list of ten essentials for each notebook write-up is found with the lab text, Mayo et al., 4th ed.

*Technique will include items such as being careful to avoid contamination of common reagents, remembering to keep your work area clean, taking care of the equipment including re-hanging automatic delivery pipets, finishing on time, *recapping reagent bottles,* returning equipment to the location found, keeping only originally inventoried drawer items, *, etc. The ability of a section to maintain the cleanliness around balances, sorting paper waste, sharps, and broken glass and then placing these items into the proper container will provide a measure for that section's average.*

Final totals are checked for individuals who are near grading boundaries and may have missed a quiz.

EXAMINATION POLICY:

Each week there is a lab quiz. The cumulative quiz total represents 30% of a student's final grade. There are no make up quizzes. If a quiz is given prior to your arrival, it is considered missed. Instructors are asked to wait five minutes before administering the quiz. In general, a single missed quiz should not alter a grade. An estimate of the potential score can be obtained by comparing the student's individual rank within the section to that section's performance on the specific test. Students are advised to keep all quizzes to aid in preparation for the last quiz.

This last laboratory quiz occurs on the last scheduled meeting of the student's section. It is usually a quiz of 10 or more questions that relate to the experiments performed during the semester. One or more questions may include stoichiometry, IR and NMR spectroscopy. A minimum grade on the final is expected for those who will receive grades higher than a B+.

The final collection of your notebook is no later than the day of the last quiz.

INCOMPLETES / WITHDRAWALS:

This course will adhere to the Department's and the University Policy regarding the last date to drop or withdraw from the course. The last date to drop is Monday, September 15.

Withdrawals can occur until a later time. For this semester this date will be Monday, 3 November. To obtain an "incomplete", the usual incomplete contract must be signed upon completion of 60% of the work. The student's accumulated total to that point should be more than 75% of the possible points. Non-attendance to the lab does not constitute "dropping" the course. Official withdraws can only be done through the Registrar's office. [Inattention to the proper procedure or failure complete the process and lab drawer "check-out" may result in the awarding of an "F" grade.]

SAFETY REQUIREMENTS:

Although the Department is sensitive to the need for demonstrating personal freedom, the laboratory can be a dangerous place for its expression. Therefore, in addition to denying you admission should you refuse to wear eye protection, the Department requires

1. long hair be tied back
2. closed footwear be worn (open-toed shoes/sandals are not acceptable)
3. scarves, veils, etc. be tied back or removed during the lab.

ATTENDANCE POLICY #1:

Simply stated, you must attend class to perform the experiments. You will be asked to leave the class if your pre-lab preparation is insufficient, if you do not dress appropriately or lack eye-protection, or arrive when there is insufficient time to perform the experiment. Missing more than one quiz and failing to write up a lab may effect your grade. One reason is that the lab you have missed will be included on the last lab quiz.

COURSE GOALS:

You will be learning experimental organic chemistry at the microscale level. This means you will be working with very small amounts of materials and may become able to observe and to learn more organic chemistry in a two semesters than many previous students learned in nearly two years. Hopefully you will find this laboratory an exciting, interesting and surprisingly pleasant adventure.

The course is structured to assist you develop skills in several areas considered in lecture and the lab.

1. Molecular modeling programs will be introduced and available to enable you to construct and manipulate structures considered in lecture or your lab texts.
2. General safety protocols for the laboratory will be enforced.
3. A formal, permanent, hardbound laboratory notebook will be maintained with a detailed pre-lab copy prepared in order for each student to perform successfully in the weekly quiz on the experiment to be performed.
4. Techniques and microscale organic lab skills will be developed to permit the flexibility of choosing you own scaling sequence without being tied to a prescribed set of quantities.
5. Methods of characterization of organic materials at the microscale will be utilized.
6. Successful completion will provide a foundation from which you can develop an expertise in microscale techniques as well as the confidence gained by mastering any challenging program.

This course is designed to allow the interested participant to rapidly develop the skills needed to slice more deeply into organic chemistry than ever before. Attendant benefits are greater confidence and independence in acquired laboratory techniques.

