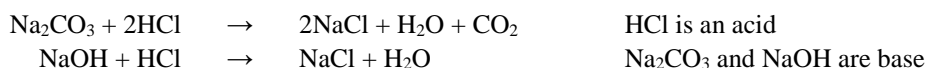


Inorganic Chemistry Experiments

1. Standardization of (approx. 0.1 N) sodium hydroxide solution by titration against a standardized solution of hydrochloric acid (approx. 0.1 N).

Introduction

According to Bronsted-Lowry acid-base theory, acid is the chemical species (either molecule or ion) which donates proton to other chemical species and base is the chemical species (either molecule or ion) which can accept proton from an acid.



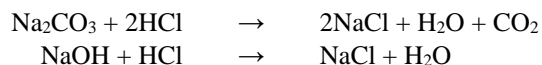
The chemical reaction involved in acid-base titration is known as neutralization reaction. It involves the combination of H_3O^+ ions with OH^- ions to form water. In acid-base titrations, solutions of alkali are titrated against standard acid solutions. The estimation of an alkali solution using a standard acid solution is called *acidimetry*. Similarly, the estimation of an acid solution using a standard alkali solution is called *alkalimetry*.

Principle

Hydrochloric acid is a secondary standard substance as it absorbs moisture from air and it cannot be obtained perfectly free from water. Firstly it is necessary to standardize it against a primary standard substance. Sodium carbonate can be used for this purpose. This experiment involves two successive steps.

- (i) Standardization of hydrochloric acid solution against a standard solution of sodium carbonate and
- (ii) Standardization of the supplied sodium hydroxide solution against the freshly standardized hydrochloric acid.

During titration equivalent amount of base reacts with equivalent amount of acid according to stoichiometric relation.



Equivalent amount of acid = Equivalent amount of base

$$V_1 \times \frac{\text{Equivalent amount of acid}}{V_1} = V_2 \times \frac{\text{Equivalent amount of base}}{V_2}$$

$$V_1 \times S_1 = V_2 \times S_2$$

If V_1 ml of S_1 N hydrochloric acid is required for the titration of V_2 ml sodium hydroxide solution, the concentration S_2 of sodium hydroxide solution can be calculated as per the above equation.

Experimental Procedure

Preparation of 100 mL 0.1 N sodium carbonate solution:

We know that, $\text{normality} = \frac{w \times 1000}{E \times V \text{ (ml)}}$

Here,

w = amount of Na_2CO_3 in g

E = gram-equivalent weight of Na_2CO_3 (53)

V = volume of the solution in ml (100 ml)

Titration

Check the apparatus for cleanliness. Wash the supplied apparatus successively with chromic acid, tap water and distilled water. Place the hydrochloric acid solution in a burette. Take 10 ml of sodium carbonate solution in a 250 ml conical flask and add 1-2 drops of methyl orange as indicator and observe the color. Take the initial burette reading and start adding HCl acid solution from burette drop wise with continuous shaking of the flask. Titrate the solution until the end point at which the pink solution turns into yellow color. Take and record the final burette reading. Repeat this at least three times and calculate the strength of HCl acid solution against Na_2CO_3 solution. Similarly titrate the supplied NaOH solution against the freshly standardized HCl acid solution keeping it in the same burette.

Experimental data collection table – 1: For the standardization of hydrochloric acid solution

No. of obs.	Volume of Na_2CO_3 solution, ml	Burette reading (volume of HCl solution), ml			Average or mean value (ml)	Conc. of std. solution (N)	Conc. of HCl soln. (N)
		IR	FR	Difference			
1							
2							
3							

Experimental data collection table – 2: For the standardization of sodium hydroxide solution

No. of obs.	Volume of NaOH solution, ml	Burette reading (volume of HCl solution), ml			Average or mean value (ml)	Conc. of std. solution (N)	Conc. of HCl soln. (N)
		IR	FR	Difference			
1							
2							
3							

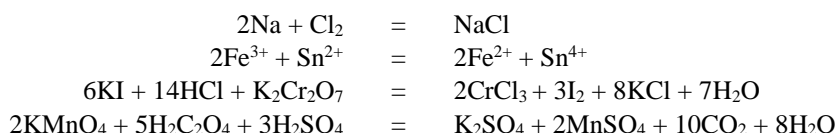
Result: The concentration of the supplied sodium hydroxide solution was found to be of N.

