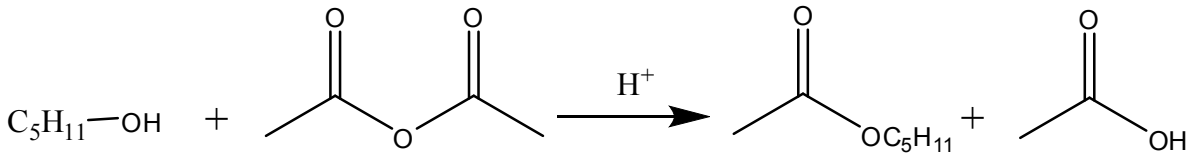
E29 Esterification: Preparation of 1-Pentyl Acetate

Abstract
The acid catalysed reaction of a carboxylic acid anhydride (acetic anhydride) and an alcohol (1-pentanol) has been used to synthesise an ester (1-pentyl acetate). As the products are miscible, they were separated using the higher solubility of acetic acid in water and distillation. The reaction is exothermic and a yield of 71% was obtained.

Introduction
Esters are important organic compounds and are found in many natural and biologically active molecules. The two main methods for the synthesis of esters are:

- reaction of a carboxylic acid with an alcohol or phenol, and
- reaction of a reactive carboxylic acid derivative with an alcohol or phenol.

The first method generally leads to an equilibrium mixture of reactants and products. The second method generally leads to complete conversion of the reactants to products. It is also rapid when catalysed by acid. The aim of this experiment is to use the second method to synthesise pentyl acetate from acetic anhydride and 1-pentanol using sulfuric acid as a catalyst:



1-pentanol acetic anhydride pentyl acetate acetic acid

The products are miscible liquids and must be separated and purified. This is achieved using the higher solubility in water of acetic acid and by distillation. Alongside the synthesis of 1-pentyl acetate, the experiment also shows how the technique of distillation can be used to purify liquids. It also shows how percentage yield can be used to quantify the efficiency of a synthetic process.

Method
The method in the laboratory handbook[1] on page E29.4 was followed.

Results
The reaction of 10.0 mL of 1-pentanol (equivalent to 8.15 g or 0.0925 mol) with 9.7 mL of acetic anhydride (equivalent to 11 g or 0.10 mol) led to the synthesis of 8.5 g (equivalent to 0.065 mol) of 1-pentyl acetate. The product was obtained as a clear liquid from the distillation between 144 and 150 °C. It had a strong smell similar to that of ripe bananas.

Discussion
The ester 1-pentyl acetate was produced by the acid-catalysed reaction of 1-pentanol and acetic anhydride.

The theoretical yield of 1-pentyl acetate from the 1:1 reaction of 0.0925 mol of 1-pentanol and 0.10 mol of acetic anhydride is 0.0925 mol. As the molar mass of 1-pentyl acetate is 130.18 g mol^{-1}, this corresponds to 12 g. The percentage yield obtained in the experiment is:

$$\text{percentage yield} = \frac{\text{yield}}{\text{theoretical yield}} \times 100\%$$

$$= \frac{8.5\text{ g}}{12\text{ g}} \times 100\% = 71\%$$

This is a reasonable yield. The procedure appeared to go well as the flask got very hot due to the exothermic reaction. It is likely that product was lost during the separation of the products. The solubility of 1-pentyl acetate in water has been reported[2] to be 10 g L^{-1} so it is possible that some of the ester was lost into the water during the separation.

Unfortunately, there was not time to identify or test the purity of the product. The boiling point of pentyl acetate has been reported[2] to be 149 °C. This could be used to identify whether the product is actually pentyl acetate . Spectroscopy could also be used. The mass spectrum of pentyl acetate would show a molecular ion peak at m/z = 13. The IR spectrum would show a peak at around 1700 cm^{-1} due to the presence of a C=O group. The ^{1}H NMR spectrum would probably be the most useful as it would in principle 6 separate signals due to the 6 groups of alkyl protons in the molecule.

The acid catalysed reaction of a reactive carboxylic acid derivative with an alcohol appears to be a good method for the synthesis of esters. Distillation appears to be a good technique for separating miscible liquids.

Conclusions

The acid catalysed reaction of a carboxylic acid anhydride (acetic anhydride) and an alcohol (1-pentanol) has been used to synthesise an ester (pentyl acetate). After purification of the products by distillation, a yield of 71% was obtained.

References

1. *First Year Chemistry Laboratory Handbook*, The University of Sydney, Australia, 2009.
2. Amyl Acetate, Wikipedia, http://en.wikipedia.org/wiki/Amyl_acetate, accessed 09/04/09.