

CHEMISTRY

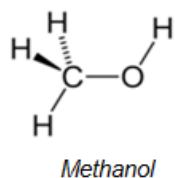
Prac Report: Esterification

Alkanol (Alcohol) + Alkanoic Acid \square Alkyl Alkanoate (Ester) + Water

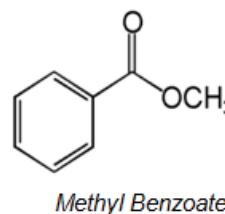
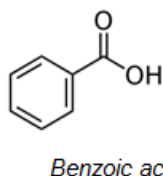
Methanol + Benzoic Acid \square Methyl Benzoate + Water

$\text{CH}_3\text{OH} + \text{C}_6\text{H}_5\text{COOH} \square \text{C}_6\text{H}_5\text{COOCH}_3 + \text{H}_2\text{O}$

sulfuric acid catalyst



+



AIM:

To synthesis an ester using a reflux apparatus.

HYPOTHESIS:

The combination of an alcohol and an acid will produce an ester and water; that is, adding methanol and benzoic acid together will create methyl benzoate and water. Methyl benzoate is said to be a colourless to pale yellow liquid which has a floral smell.

RISK ASSESSMENT:

| Risk | How to minimise the risk |
|--|---|
| Acids and Alcohols <ul style="list-style-type: none">• Corrosive• Toxic or noxious vapours/gas• May irritate eyes• Skin and respiratory sensitiser• May be combustible• Methanol is highly flammable | <ul style="list-style-type: none">• Wear safety goggles and gloves• Wear lab coat• Wear enclosed shoes• Work in a well ventilated area• Handle with care• Keep away from open flames• Clean up spills immediately |
| Bunsen burner <ul style="list-style-type: none">• May cause burns | <ul style="list-style-type: none">• Do not touch directly• Handle with care |

EQUIPMENT:

- ALKANOL [ALCOHOL]: Methanol CH_3OH
- ALKANOIC ACID: Benzoic acid $\text{C}_6\text{H}_5\text{COOH}$
- CATALYST: Sulfuric acid H_2SO_4
- Sodium Carbonate Na_2CO_3
- Calcium Chloride CaCl
- Distilled Water H_2O
- 100 mL round-bottom flask
- Boiling chips
- Conical flask
- Beaker
- Separating funnel

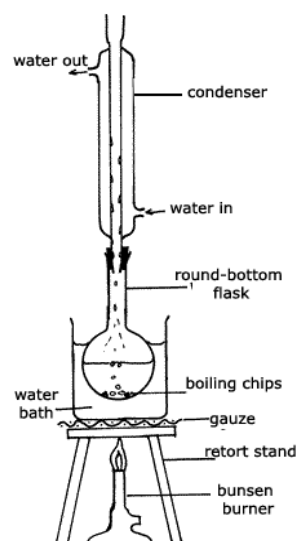
REFLUX EQUIPMENT:

- Water bath
- Retort stand
- Cooling condenser
- Bunsen burner
- Gauze

METHOD:

A. Reaction:

1. Carefully add 15 mL of methanol and 15 g (assuming 1 mL = 1 g) of benzoic acid into the 100 mL reaction flask (round-bottom flask).
2. Put 2-3 pieces of boiling chips into the flask.
3. Add about 10 drops of concentrated sulfuric acid into the flask.
4. Attach the flask to the reflux apparatus securely. Connect the tubing to the tap and condenser, and turn on the water so a uniform flow is achieved.
5. Heat in a water bath using a bunsen burner until a distinct layer is visible (approx. 30mins). Let it cool. Turn off the water.



B. Extraction:

6. Remove the flask and pour the contents into a separating funnel containing about 20 mL of distilled water. Stopper the funnel and gently shake.
7. Allow the layers to separate and discharge lower aqueous later into a clean beaker.
8. Add about 15 mL of Sodium carbonate solution (Na_2CO_3) to neutralise the excess sulfuric acid and shake.
9. Allow the layers to separate and discharge lower aqueous later into a clean beaker. Pour liquid that is left, which should be the ester into a conical flask.
10. Add a teaspoon of calcium chloride (CaCl_2) to remove the water.

RESULTS & OBSERVATIONS:



reflux apparatus

Beam Balance: 2

Beaker, round-bottom flask and methanol: 170.012 g

26 May 2008

- 10 mins: Bubbles started to form in the water bath.
- 11 mins: The ester started to boil
- 18 mins: The temperature started to rise above the recommended temperature, so I had to switch to the yellow flame. However, this led there to be soot on the outside of the water bath.
- 30 mins: Still no distinct layer and I had ran out of time. So I had to stop what I was doing and continue another time.



