

**WINTER 99  
130BL**

**Organic Chemistry Laboratory**  
*Tentative Course Syllabus*

**Instructor:** Dr. John K.M. Mouser

**Office:** Young Hall 3077B

**Phone:** 825-1853

**Office Hours:** M 5-5:30; Tu/Th 12-1 PM

**Safety:** Safety glasses are to be worn at all times in the laboratory. A lab coat must be worn while in lab. Contact lenses should *not* be worn in the laboratory. Low-heeled shoes which cover the toe and instep must be worn at all times while in the laboratory. Clogs, sandals, cloth-top sneakers and narrow-base heels are not acceptable. Tennis shoes will suffice. Shorts are not allowed. Pants that cover the ankles are required. Hair that is shoulder length or longer should be put up or tied back. Food or chewing gum is not allowed in the laboratory. Any student under the care of a physician for either acute or chronic medical reasons (e.g.; diabetes, essential hypertension, epilepsy, pulmonary disease) and/or is pregnant should advise his/her physician that s/he is taking organic chemistry lab. Also, the student should advise the Chemistry Lab Instructor immediately.

**Glass and syringe needles are never** to be disposed of in the regular trash receptacles (this poses a hazard to the janitor). All glass waste and syringe needles go into the glass waste containers (blue box labeled glass waste). Failure to observe this rule will result in dismissal from the course.

Any student whose deliberate or negligent acts or omissions endanger his/her own safety or that of others may be expelled from the laboratory. Lab safety will be discussed at the first lab lecture.

**Wait Listed:** *If you are wait-listed*, wait inside the lab section you are wait-listed for. If there is space you will be added. The department will add you if you are written in on the class roster. **Regardless, it is your responsibility to make certain that you have been added to URSA.** *If your name doesn't appear on URSA within 48 hours of your enrollment, contact Denise Mantonya (Young 1039) before the last day to add.*

**Text:** 130AL Course reader **or** Donald L. Pavia, Gary M. Lampman, George S. Kriz and Randall G. Engel, *INTRODUCTION TO ORGANIC CHEMISTRY, A Microscale Approach*, Chicago, Saunders, 1995.

**Lab Notebook, Glasses & Coat:** Bound with **duplicate (a must!)** removable pages, safety glasses and lab coat are available from Young 1275, the ASUCLA bookstore or the medical bookstore.

**Handouts and Lecture notes:** Lecture handouts will be available at the beginning of lecture. If you are unable to attend lecture then you need to make arrangements with a classmate or friend for those days notes and handouts.

**Academic Standards:** Attendance to the lab course lecture is mandatory. You will be held responsible for all material covered during lecture. On occasion, adjustments to the course syllabus during lecture may be made (e.g., change of exam date, lecture material to be covered, etc.). An unexcused failure to attend lecture or to perform experiments on schedule or to submit timely experimental write-ups may have an **out of proportion** adverse effect on your course grade. In extreme cases, the student may receive an "F" for the course regardless of the point total. Failure to turn in a laboratory notebook at the end of the term will result in a failing grade.

Failure to perform an experiment will result in an "F" for the course. In the case of an *excused* absence, the student will be allowed to make up the experiment without penalty (if you are unable to make up the experiment you will receive an Incomplete). Falsely representing that an experiment has been performed will be regarded as equivalent to cheating on an exam.

**Examination Policy:** *There will be no make-up exams or quizzes. No exams will be administered earlier than the scheduled exam time.* If the final exam is missed, the student should bring it to the *immediate* attention of the instructor. If the student has a *good* reason then they will receive an incomplete for the course, otherwise, they will receive an *automatic failure grade* (F) for the entire quarter. An established incident of cheating will also result in an *automatic failure grade* (F) for the entire quarter. Students who are aware of cheating are encouraged to report such incidents. Securing a "recommendation" after a cheating incident at the University is, needless to say, awkward at best! Do not foolishly mortgage your professional future by cheating now. A course grade of incomplete (I) will be considered only for individual cases with exceptional extenuating cause. **An incomplete will not be given in lieu of a failing grade.**

**Lab Write-ups and Grading:** Read pages 22-29 in your text for description of laboratory notebook write-ups. During week 1 in lab, your TA will *briefly* discuss lab report format and answer any questions you have concerning this matter (i.e., pre-lab and post-lab write-ups).