Precautions: (To be written by the students)

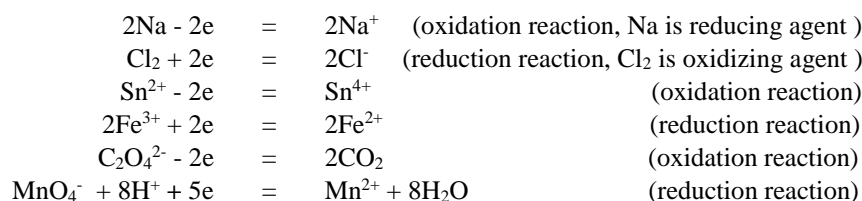
2. Standardization of approx. 0.1 N potassium permanganate solution by titration against a standard solution of oxalic acid (0.1 N).

Introduction

This is an oxidation reduction titration that means the chemical reaction taken place here is an oxidation-reduction reaction. Oxidation-reduction reaction is the chemical reaction in which electron is transferred from one species to another. An oxidation-reduction reaction can be divided into two half reactions. One is oxidation and another is reduction. The reaction in which electron is lost is called oxidation and the reaction in which electron is gained is called reduction. For example,

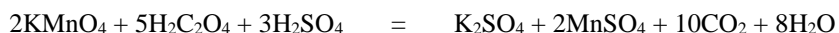


The chemical species which lose electron is called reducing agent and the chemical species which gain electron is called oxidizing agent.



Principle

In this experiment the strength of potassium permanganate solution is determined with the help of a standard oxalic acid solution. The reaction of this titration is as follows,



During titration equivalent amount of reducing agent reacts with equivalent amount oxidizing agent according to stoichiometric relation.

Equivalent amount of reducing agent = Equivalent amount of oxidizing agent

$$V_1 \times \frac{\text{Equivalent amount of reducing agent}}{V_1} = V_2 \times \frac{\text{Equivalent amount of oxidizing agent}}{V_2}$$

$$V_1 \times S_1 = V_2 \times S_2$$

If V_1 ml of S_1 N standard oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) solution is required for the titration of V_2 ml of supplied Potassium Permanganate solution, the concentration (S_2) of the Potassium Permanganate solution can be calculated as per the above equation.

Experimental Procedure

Preparation of 100 ml 0.1 N oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$) solution: Pure oxalic acid exists crystalline dihydrate form as $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. Its MW is 126 and equivalent weight is $126/2 = 63$.

We know that, $normality = \frac{w \times 1000}{E \times V (ml)}$

Here,

w = amount of $H_2C_2O_4 \cdot 2H_2O$ in g

E = equivalent weight of $H_2C_2O_4 \cdot 2H_2O$ (63)

V = volume of the solution in ml (100 ml)

Titration

Check the apparatus for cleanliness. Wash the supplied apparatus successively with chromic acid, tap water and distilled water. Place the supplied Potassium Permanganate solution in a clean burette. Take 10 ml of oxalic acid solution in a 250 ml conical flask with the help of a pipette and pipette filler bulb and acidify the solution with 10 ml of 1M sulfuric acid solution. Warm the solution to 50-60°C. Take and record the initial burette reading very carefully with two digit after the decimal point. Add 1-2 drops of Potassium Permanganate solution from the burette to the oxalic acid solution in the conical flask and the resultant solution becomes pink color. Shake well and allow to stand until the solution is colorless. Then continue titration until a faint pink color appears and persists at least 30 seconds. Read and record the final burette reading. Carry out two or three more readings for the confirmation of the end point until the readings agree with ± 0.05 ml from each other.

Experimental data collection table: For the standardization of $KMnO_4$ solution

No. of obs.	Volume of oxalic acid solution, ml	Burette reading (volume of $KMnO_4$ solution), ml			Average or mean value, ml	Conc. of std. solution (N)	Conc. of $KMnO_4$ soln. (N)
		IR	FR	Difference			
1							
2							
3							

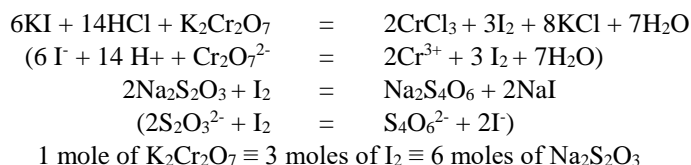
Result: The concentration of the supplied Potassium Permanganate ($KMnO_4$) solution was found to be of N.

