Fractional distillation process to separate organic liquids

Distillation is a very useful method of purifiying liquids. Simple distillation is used when a pure solvent is required to be obtained from the solution. This is usually water. On the other hand, fractional distilation is basically used for the separation of a mixture of two miscible organic liquids having different boiling points. A common example of two liquids that mix with each other are ethanol and water. Another example is petrol and paraffin.

In this experiment, a mixture of acetone and toluene was provided. Basically, the liquid mixture was boiled to evaporate the liquid that had the lowest boiling point, referred to as the first fraction. The vapour passed up through a fractionating column, which is not used in a simple distillation. As the mixture vaopur passed up the fractionating column, it continually condensed and evaporated. This caused it to become increasingly richer in the liquid with the lowest boiling point until the vapour that reached the top consisted almost entirely of the component with the lowest boiling point. The vapour is then cooled in the condenser and so it condensed back to a liquid, which was collected, hence referred to as the distillate. When almost all the liquid with the lowest boiling point was distilled over, the temperature rised rapidly showing that a mixture of both liquids was distilling over. This should be collected in a separate container and discarded. Once the temperature reached the boiling point of the second liquid, the liquid was then distilled into another container.

This basically explained the process of fractional distillation. However, there is a theory behind all this, because the process of distillation should be related in reference to an ideal liquid mixture where one is more volatile than the other. Regarding the mixture of acetone/toluene provided in this

experiment, ideal behaviour was assumed and once the process was carried out, the more volatile liquid was found by finding the boiling point of each component. It was noted that the more volatile liquid was acetone since this had a lower boiling point. This was discussed further in relation to boiling point-composition graphs.

2. Method

2. 1 Chemicals used

Reagent:

Grade:

Manufacturer:

Acetone

GPR

BDH

Toluene

GPR

Merck

2, 4-dinitrophenylhydrazine

GPR

Riedel de Haem

Sodium hydroxide

GPR

Tinstar

Iodine

GPR

BDH

Dioxane

GPR

Aldrich

A mixture of acteone (BDH, GPR) and toluene (Merck, GPR).

2. 2 Apparatus

Fractionating column, thermometer, 100 mL round bottomed flask as the distillation pot, glass beads, anti-bumping granules, cotton wool, tight clip, Leibig condenser with rubber tubings, heating mantle, connecting side-arms as part of the fractional distillation setup, retort stand with clamp, water supply, 10 mL and 100 mL measuring cylinders, electronic balance, test-tubes, distilled water.

2. 3 Procedure

Part a) The separation of the acetone/toluene mixture and the measurement of the boiling points of each.

The apparatus for fractional distillation was set-up appropriately using a 100 mL round-bottomed flask, the fractionating column provided, insulated well with cotton wool.

50 mL of the acetone/toluene mixture was placed in the 100 mL round-bottomed flask. This was measured using a measuring cylinder.

A few boiling chips or anti-bumping granules, which were small irregularly pieces of material, were added to the round-bottomed flask in order to allow prolonged boiling.

The apparatus was clamped accordingly from the neck of the round-bottomed flask and checked to be balanced and well set-up before the heating mantle was switched on.

Then round-bottomed flask was heated slowly using a heating mantle, until the reading on the thermometer reached a steady state and drops were observed to condense out of the Leibig condenser. This was the boiling point of the first fraction. This steady state temperature was recorded and the distillate was collected in a 100 mL measuring cylinder.

The distillation was allowed to proceed until no more liquid got out of the condenser into the measuring cylinder. Then the volume of the first fraction was recorded.

When all of the first fraction was distilled out, the temperature at the top of the column was observed to increase and then reached a second steady state, which was the boiling point of the second fraction. Drops of the second fraction were observed to start to condense out of the Leibig condenser. This

steady state temperature which was the boiling point of the second fraction, was recorded.

The second distillate was collected in a clean measuring cylinder and then its volume was recorded.

Part b) The usage of two different tests for the identification of the liquid distillate having a carbonyl group. (acetone)

2, 4-DNPH test was first carried out. 2-3 drops of the liquid to be tested were added to 3 mL of 2, 4-dinitrophenylhydrazine, and shaken. Any observations and inferences were recorded.

The iodofrom test was then carried out. 4 micro drops of the liquid to be tested were dissolved in 2 mL distilled water, in a test-tube. The drops were added carefully using a pipette. 2 mL of 10 % sodium hydroxide were then added together with 2 mL of iodine solution, which were added slowly by drops. The substance was insoluble in water and therefore 2 mL dioxane were added. This was done so that the substance dissolved. Any observations and inferences were then recorded.

Each test was carried out twice, for each distillate.

Diagram:

The set-up apparatus for Fractional Distillation.

Precautions

It was ensured that the thermometre was positioned accordingly at the mouth opening of the Leibig condenser, where it indicated which fraction

was being evaporated by noting the temperature readings. The thermometer position was very important because if the thermometer bulb was to be placed too high, the vapours would reach it before they pass into the side-arm to be collected, and the observed boiling point would be lower than it should be. If the thermometer bulb was to be placed too low, vapours of impurities might reach it, and a high reading for the boiling point range would be given.

It was ensured that the fractionating column was filled and packed with glass beads, for maximum possible surface area for vapour to condense on.

It was ensured that a joint clip was used and attached between the end of the condenser and the side arm so that there was complete attachment of the setup together and any spillage of the liquid distillate was prevented, but allowed to drop only from the side arm tube, where the vent was present.