*Each experiment must be written up, in a bound notebook in ink, before coming to lab*, including the summary and/or flow chart of the work to be performed. *Typed reports or word processing is not allowed (NO EXCEPTIONS) -- use a lab notebook!* The first four pages of your lab notebook should be reserved for a table of contents. TA/Instructor evaluation points will be assigned at the end of the course (5 point per lab meeting). Following is the point break-down for each lab report.

- **In-Lab Quiz** – ~10 minutes (closed book and notes; 3-5 questions 20 points/week; quiz will cover previous and current week's experiments) **There will be no quiz on week 1, 9 and 10.**
- **TA evaluation** (product evaluation, in-lab evaluation, preparedness) 10 points/week
  - Pre-write-up:** Purpose, procedure, answers to assigned questions (spot check)
  - Post-write-up:** Observations, results, conclusions, final product (spot check)
- **Lab Notebook\*\*** (end of quarter evaluation of lab notebook) 20 points
  - \*\*Note: must be bound with consecutively numbered pages and in ink; if a bound lab notebook is not used (e.g., spiral notebook; word processing generated report; ring note book) **then no points will be given**; if a table of contents is missing then there will be a 20 point deduction; missing homework assignment(s) will result in deduction of 5 points per lab; notebooks that are well-organized and complete will receive full credit. Failure to hand in a lab notebook, at best will result in an incomplete and in unexcused cases will result in a non passing grade.
- **Final Exam (during lecture of week 9)** 35% of grade
- **Lecture attendance:** (mandatory) -5 points/absence
- **Housekeeping, labeling, safety, etc.** *see below*

**Lab Preparation:** You must be prepared for the experiment before you come to lab if you wish to complete the experiment in the time allotted. There is no make-up time in the course and no space for you to work in other sections. Also study the pertinent sections in the text and then prepare a summary or flow chart of the procedures that will be performed in the laboratory. *Your teaching assistant will check your pre-lab preparation before you are allowed to begin any experiment. An open laboratory text will result in an automatic 10 point deduction. For purposes of safety, lack of preparation will result in dismissal from that lab period.*

**Housekeeping:** The laboratory can function smoothly only if left clean at the end of each lab. On leaving the lab, after an experiment, each student at his/her station and hood used should ensure the following:

1. Hot plate is unplugged and cord is neatly coiled around plate. Vacuum trap is emptied and clamped. if not then -2 points
2. Work bench area needs to be sponged down at the end of lab. if not then -2 points
3. Spills are wiped up and balances are swept clean. if not then -2 points
4. The sink is free of glass, paper towels, corks and other insoluble solid refuse. Such material belongs in the *appropriate* waste can. if not then -2 points

*Poor house keeping will result in a grading penalty of 5 points for each infraction.* Dumping of hazardous material in trash receptacles or in sinks is ground for dismissal from the course.

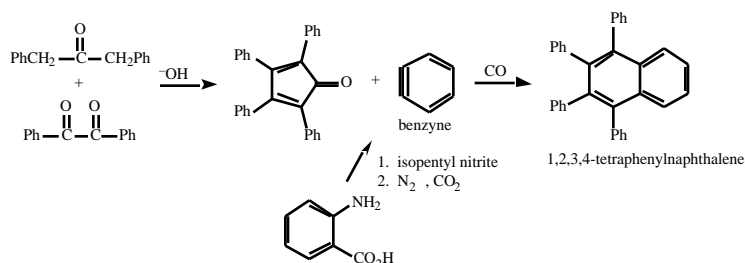
**Waste Disposal:** All chemical waste goes into the appropriate waste containers supplied in the hood. If you are not sure of how to dispose of a chemical, ask your TA or stockroom personnel for help. All products are to be stored in a labeled container. All vials (storage containers) containing product must be labeled correctly (see bottom of p. 29, lab text). Failure to correctly label a vial will result in a 5 point deduction.