Precautions: (To be written by the students).

3. Standardization of approx. 0.1 N sodium thiosulfate solution iodometrically against a standard solution of potassium dichromate (0.1 N).

Principle

In this experiment the strength of sodium thiosulphate solution is determined with the help of a standard potassium dichromate solution. This is an oxidation and reduction reaction, as well as “Iodometric reaction”. Titration involving with iodine and dealing with iodine liberated in chemical reaction is called Iodimetric and Iodometric titration respectively. This titration is iodometric because iodine is got from KI. The reactions of this experiment are:



Here, $\text{K}_2\text{Cr}_2\text{O}_7$ is an oxidizing agent and I^- is a reducing agent. Again, in the second reaction, I_2 is an oxidizing agent and $\text{S}_2\text{O}_3^{2-}$ is a reducing agent. In the 2nd step of the reaction a specific indicator, “Starch” is used which has a significant characteristic blue color on iodine.



If some starch solution is added to a solution containing a little iodine, the blue color of the starch-iodine complex will appear. If $\text{Na}_2\text{S}_2\text{O}_3$ solution is run in from the burette, the blue color of the starch-iodine complex will disappear from the solution as soon as all the iodine has been reduced to iodide ion.

During titration equivalent amount of reducing agent reacts with equivalent amount of oxidizing agent according to stoichiometric relation.

Equivalent amount of reducing agent = Equivalent amount of oxidizing agent

$$V_1 \times \frac{\text{Equivalent amount of reducing agent}}{V_1} = V_2 \times \frac{\text{Equivalent amount of oxidizing agent}}{V_2}$$

$$V_1 \times S_1 = V_2 \times S_2$$

If V_1 ml of S_1 N standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution is required for the titration of V_2 ml $\text{Na}_2\text{S}_2\text{O}_3$ solution, the concentration (S_2) of the $\text{Na}_2\text{S}_2\text{O}_3$ solution can be calculated as per the above equation.

Apparatus and chemicals used:

Apparatus used

1. Conical flask
2. Burette
3. Pipette
4. Volumetric flask
5. Stand
6. Funnel

Name of the chemicals used

1. $\text{Na}_2\text{S}_2\text{O}_3$ solution
2. $\text{K}_2\text{Cr}_2\text{O}_7$ solution
3. KI salt
4. NaHCO_3 or Na_2CO_3
5. HCl (concentrated) or H_2SO_4 (concentrated)
6. Starch (Indicator)

Experimental Procedure

Preparation of 100 ml 0.1 N potassium dichromate (K₂Cr₂O₇) solution: Its MW is 294 and equivalent weight is 294/6 = 49.

We know that, $normality = \frac{w \times 1000}{E \times V (ml)}$

Here,

w = amount of K₂Cr₂O₇ in g

E = equivalent weight of K₂Cr₂O₇ (49)

V = volume of the solution in ml (100 ml)

Titration

Take 20mL recently boiled distilled water in a 250mL conical flask (preferably glass-stoppered flask), add 1.3-1.5g of iodate-free potassium iodide and 1.0g of sodium hydrogen carbonate or sodium carbonate and shake until the salts dissolve. Then add 3mL of conc. HCl or conc. H₂SO₄ acid solution slowly while gently rotating the flask in order to mix the liquids. Run in 10mL of standard 0.1N K₂Cr₂O₇ solution, mix the solutions well, and wash the sides of the flask with a little boiled-out distilled water from the wash bottle. Stopper the flask and allow to stand in the dark for 5 minutes in order to complete the reaction. Rinse the stopper and the sides of the flask. Titrate the liberated iodine with the Na₂S₂O₃ solution contained in a burette, while constantly rotating the solution. When most of the iodine has reacted as indicated by the solution acquiring yellowish-green color, add 2mL of starch indicator and rinse down the sides of the flask. The color should change to blue. Continue the addition of Na₂S₂O₃ solution drop wise, and swirling the liquid constantly, until one drop changes the color from greenish blue to light green. The end-point is sharp, and is readily observed in a good light against a white background.

