



## 2.3.2

ICT ENABLED TEACHING SUPPORTIVE DOCUMENT

LAB MANUAL

DEPT OF CHEMISTRY (PG)



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**[Reaccredited with 'A' Grade (CG PA 3.58) by NAAC]**

**PG Department of Chemistry**

**CH 460 – Physical Chemistry Practicals – II**

**Name :**

**Subject :**

**Class :**

**Roll No :**

**Seat No :**

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## DETERMINATION OF ASSOCIATION CONSTANT OF BENZOIC ACID IN TOLUENE

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**Aim:** To determine the association constant of a carboxylic acid in an organic solvent by distribution method.

### Principle:

When the solute associates in one of the phases, say in toluene, the distribution coefficient is given by;

$$k = \frac{C_{\text{org}}^n}{C_{\text{aq}}}$$

Where  $C_{\text{org}}$  and  $C_{\text{aq}}$  are the concentrations of the acid in organic and aqueous layer separately and  $n$  is the number of molecules of the acid which associate to form a complex molecule.

The values of 'n' can be determined by giving different values for  $n$ , say 1,2,3... etc. So as to attain constant value of  $k$  alternatively, 'n' can be determined in the following way.

Taking logarithm of the above equation;

$$\log C_{\text{aq}} = \frac{1}{n} \log C_{\text{org}} - \log k$$

Monocarboxylic acids have a tendency to dimerise in non - polar solvents like benzene and toluene. The dimers are formed through hydrogen bonding.

A plot of  $\log C_{\text{aq}}$  against  $\log C_{\text{org}}$  must be a straight line. The slope of which will be  $1/n$  and the intercept on the ordinate axis will be  $\log k$ . A graph of  $C_{\text{org}}/C_{\text{aq}}$  v/s  $C_{\text{aq}}$  can be plotted from which dimerisation constant can be found.

### Procedure:

About 10g of benzoic acid was dissolved in 100 cm<sup>3</sup> of toluene. Four reagent bottles were taken and numbered as 1,2,3,4 and 10, 15, 20 and 25 cm<sup>3</sup> of benzoic acid in toluene was added respectively to the bottles 1 to 4. Similarly 25, 20, 15, and 10 cm<sup>3</sup> of pure toluene was added to each reagent bottle respectively. Then for each bottle 20 cm<sup>3</sup> of water is added and shaken well. The organic layer and aqueous layer in each mixture were separated by means of separating funnel. The concentrations of benzoic acid in organic layer was estimated by taking 5cm<sup>3</sup> of organic layer and titrating against 0.1N NaOH solution, using phenolphthalein as an indicator. Similarly concentration of benzoic acid in aqueous layer was estimated by taking 5 cm<sup>3</sup> of aqueous layer and titrating against 0.01N NaOH using phenolphthalein as an indicator. Two graphs were plotted one with  $3 + \log C_{\text{aq}}$  v/s  $2 + \log C_{\text{org}}$  which gives the number of molecules involved in association and other with  $C_{\text{org}}/C_{\text{aq}}$  v/s  $C_{\text{aq}}$ , which gives the dimerisation constant.

### Result:

- 1) Number of molecules involves in association =
- 2) The value of dimerisation constant = ----- mol/dm<sup>3</sup>

### Observations and calculations:

Sl. No.	Volume of Benzoic acid in toluene (cm <sup>3</sup> )	Volume of toluene (cm <sup>3</sup> )	Volume of water (cm <sup>3</sup> )	Volume of 0.1N NaOH used for organic layer. V <sub>1</sub> cm <sup>3</sup>			Volume of 0.01 N NaOH (cm <sup>3</sup> ) for aqueous layer . V <sub>2</sub> cm <sup>3</sup>		
				1	2	Mean	1	2	Mean
1	10	25	20						
2	15	20	20						
3	20	15	20						
4	25	10	20						

