Experiment K2

Investigation of the reaction of iodine with propanone (Titrimetric method)

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Objective

To determine the orders of reaction of iodine and propanone respectively in the iodination of propanone.

Introduction

The iodination of propanone is a special reaction at which it is catalyzed by acid and its rate order can be determined by titrimetric method. Propanone is allowed to react with iodine in the presence of sulphuric acid. At regular time intervals, concentrations of iodine will be determined by titrametric method. Thus, the rate of decrease of iodine is known and the order of reaction can be calculated.

First, known volume of standard iodine solution is allowed to react with known volume of standard propanone solution. The process is called iodination. Sulphuric acid is added to catalyze the reaction. At the same instant, a stop watch is started.

$$CH_3COCH_3(aq) + I_2(aq) \rightarrow CH_3COCH_2I(aq) + H^+(aq) + I^-(aq)$$

After certain time, a known volume of reaction mixture is withdrawn and a known volume of sodium hydrogencarbonate solution is added to quench the reaction. The known volume of reaction mixture withdrawn will titrate against standard sodium thiosulphate solution, using starch as the indicator. Excess iodine in the reaction mixture will react with $S_2O_3^{2-}(aq)$ to give $I^-(aq)$

$$I_2(aq) + 2 S_2O_3^{2-}(aq) \rightarrow 2I^-(aq) + S_4O_6^{2-}(aq)$$
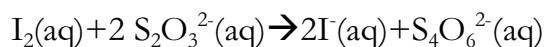
The quenching and titration steps will be repeated several times at regular time intervals. The rate equation of the reaction can be represented by

$$- \frac{d[I_2]}{dt} = k[CH_3COCH_3]^a[I_2]^b$$

Since the propanone used is in large excess, the rate equation can be

adjusted to

$$-\frac{d[I_2]}{dt} = k_1[I_2]^b, \text{ where } k_1=k[CH_3COCH_3]^a$$

The order of reaction with respect to iodine (b) can then be determined from the slope of the graph. By further plotting a rate against initial concentration of propanone graph, the order of reaction with respect to propanone can be found out as well.
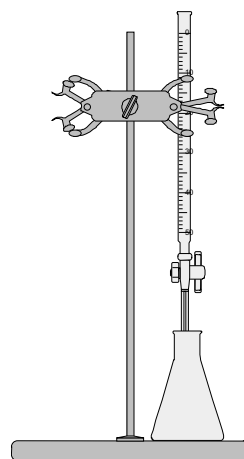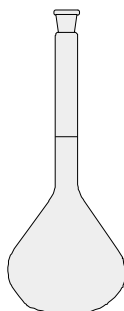
Apparatus used

A titration set up
Measuring cylinder
10 cm^3 pipette
25 cm^3 pipette
Stop watch
250 cm^3 volumetric flask

Chemicals used

0.02 M iodine solution
1 M propanone
0.1 M sodium thiosulphate solution
Starch solution
1 M sulphuric acid
0.5 M sodium hydrogencarbonate solution

Procedures

1. All apparatus was washed with deionized water.
2. 25 cm^3 of 0.1 M sodium thiosulphate solution was pipette to a 250cm^3 volumetric flask and water was added to make up the solution to the graduation mark.
3. 25 cm^3 of 1 M sulphuric was measured by a measuring cylinder.
4. 25 cm^3 of 1 M propanone solution was added into a conical flask containing 25 cm^3 of 1 M sulphuric acid.
5. 50 cm^3 of 0.02 M iodine solution was added to the propanone mixture. The stop watch was started at the same time. The contents were mixed thoroughly.
6. 10 cm^3 of the reaction mixture was pipette into another conical flask containing 10cm^3 of 0.5 M sodium hydrogencarbonate solution after 5 minutes.
7. The mixture was mixed thoroughly and titrated against the diluted 0.1 M sodium thiosulphate solution in which starch was used as the indicator.
8. Another 10 cm^3 of the reaction mixture was withdrawn from the conical flask and step 7 was carried out again after 10 minutes.
9. Steps 6 to 8 were carried out at 15th, 20th, 25th and 30th minute interval.
10. All the results were recorded.