Experimental data collection table: For the standardization of Na₂S₂O₃ solution

No. of obs.	Volume of K ₂ Cr ₂ O ₇ solution, ml	Burette reading (volume of Na ₂ S ₂ O ₃ solution), ml			Average or mean value, ml	Conc. of std. solution (N)	Conc. of Na ₂ S ₂ O ₃ soln. (N)
		IR	FR	Difference			
1							
2							
3							

Calculation: For normal solution, we know,

$$V_1 S_1 = V_2 S_2$$

$$S_2 = \frac{V_1 S_1}{V_2}$$

V ₁ (K ₂ Cr ₂ O ₇)	= mL
S ₁ (K ₂ Cr ₂ O ₇)	=
V ₂ (Na ₂ S ₂ O ₃)	= mL
S ₂ (Na ₂ S ₂ O ₃)	=	?

Result: The strength of the supplied Sodium Thiosulphate solution was normal (gram-equivalent/litre).

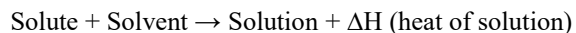
Precautions: (To be written by the students).

Physical Chemistry Experiments

4. Determination of heat of solution of a supplied sample by calorimetric method.

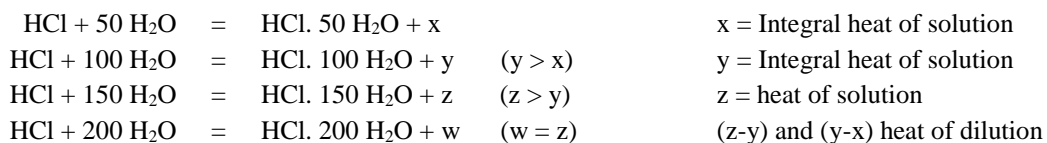
Introduction

Heat of solution is defined as amount of heat or enthalpy released or absorbed when one mole of a substance is dissolved in a sufficient amount solvent. Here sufficient amount of solvent means the amount of solvent which is required for the solute to release the maximum amount of heat.



Principle

When a substance is dissolved, heat may be either released or absorbed depending on the relative amounts of energy which is used up in breaking down the crystal lattice on the one hand and the energy liberated during the hydration of the solute on the other. The quantity of heat evolved is not constant but varies with the concentration of the final solution.



Experimental Procedure

Take the calorimeter and check the cleanliness. Weigh the calorimeter and record it. Take 50ml of distilled water in the calorimeter and put the thermometer in the solution. Record the constant temperature of the water with the help of a thermometer. Then add 0.5 g of solid KNO_3 to it and start recording the temperature of the solution at 30 second interval with continuous stirring of the solution. Continue temperature recording until constant.

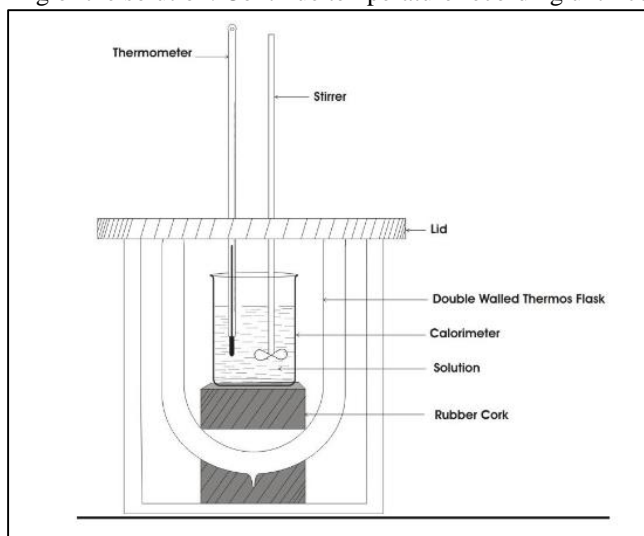


Figure: Determination of heat of neutralization

Data collection table

Time, min	0	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0
Temp, °C											

Plot a graph with temperature against time. The final temperature is obtained from the graph.

