

Organic Chemistry Laboratory
CHEM 2460 1

Winter, 2002

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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 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. 2-20 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 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 must have a bound lab notebook. You should use the style described in your lab text. All data, observations, etc. must be entered directly in your notebook. Your notebook will not be graded on neatness, only on completeness.

Prelabs: For most experiments, you must do a prelab write-up in your notebook before coming to the lab. This write-up should be similar to that shown on p. 24 of your lab text up to the "Data and Observations" part. In place of the "Separation Scheme" write a brief outline of the procedure that you will follow for that experiment. You must have your prelab write-up initialed by your TA before you can get your starting materials from the stockroom or begin the experiment.

Reports: Read p. 26 in your lab text. The report for each experiment must be typewritten. These reports should include:

- Your name and the date and title of the experiment;
- A brief description of the objective or purpose of the experiment;
- A chemical equation if applicable (Structures must be done using a computer program such as ISIS/Draw.);
- A description of the experimental procedure including pertinent observations, data (actual weights, volumes, mp, etc.), graphs, and spectra;
- All of your calculations, e.g. for the theoretical and percent yields;
- A brief discussion, analyzing your experimental data (If the experiment did not work well, comment on why this might be.);

Excluding the data, calculations, and graphs, the report should be a maximum of two pages long. See the attached guidelines and sample lab report for more details.

Unless you are told otherwise, each report is due at the beginning of the next lab after the completion of the experiment. Reports will be penalized 30% for lateness. Reports more than two periods late will not be accepted.

Products: Read p. 27 in your lab text. For synthesis experiments, submit the product in a clean, labeled 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.	80 points
Lab reports and products (if applicable) 80 pts/exp.	880 points
Lab notebook, other than pre-lab write-ups	40 points

Schedule of Laboratory Experiments Winter Quarter, 2002

Week of Jan. 7

Check In

See pp. 33-36 in your lab text.

Experiment 1 Drawing Chemical Structures on the Computer

You will download a free program for drawing chemical structures from the Internet. You will learn to use this program during this lab. (See the attached handout.) This is the program that you will use it to draw any structures in lab reports for subsequent labs. If you have a laptop computer, please bring it to this lab. Otherwise, please bring a disk so you can save the structures that you will need for your lab report. You may load the program before coming to lab if you so desire. Access www.mdli.com on the Internet. Look for the "Download Free Software" button. Click on the button and download the version of ISIS/Draw that is appropriate for your computer. Also download the appropriate Help file. While you are at this site, also download the Chime program that is appropriate for your computer. This program enables you to view molecules in three dimensions and will be very useful in class. Both of these programs are self-extracting. Go to the location on your computer where they have been saved and double-click on each to extract the program. There is no prelab for this experiment.

Week of Jan. 14

Experiment 2 Isolation of the Active Ingredient in an Analgesic Drug

Read pp. 96-99 in your lab text. Make sure that you also do the "Required Reading" listed on p. 97. The analgesic drug, either aspirin, Tylenol, Advil, Nuprin or Motrin, will be provided. Do Experiment 7.

Week of Jan. 21

Experiment 3 Acetylsalicylic Acid

Read pp. 100-105 in your lab text. Do Experiment 8. Allow the product to dry and obtain the melting point during next week's lab.

Week of Jan. 28

Experiment 4 TLC Analysis of Analgesic Drugs

Read pp. 114-119 in your lab text. Do Experiment 10. You will be given an unknown analgesic. Analyze the unknown and four other analgesic tablets of your own choice.

Week of Feb. 4

Experiment 5 Synthesis of Isopentyl Acetate

Read pp. 131-133 in your lab text. Do Experiment 12A. Use the apparatus shown in Figure 8.7B on pp. 624 to do the distillation and to measure the boiling point of the product.

Week of Feb. 11

Experiment 6 Separation of a Mixture by Extraction

Read pp. 71-72 and 76-79 in your lab text. Do Experiment 4, Part D and Part E.

Week of Feb. 18

Experiment 7 Gas Chromatographic Analysis of Gasolines

Read pp. 227-228 in your lab text. Do Experiment 24. Work in groups of four. Each member of your group should inject one of the four samples (reference mixture, regular gasoline, mid-grade gasoline, high-grade gasoline) into the gas chromatograph. No prelab writeup is required for this experiment.

Experiment 8 Stereochemistry

Read pp. 166-174 in your lab text. Do the experiment in the handout that will be provided. Bring your laptop computer to lab if you have one. Work with a partner. No prelab is required for this experiment.

Week of Feb. 25

Experiment 9 Synthesis of t-Pentyl Chloride

Read pp. 208-209 and 478-479 in your lab text. Do Experiment 53B. This experiment is done on a much larger scale than the other experiments so that you can see the difference.

Experiment 10 Reactivities of Some Alkyl Halides

Read pp. 190-192. Do Experiment 18, Parts A and B. Work with a partner.