**Breakage & Check-out:** The security of your locker is your responsibility. You will be charged for broken or missing items; your desk must be fully stocked on check-out. If you fail to check-out of a desk you have drawn, you will be assessed a \$25.00 abandoned desk penalty in addition to charges for missing or broken equipment.

# 130BL Course Overview

## Lab 1-2

### Diels-Alder/Benzyne Reaction Rxn: Synthesis of octaphenylnaphthalene

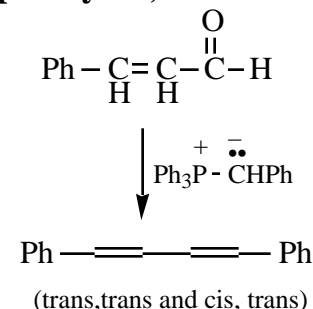


**x4 scale!**

**Read experiment 29 and 46** Do questions: 1, 2, 5&6 (pp.433-4); Scale reaction up x4. Obtain IR spectrum & mp of product; **ATTENTION:** p.433, problem 2a. should be furan + benzyne (and not benzene as shown).

## Lab 3

### Wittig Reaction: Synthesis of 1,4-diphenyl-1,3-butadiene

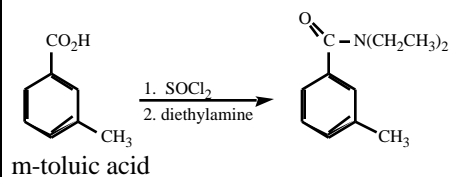


**x4 scale**

**Experiment 33 (p. 319)**  
Do questions: 1,2,4 and 5 (p. 325);  
Obtain UV Spectrum&mp of product

## Lab 4

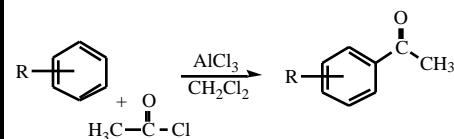
### Preparation of an Amide: Synthesis of N,N- diethyl-*m*-toluamide



**Exp. 42** (pp.387-92); Do questions: 1-6 (p.392); read technique 12 (pp. 723-749); Obtain IR spec. of product  
**DOUBLE SCALE**

## Lab 5

### Friedel-Crafts Acylation



•Vacuum Distillation•

**Exp. 36** (334-341); Do questions 1, 4-6, and 8. Obtain IR; NMR spectrum will be supplied in lecture  
**DOUBLE SCALE**

## Lab 6

### GC/MS of FC Products

### Identification of an Unknown Compound

Unknown will be issued during lab 5

Unknowns will be issued during this week.

## Lab 7-9

### Grignard Reaction

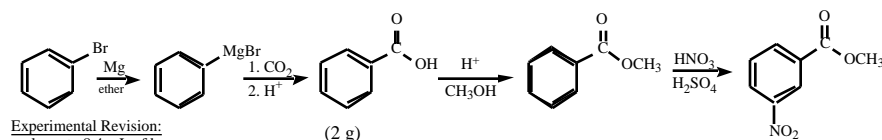
Experiment 25  
(Lab 7)

### Esterification

procedure given in lecture  
(Lab 8)

### Nitration

Experiment 35  
(Lab 9)



**Experimental Revision:**  
scale up to 8.4 mL of bromobenzene and 2.0 g of Mg.

## Cleaning Schedule

(assignment TBA by your TA):