Sl. No.	Conc of acid in toluene C <sub>org</sub> = (V <sub>1</sub> x 0.1)/5 mol <sup>-1</sup> dm <sup>3</sup> .	Conc of acid in aq layer, C <sub>aq</sub> = (V <sub>2</sub> x 0.01)/5 mol <sup>-1</sup> dm <sup>3</sup>	log C <sub>org</sub>	--+log C <sub>org</sub>	log C <sub>aq</sub>	--+log C <sub>aq</sub>	C <sub>org</sub> /C <sub>aq</sub>
1							
2							
3							
4							

From Graph 3 + log C<sub>aq</sub> v/s 2+ log C<sub>org</sub>, *Slope* = -----

n= 1/slope

Therefore number of molecules involved in association, n = -----

From Graph C<sub>org</sub>/C<sub>aq</sub> v/s C<sub>aq</sub>

$$\text{Dimerisation constant} = \sqrt{\frac{\text{slope}}{2 \times \text{intercept}}}$$

$$= \text{----- mol/ dm}^3$$

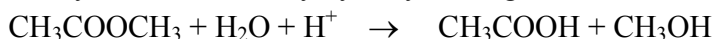
## KINETICS OF HYDROLYSIS OF ESTER

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**Aim:** To determine the rate constant of the hydrolysis of ester of two acids at the same temperature and hence to determine the relative strength of two acids.

### Principle:

Methyl acetate is readily hydrolyzed to give alcohol and acetic acid as follows



The reaction is catalyzed by hydrogen ions. Two molecules are involved in the reaction but the kinetics are first order due to the reason that the quantity of water is large and its concentration remains practically constant during the reaction, the rate depends entirely on the concentration of methyl acetate. The rate depends entirely on the concentration of methyl acetate. The rate constant of the reaction is therefore given by

$$k = \frac{2.303}{t} \log \left[ \frac{V_\infty - V_0}{V_\infty - V_t} \right]$$

The rate is approximately proportional to the concentration or more exactly the activity of hydrogen ions. Hence it may be assumed that rate constant of the reaction catalyzed by equal concentration of acids are directly proportional to the degree of dissociation of relative acids. Thus the relative strengths is given by

$$\text{Relative strength} = \frac{\text{Strength of one acid}}{\text{Strength of other acid}} = \frac{\alpha_1}{\alpha_2} = \frac{k_1}{k_2}$$

Where  $\alpha_1$  and  $\alpha_2$  are degree of dissociation of two acids and  $k_1$  and  $k_2$  are rate constants in presence of equimolar solution of the respective acids.

**Requirements:** Thermostat, 250 ml and 150 ml conical flasks, 5 ml pipette, stop watch, pure methyl acetate, 0.5N HCl and 0.5N  $\text{H}_2\text{SO}_4$ , 0.25N NaOH, phenolphthalein indicator and ice.

### Procedure:

50 ml of 0.5N HCl was taken in a reagent bottle and 10 ml of ester is in another. Both were kept in thermostat at room temperature. After attaining the same temperature, 5 ml of ester was pipetted out into the bottle containing 50 ml of HCl. The mixture was stirred well and 5 ml of mixture is immediately pipetted out into a conical flask containing crushed ice and titrated against 0.25N NaOH taken in burette using phenolphthalein indicator. When the color of the mixture changed to pale pink the burette reading was noted. This corresponds to  $V_0$ . Similar titration were carried out at 10, 20, 30, 40, 50 and 60 min. The volume of NaOH at each time is noted. Then the remaining reaction mixture was heated on a water bath for an hour so that the reaction goes for completion. The 5 ml of solution was titrated against 0.25N NaOH using phenolphthalein indicator. Then a graph of  $\log \left[ \frac{(V_\infty - V_0)}{(V_\infty - V_t)} \right]$  was plotted against time and also another graph of  $\log (V_\infty - V_t)$  against the time was plotted. Rate constant,  $k = 2.303 \times \text{slope}$  was calculated in each case.