Week of Mar. 4

Experiment 11 Dehydration and Zaitsev's Rule

Read pp. 215-216 and 222-227. Do the dehydration of 2-methylcyclohexanol using the same procedure described in Experiment 23A for 4-methylcyclohexanol. Analyze the final product mixture by gas chromatography to determine its composition.

Mar. 11, 12, or 13

Clean Up and Check Out

You must check out of the lab or you will get an incomplete. Do not forget to give your lab notebook to your TA for grading.

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ORGANIC CHEMISTRY LABORATORY
GUIDELINES FOR LAB REPORTS

The lab report should be a self-contained description of what you did in lab. By reading your report and any references you give, someone should be able to reproduce your experiment, mistakes and all. Use the past, passive voice to write your report. For example, use "The aspirin was recrystallized from 3 mL of ethyl acetate ...", not "I recrystallized the aspirin ...". Begin the report with the title of the experiment, your name and the date the experiment was done. Continue the report with the headings and sections listed below.

Introduction

This section should have a brief statement of the purpose of the experiment. If the experiment is a synthesis of a compound, a balanced equation for the synthesis should be provided.

Procedure

This section should outline the procedure you used. You may refer to your lab book. Keep it simple, but complete.

Data and Calculations

In simple experiments, where only a little data produced, the data can be included in the Procedure section. Be sure to include the amounts of all reagents used. If a lot of data is produced, you should collect the data in a table.

Show any calculations that were done, such as calculation of the percent yield. If there are a lot of similar calculations, include only a calculation for one example.

Results and Discussion

This section should include the results of the experiment along with a brief discussion of these results. Include the yield of the reaction, the results of any tests run on the product, a discussion of the purity of the product and the basis for your conclusions (i.e., test results, comparison of the mp with the literature value of the mp, etc.), a discussion of any mistakes that were made in the experiment, interpretation of any spectra obtained, and whatever else might be appropriate for a particular experiment.

References

List any references, along with footnote numbers, that you use in your report. Your lab text will be the most common reference. Use the form given below.

1. Pavia, D.L., Lampman, G.M., Kriz, G.S., Engel, R.G., *Organic Laboratory Techniques, a Microscale Approach*, Third Edition. Philadelphia: Sanders, 1999, pp. 76-77.

(Sample lab report)

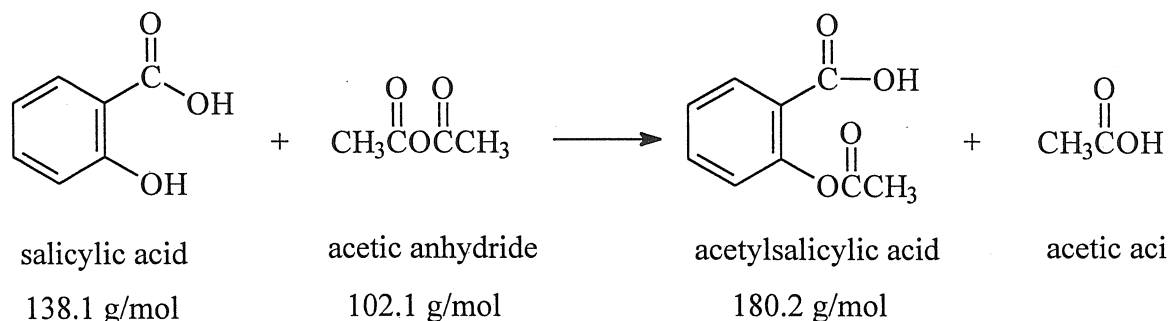
Preparation of Acetylsalicylic Acid

Amy Student

Jan. 15, 2002

Introduction

The purpose of this experiment was to prepare acetylsalicylic acid according to the equation given below.



Procedure

The procedure of Pavia et al.¹ was followed. A mixture of 0.213 g (1.54 mmol) of salicylic acid, 0.480 mL of acetic anhydride (0.518 g, 5.08 mmol) and one drop of concentrated phosphoric acid was placed in a 5-mL conical vial. A magnetic spin vane was added to the vial and it was fitted with an air condenser. The vial was heated at 50°C for a total of 14 minutes.

(Continue, describing the isolation procedure, etc.)

Data and Calculations

(Show the calculation of the percent yield here.)

Results and Discussion

This procedure produced 0.142 g (51% yield) of acetylsalicylic acid as a white solid. The melting point of the product was slightly lower than the literature mp of 135-136°C¹, indicating the presence of only minor impurities.

(Continue with a discussion of the purity, the reasons for loss of material and a low yield, etc.)

References

1. Pavia, D.L., Lampman, G.M., Kriz, G.S., Engel, R.G., *Organic Laboratory Techniques, a Microscale Approach*, Third Edition. Philadelphia: Sanders, 1999, pp. 133-134.

