

# ORGANIC CHEMISTRY LABORATORY II

## (CHEMISTRY 0124)

TENTATIVE SYLLABUS  
Main Campus

FALL '06  
TEMPLE UNIVERSITY

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OFFICE HOURS: \*during and after most labs  
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### COURSE DESCRIPTION:

This course is a continuation of Chemistry 123 in which you will utilize the microscale organic chemistry laboratory techniques introduced there. This course includes the preparation, purification, and analysis including multi-step sequences, of typical moderately complicated organic compounds. Offered year round, it places an emphasis on each student's independent skills in synthesizing and characterizing organic compounds. It meets for one session of two hours and fifty minute each week during Fall and Spring semesters. The "general guidelines" from the first semester course still apply and may be consulted at [www.temple.edu/chem-help](http://www.temple.edu/chem-help) or [www.blackboard.temple.edu](http://www.blackboard.temple.edu)

**Co requisite:** Chemistry 122 - Organic Chemistry Lecture II (Note: This course requires completion of both Organic Chemistry 121 and Chemistry 123.) The minimum grade requirement is C- or better in BOTH courses. Failure to meet these standards is grounds for dismissal from this course.

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

### SCHEDULING:

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

Your Organic Chemistry 124 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 equipment as necessary. Your 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!*

Your daily schedule is also presented on the web. You may access your account to find the work scheduled plus comments on many of the labs at [www.blackboard.temple.edu](http://www.blackboard.temple.edu) All are expected to use Blackboard and their TU URL for all communications. You are reminded of the Department's help page - [www.temple.edu/chem-help](http://www.temple.edu/chem-help)

As in Chemistry 123 this course will include the opportunity to utilize the Educational Technology Center, Ground Floor Anderson Hall. In this course we will use self pace learning software package, "Spectroscopy" in addition to the "Essential Spartan" program you learned last semester. **YOU WILL BE AT A DISTINCT DISADVANTAGE IN THE LECTURE PORTION OF THE COURSE WITHOUT THIS MATERIAL!**

Most 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!

**Laboratory Schedule:** [There is a new Lab each week. Your week begins on Tuesday morning and ends the following Monday.]

**Before you come to class**, you must read and sign the appropriate safety acknowledgment to be found in the "GENERAL GUIDELINES FOR CST LABORATORIES" - available in CONWELL 601. This RED SHEET will be collected during lab check-in and is required to continue in this course. You will not be permitted to work beyond September 12 without a current copy on file.

**Beginning Monday Experiment**

**28 August** Check-in and Discussion of regarding lab policies, notebooks, grading and safety. Review stoichiometry and first semester techniques (ADP use, recrystallization, etc.). Understand expectations of Dep't, AF, ... Begin Diels - Alder Reaction of **alpha-phellandrene and maleic anhydride** attached to this syllabus

**LAB WEEK NOW BEGINS TUESDAY AND ENDS MONDAY OF FOLLOWING WEEK**

**5 September** Check Diels-Alder Reaction. Review SPECTROSCOPY problems in the Trinity software package on ETC servers in the TECH CENTER. Use "Essential Spartan" to draw starting material and products in at least one lab. Minimize each structure. Monday, September 12 is the last day to drop courses.

**12 September** Modification of Experiment 28: Electrophilic Aromatic Substitution: Bromination of Aniline to yield 2, 4, 6-Tribromoaniline. See page attached or "photon.chem.temple.edu" What is the best way to characterize this product? Also collect and recrystallize your Diels-Alder product if it is complete. Determine product's melting point when dry. Complete review of stoichiometry and importance of knowing physical properties

**19 September** Experiment 16: Grignard Reaction with a ketone: Triphenylmethanol, p 246 NO FLAMES. Do not wash your equipment before use. Do not rinse with acetone. Dry in the oven if you ignore these instructions.

**26 September** Experiment 34B: Haloform Reaction: Substrate may be varied. Consult [www.blackboard.temple.edu](http://www.blackboard.temple.edu) Experiment 34B, p 365 to be modified

**3 October** Experiment 22: Modification of Williamson's ether synthesis: n-Propyl  $\beta$ -Naphthyl Ether. Substitute n-propyl bromide for n-propyl iodide. See attached instructions or "photon.chem.temple.edu" for other changes

**10 October** Experiment 5B: Reduction of Ketone using a Metal Hydride Reagent: cis- and trans-4-tert-Butylcyclohexanol. p 139. Run IR. What other method(s) could be used to characterize your product?

