Experiment 13

Aim:

To determine the equilibrium constant for esterification from ethanoic acid and propan-1-ol.

Procedure:

1. 0.25 mole (equals to 14.3 cm^3) of glacial ethanoic acid (density = 1.05 g cm^{-3}) and 0.25 mole (equals to 18.8 cm^3) of propan-1-ol (density = 0.8 g cm^{-3}) was put into a clean, dry pear-shaped flask. It was mixed thoroughly.
2. 1.0 cm^3 of the mixture was transferred by pipette to a 250 cm^3 conical flask containing about 25cm^3 deionized water and 2 drops of phenolphthalein indicator. It was titrated to end point with 0.50 M sodium hydroxide solution. The titre (V_1 cm^3)was recorded.
3. 8 drops of concentrated sulphuric(VI) acid was added to the remainder of the acid-alcohol solutions while continuously swirling the flask. Another 1.0 cm^3 sample was titrated immediately. The titre (V_2 cm^3)was recorded. The difference between V_1 and V_2 Represents the volume to be subtracted from subsequent titration to correct for the amount of sulphuric (VI) acid present.
4. A few anti-bumping granules were added to the flask, and it was attached to a water-cooled reflux condenser. It was refluxed for 1 hour. The flask and its contents in an ice bath was cooled. 1.0 cm^3 sample was removed from the flask for titration with the 0.50 M sodium hydroxide solution as before. The titre needed was recorded (V_3)and was corrected for the sulphuric(VI) acid.
5. Refluxing was continued for an additional half hour, and was then cooled and was titrated another 1.0 cm^3 sample. The two titres should agreed to within 0.2 cm^3. Otherwise, this step was repeated.

Some changes:

1. After adding 8 drops of concentrated sulphuric(VI) acid, titration should be started immediately. But this procedure was missed, V_2 (which should be greater than V_1) was not accurate. The volume to be subtracted from subsequent titration to correct for the amount of sulphuric (VI) acid present was then found by adding 8 drops of sulphuric (VI) acid to a beaker of water of volume same as before. This procedure was done by Joe. The volume is 0.95 cm^3.
2. As the time is not enough, step 5 was not performed.

Result

	V_1 (cm^3)	V_2 (cm^3)	V_3 (cm^3)
Initial	1.3	17.6	3.4
Final	17.6	32.6	9.5
Net	16.3	15.6	6.1

Calculation

1. A small amount of concentrated sulphuric(VI) acid was added to the reaction mixture at the beginning of the experiment in order to act as a catalyst to speed up the reaction.
2. During refluxing, the mixture may become gases bubbles. Anti-bumping granules were added to the reaction mixture before refluxing in order to prevent the splitting out of the reaction mixture.
3. The refluxing was continued in step 5 until the titre of sodium hydroxide used approaching constant in order to make sure that the reaction had reached equilibrium stage.
4. The equation for the esterification reaction between ethanoic acid and propan-1-ol is:

   $$CH_3COOH_{(aq)} + CH_3CH_2CH_2OH_{(aq)} \overset{H_2SO_4(l)}{\Leftrightarrow} CH_3COOCH_2CH_2CH_{3(aq)} + H_2O_{(l)}$$

5. The equation of the reaction between ethanoic acid and sodium hydroxide is:

   $CH_3COOH_{(aq)} + NaOH_{(aq)} \rightarrow CH_3COONa_{(aq)} + H_2O_{(l)}$

   The volume of NaOH needed for the neutralization with CH_3COOH in 1 cm^3 sample after refluxing is:

   6.1 - 0.95 = 5.15 cm^3

   mole of CH_3COOH ： mole of NaOH = 1 ： 1

   mole of NaOH used is:

   $0.5M \times 5.15cm^3 \times 10^{-3}$

   = 0.002575 mole

   mole of CH_3COOH is:

   = 0.002575 mole

   Therefore, the concentration of ethanoic acid remaining at the end of the reflux is:

   = 0.002575 mole per 1 cm^3

   =2.575 M
6. Concentration of CH_3COOH ： Concentration of $CH_3CH_2CH_2OH$

   =1 ： 1

   Concentration of $CH_3CH_2CH_2OH$ at the end of the reflux is = 2.575 M

Concentration of CH_3COOH at the beginning (after procedure 1) is:

$= 16.3\ cm^3 \times 10^{-3} \times 0.5M / 10^{-3}\ cm^3$

$= 8.15\ M$

Concentration of $CH_3COOCH_2CH_2CH_3$ after refluxing is:

$= 8.15\ M - 2.575\ M$

$= 5.575M$

Concentration of water after refluxing is:

$= 5.575M$

7. $K_C = ([CH_3COOCH_2CH_2CH_3] \times [H_2O])/ ([CH_3COOH] \times [CH_3CH_2CH_2OH])$
8. $K_C = 5.575 \times 5.575 / (2.575 \times 2.575) = 4.687\ mole^{-1}\ dm^3$
9. If the concentration of the sodium hydroxide solution is not known exactly, there will be no effect on the determination of the equilibrium constant for the esterification reaction. The initial concentration of CH_3COOH can be found {0.25 / (14.3 + 18.8) × 1000 = 7.55}. The concentration of NaOH can also be found { 7.55 / (1/1000) / (16.3/1000) = 0.46M ≈ 0.5M}

Conclusion

The equilibrium constant for the reaction (esterification) between ethanoic acid and propan-1-ol is $4.687\ mole^{-1}\ dm^3$

Discussion

1. It is assumed that the volume of 8 drops of concentrated sulphuric(VI) acid is very small which could not greatly affected the volume of the original mixture.
2. It is assumed that the volume of 8 drops of concentrated sulphuric(VI) acid added by Joe is same as that of me.
3. It is assumed that the rate of reaction was slow that when the reaction mixture is cooled down by ice water, K_C did not change.
4. After adding of 8 drops of concentrated sulphuric(VI) acid, V_2 should be found out immediately. Otherwise, the reaction may have started and the result became incorrect.