

# ORGANIC CHEMISTRY LABORATORY (CHEMISTRY 0123)

SYLLABUS  
Ambler Campus

FALL 2006  
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 THURSDAY, 31 August 2006.**

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 lab", 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!**

## PART 2

### Laboratory Schedule: Experiment

**31 August** Check in, orientation, explanation of lab procedures. Examine equipment (see pp.16-18 and Fig 3.1-9) and pipettes(pg.28; 1a,b). (Work with automatic pipette optional; pg. 27-28, Fig. 3.21). Read material thru pg 43.

**7 September** Experiment 4A, pg 123; read pp 55-64: 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  $CH_2Cl_2$ .**

**14 September** Experiment 11B, pg 204; read pg 199: Isolation of caffeine from tea. **Read the introduction on page 199 and the section on sublimation on pp 96-98.** Save the weighed sample in a labeled vial.

**21 September** GC of gasoline; **read the GC discussion (pp 43-49) and the supplemental material for today's experiment (handouts).**

**28 September** Experiment 3B, pg 115; read pp 50-55: Fractional Semi microscale Distillation. You will be separating hexane (bp 66°C) from toluene (bp 110°C). You will not measure refractive index. Use GC to check the purity. Each student of pair will work with her/his own Hickmann Still (Fig 5.12,pg 55) 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. Copies of GC chromatograms will be made by pair and then taped into notebooks

**5 October** . Experiment 9, pg 190; read pp184-189: 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.\*\**

**12 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

**19 October** Experiments D2, pg 481; read pp 478-480: Bromination of *trans*-Cinnamic Acid: *ortho*-2,3-Dibromo-3-phenylpropanoic Acid, Use dichloromethane for recrystallization solvent. Crystallization procedures found on pages 71-75. Read pp 71-75.

**26 October** Experiment 12, pg 218; read pp 218-223: Reductive Hydrogenation of an Olefin: *n*-Decane. . GC or IR spectroscopy may 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)

**2 November** Experiment 13, pg 227; read pp 224-229: Oxidative Hydroboration of *1-Decene*: *1-Decanol* Perform the Column chromatography and obtain an IR spectrum.

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

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

**21 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.

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 submit 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 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 for this 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 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 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 FOUR (4) experiments for which product (in labeled vials) can be submitted. To receive full credit for them, you must turn in products, stating the yield in which they were obtained. The experiments are those entitled: Caffeine (Experiment 11B) , the bromination experiment, the hydrogenation experiment and the hydroboration 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 considered simultaneously in lecture.

### Other Observations and Comments:

This syllabus was prepared 25 August 2006 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.

**POST-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. Information helpful to you in answering these questions may be found in the experimental introductory material in your laboratory text or in McMurry. It is important that the correct answers be supplied. Failure to do so will result in a request to redo the exercise, i.e. answer the questions appropriately and resubmit the questions for checking. Answers to the questions are to be provided on a **SEPARATE** sheet and submitted at the END of EACH laboratory period during which the experiment was carried out. These will be checked and returned during the following week and **taped** into your notebook on the last page of the experiment. 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](mailto:alfred.findeisen@temple.edu)) at 215-204-7161 in Beury Hall 404 or Dr. Hill ([hill@temple.edu](mailto:hill@temple.edu)) or Dr. MacMillan ([JHM@ix.netcom.com](mailto:JHM@ix.netcom.com)).

**MAKE UP LABORATORIES:** Unlike the main campus Chemistry 123 laboratory course, there is no separate week set aside at the end of the semester in which you may make up any missed laboratories. Therefore it is essential that you attend ALL laboratory sessions in which you are enrolled. If, for some legitimate reason, you cannot attend a specific laboratory, speak to me and an alternate arrangement may be made.

**POLICY ON STUDENT AND FACULTY ACADEMIC RIGHTS AND RESPONSIBILITIES:** Freedom to teach and freedom to learn are inseparable facets of academic freedom. The University has adopted a policy on Student and Faculty Academic Rights and Responsibilities (Policy #03.70.02) which can be accessed through the following link:

[http://policies.temple.edu/getdoc.asp?policy no=02.78.13](http://policies.temple.edu/getdoc.asp?policy%20no=02.78.13)

## CHEMISTRY 123

### POST-LABORATORY QUESTIONS: FALL, 2006

Laboratory experiments are experiential exercises in learning. They are meant to broaden your chemistry experience and knowledge thru hands-on activities. To help you further, to accomplish this, several questions found in your laboratory text or handouts at the end of each experiment and relevant to the laboratory exercise just completed, are assigned for you to consider and to supply answers or solutions. These questions are also pertinent to the lecture material. The questions (in most instances limited to 3 or 4) are to be answered on a separate sheet of paper and turned in at the END of the laboratory experience. These will be checked and (ideally) returned to you at the next laboratory period. The sheets should then be taped into your laboratory notebook at the end of the experiment for which they are assigned. A grade may be attached to them so it is important that they be completed accurately. Indeed failure to answer the questions appropriately may result in a request for you to redo them. The questions are listed below. The page numbers refer to the laboratory text. To help you answer the questions you may refer to the introductory material for the experiment found in Mayo or use your McMurry text.

**NOTE:** The order of the assigned questions differs from the order of the experiments.

**Gasoline:** pg 49; 5-1 thru 5-5.

**Partition Coefficient:** Questions 1 thru 4 on the handout; pg 132: 6-23, 6-24.

**Fractional Distillation:** pg 122; 6-12, 6-16, 6-17.

**Caffeine Isolation:** pg 211; 6-71, 6-73.

**2-Butanol Dehydration:** pg 191; 6-55, 6-56, 6-57.

**Bromine Addition:** pg 445; 8-23, 8-25, 8-26.

**Hydroboration:** pg 229; 6-87, 6-90, 6-91.

**Hydrogenation:** pg 223; 6-82, 6-84, 6-85.

## **NOTEBOOK REQUIREMENTS FOR CHEM123 AND CHEM 124**

Items 1 thru 6 should be in your prelab, done before coming to class ! (I will check.)

1. **Title/Date**: self-explanatory.
2. **Objective**: a concise reason for doing the experiment.
3. **Physical Table Data**: molecular weights, gram moles, melting points, boiling points, densities etc.
4. **Reaction Schemes**: balanced equations for that day's synthesis when applicable. Data (masses, moles etc) germane to the experiment may be placed under each reactant in the scheme for quick reference.
5. **Procedure, Outline**: detailed description of that day's work. It must be written in the book, not stapled in or referenced.
6. **References**: use the lab text if necessary, otherwise use references cited in the experiment or handout or the handout itself.
7. **Experimental Data**: weights obtained, melting points, gas chromatograms, infrared spectra etc. Gas chromatograms and infrared spectra must be stapled into the book and clearly labeled with column conditions, band assignments etc.
8. **Calculations**: theoretical yields, limiting reagents, percent yields etc.
9. **Conclusions**: What went right or wrong with the experiment? What did you learn from the experiment? What principles were demonstrated?
10. **Post-laboratory Questions**: Separate sheets containing the answers to the post lab questions must be taped into the notebook.
11. Leave several pages at the beginning of the notebook for a table of contents.
12. For additional information regarding notebooks see Mayo, pp 29-31.
13. Lastly ALL notebooks are to be bound notebooks. Spiral notebooks are not acceptable nor are "professional" laboratory notebooks containing carbon or tear off sheets.
14. A sample notebook page is on the back.