An experiment to produce pure ethyl ethanoate

Introduction

The aim of the experiment is to produce pure ethyl ethanoate using a variety of distilling methods. Ethyl ethanoate is an organic compound with the formula of $CH_3COOCH_2CH_3$. Ethyl ethanoate is a colourless liquid and smells similarly to pear drops. It is mainly used as a solvent for example in nail varnishes, and is also used in chromatography (wikipedia). It's a highly flammable liquid and therefore must be treated with caution (cameochemicals.noaa.gov). An organic compound contains a carbon based molecule. It is a compound which can be found either naturally, being produced by plants or animals, or can be synthesised in a laboratory. Distillation as the main method to produce pure ethyl ethanoate is ideal for this experiment as it allows for mixtures to be separated, until the pure substance is obtained, by heating and getting them to its high boiling point, turning it into a gas and condensing it back to a liquid. If this process is repeated several times, it ensures that it is pure, this is known as double distillation (chemistry.about.com).

Hypothesis

It is expected that it will be possible to produce pure ethyl ethanoate by distilling.

Safety assessment

Goggles	Prevents from any splashes getting in the eyes.
Lab coats	Prevents staining of clothes.
Stools and bags	Should be put away, preventing trips and falls.
Pipettes	Must be handled carefully, as the tip is fragile and can break
Liquids	Care must be taken when measuring and the correct procedures must be carried out when cleaning up any spillages. * Glacial ethanoic acid is corrosive and flammable. * Ethanol is flammable. * Concentrated sulphuric acid is very corrosive and reacts violently with water.
Glassware	Handle carefully. Follow procedures for cleaning any breakages.
Gloves and hands	Both must be washed thoroughly with soap and water and gloves disposed correctly.

Method

1. $10cm^3$ of ethanol was measured into a $50cm^3$ pear shaped flask in a fume cupboard. Then 10 cm^3 of glacial ethanoic acid was gradually added, and carefully swirled.
2. Anti – bumping granules were added to the flask in the fume cupboard.
3. 2 cm^3 of concentrated sulphuric acid was then added, drop-by-drop, which was carefully mixed by swirling after each drop, again in the fume cupboard.
4. The pear shape flask was added to the set up experiment as shown below and was gently heated for 15minutes.
5. After 15minutes the apparatus was allowed to cool down in order for the apparatus to be re-arranged for the distillation process.
6. The mixture was then distilled, ensuring that it was collected at 82°C
7. The distillate was then transferred to a separating funnel and 6 cm^3 of 30% sodium carbonate solution was then added. The funnel then had a stopper in place and was then shaken, allowing for the stopper to be removed to vent for short frequent periods.

8. The two layers were left to separate in the funnel. The lower aqueous layer was then ran off and discarded, leaving 1 layer left in the funnel.
9. 5g of anhydrous calcium chloride were then dissolved in 5 cm^3 of water. This was then added to the funnel contents and shaken vigorously. The lower layer was then ran off and again discarded.
10. A few small pieces of solid anhydrous calcium chloride were then added, and the flask was then bunged, shaken gently and left until the liquid becomes clear.
11. The liquid was then decanted into a clean flask, with a few more anti-bumping granules added to the flask.
12. The liquid was again distilled and collected, ensuring that it remained at a temperature between 74°C and 79°C. The flask was then weighed.

Set up of experiment

Nos. 1-5

Nos. 6-13

Results

Density = $\frac{\text{mass}}{\text{Volume}}$ Volume = $\frac{\text{mass}}{\text{density}}$ $\frac{\text{volume of product}}{20}$ x 100

$\frac{10.7}{0.92 = \text{density}}$ $\frac{11.63}{20}$ = 0.58 x 100 = 58.15%

The known density of ethyl ethanoate is 0.92grams per cm^3.

Interpretation

From the results it shows that a 58.15% yield was gained. At the start of the experiment I started off with 10 cm^3 of ethanoic acid and 10 cm^3 ethyl ethanoate, giving a total of 20 cm^3 of reactants. Therefore the overall result should end up with having 20 cm^3 of product equalling to 100% yield gained.

Evaluation

There are several reasons for not having a full percentage volume of ethyl ethanoate. One of these could be some residue was left in the beaker, this could be prevented by ensuring that all drops are removed, then weighing the beaker, checking it against a dry beaker for a comparison in weight. Another reason for a loss in percentage volume is due to the liquid spitting, when being heated, to prevent this from happening again; I would ensure that the Bunsen flame is low to prevent any spitting. When heating, the liquid vaporises, this can be proven by the smell. Using bungs to create and airtight seal is not adequate and is also a safety hazard, with a build up of gases. Instead, by using Vaseline you are able to prevent any fumes leaking, preventing any inhalation of toxic fumes and a loss in percentage volume. What seemed like a disadvantage during the experiment at the beginning of the experiment turned out to be an advantage. When heating the distilled mixture at a temperature of 82°C, it took a long time, as our experiment set up was next to the door, and it was a cold day, so every time the door was opened, the temperature dropped, causing it to take over half an hour to reach temperature. This however was advantageous to our experiment as it ensured that the remaining liquid was as pure as it can be. The whole process, from mixing hazardous substances to setting up the experiment required on the method being very precise, to ensure that I gained an accurate result, I would repeat the experiment again, or by practising the techniques used, to ensure of a reliable and accurate result. The use of anhydrous calcium chloride removes any water, by adding extra to the collection it's ensuring that there could possibly be no more water left – just to be sure.

REFERENCE:

http://www.wikipedia.co.uk

http://www.cameochemicals.noaa.gov/chemicals/665

http://www.chemistry.about.com/cs/5/f/bldistillation.htm