Calculation

The weight of calorimeter	=	m_1 g
weight of total solution	=	m_2 g
Initial temperature	=	t_1 °C
Final temperature	=	t_2 °C
Specific heat capacity of calorimeter (glass)	=	S_1 J/K-g
Specific heat capacity of solution	=	S_2 J/K-g
Temperature difference	=	$(t_1 - t_2)$ °C
Amount of heat absorbed by calorimeter	H_1	= $m_1 S_1 (t_1 - t_2)$ J
Amount of heat absorbed by solution	H_2	= $m_2 S_2 (t_1 - t_2)$ J
Total heat absorbed/released	H	= $(H_1 + H_2)$ J
Heat of solution	ΔH	= $\frac{H}{V(\text{litre}) \times N}$ J/mole
		= $\frac{H}{V(\text{ml}) \times N \times 1000}$ KJ/mole

Result: The heat of solution of the supplied salt (KNO_3) was determined as

Precautions: (to be written by the students).

5. Determination of heat of neutralization of strong acid and strong base calorimetrically.

Introduction

Generally the reactions taking place in the chemical sciences are breaking and making of chemical bonds. This is accompanied by some heat effects. Formation of chemical bonds releases energy in the form of heat and hence known as an exothermic reaction. The reaction which is accompanied absorption of heat is known as endothermic reaction. Calorimetry is a scientific term dealing with the changes in energy of the system by measuring the heat exchanged with the surroundings. In a broader sense it is defined to determine the heat released or absorbed in a chemical reaction. A calorimeter is a device designed to measure heat of reaction or physical changes and heat capacity. The device can be sophisticated and expensive or simple and cheap.

A calorimeter consists of two vessels, outer vessel and an inner vessel. The space between these vessels acts as a heat insulator and hence there is very little heat exchange in between the inner and outer vessels. Thermometer measures the temperature of the liquid in the inner vessel. The stirrer functions in such a way to stir the liquid to distribute the heat in the entire vessel. The fibre rings in the calorimeter helps to hold the inner vessel hanging in the center of the outer vessel. It also has an insulating cover or lid with holes for attaching the stirring rod and thermometer.

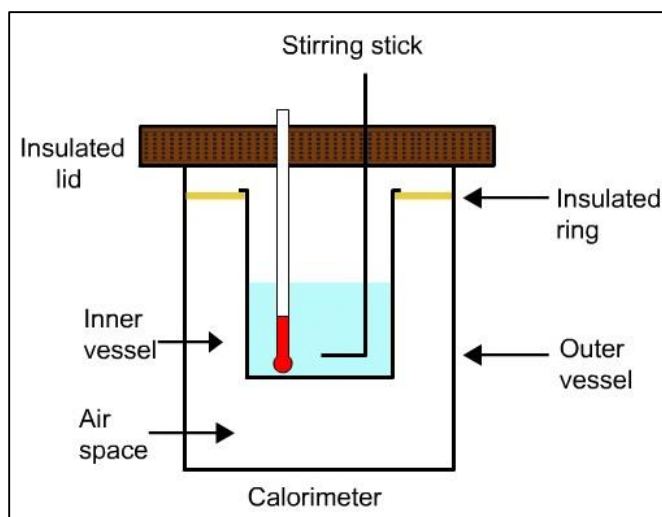
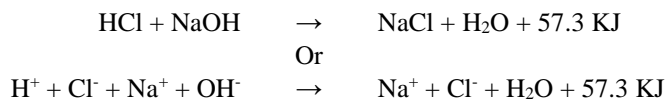


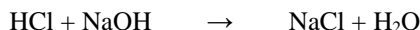
Figure: Calorimeter

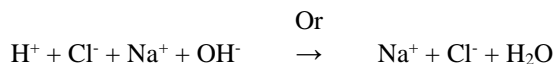
The heat of neutralization of an acid is defined as the amount of heat evolved when one gram-equivalent of an acid and one gram-equivalent of a base undergo a neutralization reaction to form water and a salt. Similarly the heat of neutralization of a base is the amount of heat evolved when 1 gram-equivalent of the base is completely neutralized by a strong acid in a dilute solution.



Principle

A known volume (V ml) of HCl solution of exactly known concentration (N normal) is allowed to neutralize completely with a strong alkali in dilute solution in a calorimeter. The temperature change is then noted. With the known volume of HCl solution, the heat of neutralization can be calculated.





