Preparation and isolation of an ester essay sample

Food & Diet, Alcohol

Background Theory:

Esters are derived from carboxylic acids and alcohols with the presence of a catalyst. A carboxylic acid contains the -COOH group, and in an ester the hydrogen in this group is replaced by a hydrocarbon group of some kind. Sulfuric acid (H2SO4)is used as a catalyst for this reaction in order to accelerate the rate at which the product is formed. The general formula of an ester is RCOOR’ in which R is the alkyl group that comes from the carboxylic acid while R’ is the alkyl group that comes from the alcohol and COO is the functional group of the ester known simply as the ester group. The general structural formula of an ester is as follows,

The general equation for esterification is as follows,

RCOOH + R’OH ⇌ RCOOR’ + H2O

This type of reaction is known as condensation reaction because water is produced. Water is produced from the hydroxyl group of the carboxylic acid and the hydrogen atom from the hydroxyl group of the alcohol as can be seen in the following formula. R’-O

-H

+

HO-

OC-R

H+

→

R’-OOC-R (ester)

+

H2O

Since a catalyst is not consumed during the course of a reaction, you need to use only a small amount of sulfuric acid in order for it to be effective. The name of the ester formed is butyl ethanoate. This is because the ‘-anol’ part of the alcohol name becomes ‘-yl’ and the carboxylic acid part which has the name of ‘-ic acid’ will end with a ‘-ate’

1-butanol + ethanoic acid butyl ethanoate + water.

C4H9OH(aq) + CH3CO2H(aq) CH3CO2C4H9(aq) + H2O(l)

CH3CH2CH2CH2OH + CH2COOH ☾ CH3COOCH2CH2CH2CH3 + H2O

The preparation of an ester involves three stages which are synthesis of the ester, isolation of the ester and lastly purification of the ester.

The first process is synthesis of the ester. This is done by reflux. Reflux is a process of heating the chemical reaction for a specific amount of time, while continually cooling the vapour produced back into liquid form, using a condenser. The vapours produced above the reaction continually undergo condensation with the aid of a liebig condenser, returning to the flask as a condensate. Concentrated H2SO4 was added as a catalyst to lower the activation energy and thus increasing the rate of reaction.

The isolation process involves the washing of the produced ester after the reflux. This is done by adding distilled water two times and sodium carbonate solution, Na2CO3 and anhydrous calcium chloride, CaCl. Distilled

water is added to remove most of the water soluble substances such as alcohol, carboxylic acid and sulfuric acid. The ester is not water soluble and this causes it to separate into two layers, one that is cloudier than the other being the ester. The traces of acid is removed in the ester by adding sodium carbonate solution. The equation of the reaction is as follows

$CO_3^{2-}(aq) + 2H^+(aq) \rightarrow CO_2(g) + H_2O(l)$

Distilled water is added for a last time after to remove any left behind salts and finally, the anhydrous calcium chloride is added to remove excess water.

The purification of the ester is the final stage where the ester is purified through a distillation process. The impure ester formed after the washing process is the decanted into a round bottomed flask to be distilled. Different conical flask are used to collect the different distillates formed based on the boiling ranges provided to us.

Reagent:

– 1-butanol, $CH_3CH_2CH_2CH_2OH$ (l)
– Ethanoic Acid (acetic acid), CH_3COOH (l)
– Sodium Carbonate, Na_2CO_3
– Calcium Chloride (anhydrous), $CaCl_2$ (s)
– Concentrated Sulfuric Acid, H_2SO_4

Apparatus and Material:

– Distillation apparatus
– Reflux apparatus

– Separating funnel (250mL)

– 10mL, 50mL measuring cylinder

– 250mL conical flask

– 100mL beaker

– 0-3600C thermometer

– Boiling chips

Procedure:

(session 1)

1. About 30mL of the 1-butanol and about 30mL of the ethanoic acid was placed in the 250mL round-bottom flask. 2. 2mL of concentrated sulfuric acid and some boiling chips were added slowly in the round-bottom flask 3. The reflux apparatus was set up and the mixture was refluxed for about 30 minutes. The flame was kept to maintain a steady boil. 4. After completion of the reflux, the mixture was put into a container and covered with a stopper. 5. Observation for reflux were recorded.

