# Organic Chemistry Laboratory CHEM 2462 Majors Lab Section

Spring, 2004

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**Teaching Assistant:** The name of the teaching assistant, his/her office, phone number, and e-mail address is on Blackboard.

**Text**: "Introduction to Organic Laboratory Techniques: A Microscale Approach", Third Edition, by Pavia, Lampman, Kriz, and Engel.

**General**: Lab is scheduled from 2:00 to 5:30 on Monday. Please come on time. Attendance will be taken. Each lab will begin with a brief introduction to the experiment to be done that day.

The schedule of experiments for the quarter is attached. Please read the assigned sections in your lab book before coming to lab. Also read the "Required Reading" sections that are indicated for each experiment. This will enable you to follow the introduction more easily. If you read ahead and plan your time well, you will find it easy to complete the experiments in the allotted time or less. Planning ahead will also enable you to use your time most effectively. For example, an experiment might require that a reaction mixture be heated for one hour. The best use of your time would be to get the reaction started immediately. Then, while the reaction is being heated, you could distill the product from the preceding experiment, take melting points, wash glassware, etc. In addition, you will find it advantageous to clean your glassware before you leave for the day. When you come for the next lab, your glassware will be clean, dry, and ready to use.

# Safety: READ PP. 4-20 IN YOUR LAB TEXT.

The organic laboratory is a potentially dangerous place. However, no accidents need occur if you are careful and are constantly aware of what you are doing and why. Read the directions given in each experiment carefully and in advance. Pay particular attention to the "Special Instructions" that are described for each experiment. Make sure you understand each step of the experimental procedure and any potential dangers. If you have questions, ask! Use your common sense and, above all, THINK!

# The following safety rules will be in effect at all times:

1. Safety glasses must be worn at all times (no contact lenses).

- 2. Wear proper attire; gloves and lab coats when necessary; shoes (no sandals); no shorts.
- 3. No flames or smoking allowed in the lab.
- 4. No food or drink allowed in the lab.
- 5. No horseplay allowed in the lab.
- 6. Keep all work areas clean and orderly. This includes your bench, the balance table and the hoods. Clean up all chemical spills immediately.
- 7. Use proper disposal procedures, as specified by your TA, for all chemicals and solutions.
- 8. Be careful to avoid contaminating the reagents. Close all containers snugly after use.
- 9. Be sure to read the labels on chemical containers carefully. Many chemical names are very similar.
- 10. Most organic chemicals are toxic to some extent. Treat them all with respect. Avoid getting them on your skin or clothing and avoid extensive breathing of their fumes. Work in the hoods when the experimental directions so instruct.
- 11. No students are allowed in the lab unless the TA is present. Only students assigned to that lab section are allowed in the lab.
- 12. No chemicals, glassware or equipment are to be removed from the lab. No unauthorized experiments are to be done.

#### Notebooks: READ PP. 20-26 IN YOUR LAB TEXT.

You may use the same notebook as last quarter, but it must be **bound.** Your notebook will not be graded on neatness, only on completeness.

**Prelabs:** Prelab write-ups are required as last quarter.

**Reports:** The style for lab reports is the same as last quarter. Reports will be penalized 30% for lateness. Reports more than two periods late will not be accepted.

Grading: Your grade will be based on a total of 900 points, distributed as follows:

Pre-lab write-ups, 10 pts./exp. 60 points Lab reports and products (if applicable) 100 pts./exp. 700 points Lab notebook, other than pre-lab write-ups 40 points

# Schedule of Laboratory Experiments Spring Quarter, 2004 Majors Lab Section

#### Mar. 22

# **Experiment 1** An Oxidation-Reduction Scheme

Read pp. 266-277 in your lab text. Do Experiment 28. This is a two-week experiment. Do Part A during the first week and Part B during the second week. Determine the percentages of borneol and isoborneol by gas chromatography. The report is due on Apr. 5.