Observation

-When the reaction mixture was withdrawn and added to the conical flask containing sodium hydrogencarbonate solution, colourless effervescence occurred.

-At the end point of the titration, the blue black solution turned colourless.

Data Analysis

Group A

Time when reaction mixture was added to $NaHCO_3$ (min)	5	10	15	20	25	30
Initial burette reading (cm³)	0	4.1	5.0	22.8	~~6.0~~	4.6
Final burette reading (cm³)	19.8	22.9	29.8	39.8	~~22.6~~	37.7
Volume of $Na_2S_2O_3$ used (cm³)	19.8	18.8	17.8	17.0	~~16.6~~	15.1

Since the value of volume of $Na_2S_2O_3$(aq)obtained at 25th minute does not follow the trend, it is neglected and considered to be experimental error. Thus, the graph of the volume of $Na_2S_2O_3$ (aq) needed to react with the remaining iodine against the time at which 10.0 cm³ samples of the reaction mixture were added to the $NaHCO_3$(aq) is obtained below.
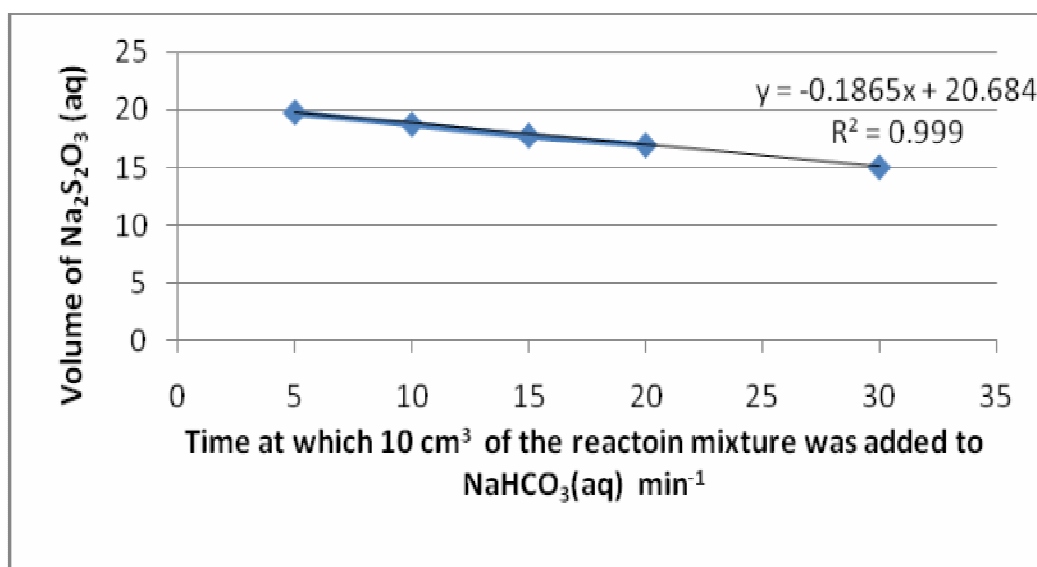
Group B

| Time when reaction mixture was added to NaHCO$_3$ (min)	5	10	15	20	25	30
Initial burette reading (cm^3)	9.5	~~9.4~~	9.5	9.6	9.4	9.5
Final burette reading (cm^3)	28.3	~~26.3~~	26.8	27.7	27.0	25.9
Volume of Na$_2$S$_2$O$_3$ used (cm^3)	18.8	~~16.9~~	17.3	17.8	17.6	16.4

Group C

Time when reaction mixture was added to NaHCO$_3$ (min)	5	10	15	20	25	30
Initial burette reading (cm^3)	8.0	1.7	2.0	7.0	2.7	2.2
Final burette reading (cm^3)	31.8	24.8	24.7	29.2	24.0	22.9
Volume of Na$_2$S$_2$O$_3$ used (cm^3)	23.8	23.1	22.7	22.2	21.3	20.7

