

# ORGANIC CHEMISTRY LABORATORY (CHEMISTRY 0123)

TENTATIVE SYLLABUS  
Main Campus

FALL '05  
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](mailto:afindeis@temple.edu)

## 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: Both Chemistry 121 and 123 courses require completion of General Chemistry 072 and 074 with a C- or better. 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 meeting ANSI standards [and other PPE (gloves, lab coat, etc.) as you desire].
- 3) "General Safety Guidelines for CST Labs" including the release waiver\*\*
- 4). A hardbound composition book to serve as permanent lab notebook.

## SCHEDULING:

The **FIRST LABORATORY DAY IS MONDAY, 29 August 2005.**

Your Organic Chemistry 123 lab is scheduled for only one meeting per week. Students are expected to arrive on time with a carbon copy of the pre lab 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 obtained for use that period. 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 where you will learn to work with the molecular modeling program. That program 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 THAT 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](http://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](http://www.temple.edu/chem-help)

PART 1  
The curriculum for the first three sessions...

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

## Beginning Monday Experiment

**29 August** Lecture/Demo on an introduction to molecular modeling; Turning on the PC; Finding "Spartan"; Practicing with "Essential Spartan". Getting the program "up-and-running". Leaving "Essential Spartan". Fundamentals of molecule building. Will be detailed as we construct ethane ( $\text{CH}_3\text{CH}_3$ ) and seek to find its "preferred state".

### ROTATING TO TUESDAY

**6 September** "Essential Spartan" Supplement #2 is available at <http://photon.chem.temple.edu> from any terminal on campus. Building molecules: Butane. Measure energy of the conformations of butane. Construct a plot of energy versus dihedral angle. Download your copy of supplement for class. Read Chapter 4 in lecture text.

### LAST DAY TO DROP, MONDAY - SEPTEMBER 12<sup>th</sup>

**13 September** "Essential Spartan" Supplement #3. Build cyclic molecules. Examine cyclopropane, cyclobutane, cyclopentane and the conformers of cyclohexane. Introduce substituents onto cyclic hydrocarbons. Submit your plot of energy vs. dihedral angle. Submit your calculations of cyclic molecules.

### 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

**20 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. Calibrate pipettes (p. 26; 1a, b). [Work with automatic pipette optional; p.27 - 28; Fig.3.21.]

**27 September** Experiment 4A: 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 dichloromethane,  $\text{CH}_2\text{Cl}_2$ .**

**4 October** Experiment 3B: Fractional Semi microscale Distillation. You will be separating cyclohexane (bp  $80.7^\circ\text{C}$ ) from toluene (bp  $110^\circ\text{C}$ ). Adapt procedure on lab text 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.

**11 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\*\*\***

**18 October** Experiment 9: 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.\*\*\*

**25 October** Experiments D2: Bromination of *trans*-Cinnamic Acid: *erthyro*-2,3-Dibromo-3-phenylpropanoic Acid, Use dichloromethane for recrystallization solvent. Procedures found on pages 441 ff and 75 respectively.

### LAST DAY TO WITHDRAW, MONDAY, OCTOBER 31<sup>st</sup>.

**1 November** Experiment 13: Oxidative Hydroboration of *1-Octene*: *1-Octanol* You must have read the material pp. 223 ff before coming to class. Perform the Column chromatography and obtain an IR spectrum.

**8 November** Experiment 12: Reductive Hydrogenation of an Olefin: *n-Octane*, p. 218 ff. GC will be used to determine if the reduction has succeeded. How will you know?\*\*\***H & M variation presented at [www.temple.edu/chem-help](http://www.temple.edu/chem-help)** Sign up for catch-up / Make-up

**15 November** Catch-up / Make up [with your instructor during your regularly scheduled period].

**22 November** "Spectroscopy" Revisit the ETC and begin using the Trinity Software self instruction package during the "Temple Time days" around Thanksgiving.

**29 November** **CHECK OUT. LAST LABORATORY QUIZ. [Your notebook is due at your test time].**

**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.**

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 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., 4<sup>th</sup> 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 12. Details may be found at [http://policies.temple.edu/getdoc.asp?policy\\_no=02.10.14](http://policies.temple.edu/getdoc.asp?policy_no=02.10.14).

Withdrawals can occur until a later time. For this semester this date will be Monday, 31 October. 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 withdrawals 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.] Details may be found at [http://policies.temple.edu/getdoc.asp?policy\\_no=02.10.13](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.

**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 two years. Hopefully you will find this laboratory an exciting, interesting and surprisingly pleasant adventure.

### **COURSE GOALS (continued):**

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 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 your 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 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.]

b) **The carbon copy of the preliminary write-up will 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.

#### **THE QUIZ:**

(a) **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:**

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.

(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.

(c) On behalf of the faculty of the Department, Drs. Dalton, Davis, Krow, Sieburth, and Williams 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.

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 macro-scale, 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.

#### **ASSISTANCE:**

The Math and Sciences Resource Center will be open to support Organic Chemistry this semester. It's URL is [www.temple.edu/msrc](http://www.temple.edu/msrc) **It is free and offers individual as well as group tutoring.** Its hours are -

Monday	8:30 to 8:30
Tuesday	8:30 to 8:30
Wednesday	8:30 to 7:30
Thursday	8:30 to 7:30
Friday	8:30 to 2:30

#### **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](http://www.temple.edu/disability) ; 215.204.1280; TTY at 204.1786; FAX at 204.6794 Students intending to utilize these services should introduce themselves to Dr. F. early in the semester in order to utilize the fullest capabilities of these resources.

#### **Other Observations and Comments:**

This syllabus was prepared 9 August 2005 and before the final 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.

Attached to this document is a hand-in from the first day's lab, a commitment sheet, and a check-in sheet to be used during the 4<sup>th</sup> 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.

Course syllabi do not constitute a contract with students and do not supercede University policies.

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