#### Apr. 5

# **Experiment 2** Identification of Antihistamine Drugs by GC-MS

Read pp. 500-501 in your lab text. Do Experiment 59. The report is due on Apr. 12.

## Apr. 12

### **Experiment 3** Catalytic Hydrogenation

Read pp. 244-248 in your lab text. Do Experiment 25. The report is due on Apr.

Apr. 19

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# **Experiment 4** Unknown Identification by Spectroscopy

There is no pre-lab for this experiment. You will be given two unknown samples. You will identify one of them using its IR spectrum. You will use both the IR and <sup>1</sup>H-NMR spectra of the other compound to identify it. The report is due on May 3.

**IR Unknown**: You will be given an unknown compound from the list below. Obtain the IR spectrum of your unknown and identify it. The report should contain an interpretation of the IR spectrum and the reasons behind your identification of the unknown.

t-butanol benzene benzyl alcohol o-nitrotoluene ethyl acetate cyclohexane ethyl benzoate nitrobenzene acetophenone aniline 3-pentanone p-nitroaniline benzophenone cyclohexylamine acetic acid benzonitrile benzoic acid acetonitrile toluene benzaldehyde

**IR/NMR Unknown**: You will be given an unknown from the list below. Obtain an IR and a NMR spectrum of your unknown. The report should contain a complete interpretation of both the IR and NMR spectra.

#### **Aldehydes**

2-methylpropanal 4-nitrobenzaldehyde (E)-3-phenyl-2-propenal (cinnamaldehyde)

#### **Acids**

diphenylacetic acid
1,4-butanedioic acid
(succinic acid)
propanoic acid
(E)-2-butenoic acid
(crotonic acid)

#### **Amines**

4-methylaniline dibutylamine triethylamine

#### **Esters**

ethyl formate
ethyl acetate
methyl butyrate
3-methylbutyl acetate
methyl benzoate
diethyl phthalate
methyl m-nitrobenzoate
2-propenyl acetate

#### Ketones

3-methyl-2-butanone
3-pentanone
3,3-dimethyl-2-butanone
(pinacolone)
2,5-hexanedione
propiophenone
(ethyl phenyl ketone)
acetophenone
(methyl phenyl ketone)
4-heptanone

#### **Alcohols**

2-isopropyl-5-methylphenol (thymol) ethanol 3-methyl-1-butanol 2-phenylethanol E-3-phenyl-2-propene-1-ol 2-propanol 2-propanol benzyl alcohol (phenylmethanol) diphenylmethanol (benzhydrol)

# Experiment 5 Identification of Unknowns by Gas Chromatography-Mass Spectrometry

Read pp. 608-627 in your <u>lecture text</u>. The procedure for this experiment is attached to this syllabus. The report for this experiment is due on May 10.

# Apr. 26

# Experiment 6 Isolation and Identification of Spice Ingredients

Read pp. 487-492 in your lab text. Do Experiments 57A and 57B. The report for this experiment is due on May 3.

### May 3

# **Experiment 7** The Addition of HBr to Unsymmetrical Alkenes

Read pp. 403–408 in your <u>lecture text</u>. The procedure for this experiment is attached to this syllabus. Each person should use a different one of the alkenes, 3-propen-2-one (methyl vinyl ketone), methyl 2-propenoate (methyl acrylate), vinyl acetate, or phenylethene (styrene). Obtain a <sup>1</sup>H-NMR spectrum of your product. You should discuss the regiochemistry of addition to all of the alkenes in your report. The report for this experiment is due on May 17.

# **May 10**

# **Experiment 8** The Grignard Reaction

Read pp. 292-299. You will do a procedure very similar to Experiments 31 and 31A, except on a larger scale. The procedure for this experiment is attached to this syllabus. This is a two-week experiment. The report for this experiment is due on May 24.

# May 24 Finish any experiments, clean up, and check out.

You must check out of the lab at this time or you will receive an incomplete in the lab. Do not forget to turn in your lab notebook for grading.