

ORGANIC CHEMISTRY LABORATORY (CHEMISTRY 0123)

TENTATIVE SYLLABUS
Main Campus

Fall '06
TEMPLE UNIVERSITY

Dr. A L Findeisen
Organic Coordinator
Beury 406B; phone 1-7161

OFFICE HOURS: *during and after most labs
and by appointment
E-MAIL: afindeis@temple.edu

COURSE DESCRIPTION:

This course provides an introduction to microscale laboratory techniques in organic chemistry. Offered in the Spring, First Summer and Fall sessions, Organic Chemistry Lab I places an emphasis on each student's independent learning to manipulate equipment, to efficiently separate, purify and characterize organic compounds. Chemistry 123 meets for one session of two hour and fifty minute each week during the Fall and Spring semesters.

Pre- or Co-requisite: Chemistry 121 - Organic Chemistry Lecture (Note: This course requires completion of General Chemistry 072 and 074 with a C- or better. Students without these pre-requisites will be de-enrolled from the course. Knowledge of stoichiometry including determination of limiting reagents, solution preparation and the proper 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. Purchase these materials two days before class so you may be prepared for lab. This is to be submitted prior to 19 September.

SCHEDULING:

The **FIRST LABORATORY DAY IS MONDAY, 28 August 2006.**

Your Organic Chemistry 123 lab is scheduled for only one meeting per week. Each student is expected to arrive on time with a carbon copy of the "pre-lab" write up as it appears in her/his 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 equipment as necessary. The timely attention to these responsibilities will be rewarded.

Although there will a catch-up make-up session scheduled for the "wet lab", 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. THE FIRST THREE CLASSES WILL BE DIFFERENT THAN THE LAST ELEVEN. During the FIRST THREE classes, you will be expected to meet in the laboratory room for discussion prior to adjourning, with your instructor, to the EDUCATIONAL TECHNOLOGY CENTER, Ground Floor, Anderson Hall or to 640 Weiss Hall where you will learn to work with the molecular modeling program *Essential Spartan*. This software will be available for your use throughout the entire semester. **YOU WILL BE AT A DISTINCT DISADVANTAGE IN THE LECTURE PORTION OF THE COURSE WITHOUT THIS MATERIAL.**

The last eleven meetings of the term will be 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 GLASSES READY IN THE LABORATORY UNLESS YOU ARE EYE PROTECTION!**

Your daily schedule is also presented on the web. If you are using www.blackboard.temple.edu, you may access your account to find that are scheduled plus comments on many of the labs. For those not using Blackboard, you are reminded of the Department's help page - www.temple.edu/chem-help

PART 1

The curriculum for the first three sessions...

Text: Temple University, Organic Chemistry 121 Lecture (which incorporate, with permission, portions of the manual for "Essential Spartan", copyright Wavefunction, Inc.).

Laboratory Schedule: [There is a new Lab each week.]**Beginning Monday Experiment**

28 August Spartan Supplement #1. Lecture/Demo on an introduction to molecular modeling; Turning on the PC; Finding and Practicing with Spartan; getting the program "up-and-running". Leaving Spartan. Fundamentals of Molecule Building. Finding the menu bar, combining key strokes to manipulate your molecule. Building ethane (CH_3CH_3). Remember your 3.5" diskette. Download the Supplement 1 instructions from "photon.chem.temple.edu" using any terminal on campus.

The remaining weeks begin on Tuesday and conclude the following Monday

5 September "Essential Spartan" Supplement #2. Building BUTANE molecule. Measure energy of the conformations of butane. Construct a plot of energy *versus* dihedral angle. Read Chapter 4 in lecture text.

12 September "Essential Spartan" Supplement #3. Build cyclic molecules. Examine the conformers of cyclohexane. Introduce substituents onto cyclic hydrocarbons. Submit your plot of energy vs. dihedral angle. Turn in your calculations of cyclic molecules. Remember your 3.5" diskette. The last day to drop is Monday, 11 September.

Prepare GASOLINE SUPPLEMENT FOR NEXT WEEK!!

NEXT WEEK, AS YOU GO TO YOUR ASSIGNED LABORATORY IN BEURY HALL

>>> REMEMBER <<<

NO ONE IS PERMITTED IN THE LABORATORY WITHOUT SAFETY EYE WEAR.
THERE ARE NO EXCEPTIONS.

WITHOUT YOUR SAFETY GLASSES YOU WILL NOT BE PERMITTED TO STAY

PART 2

Laboratory Schedule: Experiment

19 September Check in and 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!! Examine equipment especially pipettes (p. 26; 1a, b). [Work with automatic pipette optional; p.27 - 28; Fig.3.21.]

26 September Experiment 4A and 4B: 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 detailed in 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 a portion of the CH_2Cl_2 .**

3 October Experiment 3B: Fractional Semi microscale Distillation. Modify 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 re-inject "re-distilled combined cuts" as directed by instructor to check purity. We will may separate something other than hexane and toluene. Check Blackboard to see if we will isolate *cyclohexane* from toluene.