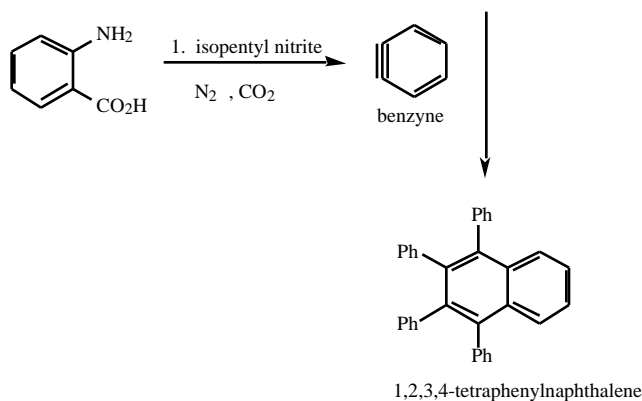
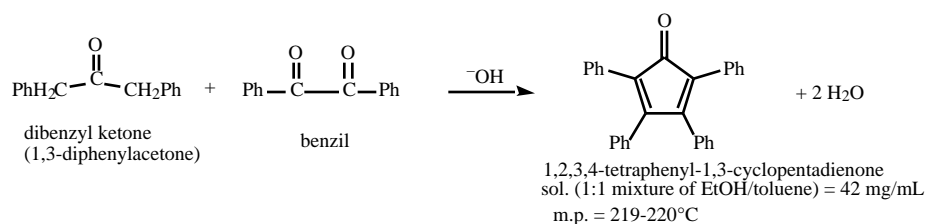
During your assigned week, you will be responsible for making sure that the lab is *left* clean. A clean-up checklist can be obtained from your TA. If any portion of the lab is left unclean, then a 4-5 page report paper (single space, font size 12, font type times, 1 inch margin on all sides) will be assigned to all the individuals responsible for that week.

**Lab 7. Exp. 25** (p.266): Do questions: 1,2,4,5 (p.277); **scale up rxn:** use 8.4 mL of bromobenzene; obtain mp of product; **optional:** IR (use methylene chloride)

**Lab 9. Obtain IR of Methyl benzoate; Lab 9. Exp. 35** (p.330); Do questions: 1-5 (p.333); Obtain mp & IR spectrum of product

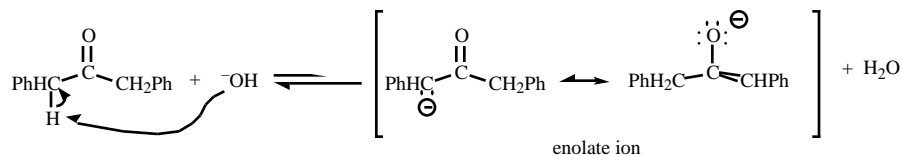
**Lab Check-out during last hour of Lab 10**