The above procedure was repeated by taking 0.5N H<sub>2</sub>SO<sub>4</sub> and ester against 0.25N NaOH. Again the rate constant is calculated as before. Then the relative strength of two acids can be given by;

$$\text{Relative strength} = k_1/k_2$$

**Result:**

- 1) Rate constants of the reaction with 0.5N HCl at lab temperature:
  - a) By calculation = -----min<sup>-1</sup>
  - b) By graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = -----min<sup>-1</sup>
  - c) By graph  $\log (V_{\infty}-V_t)$  v/s time = -----min<sup>-1</sup>
- 2) Rate constants of the reaction with 0.5N H<sub>2</sub>SO<sub>4</sub> at lab temperature:
  - a) By calculation = -----min<sup>-1</sup>
  - b) By graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = -----min<sup>-1</sup>
  - c) By graph  $\log (V_{\infty}-V_t)$  v/s time = -----min<sup>-1</sup>
- 3) Relative strength of two acids  $\left( \frac{k_{\text{HCl}}}{k_{\text{H}_2\text{SO}_4}} \right) = \text{-----}$

**Observation and calculation:**

a)Hydrolysis of ester with 0.5N HCl at lab temperature:

Time (min)	Volume of NaOH added in $\text{cm}^3$	$(V_{\infty}-V_t)$	$\log (V_{\infty}-V_t)$	$\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$	$k_1 = \frac{2.303}{t} \log \left[ \frac{V_{\infty}-V_0}{V_{\infty}-V_t} \right] (\text{min}^{-1})$
0					
10					
20					
30					
40					
50					
60					

 $V_{\infty} = \text{---- cm}^3$ Mean  $k_1 =$ From graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time,  $k_1 = \text{----- min}^{-1}$ From graph  $\log (V_{\infty}-V_t)$  v/s time,  $k_1 = \text{----- min}^{-1}$ b)Hydrolysis of ester with 0.5N  $\text{H}_2\text{SO}_4$  at lab temperature:

Time (min)	Volume of NaOH added in $\text{cm}^3$	$(V_{\infty}-V_t)$	$\log (V_{\infty}-V_t)$	$\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$	$k_2 = \frac{2.303}{t} \log \left[ \frac{V_{\infty}-V_0}{V_{\infty}-V_t} \right] (\text{min}^{-1})$
0					
10					
20					
30					
40					
50					
60					

 $V_{\infty} = \text{---- cm}^3$ Mean  $k_2 =$ From graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time,  $k_2 = \text{----- min}^{-1}$ From graph  $\log (V_{\infty}-V_t)$  v/s time,  $k_2 = \text{----- min}^{-1}$ Therefore relative strength of two acids =  $k_1/k_2$



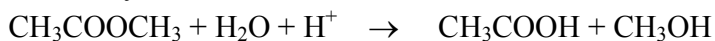
## KINETICS OF HYDROLYSIS OF ESTER

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**Aim:** To determine the rate constants of hydrolysis of ester at two different temperature and hence to determine the energy of activation.

### Theory:

Methyl acetate is readily hydrolyzed in the presence of mineral acids like HCl to form acetic acid and methyl alcohol. .



During the reaction, the concentration of water being very large, it practically remains constant. Hence the above reaction becomes pseudomolecular and follows first order kinetics. The rate depends entirely on the concentration of methyl acetate. The rate constant of the reaction is therefore given by

$$k = \frac{2.303}{t} \log \left[ \frac{V_\infty - V_0}{V_\infty - V_t} \right]$$

The rate is approximately proportional to the concentration or more correctly activity of hydrogen ions. Hence it may be assumed that rate constants of the reaction catalysed by equal concentration of acids are directly proportional to the degree of dissociation of the reactive acids. The variation of velocity constant is expressed in terms of an exponential equation known as Arrhenius equation,  $k = Ae^{-E_a/RT}$ , Where  $E_a$  is the energy of activation (Joules/mol) and  $A$  is a constant.

