Experiment 03 (16-10-09)

Determination of the Partition Coefficient of Ethanoic Acid between Water and Butan-1-ol

Objective

To determine the partition coefficient of ethanoic acid between water and butan-1-ol by titration.

Theory

When a solute(X) is added into two immiscible liquids A and B, it can dissolve in both liquids with different solubilities. After shaking, solutions of X in A and X in B can be obtained. X is said to distribute between immiscible liquids A and B.

This phenomenon is called the distribution or partition of the solute in two immiscible liquids. The two liquid layers can then be separated by a separating funnel.

At constant temperature, the concentrations of solute X in these two liquids are governed by the distribution or partition law:

K_d = Concentration of solute in solvent A / Concentration of solute in solvent B,

where K_d is the distribution coefficient or partition coefficient.

Procedures

1. Room temperature was recorded.

2. 25 cm^3 of 0.2000 M aqueous Ethanoic acid and 25 cm^3 of butan-1-ol was poured into a 250 cm^3 separating funnel by using a 100 cm^3 measuring cylinder.

3. The funnel was stoppered and shook vigorously for 1 to 2 minutes. The tap was opened occasionally to release pressure in the funnel.

4. 20 cm^3 of each layer was separated approximately into two beakers respectively. The fraction near the junction of the two layers was discarded.

5. 10 cm^3 of the aqueous layer was pipetted into a conical flask and it was then titrated with 0.1000 M sodium hydroxide solution using phenolphthalein.

6. 10 cm^3 of the alcohol layer was pipetted into a conical flask using another pipette. it was then titrated with 0.1000 M sodium hydroxide solution using phenolphthalein. Some water was added during titration to extract the Ethanoic acid from butan-1-ol layer to react with sodium hudroxide.

7. Procedures (2) – (6) were repeated with another separating funnel using 35 cm^3 of aqueous Ethanoic acid and 25 cm^3 of butan-1-ol.

Results

Room temperature : 24.7℃

Volume of butan-1-ol used : 25 cm^3

Volume of 0.2000 M Ethanoic acid / cm^3	Volume of 0.1000 M NaOH required for the aqueous layer / cm^3	Volume of 0.1000 M NaOH required for the alcohol layer / cm^3
25.00	9.10	11.20
35.00	10.50	13.20

Calculations

Volume of 0.2000 M Ethanoic acid / cm^3	Partition coefficient K_d
25.00	$\frac{9.10}{11.20} = 0.8125$
35.00	$\frac{10.50}{13.20} = 0.7955$

Average K_d = $\frac{0.8125 + 0.7955}{2}$ = 0.8040

Conclusion

The average partition coefficient of ethanoic acid between water and butan-1-ol is 0.8040.

Discussion

Sources of errors

- Temperature has changed during the experiment. Although the experiment is carried out in an air-conditioned laboratory, there are still some minor things which affect the temperature of the solution mixture. The shaking process of the separating funnel will increase the kinetic energy of

the solutions' molecules and thus will increases the temperature. Also, when touching the funnel, warmth of human palms may increase the temperature of the solutions as well. As the partition coefficient is temperature dependent, increasing the temperature will increase the partition coefficient.

- Butan-1-ol and water are not completely immiscible. Butan-1-ol is slightly soluble in water.

- Ethanoic acid dissociates in water. Ethanoic acid ionizes to some extent in water, so the concentration of free ethanoic acid in water is lowered, leaving less ethanoic acid molecules in butan-1-ol.

Suggestion for the improvement

- Avoid holding the separating funnel with palms which greatly affects the partition coefficient.

- Two totally immiscible solvents should be used instead, e.g. water and ether.

- It's hard to prevent the dissociation of ethanoic acid in water. But this error can be minimized by shaking the separating funnel again to force some ethanoic acid molecules back to butan-1-ol before running the solution to the beaker.

Question

1. Shaking can increase the surface area for ethanoic acid to contact with both water and butan-1-ol. Also, by shaking, equilibrium state can be attained faster.

2. As butan-1-ol evaporates, pressure is built up in the separating funnel. The tap should be opened occasionally to release pressure built up inside to avoid the breakage of the funnel.

3. Because butan-1-ol is slightly soluble in water.

4. The purpose of adding water and butan-1-ol is to leave the mixture to equilibrium and provide enough solvent for the titration. Also, volumes of water and butan-1-ol used are in the calculation of partition coefficient. Therefore the amounts of aqueous ethanoic acid and 2-methypropan-1-ol need not be measured out accurately.
   However, as volumes of the aqueous and alcohol solution determine the partition coefficient, volumes of these solutions must be accurate.

5. The partition coefficient calculated is not accurate as assumptions made in this experiment are invalid.(See question 7)

6. Yes, partition coefficient is temperature dependent, i.e., ln K_d = constant - $\frac{\Delta H}{RT}$.

   Increasing the temperature will increase the partition coefficient.

7. A few assumptions are made during the experiment:

   a) Temperature is constant. This assumption is not valid as temperature of the solutions may change when shaking the separating funnel. Shaking can increase the kinetic energy of the solutions and thus increases the temperature. Also, when touching the funnel, warmth of human palms may increase the temperature of the solutions as well.

   b) The two solvents are immiscible. This assumption is also invalid as butan-1-ol, being an alcohol, is slightly soluble in water.

   c) The solute, ethanoic acid has no dissociation or association in both solvents. This assumption is invalid as ethanoic acid dissolved in water ionizes to some extent in water and it also dimerizes to some extent in butan-1-ol.

8. Solvent extraction is more efficient if the extraction solvent is added in small portions several times instead of all at once.

   <u>One portion of 50 cm^3 butan-1-ol:</u>

   No. of moles of ethanoic acid originally = 25 x 10^{-3} x 0.2000 = 5 x 10^{-3} mol

   Let x be the no. of moles of ethanoic acid extracted in butan-1-ol:

$$\frac{\frac{5\times10^{-3}-x}{0.025}}{\frac{x}{0.05}} = 0.8040$$

   x = 3.57 x 10^{-3} mol

   <u>Two portions of 25 cm^3 butan-1-ol:</u>

   No. of moles of ethanoic acid originally = 25 x 10^{-3} x 0.2000 = 5 x 10^{-3} mol

   Let y be the no. of moles of ethanoic acid extracted in butan-1-ol in the first 25 cm^3 butan-1-ol:

$$\frac{\frac{5\times10^{-3}-y}{0.025}}{\frac{y}{0.025}} = 0.8040$$

   y = 2.77 x 10^{-3} mol

Let z be the no. of moles of ethanoic acid extracted in butan-1-ol in the second 25 cm^3 butan-1-ol:

$$\frac{\dfrac{(5\times10^{-3}-2.77\times10^{-3})-z}{0.025}}{\dfrac{z}{0.025}} = 0.8040$$

z = 1.24 x 10^{-3} mol

∴Total no. of moles extracted = 2.77 x 10^{-3} + 1.24 x 10^{-3} = 4.01 x 10^{-3} mol,

which is more than that extracted in one portion of 50 cm^3 butan-1-ol.

9. Paper chromatography and solvent extraction.