Let,

The weight of calorimeter	=	m_1 g
weight of total solution	=	m_2 g
Initial temperature	=	t_1 °C
Final temperature	=	t_2 °C
Specific heat capacity of calorimeter (glass)	=	S_1 J/K-g
Specific heat capacity of solution	=	S_2 J/K-g
Temperature difference	=	$(t_1 - t_2)$ °C
Amount of heat absorbed by calorimeter	H_1	= $m_1 S_1 (t_1 - t_2)$ J
Amount of heat absorbed by solution	H_2	= $m_2 S_2 (t_1 - t_2)$ J
Total heat released by the reaction	H	= $(H_1 + H_2)$ J
Heat of neutralization	ΔH	= $\frac{H}{V(\text{litre}) \times N}$ J/equivalent
		= $\frac{H}{V(\text{ml}) \times N \times 1000}$ KJ/equivalent

Experimental Procedure

Take the calorimeter and check the cleanliness and dryness. Weight the calorimeter. Take 50 ml of HCl solution into the calorimeter and put the thermometer in the solution. Record the constant temperature of the acid solution. Then add 50 ml of NaOH solution of same concentration as acid solution to it, record the temperature of the solution at 30 second interval with continuous stirring of the solution. Continue temperature recording until constant.

Time, min	0	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0	4.5	5.0
Temp, °C	28	28	28	28.5	29.0	31	32	33	33	33	31

Plot a graph with temperature against time. The final temperature is obtained from the graph.

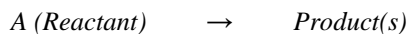
Result: the heat of neutralization determined was KJ/mole.

Precaution: (to be written by the students)

6. Determination of the rate constant and justification of the order of a chemical reaction.

Introduction

Rate of reaction is defined as the change in concentration of any reactant or product per unit time.



According to the law of mass action, rate of the reaction,
 $r \propto [A]$
 or $r = k. [A]$

Where,
 [A] is molar concentration of reactant A.
 k is a proportionality constant and it is called rate constant of the reaction.

Order of reaction is defined as the sum of the powers of the concentration terms used in the rate law of the reaction concerned. Let us consider the examples,

Reaction			Rate law	Order of reaction	
A	→	Product(s)	$r = k. [A]$	1	first
A + B	→	Product(s)	$r = k. [A].[B]$	1+1 = 2	second
2A + B	→	Product(s)	$r = k. [A]^2. [B]$	2+1 = 3	third
A + 2B	→	Product(s)	$r = k. [A]. [B]^2$	1+2 = 3	third
aA + bB	→	Product(s)	$r = k. [A]^a. [B]^b$	a+b = n	n th

Principle

Consider a second order reaction as follows

	A	+	B	→	Product(s)
Initially molar conc. of reactants (at time $t = 0$)	a		b		0
molar conc. of reactants after time t	a-x		b-x		2x

Rate of the reaction,

$$\frac{dx}{dt} = k. (a - x)(b - x)$$

$$\text{or, } \frac{dx}{(a-x)(b-x)} = k. dt$$

$$\text{or, } \frac{1}{(a-b)} \left\{ \frac{1}{(b-x)} - \frac{1}{(a-x)} \right\} dx = k. dt$$

After integrating we get,

$$\frac{1}{(a-b)} \{ -\ln(b-x) + \ln(a-x) \} = k. t + C \quad \dots\dots\dots(i)$$

Here C is a integral constant. When $t = 0$, $x = 0$, then

$$\frac{1}{(a-b)} \{ -\ln(b-0) + \ln(a-0) \} = k. 0 + C$$

$$\text{or, } \frac{1}{(a-b)} \ln \frac{a}{b} = C$$

Putting the value of C in equation (i) we get,

$$\frac{1}{(a-b)} \{-\ln(b-x) + \ln(a-x)\} = k.t + \frac{1}{(a-b)} \ln \frac{a}{b}$$

$$\text{or, } k.t = \frac{1}{(a-b)} \{-\ln(b-x) + \ln(a-x)\} - \frac{1}{(a-b)} \ln \frac{a}{b}$$

$$\text{or, } k.t = \frac{2.303}{(a-b)} \log \frac{b(a-x)}{a(b-x)} \dots\dots\dots(\text{ii})$$

