Investigating Esters

Background

Esters are organic compounds with characteristic pleasant odours. They are among the compounds that give fruits and flowers their flavours and smells. They can be prepared by reacting an alcohol with a carboxylic acid.

\[
R-\text{OH} + R'-\text{C}=\text{O} \rightarrow R'-\text{C}=\text{O} + \text{H}_2\text{O}
\]

The reaction is reversible and the reaction proceeds very slowly towards an equilibrium. It is difficult to achieve 100% conversion and the yield of the ester will not be high. It may possible to improve the yield by changing the reaction conditions. The methods of changing the position of the equilibrium are well known. This equilibrium can be displaced in favour of the ester by the use of excess of one of the reactants.

Practical Techniques

Techniques that you need to find out about are reflux and distillation and the use of separating funnels. You also need to find out about how to find the boiling point of a liquid. (There are semi micro techniques that can be used if the yield is small.)

Where to start

Plan to prepare a sample of butyl ethanoate, ethyl ethanoate or methyl ethanoate.

Possible Investigations

- How is the yield affected by the use of excess of one of the reactants? What is the maximum yield that can be obtained?

- How is the yield affected by removing the product? This can be done by distilling out the product as the reaction proceeds effectively creating non-equilibrium conditions which would allow the reaction to go to completion. This is only feasible if the ester has the lowest boiling point of the reaction components. In the preparation of butyl ethanoate this does not apply but it is the case with methyl ethanoate.

- The heat change in the formation of an ester is almost zero, and changes to temperature have very little effect on the position of equilibrium.
However as the reaction is very slow, the length of reflux time will have an affect. You could investigate the effect of this on yield.

- Esters can also be prepared by reacting an acyl chloride or an anhydride with the carboxylic acid. For example if you were preparing an ester using ethanol as the alcohol this could be replaced by ethanoyl chloride or ethanoic anhydride. The methods of preparation using these reactants are different and you will need to research these before you start any practical work. You could investigate the effect of using these reactants on the yield of an ester.

- The properties of organic compounds change as you go along a homologous series. You could prepare a series of esters and investigate how their properties (e.g. boiling point, density, viscosity) differ.

Sources of Information


- Unknown author, Measuring the boiling point of a liquid, *Chemistry Review, May 1998*

- Unknown author, Using a separating funnel, *Chemistry Review, January 1994*

- Lindsey D, Refluxing and Distillation, *Chemistry Review, November 1999*

- [http://ull.chemistry.uakron.edu/genobc/chapter_15/](http://ull.chemistry.uakron.edu/genobc/chapter_15/)

- [http://www.iit.edu/~smile/ch9205.html](http://www.iit.edu/~smile/ch9205.html)

- Thorpe A, Assessing the risks in practical work, *Chemistry Review, September 2000*

- Thorpe A, Experimental error and error analysis: just how good are those results, *Chemistry Review, November 2001*
Teachers’ Notes

General Tips

This investigation is largely based around the preparation of esters and the factors that affect the yield of the ester. Each preparation is potentially quite time consuming and technically challenging. However competent students do tend to work more quickly with each new preparation. Therefore students will need to be organised and technically competent in order to collect sufficient data to analyse.

When equimolar quantities of acid and alcohol are used only about two thirds of the theoretically possible yield can be obtained. If the yield is small it may not be possible to determine the boiling point by the distillation method and a semi micro method should be employed.

The suggested method in the starter experiment is a generic one and may need to be slightly adjusted for each ester. Students should be encouraged to find details from the literature and compare methods.

Chemical Principles

Alcohols, Organic Acids, Esters, Equilibrium, Homologous Series, Percentage Yields.

Essential Equipment

Quickfit apparatus for refluxing and distillation, separating funnel, fume cupboard.

Essential Chemicals

Organic acids, alcohols, concentrated sulphuric acid, drying agent (e.g. anhydrous sodium sulphate), sodium hydrogen carbonate.

Safety

No risk assessment has been given. It is essential that students prepare a detailed risk assessment before they start. Teachers must be satisfied that this is suitable for the proposed investigation.
Starter Experiment - Investigating Esters

Prepare a sample of butyl ethanoate, ethyl ethanoate or methyl ethanoate.

Start with equimolar amounts of the alcohol and the acid (0.5 mol may be suitable). Cautiously add 2 cm³ of concentrated sulphuric acid. Reflux the mixture for 45 minutes. Allow to cool.

Pour the mixture into 50 cm³ of water in a separating funnel. Shake vigorously and remove the lower aqueous layer. Keep the upper layer of crude ester. Add a further 50 cm³ of water and about 25 cm³ of saturated sodium hydrogen carbonate solution to the ester. Repeat the washing process. Transfer the crude ester to a small conical flask and add anhydrous sodium sulphate. Swirl until the liquid appears clear. Decant the dried ester into a weighed bottle and reweigh. Record the yield.

You can check the purity of your ester by distilling the product and collecting the distillate at the boiling point of the ester. Record the yield again.