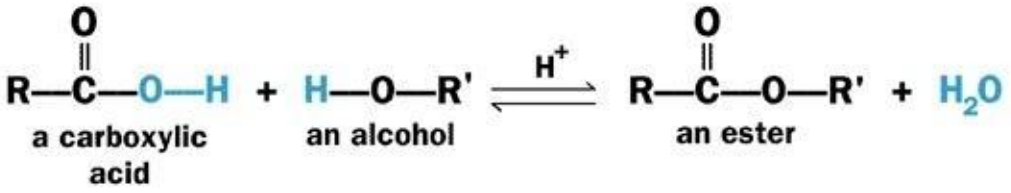
The objective of this experiment is to synthesize an ester and determine its structure.

All products have distinctive aromas, as do the starting materials. All products are currently in use in the United States in the flavor and fragrance industry. A commercial flavor and fragrance catalog will be available in the laboratory for reference.

Chemistry Background

In the presence of an acid catalyst, an alcohol and a carboxylic acid will form an ester. The reaction is an equilibrium.



(Where R and R' are general hydrocarbon groups)

Fun Fact

Clean all glassware with acetone, as you are making a **super glue** (ester compound) and failure to remove any materials may result in glassware becoming **fused** together.

Waste Disposal

Dispose of the aqueous filtrate in the aqueous waste container.

Please wash glassware with soap and water, rinse and let dry in drying rack or dry with a paper towel. Use acetone rinse for microkit and glassware that has difficulty air drying. Use the acetone sparingly.

Unknown Selection

In the table that follows, select ONE (1) unknown combination that you will be using in this lab (e.g. A1). Mark your selection both on Paper as well as on the instructor provided spreadsheet.

Combinations that are **NOT** permitted have been **greyed** out for your convenience. These have been shown to not work in practice.

	Organic Acid:		
Alcohol:	1	2	3
A			
B			
C			
D			
E			
F			

Procedure:

Reaction mixture: The unknown acids are in bottles labeled **1–3**; the unknown alcohols are in bottles labeled **A-F**. Each student will be assigned a combination such as **A3** (alcohol A and carboxylic acid 3). YOU MUST WRITE DOWN YOUR COMPOUND CODE.

1. Set the following reaction mixture up for heating under reflux for 45 minutes in a 10 mL Round Bottom Flask:
    a. 2.00 mL of the unknown alcohol - record the mass of alcohol added to the RB flask.
    b. 4.00 mL of the unknown acid (If your acid is a solid, use about 4g)- record the mass of carboxylic acid added to the RB flask
    c. Add 10-12 drops of concentrated sulfuric acid and a bar magnet.

2. When cool enough to handle, transfer mixture to a centrifuge tube.

NOTE: Do not re-attach the condenser to the RB Flask, as this will glue these together.

3. Use the following sequence of solutions to work up and extract the organic mixture - ***Each time, you are discarding the lower aqueous layer:***
    a. First extract with ~4 mL Saturated Salt (NaCl) Water
    b. Then extract with ~4 mL 10% sodium bicarbonate solution
    c. Perform this **SLOWLY**, as you may have an aggressive initial reaction.
    d. Stir/shake gently - making sure to relieve any pressure in your centrifuge tube. You may wish to stir using a glass stir rod, to avoid spillage.
    e. Remove the aqueous layer, using a pipette
    f. Repeat b-e until the solution no longer reacts with bubbles, or a max of three times.

4. Dry the resulting organic layer with anhydrous magnesium sulfate and filter – using filter tip pipet into a 3mL conical vial.

5. Record crude mass.

6. Set up apparatus for microscale distillation (*warning: if you have more than 3 mL crude product, remove some of it until you have a maximum of 3 mL product in the conical vial before starting distillation*). Distill compound, you should be able to collect at least 1 mL of distillate. Transfer distillate from Hickman Head to a pre weighed sample bottle. Note fragrance of distilled ester.

7. Weigh final product. Determine mass yield.

8. A data packet is provided via the links that follow, locate and then print your copy:

	Organic Acid:		
Alcohol:	1	2	3
A	A1		A3
B	B1		B3
C		C2	
D	D1		D3
E	E1		E3
F	F1		F3

9. On your IR spectrum, label spectrum appropriately and assign functional group peaks.

10. ^{1}H NMR of your product, determine structure of the ester.

11. Then compare your boiling point, IR spectrum and NMR to those found on the following website;

SDBS Website

or

any other web databases you can find.

Prelab:

1) Write out the mechanism for the acid catalyzed esterification reaction between ethanoic acid (CH_3CO_2H) and propanol. (Lookup Fischer esterification). Note that the alcohol functions as a solvent as well as a reagent.

(5 Points)

Postlab:

1. For a reaction to take place the reactants need to collide with enough energy and in the correct orientation. In this experiment we used both heat and an acid catalyzed to increase the rate of the reaction. With collision theory in mind, briefly explain how each of these factors(heat and acid catalyst) increase the reaction of rate. Be specific, they each affect the rate in a different, yet related, way.

(1pts)

- Heat: _____

- Acid: _____

2. How would removing water from the reaction vessel affect the equilibrium?

(1pt)

3. Explain how adding concentrated H_2SO_4 catalyzes the reaction. Be specific and **refer to structural drawings**. (Draw a Picture)

(1pt)

4. What is the purpose of washing the reaction mixture with sodium bicarbonate?

(1pt)

5. Why is it necessary to add anhydrous magnesium sulfate to the extracted ester product?

(1pt)

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PreLab
PostLab