10 October 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*****

17 October Experiment 10: Dehydrohalgenation of 2-Bromobutane: "the butenes" p. 192. GC needed. **WARNING: Concentrated alkaline solution will be used.** Note: the gas delivery tube will be modified. Your instructor will provide you with the new arrangement.***

24 October Experiment D2: Bromination of (E-Cinnamic Acid: *erythro-2,3-3 phenylpropanoic acid.*, Recrystallization procedures found on pages 478 and 75 respectively. Report yield and melting point.

31 October Experiment 13: Oxidative Hydroboration of **1-Decene: 1-Decanol** You must have read the material pp. 223 ff before coming to class. Perform the column chromatography and obtain an IR spectrum. Monday, 30 October is date of last withdrawal for undergraduate classes.

7 November Experiment 12: Reductive Hydrogenation of an Olefin: **n-Decane**, p. 218 ff. GC will be used to determine if the reduction has succeeded. How will you know?

14 November Catch-up/Make up;

21 November "Spectroscopy". Re-visit the ETC and begin using this Trinity Software self instruction package.

28 November **CHECK OUT. LAST LABORATORY QUIZ. Your notebook is due. Times of last quiz are fixed by the University Matrix and your schedule. These times are not to be changed by negotiation.** Performance on the last quiz above minimum standards is required to pass the course

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%

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

There is no make-up for missed quizzes.

The requirements for your LABORATORY NOTEBOOK are noted below in the GENERAL INFORMATION SECTION. This notebook is to accompany you to lab every meeting and is due no later than the day of the last quiz.

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, returning equipment to the location found, keeping only originally inventoried drawer items, *recapping reagent bottles, 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 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 will 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.

INCOMPLETES / WITHDRAWALS:

Withdrawal:

Please note that a withdrawal (W) is an institutional procedure which is not complete until the withdrawal form has been signed and submitted to the Registrar's office. This course is governed by the Temple University Policy (#03.12.12) on Withdrawal. Details may be found at http://policies.temple.edu/getdoc.asp?policy_no=02.10.14.

Incomplete:

Please note that an Incomplete (I) is only to be given in accord with institutional procedures and which is not fulfilled until the specific requirements have been met; the appropriate forms signed and submitted. This course is governed by the Temple University Policy (#03.12.13) on "Incompletes". Details may be found at http://policies.temple.edu/getdoc.asp?policy_no=02.10.13.

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:

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 of 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.

RECYCLING:

In an attempt to offer the best equipment at your lowest cost, you are instructed to:

a) Return items "to where you found them". Items "from the basket" are to be returned "to the basket in the lab period they are obtained". If sufficient quantities or the need existed for each student to have her or his own piece of equipment it will be provided. **To reduce costs we recycle equipment.**

b) Re-hang the automatic delivery pipets to the funnel racks provided.

c) Dry solid products on watch glasses. Store liquid yields in shell vials and not conical reaction vials.

Each drawer should need no more than two conical vials.

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 WHILE MAKING A CARBON COPY TO BE TURNED IN**) 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.]

YOUR NOTEBOOK

continued

b) You will present a carbon copy (preliminary write-up) to be examined by your instructor 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. Dr. Findeisen may ask to see them at any time.

THE QUIZ:

At the beginning of every laboratory period, while your teaching assistant is looking through the carbon copy of the pre-lab 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:

- (a) Your registration is section, room and time specific. It is expected that you attend the section for which your instructor has been assigned and only perform work to be graded under her/his supervision.
- (b) You will be given the opportunity to make up and complete a missed lab during the scheduled catch up make up week. Students who are found in the lab at other times **will be asked to leave and penalized.**
- (c) *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!

- (d) 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

DISABILITY RESOURCES and SERVICES:

Located in 100 Ritter Annex, this Office of Empowerment arranges accommodations and provides information and support in accessing University programs, facilities, and activities for students with 'certified' disabilities. Services include assisting with academic adjustments and accommodations including sign language interpreters, test proctoring, library research, note taking, and reader services. Information on mobility, wheel chair storage, adaptive computing, small equipment loan, specialized scholarship, and career/internship resources is also available. URL - www.temple.edu/disability ; 215.204.1280; TTY at 204.1786; FAX at 204.6794

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 levels, 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 considered simultaneously in lecture.

On behalf of the faculty of the Department, Drs. Dalton, Davis, Krow, Sieburth, Williams, Cross, Hill, and Przeslawski have agreed to participate in this laboratory experience with you and, on any given day, you may expect to find **at least one of them** in your laboratory sometime during the period.

This syllabus was prepared 1 August 2006 and before precise lecture syllabus determinations. As a result all information is **tentative** and subject to change. Visit your "**Blackboard**"™ site or www.temple.edu/chem-help for more recent announcements.

Attached to this document are a worksheet/hand-in to be used at the first day's lab and a commitment page to be submitted the first lab meeting as well as a locker check-in for the 4th week of classes. [One observation we make from the use of these attached materials is a determination of who can keep her/his syllabus for this period of time.]

Questions concerning this syllabus may be addressed to Dr. Findeisen (afindeis@astro.temple.edu) at 215-204-7161 in Beury Hall 406B.