CHEMISTRY 344 - Organic Chemistry Laboratory II – Spring 2012
Lab #7: Esterification of acetic acid with an unknown alcohol (two weeks)

THIS IS A TWO-WEEK LAB AND YOU WILL CHECK-OUT IN WEEK #2.

In this lab, you will prepare an ester from acetic acid and an unknown C-4 or C-5 alcohol. You will evaluate your product by its percent yield, IR and NMR spectroscopy.

Pre-lab Reading: You will follow the microscale experimental procedure found on the next page. Microscale distillation with a Hickman head will be used.

Pre-Lab Notebook Preparation: Complete the following information in your lab notebook before your pre-lab recitation. Use the numerals and headings as given.

Table of Contents: You will need to update the Table of Contents for each new lab with the date and brief title of each experiment, and the notebook page number on which the lab begins.

I. Title of the Experiment and Date:

II. Purpose of the experiment: One- or two-sentences describing what you are going to do and why. State your experimental goals.

III. Equations: Write the balanced equation for this reaction, ***including structures***.

IV. Table of Reagents: In your lab notebook create a table, like the one shown below, for the following reagents used in this lab: **acetic acid, 1-butanol, 2-butanol, 2-methyl-1-propanol, cyclopentanol, 1-pentanol, 2-pentanol, 3-pentanol, 3-methyl-1-butanol.** Your table must include the name, structure, and all physical data. You can find this information in the Aldrich Chemical Catalog, the Merck Index, or on-line from the **ChemIndex** web site. The link to ChemIndex and instructions can be found on Dr. Brush's Lab web site: **http://webhost.bridgew.edu/ebrush/CH343%20Lab.htm**.

Chemical name and formula	structure	molecular mass (g/mole)	melting point (solids) (^{o}C)	boiling point (liquids) (^{o}C)	density (liquids) (g/mL)

V. Procedure: Summarize the lab procedure in your notebook. Do not copy the procedure word-for-word.

VI. Health, Environmental, and Waste disposal: Look up and summarize some of the health and environmental hazards for **acetic acid** and **all of the alcohols listed** at the MSDS database: **http://hazard.com/msds/index.php**

***Laboratory Book*: The following should be completed before leaving lab, initialed and dated by you.**

VII. Data and Observations: Record a rough transcript of your experimental method in your lab notebook, indicating what you actually did and what you actually observed. Do not prepare this section in advance. This section should be written in a paragraph format and include: **experimental procedure, all reagent mass and volume measurements, observations, crude and pure product mass or volume, % yield calculations, and product analysis by melting point, chromatography analysis, or instrument analysis.** You will be judged on the depth of your observations and technical success (yields, purity, etc.).

VIII. Discussion and Conclusions: A brief, but critical evaluation of your results, and the success of your experiment. Address the experiments goals and purpose!

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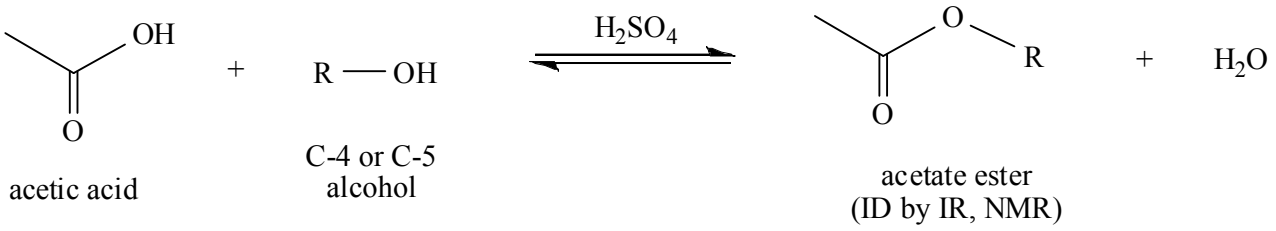
Lab #7: Esterification of acetic acid with an unknown alcohol

You will be doing the acid catalyzed esterification of acetic acid with an unknown C-4 or C-5 alcohol. Either your instructor will assign or you will choose one of the following C-4 or C-5 alcohols to react with acetic acid:

1-Butanol	1-Pentanol
2-Butanol	2-Pentanol
2-Methyl-1-propanol	3-Pentanol
Cyclopentanol	3-Methyl-1-butanol

The general chemical reaction for this esterification is:



acetic acid

C-4 or C-5 alcohol

acetate ester (ID by IR, NMR)

Remember to write down the number of your alcohol unknown.

Experimental Procedure

Step 1. Assembly the apparatus
Using a 5 ml conical vial assemble a reflux apparatus with **air condenser** and **drying tube**. Top the condenser with a drying tube that contains a loose plug of cotton. The purpose of the drying tube is to control odors in this case. Use a hot plate and an aluminum block for heating.

