Organic Chemistry Laboratory CHEM 2462

Spring, 2005

Instructor:

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Teaching Assistants: A list of the teaching assistants, their offices, phone numbers, and email addresses 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 M, Tu, W, Th or 6:00 to 9:30 Tu. Please come, on time, to your assigned lab section. 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:

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. 800 points Lab notebook, other than pre-lab write-ups 40 points

Schedule of Laboratory Experiments Spring Quarter, 2005

Week of Mar. 21

Check In

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 during the week of Apr. 4.

Week of Apr. 4

Experiment 2 Bromination of Stilbene

Read pp. 413-419 in your <u>lecture text</u>. The procedure for this experiment is available on Blackboard. The report is due during the week of Apr. 11.

Week of Apr. 11

Experiment 3 Catalytic Hydrogenation

Read pp. 244-248 in your lab text. Do Experiment 25. The report is due during the week of Apr. 18.

Week of Apr. 18

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 ¹H-NMR spectra of the other compound to identify it. The report for this experiment is due during the week of May 2.

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-propen-1-ol 1-propanol benzyl alcohol (phenylmethanol) diphenylmethanol (benzhydrol)

Experiment 5 Identification of Unknowns by Gas Chromatography-Mass Spectrometry

Read pp. 617-633 in your <u>lecture text</u>. The procedure for this experiment is available on Blackboard. No prelab is required. The report for this experiment is due during the week of May 9.

Week of Apr. 25

Experiment 6 Oil of Cloves

Read pp. 135-143. Do Experiment 13A. The report is due during the week of May 2.

Week of May 2

Experiment 7 Acetaminophen

Read pp. 105-113. Do Experiment 9B. The report and the solid product you prepared are due during the week of May 16.

Week of May 9

Experiment 8 The Grignard Reaction

Read pp. 292-299. Do Experiments 31 and 31A. This is a two-week experiment. The report is due during the week of May 23.

Week of May 23 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.