Group D

Time when reaction mixture was added to NaHCO$_3$ (min)	5	10	15	20	25	30
Initial burette reading (cm^3)	1.4	1.6	2.0	21.4	1.7	2.6
Final burette reading (cm^3)	22.6	24.5	21.4	45.0	26.0	48.4
Volume of Na$_2$S$_2$O$_3$ used (cm^3)	21.2	22.9	19.4	23.6	24.3	22.4
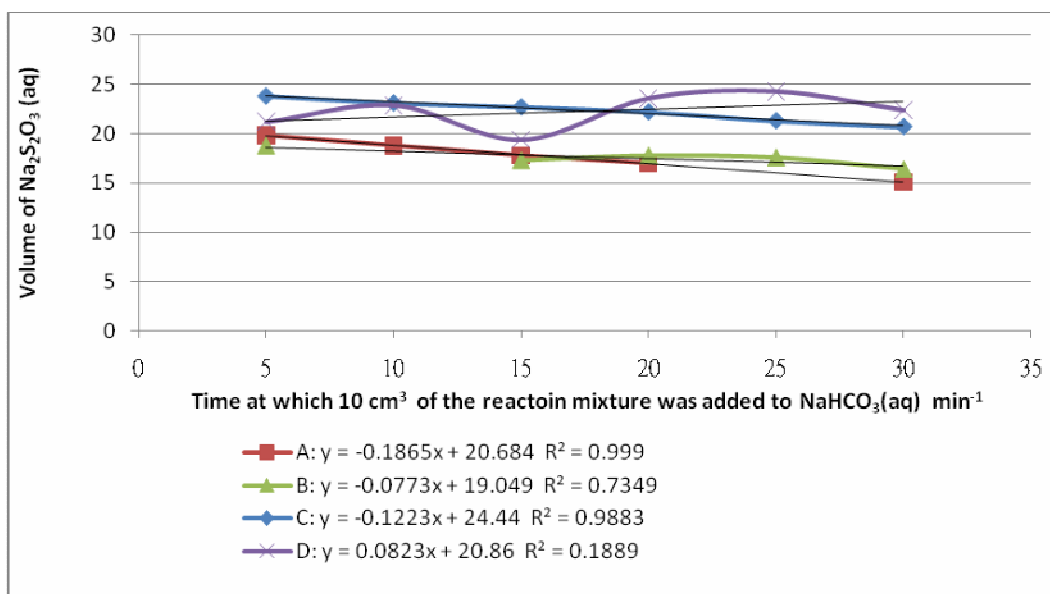
| Group	A(25 cm^3)	B(20 cm^3)	C(15 cm^3)	D(10 cm^3)
Gradient of graph (cm^{3}min^{-1})	-0.1865	~~-0.0773~~	-0.1223	0.0823

The data from group B is not to be considered.

Graph of gradients of graphs against initial volume of propanone solution added:
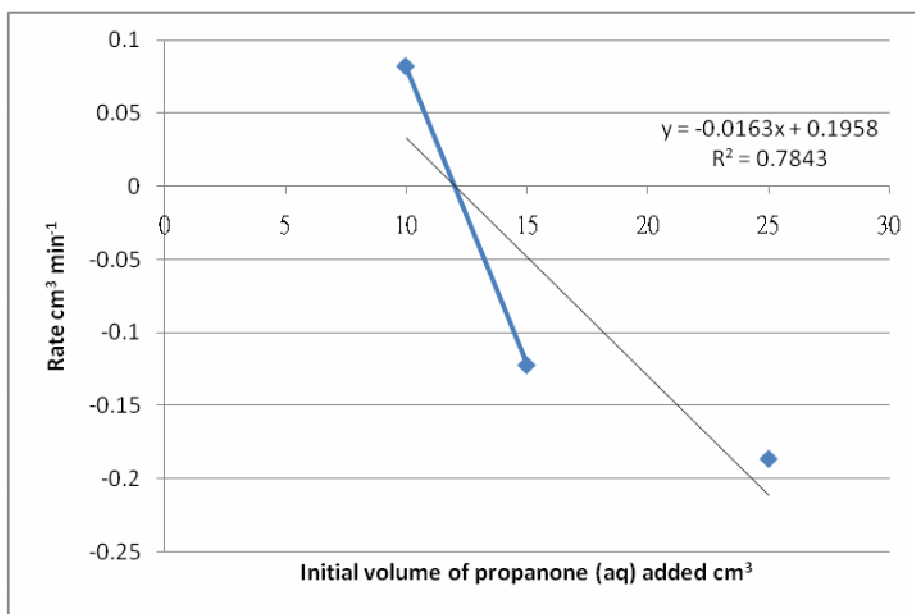
Discussion

