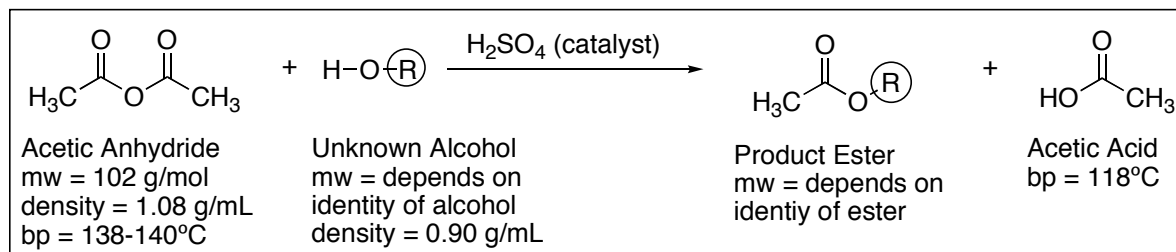


**ALCOHOL TO ESTER**  
**Acid-Catalyzed Esterification of an Unknown Alcohol**

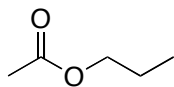


**Summary:** You will be given an unknown alcohol, you will convert it to an ester, and you will identify both the original alcohol and the derived ester.

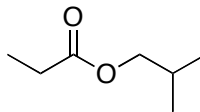
**Some Learning Goals:**

1. Observe the dramatic impact of acid catalysis
2. Understand the construction of esters
3. Review the distillation process
4. Use NMR combined with boiling point to identify the product ester

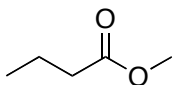
**Procedure:** To a single-necked 50-mL round-bottomed flask with a stir bar add 7.5 mL of acetic anhydride from a buret in the hood. Add 5.0 mL of your unknown alcohol, via syringe. (Notice that nothing happens!) Attach a Claisen adapter to the flask. Place a thermometer adapter with a thermometer in the main arm of the Claisen adapter so that the thermometer bulb is immersed in the liquid (but not so deep that it interferes with the stir bar.) Place a reflux condenser in the side arm of the Claisen adapter. Monitor the temperature for 1-3 minutes. Then remove the thermometer and adapter and add one drop of concentrated sulfuric acid (may be strong exotherm). If nothing happens, add a 2<sup>nd</sup> drop, or a 3<sup>rd</sup>. Rapidly replace the thermometer adapter and thermometer and magnetically stir the solution while monitoring the temperature of the reaction mixture. After the internal temperature has reached its maximum, wait an additional 3-5 minutes, and then transfer the mixture to a separatory funnel using a 25-mL ether rinse to aid the transfer. Extract the mixture three times with 20-mL portions of cold 5% NaOH solution. (Be sure to shake things up vigorously each time.) Check the last extract with pH paper to be certain that it is basic. Pour the organic layer into an Erlenmeyer flask and dry it over anhydrous sodium sulfate. Filter the solution into a clean, dry, 50- or 100-mL round-bottomed flask. Have three receiver flasks (**A**, **B**, and **C**) ready, with both **B** and **C** pre-weighed. Carefully distill (simple distillation) the ether and the product, using a heating mantle as heat source. The ether will boil off at relatively low temperature (35°-95°) and should be collected in flask **A**. The ester will boil at higher temperature, at least 90°, so after the temperature has hit 90° make flask **B** your receiver. If the product ester has a relatively low boiling point (<130°C), the boiling temperature should quickly plateau, and you can collect your ester in flask **B**. If you have a higher-boiling ester, switch to flask **C** by the time your temperature rises to 130°C. (Flask **B** will have some impurities but not desired material in it.) Record the “plateau” temperature when most of your ester boils off. Because you are using simple and not fractional distillation, your boiling point will have some uncertainty, and depending on which ester you have, there may be coincidental contaminants. Weigh your product ester. Prepare a GC-MS by adding one drop and diluting. Run a proton NMR on your starting alcohol to verify its identity. Between the bp information about the product ester and the NMR information about the alcohol, determine the structure of both the product ester and the starting alcohol. Note: the NMR info may be essential in some cases where bp's are close.

Ester Candidates

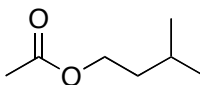
Propyl Acetate, 100-105° ± 10°



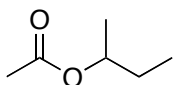
Isobutyl Propionate, 132-147°



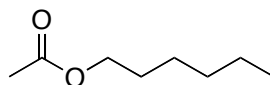
Methyl Butyrate, 100-105° ± 10°



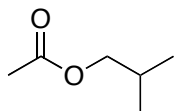
Isopentyl Acetate, 132-147°



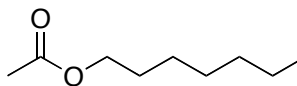
s-Butyl Acetate, 112-120°



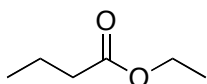
Hexyl Acetate, 167-177°



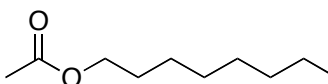
Isobutyl Acetate, 114-120°



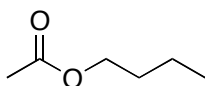
Heptyl Acetate, 187-197°



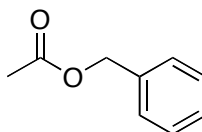
Ethyl Butyrate, 117-125°



Octyl Acetate, 202-220°



Butyl Acetate, 114-126°



Benzyl Acetate, 202-220°

**Lab Report:** This week, we'll skip the usual procedure writeup. Instead, report:

1. mass yield
2. approximate observed boiling range of ester
3. one H-NMR spectra of starting alcohol (experiment "Proton-8"). (0.07 mL sample, 0.8 mL of CDCl<sub>3</sub>). See page 30 for some tips on H-NMR complications for alcohols.
4. one GC chromatogram of your distilled product. Graph/% Report only, not mass specs.
5. based on the boiling point and/or your NMR, and the identity of the acetic anhydride reactant, identify the ester you made (the lab manual has NMR-interpretation summaries)
6. based on your product ester and/or your NMR, identify the alcohol you began with
7. determine the % yield [Note: this must be based on alcohol that you began with, so you need to deduce that first. Assume each starting alcohol had a density of 0.90 g/mL (not exactly true, but close enough) for your volume-mass-mole calculation.]
  - tip: To determine the theoretical, yield, you'll need to add up the molecular weight of both your alcohol and your product ester so that you can do mass/mole interconversions.