Organic Chemistry Laboratory
CHEM 2463
Fall, 2003
MAJORS LAB SECTION

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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.

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.

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 reagent 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. No student is allowed in the stockroom without explicit permission of the TA.

12. No chemicals, glassware or equipment are to be removed from the lab. No unauthorized experiments are to be done.

Notebooks:

You must have a bound lab notebook. You may use the same notebook and the same style as last quarter.

Prelabs:

Prelabs are to be done in the same manner as last quarter.

Reports:

Reports are to be done in the same manner as last quarter.

Products:

For synthesis experiments, submit the product in a clean, labelled vial along with the report. The label should have your name and the structure, weight, percent yield and mp or bp range of the product.

Grading:

Your grade will be based on a total of 1000 points, distributed as follows:

Pre-lab write-ups, 10 pts./exp.	50 points
Completion of experiment, 20 pts./exp.	180 points
Lab reports and products (if applicable) 80 pts./exp.	720 points
Lab notebook, other than pre-lab write-ups	50 points

Schedule of Laboratory Experiments
Fall Quarter, 2003

Sept. 8
Check-In
Experiment 1 Synthesis of Benzocaine

Read pp. 346-351. Do Experiment 41. Do not do the recrystallization nor obtain the spectra of the benzocaine.

Sept. 15
Experiment 2 Esterification Reactions of Vanillin

Read pp. 507-509. Do Experiment 62. One of you do the reaction in base (Preparation of 4-acetoxy-3-methoxybenzaldehyde) and the other do the reaction in acid (esterification of vanillin in the presence of acid).

Sept. 22
Experiment 3 Michael and Aldol Condensation Reactions

Read pp. 504-507, 316-318, and 320-323. Do Experiment 61. You will be assigned one of the substituted benzaldehydes as the starting material. Devise an experimental procedure for the entire experiment before lab. Your procedure must be approved before you start the experiment. Make the chalcone during this lab period. Save the product and use it as the starting material for the Michael/aldol product during the week of Oct 14.

Sept. 29
Experiment 4 Preparation of 1,4-Diphenyl-1,3-butadiene

Read pp. 333-338. Do Experiment 38. Do Part A, the preparation of benzyltriphenylphosphonium chloride (Wittig salt) this week. Save the Wittig salt and use it to complete the synthesis next week. During the reflux period you will have time to do Experiment 5.

Experiment 5 Simulated Identification of an Unknown

Read pp. 428-434. There is no prelab required for this experiment. This lab is a computer simulation of the real experiment you will do at the end of the quarter. You will be assigned two unknown organic compounds to identify on the computer. In order to identify each compound you must tell the computer which test to run on the unknown. The computer provides you with the result of the test. You then have to decide what test to run next. You continue this process until you have identified the compound. Since the "tests" on the computer can be done easily, this experiment allows you to hone your skills before undertaking the real thing. Make sure to interpret the IR and NMR spectra of your unknown if they are available. Record the results of each test just as though this is an experiment done in the lab. Your report should include a complete write-up of the identification of one unknown, just as thought the procedures were actually done in the lab. Also include the identity of the other unknown that you were assigned.

Oct. 6
Complete Experiment 4

Read pp. 333-338. Do Experiment 38, Part B.

Oct. 13
Complete Experiment 3

Oct. 20
Experiment 7 Friedel-Crafts Acylation

Read pp. 493-499. Do Experiment 58, using toluene, ethylbenzene, or anisole as the starting material. Do not do the vacuum distillation. You will use NMR spectroscopy to identify which position has been acylated in your product.

Oct. 27
Experiment 8 Identification of an Unknown

There is no prelab for this experiment. This time you will be given a sample of a real unknown to identify. It will be one of the compounds listed in the tables in Appendix 1 (pp. A2-A10). Follow the procedure outlined on pp. 428-468 in your lab text. You must do PART ONE, steps 1, 2, 4, and 6 (at least one positive and definitive functional group test), PART TWO, and PART THREE step 9 (prepare one solid derivative whose mp confirms the identity of your unknown) and step 10. This is a two-week experiment and the report counts twice the normal amount.

Nov. 10
Clean Up, Check Out

You must check out of lab at this time, turn in your breakage card and pay for any equipment you have broken or you will receive an I as your lab grade. **Do not forget to turn in your lab notebook.**