Name: _____

Locker Number: _____

Experiment 56

PREPARATION OF A C-4 OR C-5 ACETATE ESTER

1. Write a **balanced** chemical equation for the preparation of your ester. Use structural formulas, showing the particular alcohol that you selected as a starting material.

PURE C-4 OR C-5 ACETATE ESTER

2. Report the following for your distilled sample of C-4 or C-5 acetate ester:

   a) the complete IUPAC name of your product: _____

   b) the mass (**grams**) of your product: _____

   c) the **boiling point range** of your product: _____

   d) the *literature value* of the boiling point of your product: _____

   e) the *source* of the information provided in **2d**, above: _____

3. Report the **percentage yield** of your product. Show your calculation, including a complete setup with cancellation of units. Use the actual yield that you reported in **2b**, above. Be sure to include a calculation of the correct molecular weights of your starting materials and product. Also, be sure to use the mass of the ester that you obtained – do not use the density for this calculation.

4. Outline a separation scheme for isolating your ester from the reaction mixture.

5. Submit your ester in a labeled conical vial with your laboratory report (if requested by your instructor).

6. Attach the infrared spectrum of your ester to this report. Be sure to label the C-H, C=O, and C-O peaks on the spectrum. Also label the CH_2 and CH_3 bending peaks.

7. Attach your gas chromatography results to this report. Using the results of the gas chromatography, complete the following table. Include alkene (if any), unreacted alcohol, and ester product in the table.

FRACTION (NAME OR STRUCTURE)	RETENTION TIME	OBSERVED PEAK AREA	CORRECTED PERCENTAGE

8. If you obtained on, attach the nuclear magnetic resonance spectra that correspond to your choice of starting alcohol and the acetate ester that you made to this report. Provide complete peak assignments for both spectra, indicating which protons give rise to each of the peaks on the spectra. You may make these assignments directly on the spectra.

ANSWER THE FOLLOWING QUESTIONS

1. One method for favoring the formation of an ester is to add excess acetic acid. Suggest another method, involving the **right-hand** side of the equation, that will also favor the formation of the ester.

2. How is the **bulk** of the excess acetic acid and sulfuric acid removed in the extraction step?

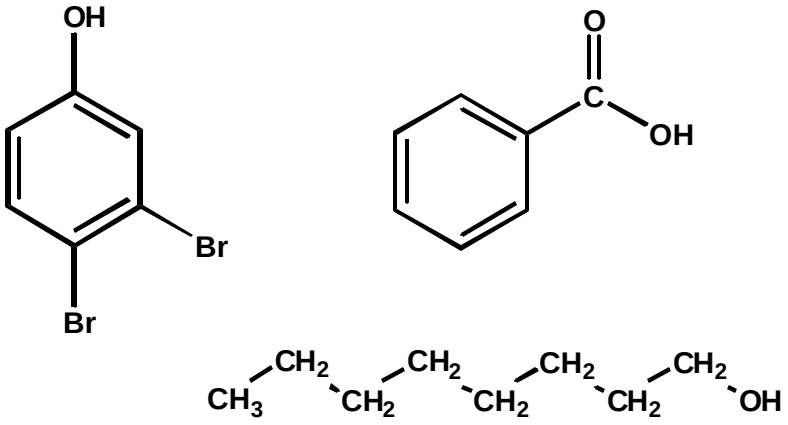
3. How is the **remainder** of the acetic acid removed? Include a chemical equation in your answer.

4. Which starting material is the **limiting reagent** in this procedure? Which reagent is used in excess? How great is the molar excess (how many times greater)?

5. What is the purpose of the extraction with aqueous sodium chloride?

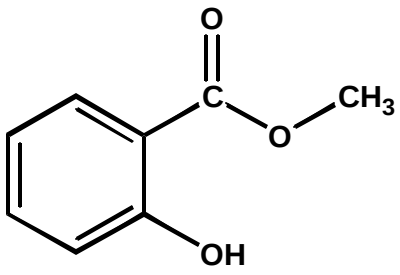
6. Tertiary alcohols do not work well in the procedure outlined for this experiment; they give a different product than the one you might expect. Explain this observation and draw the structure of the product formed when *tert*-butyl alcohol (2-methyl-2-propanol) is used in the esterification experiment.

7. Using aqueous sodium bicarbonate and sodium hydroxide solutions, devise a separation scheme (use the style shown in Figure 12.10, page 687) to separate the following three-component mixture. ***Be sure to show how you recover each component of the mixture.***



8. Draw the structures of the starting materials needed to prepare each of the following compounds:

a)



b) Start with a dicarboxylic acid