Step 2. Preparing the reaction mixture
Record the weight of the empty 5 ml conical vial. To deliver the two reagents you need either a dispensing pump or an automatic pipet. Add 1.0 ml of your unknown alcohol then determine the exact mass of alcohol added by reweighing the vial. Add 1.5 ml glacial acetic acid (MW= 60.1 g/mol, d=1.06 g/ml). Using a Pasteur pipet, add 2-3 drops of concentrated sulfuric acid (CAUTION!). Swirl the liquid to mix. Add a small magnetic spin vane and reattach the vial to the apparatus.

Step 3. Reflux
Bring the mixture to a boil (150-160^{0}C) and use continuous stirring. Continue heating under reflux for 60-75 minutes. Remove the heating source and let the reaction mixture cool to room temperature.

Step 4. Workup
Take the apparatus apart, remove the spin vane. Using a Pasteur pipet slowly add 1.0 ml 5% aqueous sodium bicarbonate ($NaHCO_3$) over the cooled reaction mixture. Stir the mixture in the vial with a spatula until CO_2 is not produced vigorously. Then cap the vial and shake gently with venting until the evolution of gas is complete. Remove the lower aqueous layer with a Pasteur pipet and discard it. Repeat the extraction two more times, using 1 ml fresh $NaHCO_3$ each time.
If the vial containing the ester has droplets of water, with a dry Pasteur pipet, transfer the ester to a dry 3 ml conical vial. If it is the case dry the ester with anhydrous sodium sulfate.

Step 5. Distillation
For distillation be sure to transfer the crude product to a clean, dry 3.0 mL conical vial. Determine the mass of crude product, and then add a spin vane. You will purify your ester product by distillation using a Hickman head. The **Hickman head** will be inserted between conical vial and **air condenser**. In order to control odors, rather than keep the reaction dry, top the apparatus with a **drying tube** packed loosely with calcium chloride and cotton. During distillation the conical vial and the base of the Hickman head should be wrapped on Aluminum foil. Begin the distillation by turning on the hot plate (180^{0}C) and the stirring. Continue the distillation until only 1-2 drops of liquid remain in the distilling vial.

DO NOT DISTILL ALL OF YOUR CRUDE PRODUCT! IT IS ALWAYS BETTER TO LEAVE A FEW DROPS BEHIND RATHER TO DISTILL OVER HIGH BOILING CONTAMINANTS!

If the Hickman head fills before the distillation is complete, it will have to be emptied using a Pasteur pipet and transfer the distillate to a preweighed conical vial. When the distillation is complete, transfer the final portion of the distillate to the same vial.

Step 6. Analysis of product
Determine the mass of your purified ester and calculate the percentage yield of it.
If time allows determine the IR and NMR. If you don't get this far then store your product in a tightly capped vial until your next lab session. In Week #2, you will determine the IR and NMR of your product, and use this information to determine the structure of your ester and identity of the unknown alcohol.

Name:_____ **Lab Partner**_____

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Lab Report Grade _____(100 points)

The following questions are based on your pre-lab reading and the results of your experiment. You and your lab partner are encouraged to work together, but you must each turn in your own report! Answer each question in the space provided, and **show all calculation work below or on a separate sheet of paper**.

1) glacial acetic acid _____ (g) _____ (moles)

2) unknown number of alcohol _____

3) IUPAC name of alcohol unknown (from below)_____

4) alcohol _____(g) _____ (moles)

5) limiting reagent_____

6) IUPAC of ester produced (from below)_____

7) theoretical yield of ester_____ (g) _____ (moles)

8) yield of crude ester _____(g)….you will NOT use this value for further calculations

9) actual yield of purified ester _____(g) _____ (moles)

10) percent yield of ester _____

11) Acid-catalyzed esterification is an equilibrium reaction. One method for favoring ester formation is to add excess acetic acid. Suggest another method, involving the right-hand side of the equation, that will favor the formation of ester.

12) Why is the reaction mixture extracted with sodium bicarbonate? Give a chemical equation and explain its relevance.

13) Why are gas bubbles observed during extraction with sodium bicarbonate?

14) Interpret the principal absorption bands in the IR spectrum of your ester. Staple a copy of your IR to this report form.

15) Interpret the NMR spectrum of your ester. Enter the name of your ester on Line #6, and the name of the alcohol unknown on Line #3. Draw the structure of your ester on the NMR spectrum, assign all signals to appropriate protons, and staple a copy of your NMR to this report form.

16) Evaluation. Was your experiment a success? Consider your goals, observations, and spectral data. A short paragraph is expected.