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Organic Chemistry Laboratory

Experiment 1, Drawing Structures on the Computer

In this experiment, you will learn to use a computer program to draw chemical structures. This program is available on the Internet and is free of charge for educational use. Once you have gained some experience with this program, you can use it to draw structures for all of your lab reports.

Download the Program

Access the Internet. Go to www.mdli.com. Select "Download Free Software". Follow the instructions to download the version of ISIS/Draw that is appropriate for the computer that you are using. Also download the help file. Then extract the ISIS/Draw program on your computer.

Drawing Structures

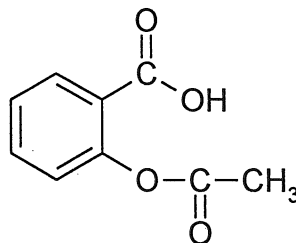
Load ISIS/Draw. From the help menu, click on "Quick Start". Step through the instructions and practice drawing some structures.

Once you have some familiarity drawing structures, draw the structure of acetylsalicylic acid shown below. Some useful hints are:

To show the hydrogens on OH and CH₃, click on "Options", "Settings", "Chemical Drawing", "Show Hydrogen Labels" and set "on hetero or terminal";

To make a carbon that is shown only as an intersection of lines visible, select the carbon, click on "Object", "Edit Atom", "Symbol", and select "C(show)";

Once you have drawn a structure and added the name below it using the text command, it is a good idea to select the entire structure and text and "Group" them under the "Object" menu. This keeps the structure and name together.

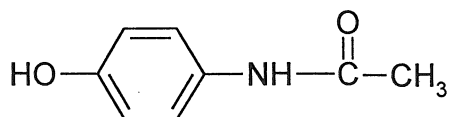


Acetylsalicylic acid

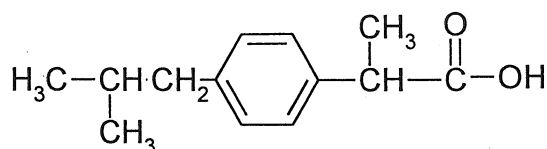
A structure can be placed in a Word document by copying the structure in ISIS/Draw and then pasting it into your document.

Your lab report for this lab should include all of the structures and equations that you draw for this experiment. You will use these structures for the next two lab reports, so you will not have to redraw them. If you are not using your own computer to draw the structures now, copy them into a blank Word document and save that document to your disk so that you can transfer them to your lab report.

Next, draw acetaminophen and ibuprofen.

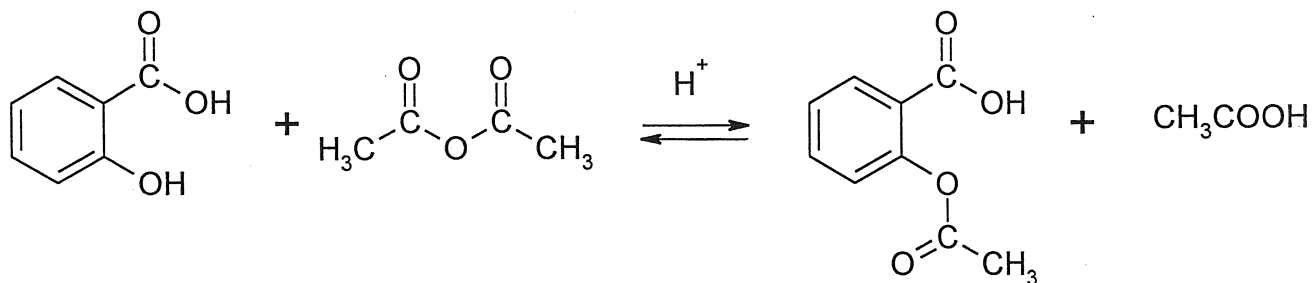


Acetaminophen



Ibuprofen

Draw the equation for the preparation of acetylsalicylic acid (aspirin) that you will do in Experiment 3.



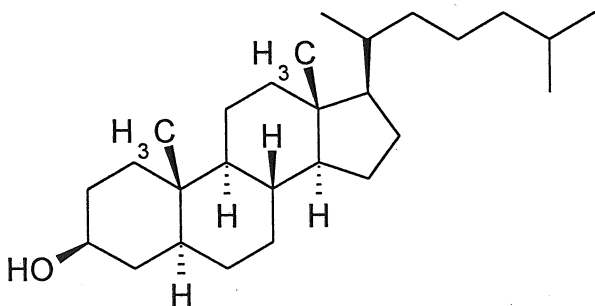
Salicylic acid

Acetic Anhydride

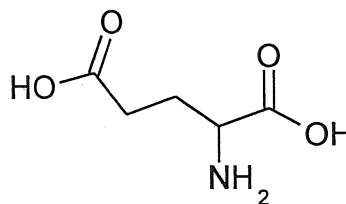
Acetylsalicylic Acid

Acetic Acid

Finally, draw the following structures. Use "Templates" in ISIS/Draw to draw glutamic acid.



cholesterol



glutamic acid