(session 2)

6. The cool mixture was poured into a separating funnel which contained about 60mL of distilled water. 7. The mixture was allowed to undergo the process of stopper, shake and allowed the layers to separate. The aqueous layer formed was discarded. 8. The washing process of the organic layer in the separating funnel was repeated, this time with about 60mL of sodium carbonate solution. The aqueous layer formed was discarded. 9. The washing process with distilled water was repeated. The aqueous layer formed was discarded. 10. The organic product was discarded to a conical flask and

some anhydrous calcium chloride was added. The mixture was left to swirl and allowed to stand for 10-15 minutes. 11. The organic liquid was poured into a clean, dry 250mL round bottom flask. Some boiling chips were added. 12. The distillation process was set up and the ester was distilled. 13. The observation during isolation with water and with sodium carbonate solution were recorded. 14. The odour, appearance and the boiling range of the fraction collected was recorded.

Safety Precautions

The first safety precaution that was taken was that our lab coats were worn and completely buttoned up at all times to ensure no harmful substance comes in contact with our skin . Our lab goggles were also worn at all times to prevent any harmful substances from splashing into our eyes. Covered shoes were worn to ensure that substances would not harm our feet if an apparatus was leaking or if someone dropped a harmful substance. Appropriate and comfortable clothing was worn on the day of the experiment. Washing process for all the apparatus were not necessary as these are organic compounds that we are dealing with. In order not to spill the mixtures each time they were transferred, a filter funnel was used. The filter funnel was used so that the mixture that had undergone reflux in session 1 would be transferred without spillage into the reagent bottle, when we transferred the 60mL of water and sodium carbonate solution to the separating funnel and finally after the washing process, from the separating funnel to the conical flask.

During the washing process with sodium carbonate solution, carbon dioxide gas is formed and the stop cork at the tip had to be opened to allow the carbon dioxide to escape out. This would prevent the pressure build up in the separating funnel causing the lid to burst open or the separating funnel to break open which will lead to the mixture pour out of the separating funnel. During the opening of the stop cork to release the carbon dioxide, the nozzle of the separating was faced away because the gas that comes out could be harmful if comes in direct contact with the face. In order to ensure an airtight fit into all the reflux and distillation apparatus, petroleum jelly (Vaseline) was used. Vaseline is highly flammable and only a bit of petroleum jelly was put because the excess petroleum jelly could seep down and touch the flame coming out of the bunsen burner and catch fire.

The 1-Butanol was handled with care because it is highly flammable which means that it was kept away from any flame at all times except for when the reflux process was going on with the presence of the Bunsen burner to heat up the mixture to undergo reflux. 1-Butanol is an alcohol that will cause skin irritation, eye irritation and very harmful is ingested or inhaled. Hence, precautions were made to not inhale, ingest and to avoid skin contact as it could cause serious problems. Acetic acid is also a flammable chemical which means it can explode easily and therefore it is kept away from any source of direct flame. The acetic acid was poured carefully so that the acid would not come in contact with our hands causing irritation or mild burns to the skin.

When handling the concentrated sulfuric acid, it was kept in a fume chamber to limit the hazardous exposure, as it was concentrated. A dropper was used to transfer the concentrated sulfuric acid instead of directly pouring the acid into the measuring cylinder. This ensures that no concentrated sulfuric acid spills out and it doesn't come in contact with our hands which will cause corrosion to our skin. The 1-Butanol, Acetic acid and the Sulfuric acid were all mixed together in a fume chamber so that no harmful gas was produced that would be inhaled by us. Lastly, when the ester was formed and it was time to smell the ester, the ester was fanned towards us rather than smelling it directly from the conical flask. This is because the smell produced by ester could be harmful to our respiratory tract.

Observations:

Soot is produced at the bottom of the round bottomed flask if the flame from the Bunsen burner is orange in colour A cloudy layer was formed and an clear layer was formed during the washing of the ester with distilled water. During the washing with sodium carbonate, , Na2CO3, a colourless gas was released Thermometer remains constant at 90 and 120 degree celcius.

The distillate is cloudy and smells like alcohol at 90 degree celcius. The distillate at 120 degree celcius has a sweet and fruity smell. Both the distillates are liquid at room temperature.