Taking logarithm of the above equation, we have;

$$2.303 \log k = -\frac{E_a}{RT} + \text{Constant}.$$

If  $k_1$  and  $k_2$  are specific rates at two temperatures,  $T_1$  and  $T_2$  then,

$$\log \left( \frac{k_1}{k_2} \right) = \frac{E_a}{2.303R} \left[ \frac{1}{T_1} - \frac{1}{T_2} \right]$$

$$\log \left( \frac{k_1}{k_2} \right) = \frac{E_a}{2.303R} \left[ \frac{T_2 - T_1}{T_1 T_2} \right]$$

$$E_a = \frac{2.303 R \log(k_1/k_2) T_1 T_2}{T_2 - T_1}$$

### Procedure:

a) Hydrolysis of methyl acetate at lab temperature:

50 ml of 0.5 N HCl was taken in reagent bottle and 10 ml of methyl acetate in the other. Both were placed in a thermostat kept at lab temperature. When both the acid and the ester attains the same temperature, 5 ml of the ester was pipetted out into the bottle containing 0.5 N HCl, the mixture was stirred well and 5 ml of the reaction mixture was immediately pipetted out into a conical flask containing crushed ice which stops the reaction of the mixture. The mixture was titrated against 0.25 N NaOH taken in the burette followed by phenolphthalein indicator. When the color of the mixture changed to pink, burette reading was noted. The reading was taken as for zero minute. Similar titrations were made at successive intervals of 10, 20, 30, 40, 50 and 60 minutes and the volume of NaOH consumed at each time was noted. The bottle containing the remaining reaction mixture was placed in a water bath at about 80°C for about an hour to complete the reaction. The flask was cooled

to the lab temperature. The flask was cooled to the lab temperature. Then the 5 cm<sup>3</sup> of the solution was titrated as above, to get the V<sub>∞</sub> value.

b) Hydrolysis of methyl acetate at 35°C :

The above experiment was repeated at 40°C and titrations were conducted at successive intervals of 10, 20, 30, 40, 50 and 60 min.

Then a graph of  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  was plotted against time and also another graph of  $\log (V_{\infty}-V_t)$

against the time was plotted. Rate constant  $k = 2.303 \times \text{slope}$  was calculated in each case. Energy of activation was also calculated knowing the rate constants at different temperature by

$$E_a = \frac{2.303 R \log (k_1/k_2) T_1 T_2}{T_2 - T_1}$$

### Result:

1) Rate constants of the reaction with 0.5N HCl at lab temperature:

a) By calculation = -----min<sup>-1</sup>

b) By graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = -----min<sup>-1</sup>

c) By graph  $\log (V_{\infty}-V_t)$  v/s time = -----min<sup>-1</sup>

2) Rate constants of the reaction with 0.5N HCl at lab temperature:

a) By calculation = -----min<sup>-1</sup>

b) By graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = -----min<sup>-1</sup>

c) By graph  $\log (V_{\infty}-V_t)$  v/s time = -----min<sup>-1</sup>

3) Energy of activation;

a) By calculation = -----kJ/mol

b) By graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = -----kJ/mol

c) By graph  $\log (V_{\infty}-V_t)$  v/s time = ----- kJ/mol

**Observation and Calculations:****a) Hydrolysis of ester with 0.5N HCl at lab temperature:**

Time (min)	Volume of NaOH added in cm <sup>3</sup>	(V <sub>∞</sub> -V <sub>t</sub> )	log (V <sub>∞</sub> -V <sub>t</sub> )	$\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$	$k_2 = \frac{2.303}{t} \log \left[ \frac{V_{\infty}-V_0}{V_{\infty}-V_t} \right] (\text{min}^{-1})$
0					
10					
20					
30					
40					
50					
60					

$$V_{\infty} = \text{---- cm}^3$$

$$\text{Mean } k_2 =$$

$$\text{From graph } \log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right] \text{ v/s time, } k_2 = \text{----- min}^{-1}$$

$$\text{From graph } \log (V_{\infty}-V_t) \text{ v/s time, } k_2 = \text{----- min}^{-1}$$

**b) Hydrolysis of ester with 0.5N HCl at 35°C temperature:**

Time (min)	Volume of NaOH added in cm <sup>3</sup>	(V <sub>∞</sub> -V <sub>t</sub> )	log (V <sub>∞</sub> -V <sub>t</sub> )	$\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$	$k_1 = \frac{2.303}{t} \log \left[ \frac{V_{\infty}-V_0}{V_{\infty}-V_t} \right] (\text{min}^{-1})$
0					
10					
20					
30					
40					
50					
60					

$$V_{\infty} = \text{---- cm}^3$$

$$\text{Mean } k_1 =$$

$$\text{From graph } \log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right] \text{ v/s time, } k_1 = \text{----- min}^{-1}$$

$$\text{From graph } \log (V_{\infty}-V_t) \text{ v/s time, } k_1 = \text{----- min}^{-1}$$

$$\text{Therefore activation energy, } E_a = \frac{2.303 R \log(k_1/k_2) T_1 T_2}{T_2 - T_1}$$