When the concentrations of the reacting molecules/species are same (a = b), under these conditions,

$$\frac{dx}{dt} = k.(a-x)^2$$

$$\text{or, } \frac{dx}{(a-x)^2} = k.dt$$

After integrating we get,

$$\frac{1}{a-x} = k.t + C \dots\dots\dots(\text{iii})$$

Here C is a integral constant. When t = 0, x = 0, then

$$\frac{1}{a-0} = k.0 + C$$

$$\text{or, } C = \frac{1}{a}$$

Now putting the value of C in the above equation (iii) we get

$$\frac{1}{a-x} = k.t + \frac{1}{a}$$

$$\text{or, } k.t = \frac{1}{a-x} - \frac{1}{a}$$

$$\text{or, } k.t = \frac{x}{a(a-x)}$$

$$\text{or, } k = \frac{1}{t} \cdot \frac{x}{a(a-x)} \dots\dots\dots(\text{iv})$$

If a chemical reaction is allowed to proceed for different period of time and right hand side of the above equation is found constant for each period of time, then the value is the value of the rate constant of the reaction and the reaction is of second order.

Required Chemicals:

1. 0.2 N Ethyl Acetate solution.
2. 0.2 N Sodium Hydroxide (NaOH) solution.
3. 0.2 N Hydrochloric (HCl) acid solution.
4. Indicator, phenolphthalein and/or methyl orange, litmus paper (blue and red).

Experimental procedure

Preparation of 0.2 N Ethyl Acetate solution: Take 17.6 g of ethyl acetate ester in a clean and dry 1.0 L volumetric flask and fill it up to the mark with distilled water.

Preparation of 0.2 N Sodium Hydroxide (NaOH) solution: Take 8.0 g of NaOH pellets in a clean and dry 1.0 L volumetric flask and fill it up to the mark with distilled water.

Take 100 ml of 0.2 N NaOH solution in a 100 ml beaker and 100 ml of 0.2 N ester solution in a 250 ml beaker. Mix the base solution to the ester solution quickly, shake gently and start the stop watch. Allow the solution stand still for reaction. After 5 minutes, take 30 ml of the solution in a 250 ml conical flask and titrate it against 0.2 N HCl solution (from burette) using phenolphthalein as indicator. Record the volume of 0.2 N HCl solution from burette. The amount of HCl solution represents the amount of un-reacted NaOH solution present in the 30 ml reaction solution. Similarly take 30 ml solution from the reaction solution after 10 min, 15 min, 20 min, and 25 min and titrate them against 0.2 N HCl solution as above.

$\text{CH}_3\text{COOC}_2\text{H}_5 + \text{NaOH}$	\rightarrow	$\text{CH}_3\text{COONa} + \text{C}_2\text{H}_5\text{OH}$
$\text{NaOH} + \text{HCl}$	\rightarrow	$\text{NaCl} + \text{H}_2\text{O}$

Experimental data table

Obs. No.	Time, t min	Volume of reaction sol ⁿ . ml	Burette reading, ml			a-x	x	$k = \frac{1}{t} \cdot \frac{x}{a(a-x)}$	Comments
			Initial	Final	Diff				
1	00	30							
2	05	30							
3	10	30							
4	15	30							
5	20	30							
6	25	30							

Calculation:

- When $t = 00$, $a-x = a$ then $x = a-(a-x) = 0$, $k = 0$. Reaction does not start.
- When $t = 05$, $a-x = \dots\dots\dots$ then $x = a-(a-x) = \dots\dots\dots$, $k = \dots\dots\dots$
- When $t = 10$, $a-x = \dots\dots\dots$ then $x = a-(a-x) = \dots\dots\dots$, $k = \dots\dots\dots$
- When $t = 15$, $a-x = \dots\dots\dots$ then $x = a-(a-x) = \dots\dots\dots$, $k = \dots\dots\dots$
- When $t = 20$, $a-x = \dots\dots\dots$ then $x = a-(a-x) = \dots\dots\dots$, $k = \dots\dots\dots$
- When $t = 25$, $a-x = \dots\dots\dots$ then $x = a-(a-x) = \dots\dots\dots$, $k = \dots\dots\dots$

Result and discussion: The rate constant of the reaction is found constant for each period of reaction time and the value is $\dots\dots\dots$. The value is calculated on the basis of a second order reaction. So the reaction (hydrolysis of ethyl acetate by sodium hydroxide base) is a second order reaction.

Precautions: (to be written by the students).

$\dots\dots\dots$ The End. $\dots\dots\dots$