Extraction process using a separating funnel

27 May 2008

There were no distinct layers after the solution was left overnight. Though the solution could have reversed itself because it is an equilibrium reaction. A stopper was used to prevent loss of water.

- Used the separating funnel with water, and there were no real distinct layers. It was a cloudy mixture and it smelt heavily of methanol, this could be due to insufficient heating time to decompose the benzene.
- Gently shaking and inverting the separating funnel aided in the solution in mixing with the water.
- The aqueous solution released was insoluble in water.
- Sodium carbonate solution was added and there was a gas reaction – the solution started to fizz. With an added carbonate solution, it can be assumed that the gas released was carbon dioxide. When the stopper was removed after mixing, there was a pop sound with the release of gas.
- The layer was not distinct, but can be distinguished from the ester solution through its observable texture. It looked as if it was gelatinous and had tiny air bubbles.
- This lower aqueous layer was disposed, though the alcoholic smell did not change.

DISCUSSION:

Possible explanations for unexpected results and observations can be found in the procedure of preparing an ester. Because I had chosen to use different chemicals to that of my cohort, I could not tell if my procedure was correct. This was especially important when heating the mixture as each groups' chemicals had different boiling points. In this case, methanol has a relatively low boiling point (simple compound) while benzoic acid had a relatively high boiling point (complex compound), so my heating method had to be using a controlled temperature. This was achieved through using a water bath and controlling the heat received from the Bunsen burner. The cooling condenser used is to prevent loss of any volatile reactant or product, and also allows any alcohol vapour which rises from heating to condense and drip back into the reaction mixture.

Because equilibrium at room temperature is a relatively slow procedure, sulfuric acid (H_2SO_4) was used to act as a catalyst. Another reason sulfuric acid was used was to absorb the water produced, allowing the equilibrium to move to the right. However, a disadvantage to using this substance was that it had to be handled with extreme caution in the ventilation cupboard because it easily dehydrates cells (as seen dehydrating the cellulose of the paper towel).

At the end of the esterification process, sodium carbonate (Na_2CO_3) was used to remove any excess sulfuric acid, and when combined with the ester, could be easily removed as it formed a distinct layer. This was similar to the use of calcium chloride (CaCl_2), however this acted as a dehydrating agent by removing water after adding distilled water. This was performed using a separating funnel, a device which allowed distinct layers of the solution to be discarded.

Other results classmates had were:

Esters which smelt like pear, apricot and strawberry.

An interesting result were that two groups used the same chemicals, however the ester that they produced were of different scents. Reasons for this could be that they heated their mixture for different amounts of time; decomposing different amounts of chemicals (such as the benzene molecule is very complex, therefore would have to be heated for a longer amount of time).

To further investigate, this procedure could be reattempted to obtain the desired ester or to test the theory of how the smell of an ester can be affected by the amount of time it is heated for. Groups using the same chemicals can compare their results through the procedures they went through. Such information could be applied in many perfume and food industries to create artificial smells, but a pure ester would need to be obtained (via distillation). However, it is an expensive and time consuming procedure.

CONCLUSION:

I am unsure if I was successful in creating my desired ester, but it can be observed as not having the smell I had hypothesised. Instead of a floral smell, there was a distinct methanol-type smell. But this was performed using a reflux apparatus successfully.

EVALUATION:

I did not have much time working individually, so when I started to perform the reflux, my benzoic acid powder was not fully dissolved in the methanol. When I had started heating, I had to change the gauze mat I was using because there was some type of substance on the gauze mat which caused smoke.

My beaker that I used as a water bath was originally too small and I had to switch to an 800 mL one, though I only used 600 mL water. It was best to fill the water bath up with hot/warm water before I started heating so there would not be a drastic temperature change to the beaker.

The refluxing process was successful as there was alcohol vapour condensing and dripping back into the reaction mixture. My solution did not achieve a distinct layer and a possible reason for this is that it was not heated for long enough. Though it was also difficult to see if there was a distinct layer because it was boiling so rapidly.

The smell that I had achieved was not what I expected, and possibly due to the insufficient heating time, it smelt strongly of methanol. When it was added to distilled water, it was also difficult to see any distinct layers. Though there was a slightly darker lower layer and I assumed that that was the lower layer. The lower layer which was discarded was insoluble in water. This was the same for when sodium carbonate was added to the remaining solution; though I had assumed that the lower layer was the gelatinous-looking substance.

The esterification process is best undertaken as a team due to the advantages in time management to complete this task. Another advantage is to ensure that the equipment was properly set up and that the procedure was correct. Working individually was difficult in these aspects, but it was easier as a learning experience and knowing exactly what was done in the procedure.