It was ensured that the Leibig condenser provided was set-up accordingly with opening below meant for water to be pumped in while the opening at the top meant for water to be pumped out, and vice-versa. Although water pressure transfers from a higher to a lower height, if the condenser had to be the other way round the liquid might not be cooled completely as it would only condense the top portion of the condenser. Therefore if the rest of the part of the Leibig condenser was not cooled, the liquid would might evaporate into gas again at the bottom part of the condenser. This explained the importance of correct set-up.

It was ensured that as much of the second fraction as possible was collected, however at the same time care was taken so as not to allow the distillation

pot, i. e. the 100 mL round-bottomed flask, to boil dry otherwise the residues might ignite or expode.

It was ensured that anti-bumping granules were used. These were placed in the 100 mL round-bottomed flask with the 50 mL of the acetone/toluene mixture. The granules were important since they allowed prolonged, smoother boiling without bumping and continuous even formation and release of vapour bubbles were observed.

It was ensured that cotton wool was used so that the whole fractionating column was completely wrapped and covered for insulation or lagging. This was important so that the apparatus remained as warm as possible and excessive cooling was avoided, but occurred very slowly.

It was ensured that parallax errors were avoided as much as possible by looking normally to the scale of the measuring cylinder were when taking readings of the volumes of liquids, or when taking temperature readings from the thermometre.

For safety measures, it was ensured that care was taken when distilling organic solvents in order to avoid explosions and fires. Hence, it was ensured that the vapour did not come into contact with flames, sources of sparks or very hot surfaces such as hot plates.

It was ensured that the apparatus was not completely sealed. A vent in the system was required so as to prevent pressure build up in the system as heating was carried out. Otherwise the apparatus would simply blow apart.

Therefore, for safey measures, it was ensured that heating in a closed system was avoided.

3. Results

Volume of acetone and toluene mixture used was: 50. 0 mL

Observations & Inferences from the 2, 4-DNPH test

A red-orange precipitate was formed. This positive result means that a carbonyl group, was present in the formula of the substance.

No precipitate was formed. The substance contained no carbonyl group, in its formula, hence a negative result was obtained.

Observations &Inferences from the Iodoform test

A pale yellow precipitate was formed. This means that the substance contains a

in its formula, hence it gave a positive iodoform with an antiseptic smell.

No precipitate was formed. No group was present in the formula of the substance, hence a negative result was obtained.

Suspected Identity of Fraction

4. Discussion:

At any given temperature a pure liquid in a close container will establish an equilibrium with its vapour:

liquidvapour

The equilibrium vapour pressure above the liquid will depend upon the temperature. Considering mixing two liquids in different proportions, Raoult’s Law states that for an ideal mixture at a fixed temperature, the vapour pressure of each component is proportional to its mole fraction. This means that the graph of the vapour pressure of each component against its mole fraction will be a straight line passing through the origin.

Considering the case of two volatile liquids, as in this experiment, each of which contributes to the total vapour pressure, by Dalton’s Law of partial pressures it is known that the total vapour pressure of the mixture is the sum of that of the components and this will also give a straight line when plotted against molar composition. Therefore it could be said that it is more convenient to plot boiling point of the mixture against molar composition.

The boiling point of a liquid is the temperature as which its vapour pressure reaches the external atmospheric pressure. Since the less volatile component will have the highest boiling point, being toluene in this case, the vapour pressure curves in figure 1 lead to boiling point-composition graph as shown in figure 2. Two curves were drawn in this diagram since the liquid mixture and the vapour in equilibrium with it do not have the same composition. The vapour will always contain a higher proportion of the more volatile (lower boiling point) component.

This difference in composition between the liquid and vapour phases in equilibrium enables such a liiquid mixture to be separated by disitllation.

To separate a liquid mixture which obeys Raoult’s Law, one must repeatedly distill, i. e. boil the liquid and condense the vapour. This is fractional

distillation, and rather than carrying out each stage separately, it is convenient to use a fractionating column designed to allow many such steps to occur simultaneously.

Fractional distillation is widely used in industrial chemistry for separating mixtures of miscible liquids which boil at different temperatures. For example pure oxygen, nitrogen and noble gases may be obtained from liquid air by fractional distillation. Another example is the hydrocarbons in crude oil which can be separated into useful fractions.

In order to discuss the results obtained, one should say that these were clearly explained in the table of results tabulated. It was observed that the boiling points measured were very reliable since these were checked using the organic compounds database website, maintanied by Colby College and the components of the mixture provided i. e acetone and toluene were listed. One should say that there were many readings, as can be observed from the table of results, where the temperature remained constant as more drops of distillate were collected. This confirmed the boiling point of the organic liquid.

Some modifications could be made for the procedure of the fractional distillation process. For instance, rather than using a fractionating column packed with glass beads to give the maximum possible surface area for vapour to condense on, a fractionating column with spikes of glass sticking out from the sides could be used, and this would serve the same purpose.

Sources of error

Once the temperature on the thermometre was observed to rise rapidly, this meant that a mixture of both organic liquids were distilling over. Hence, these few drops should have been collected in a separate container and discarded since they did not consist of an individual liquid. However, this was not done. Hence the volume of the first fraction (acetone) collected was slightly greater than it was supposed to be and was not of the pure organic liquid but had some drops of the second fraction (toluene) in it. this is the container was replaced once the temperature was raised.

The thermometer was repeatedly being moved up and down when the temperature readings were taken. This is because some of the scale was hidden by the set-up itself. This movement of the thermometre might affected the results slightly because the boiling temperature range obtained might varied a bit due to this action.

5. Conclusion:

It was concluded that fractional distillation was carried out and therefore separation of two organic liquids acetone/toluene mixture occurred. It was also concluded that the boiling point of each component was found and tests on each liquid component, once distillated and separated, were carried out in order to prove the identity of the component.