GENERAL INFORMATION FOR THE CHEMICAL LABORATORY

Recognizing its obligation to your safety and the environment, and noting the general reduction in the scale on which organic reactions are run in industrial research laboratories that has accompanied the revolution in analytical procedures, the Department of Chemistry has obtained funding from the University to convert your Organic Chemistry Laboratory from one that uses relatively large quantities of material and large (or macro scale) equipment to one that uses small quantities of material and small (or microscale) equipment. In the microscale laboratory reduction in the quantities of materials used is dramatic and, generally, the time required to carry out reactions is also reduced. **YOUR SECOND "WET" EXPERIMENT WILL COMPARE THE TWO DIFFERENT SCALES**. Your manipulative skills will be tried.

Our earlier experiences with this course have taught us that we must tell you that it is *critical* that you **read, outline, and understand the manipulations you are to perform before you come to class.** Processes on a small scale occur with rapidity. There is no time to study the book while reactions are taking place.
that has accompanied the revolution in analytical procedures

YOUR NOTEBOOK:

To help you understand the need to know what you are going to do before you begin...

a) You must write-up (in **INK AND IN YOUR HARDBOUND (NOT SPIRAL) NOTEBOOK** what you anticipate doing in the laboratory before class time. A typical write-up will be found in your text [p. 30 - 31]. Clearly, you will not be able to write-up the results, but you can, and should, indicate quantities of materials to be used, what the reaction or procedure is, and how the equipment set-up will appear. [First six "Key component of Lab Experiment write up items", p. 29.]

b) **The preliminary write-up will be examined by your instructor during the lab quiz before you start the experiment. If you do not have that material when you come to lab you will not be permitted to begin the experiment!** Laboratory notebooks will be collected and graded at least twice during the term. Your instructor will work out a schedule with you.

THE QUIZ:

(a) **At the beginning of every laboratory period**, while your teaching assistant is looking through the carbon copy of the prelab write-up, you will be taking a short (*ca.* 10 min) **QUIZ** dealing with the manipulations you have prepared to do. The quiz will be given about five (5) minutes after the laboratory period is scheduled to start. If you are late, you will miss the quiz. **There are no make-ups.** The sum of all quizzes (see below) will account for 30% of your grade.

ATTENDANCE POLICY #2:

(a) *When you return from an absence, come prepared to undertake the laboratory work scheduled for the time noted...not for what you missed!*
IF YOU ANTICIPATE BEING THIRTY (30) MINUTES OR MORE LATE TO THE LABORATORY, DO NOT BOTHER

COMING. YOU WILL NOT HAVE TIME TO DO THE EXPERIMENT!

(b) There are TWO (2) experiments for which product (in labeled vials) can be submitted. To receive full credit for both of them, you must turn in products, stating the yield in which they were obtained. The experiments are those entitled: Caffeine (Experiment 11B) and the Bromination experiment.

To Summarize:

This laboratory course, the companion course to CHEM 121: Organic Chemistry Lecture I, introduces the practice of organic chemistry in the laboratory. In this first semester course the primary emphasis is on learning basic laboratory techniques such as extraction, recrystallization, fractional distillation, reflux, gas chromatography. Experiments, performed at both the micro- and macroscale, will also include the preparation and reactions of alkanes and alkenes. Students will use software to learn molecular model building fundamentals. An attempt is made to correlate the syllabus topics with those being consider simultaneously in lecture.

Other Observations and Comments:

This syllabus was prepared 29 August 2005 and before the determination of Chem 121 lecturers. As a result all information is **tentative** and subject to change. Visit your "***Blackboard***™ site or ***/chem-help*** for more recent announcements.

LABORATORY QUESTIONS: At the end of each experiment there will be an assignment of 3 or 4 questions germane to the laboratory experiment. These questions are also relevant to the lecture discussion in Chemistry 121. Answers to the questions are to be provided in the notebook and will be checked at the beginning of the laboratory period during which the experiment was carried out. The specific questions for each experiment will be assigned at the beginning of laboratory or on an addendum to the syllabus.

Questions concerning this syllabus may be addressed to Dr. Findeisen (alfred.findeisen@temple.edu) at 215-204-7161 in Beury Hall 400 or Dr. Hill (hill@temple.edu) or Dr. MacMillan (jhm@ix.netcom.com).