## Aldol condensation:

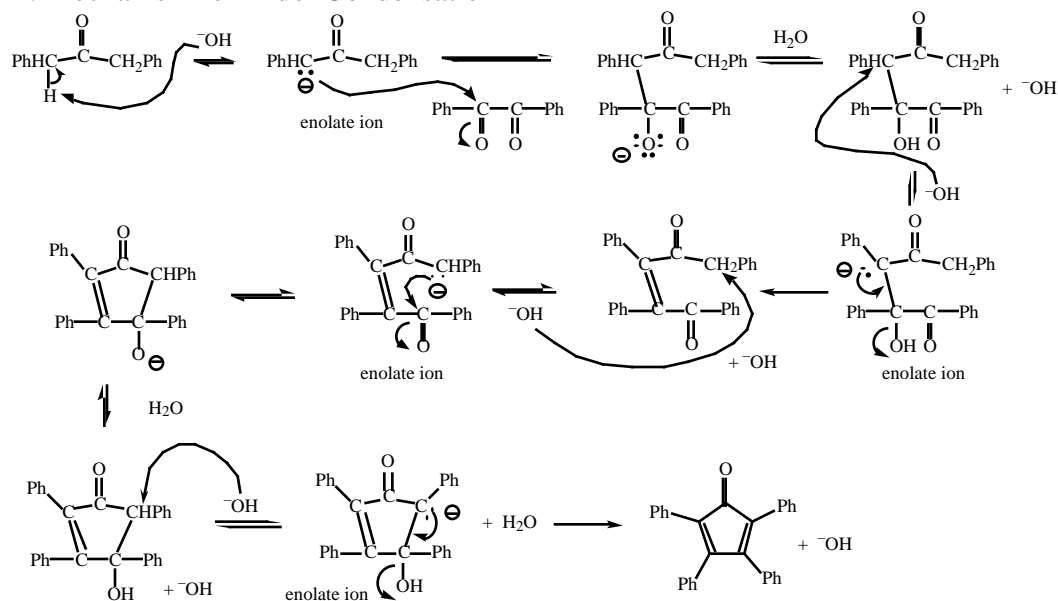


### Mechanism:

#### I. Generation of the Enolate Ion:



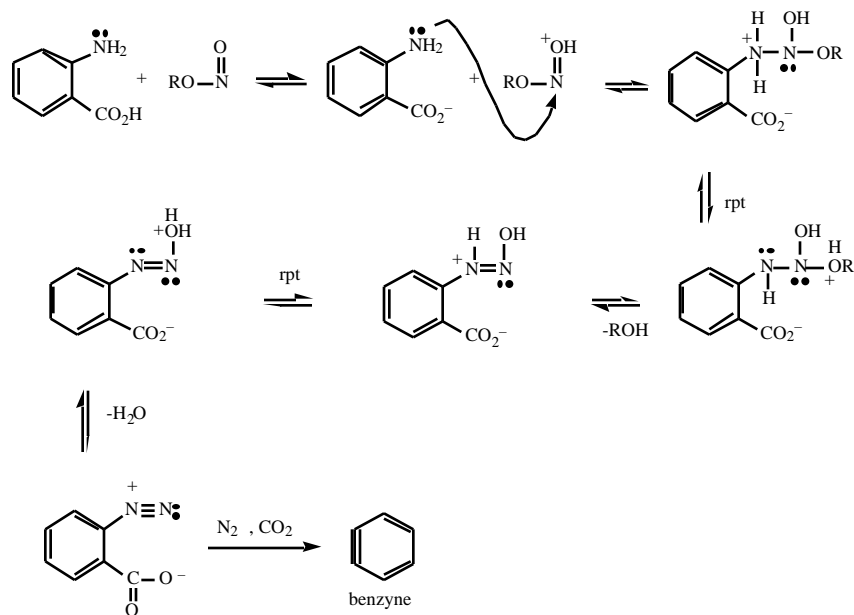
#### II. Mechanism for Aldol Condensation



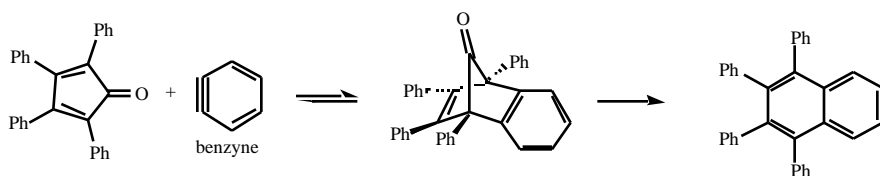
#### Drives Reaction to Right

- last step is intramolecular
- highly conjugated system
- Reflux! (entropy driven)  $G = H - T S$

### III. Generation of Benzyne:



### IV. Final Step



### Notes:

- ✳ reaction scale x4; use a 10 mL round bottom for the aldol reaction
- ✳ Record crude weight of the isolated tetraphenylnaphthalene
- ✳ In a labeled vial, turn in the recrystallized final product. Include amount isolated, crude yield, and overall yield.

Joe Ether, 1/20/98  
 TA: Nancy Doe  
 Crude 1,2,3,4-tetraphenylnaphthalene  
 Wgt: 350 mg; Crude yield = ???%  
 Recry yield = ???%  
 (draw structure)