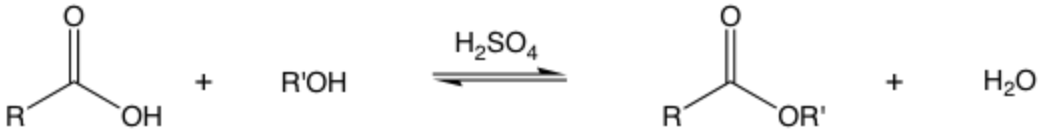
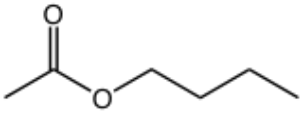
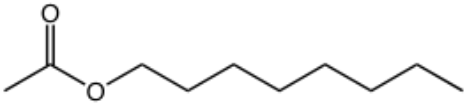
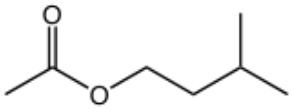
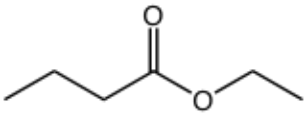
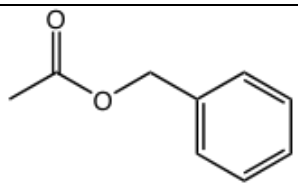
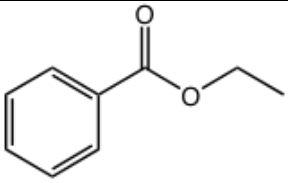
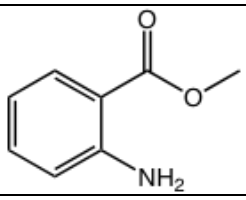
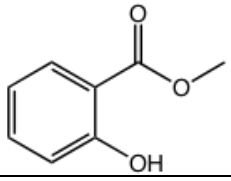
Fragrant Esters

Esters are prepared in a reaction between a carboxylic acid (RCOOH) and an alcohol (R'OH) which is known as an esterification. Esters are known for their pleasant fragrance and since we have access to a huge variety of acids and alcohols (varying the R and R' groups), we can prepare esters that produce different scents. Sometimes only small changes are needed to get a completely different fragrance. For example experiment 1 (see below) yields an ester that smells like apple. A slightly longer chain produces an orange scent. Having a branched chain leads to banana oil. While there are an infinite number of combinations of acids and alcohols, we have selected some of the most distinct fragrances. The reaction between a carboxylic acid and an alcohol is very slow so we will add sulfuric acid as a catalyst to speed up the reaction.



You will be synthesizing one of the following esters in the lab from their corresponding carboxylic acids and alcohols:

Exp	Ester	Structure	Fragrance
1	Butyl acetate		apple
2	Octyl acetate		orange
3	Isoamyl acetate		banana
4	Ethyl butyrate		strawberry

5	Benzyl acetate		peach/jasmine
6	Ethyl benzoate		perfume
7	Methyl anthranilate		grape
8	Methyl salicylate		wintergreen

Materials

- 25 mL Round Bottom Flask
- 25 mL Thermowell and Controller
- Magnetic Stirbar
- Reflux Condenser
- Tubing
- Graduated Cylinders
- Pipettes

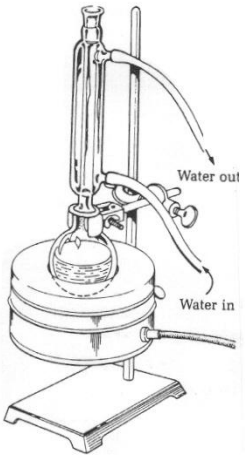
Safety

Personal protective equipment including safety goggles, gloves and a lab coat must be worn at all times during the experiment. Long pants must be worn along with close-toed shoes. No food or drink is allowed in the lab. Take special care when using sulfuric acid, it is corrosive and can cause severe skin and eye burns if it comes in contact.

Procedure

1. In a clean, dry 25 mL round bottom flask, add a magnetic stirbar, 2 mL of the alcohol and 3 mL of the carboxylic acid using graduated cylinders.

2. Slowly add approximately 10 drops of sulfuric acid.
3. Set up a reflux apparatus (shown below). Ask a demonstrator to check your set up before proceeding to the next step.



4. Heat the flask to boiling for 30 minutes. The Thermowell Controller should be around 45 and the stir knob should be around 3 on the stirrer/hotplate.
5. After 30 minutes, lower the Thermowell and allow the reaction to cool for a few minutes. Cool the reaction further in an ice-water bath for several minutes.
6. Slowly add 5 mL of saturated $NaHCO_3$.
7. Check the reaction mixture with pH paper. If the pH is below 7, add more $NaHCO_3$ until a neutral or slightly basic pH is obtained.
8. **Ask a demonstrator** to help you waft the reaction and note the odor

References: this set of experiments has been adapted from:

D.M. Birney and S.D. Starnes; *J. Chem. Educ.*, 1999, 76 (11), p 1560.