Results :

Boiling ranges (oC)

Observations

Sight

Smell

80 – 90

Cloudy

Pungent

120 – 130

Clear

Sweet and fruity

Discussion:

The reaction equation is $CH_3CH_2CH_2CH_2OH + CH_2COOH \rightleftharpoons CH_3COOCH_2CH_2CH_2CH_3 + H_2O$ which is 1-butanol reacting with acetic acid to form 1-butyl ethanoate and water. The reflux process is a process whereby the reactants must be heated for an extended period of time. This is a problem because the liquids are volatile and will evaporate and this will mean that not much product will form. Safety will also be a problem. By heating under reflux conditions the vapours condense on the sides of the water-cooled condenser so that the liquids fall back into the round bottomed flask so that no reactants or products are lost during the heating process. The steps used in recovering the ester is called the isolation process. This includes washing the ester in several different ways.

The first washing process is done with water. Water is used first because at the end of the heating period, all reactants and products are present in the mixture. This is because the system reaches a state of chemical equilibrium and the concentrations of all species remain unchanged. Washing with water

will remove most of the water-soluble substances. The aqueous layer was identified by adding some distilled water and seeing which layer would separate. This layer was removed and the ester layer left in the separating funnel. The following washing process is with sodium carbonate solution (Na2CO3). This is done because after washing the ester with water, there will be traces of acid left in the separating funnel. The sodium carbonate solution will remove the acid traces by reacting with it. The equation is as follows:

2H+ + CO32- 👉 CO2 + H2O.

The last washing process involves washing with distilled water once again. This removes any traces of water soluble materials from the separating funnel. After the washing process is complete, Calcium chloride (CaCl2) is added. Calcium chloride is a dehydrating agent and will remove any traces of water trapped in the ester layer. Too much of water was present that a new a new layer of water was formed. Finally, the ester underwent distillation process. The distillation process allows the separation of liquids that have different boiling points as each liquid have their own boiling points. The ester has the highest boiling point and the final product was collected the last. The yield that was produced was low because the reaction reaches equilibrium so that 100 percent of product yield cannot be obtained. This means that the concentration of all species, reactants and products will remain at constant and not change. Furthermore, another reason why the yield was low was because the reacting was not refluxed for long enough period of time. There was a time limit of half an hour for reflux. Lastly, the chances of loosing a bit of the ester during washing is very high.

Some of the ester had been let to flow out through the stop cork of the separating funnel with the aqueous layer formed. Some of the modifications to increase the purity of the ester is by using a larger liebig condenser during the distillation process. This would help in the condensation process giving out a higher yield of ester. Secondly, increasing the concentration of one of the reactants for example the 1-Butanol or the Acetic acid. This would help because the excess of reactant will increase the production of the ester. Lastly, the final way to increase the yield is by keeping the washing process at the least amount of time. This is because each time the mixture is washed, abit of the ester is drawn out from the mixture as well. To create an ester with less impurities, the preparation could have been carried out more carefully to get a better boiling point and a clearer ester.

This can be done by using three conical flask at the end when collecting the ester. The first conical flask is to collect the impurities between temperature 0-120 degree celcius, the second conical flask for impurities just before the ester comes out and the final conical flask for the main ester itself. Overall the Experiment was a success. My lab partner and I discussed the many ways in which to work most efficiently. This included my lab partner setting up the apparatus of distillation and reflux while I prepare the mixture and do the washing process. During the first fifteen minutes of the reflux, my partner was watching the process making sure it was fine while I was recording any observation. The second fifteen minutes of reflux was watched by me and my partner was taking down the necessary observations.

The second part of the experiment which was the distillation process, I would have been watching out for the thermometer and calling out the temperatures while my lab partner was the one changing the conical flask depending on the temperature callout. Both of us worked very well together and we kept the area clean at all times. The final cleansing of apparatus and materials were shared amongst us in a way where after I finish cleaning the apparatus and material, my partner would go place them back in the rightful position. We were both very happy to get a clear ester with a sweet and fruity smell.

Conclusion:

The ester that was produced and purified from this experiment was butyl ethanoate. It was produced from 1-butanol and ethanoic acid also known as acetic acid. This is proven as the second fraction of the distillate that was suspected to be ester exhibited all of the properties of the ester that has been produced such as having a sweet and fruity smell as well as being a clear liquid. The boiling range at which the distillate was obtained also confirmed that it was an ester. Therefore, the ester, butyl ethanoate, has successfully been isolated and purified