In the experiment, sodium hydrogencarbonate solution was used as a quenching agent. It acted by neutralizing the catalyzing hydrogen ions from the acid in the reaction mixture to quench the reaction.

$$H^+(aq) + HCO_3^-(aq) \rightarrow H_2O(l) + CO_2(g)$$
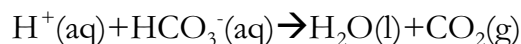
The presence of $CO_2(g)$ from the reaction can be proven by the occurrence of effervescence at the point which the reaction mixture was added to sodium hydrgencarbonate solution.

From the graph obtained by plotting the volume of $Na_2S_2O_3(aq)$ needed in the titration against the time at which 10 cm^3 of the reaction mixture were added to $NaHCO_3(aq)$, the iodine concentration fell throughout the reaction since the volume of $Na_2S_2O_3(aq)$ required decreased. The reaction between $Na_2S_2O_3(aq)$ and $I_2(aq)$ can be represented by the following equation

$$I_2(aq) + 2\ S_2O_3^{2-}(aq) \rightarrow 2\ I^-(aq) + S_4O_6^{2-}(aq)$$
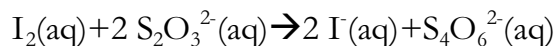
The ratio of stiochemtric coefficient between I_2 and $S_2O_3^{2-}$ is 1:2. Therefore, one mole of I_2 would react with 2 mole $S_2O_3^{2-}$. The concentration of I_2, i.e. $[I_2]$ can be calculated by molarity of $Na_2S_2O_3(aq)$ ×Volume of $Na_2S_2O_3(aq)$× 0.5. $[I_2]$ fell as the volume of $Na_2S_2O_3(aq)$ dropped. The rate of the reaction is then equal - $\dfrac{d[I_2]}{dt}$, i.e. the slope of the graph. The volume of $Na_2S_2O_3(aq)$ used decreased by almost the same amount at regular time intervals. Thus, we can conclude that $[I_2]$ decreased uniformly throughout the experiment as well, despite the decrease in $[I_2]$. The rate of change of $[I_2]$ is always the same irrespective to the change in $[I_2]$. Therefore, the rate of change of $[I_2]$ is independent of $[I_2]$. The order of the reaction with respect to iodine is zero, which means iodine does not take part in the rate determining step. The y-intercept of the graph helps to find the initial $[I_2]$ at the beginning of the reaction.

Since the order of reaction with respect to iodine is zero,

$$-\frac{d[I_2]}{dt} = k[CH_3COCH_3]^a$$

From the graph of rate against the initial volume of propanone solution added, we can determine the order of reaction with respect to propanone. When $[CH_3COCH_3]$ increased, the rate of reaction increased as well. The reaction rate is dependent of $[CH_3COCH_3]$. As the graph obtained is a straight line, the order of reaction with respect to propanone is one. The overall rate equation can then be represented by

$$Rate = k[CH_3COCH_3]$$

From the result of group D, a significant experimental error can be seen. This may due to slow quenching process. The reaction was not quenched exactly at the given time interval, thus the actual concentration of certain product or reactant at that time interval is different from the calculated value. An improvement can be made by withdrawing the reaction mixture 5-10 seconds in advance of the desire time such that the reaction is quenched at the desire instant but not later. This minimizes the error caused by delayed titration.