**17 October** Experiment 32: Hypochlorite Oxidation: Cyclohexanone. p 353. GC used.

**24 October** Preparation of Piperonylnitriles from Piperonal, to be distributed in lab; as adapted from J Chem Ed (6). Consult [www.blackboard.temple.edu](http://www.blackboard.temple.edu). Monday, October 30 is the last day to withdraw from courses.

**31 October** Experiments 24A and 24B: Imide Synthesis : N-Phenylmaleimide. p. 309

**7 November** Experiment 20: Aldol Condensation: A substituted benzaldehyde may be used. p 279. Recrystallize your product, then run the IR. Catch-up/Make-up sign-up. . Hand-out for laboratory practical.

**14 November** Catch-up/Make up

**21 November** Laboratory Practical Examination on Temple time [Tuesday = Thursday, Wednesday = Friday, M,T, W] NOTE: If you have Thursday lab, your practical will occur on the Tuesday BEFORE Thanksgiving. If you have Friday lab, your practical will occur on the Wednesday BEFORE Thanksgiving. Make appropriate plans NOW.

**28 November** **CHECK OUT. LAST LABORATORY QUIZ begins Thursday, November 30. Your notebook is due when your quiz is scheduled. Last quizzes end Wednesday, December 6.**

**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.	30%
Laboratory Notebooks...(due at Last Quiz)	30%
Laboratory Last Quiz (written)....	20%
Laboratory Final (practical)....	<u>20%</u>
	100%

## Grading:

continued:

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. 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 completed 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. Students must see Dr. F to take the quiz at another time without penalty. 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+.

## INCOMPLETES / WITHDRAWALS:

### Withdrawal:

Please note that a withdrawal (W) is an institutional procedure that is not completed 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](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](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 the lab you have missed will be included on the last lab quiz.

*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!

## 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 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 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.

There is **ONE** catch-up/make-up laboratory scheduled. If you are absent from your laboratory class, be prepared to perform the current lab on the syllabus for the day you return not the missed work. You will have missed a lab, its quiz, and the opportunity to score the associated points.

## YOUR NOTEBOOK:

The notebook will be brought to every lab so 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 your original is misplaced or lost. The format and list of ten essentials for each notebook write-up are found within the lab text, Mayo et al., 4<sup>th</sup> ed. Only those notebooks completed and submitted in a timely manner will receive maximum points.

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 QUIZZES:

**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 perform. 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 will account for 30% of your grade.

## GRADING FOR THE PRACTICUM:

Determination of grades for the laboratory. practicum:	
Answers to assigned questions	25%
Calculations	10%
Sketch of the Apparatus	10%
Product	25%
Lab technique/safety/questions by instructor	30%
TOTAL	100%

## ASSISTANCE:

The Math and Sciences Resource Center will be open to support Organic Chemistry this semester. Its URL is [www.temple.edu/msrc](http://www.temple.edu/msrc) It is free and offers individual as well as group tutoring. Its hour for Fall 05 were:

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:

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 26 July and before the determination of Chemistry 124 instructors. As a result all information is **tentative** and subject to change. Visit your "**Blackboard**"™ site or **/chem-help** for more recent announcements.

Questions concerning this syllabus may be addressed to Dr. Findeisen ([afindeis@temple.edu](mailto:afindeis@temple.edu)) at 215-204-7161 in Beury Hall 406B. During the first three weeks of each semester, Dr. F. conducts (Molecular Modeling) instruction when Chemistry 123 sections meet. He can often be located in fourth floor teaching labs within Beury Hall if you can not find him in his office. If you wish to find him for a "Green Card" for a closed section, it is best to arrive at the section you are seeking, so "noses" can be counted and compared to the class list. Your patience and cooperation are appreciated.

Our experiences have found well prepared students to be more successful in this course than in the co requisite lecture. For this reason a commitment agreement has been attached to this syllabus whereby each student earns credit for acknowledging the expectations in lab and lecture courses in a timely manner. Return your completed form to Dr. F. this week. Typically a quarter to one third of the students beginning Organic Chemistry each semester do not receive the grade of C- or higher. It is our desire to increase the success rate by alerting all students to our expectations.

To Summarize:

This laboratory course, the companion course to CHEM 122: Organic Chemistry Lecture II, introduces the practice of organic chemistry in the laboratory. In this second semester course the primary emphasis is on applying basic laboratory techniques such as extraction, recrystallization, steam distillation, reflux, gas to synthetic chromatography as well as FT-IR spectroscopy. 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.