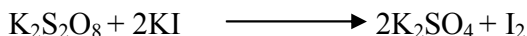
$$= \text{----- kJ/mol}$$

## KINETICS OF REACTION BETWEEN KI AND K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>

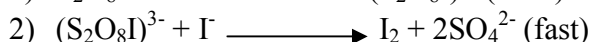
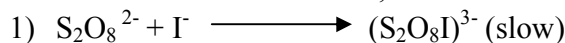
**Aim:** To study the first and second order kinetics of reaction between potassium persulphate and potassium iodide solution and to find the energy of activation.

### Principle:

The reaction between potassium persulphate and potassium iodide takes place as follows.



The mechanism of reaction is;



The reaction rate can be studied by measuring the rate of liberation of iodine. As the slowest step determines the reaction rate, step (1) gives the rate expression which can be written as

$dx/dt = k(b-2x)(a-x)$  ----- (1) Hence the reaction is second order, if we integrate above equation,

we get;  $k = \frac{1}{(2at)(a-x)}$  where  $b = 2a$  ----- (2)

The term 'a' and 'b' represents the initial concentration of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and KI in moles/litre in the reaction mixture. According to equation (1), the concentration of KI is to be made double as that of K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>. The energy of activation  $E_a$  can be determined by applying Arrhenius equation  $k = Ae^{-E_a/RT}$ , where A is frequency factor. On taking logarithm  $\log k = \log A - E_a/RT$  ----- (3)

Differentiating equation (3) wrt T, we get

$$\log\left(\frac{k_2}{k_1}\right) = \frac{E}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2}\right) \log(k_2/k_1) = E_a/2.303R[1/T_1 - 1/T_2]$$

If the values of  $k_1$  and  $k_2$  are known for two temperatures  $T_1$  and  $T_2$ , then the values of E can be calculated. The plot of  $\log_{10} k$  v/s  $1/T$  gives a straight line having slope =  $-E/2.303R$ .

By the value,  $E_a$  can also be calculated. In the 1<sup>st</sup> order reaction if 'a' is the initial concentration of the reactant and (a-x) is the concentration after a time 't' then the rate of the reaction is,  $dx/dt = kx(a-x)$ . Integrating and simplifying this equation, gives;  $k = 2.303/t \log(a/a-x)$

Since I<sub>2</sub> is liberated in the reaction between K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> and KI, the reaction can be studied by titrating sodium thiosulphate solution at various intervals of time. If this volumes are ( $V_\infty - V_t$ ) and ( $V_\infty - V_0$ ), then

the rate equation becomes,  $k = \frac{2.303}{t} \log \frac{(V_\infty - V_0)}{(V_\infty - V_t)}$ . If a graph of  $\log(V_\infty - V_0)/(V_\infty - V_t)$  is

plotted against time, we get a straight line passing through origin having slope =  $k/2.303$ .

In the second order reaction between KI and K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, the progress of the reaction can be studied by titrating liberated iodine with sodium thiosulphate using starch as an indicator. The titration value at anytime will be proportional to the amount of I<sub>2</sub> liberated, ie it gives the value of x at that time. For such reaction,  $dx/dt = k(a-x)^2$  where a is the initial concentration of the reactant. Integrating this, we get,  $k_2 = x/at (1/a-x)$ .

If  $V_t$  is the volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> consumed at time t,  $V_\infty$  that after the completion of the reaction. Then,  $k_2 = (V_\infty - V_0) / [(V_\infty - V_0)(V_\infty - V_t)]$ . The plot of  $1/(V_\infty - V_t)$  v/s time gives a straight line.

**Procedure:**First order reaction between 0.2M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.005M Hypo at lab temperature:

About 25ml of 0.2M KI and 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> solutions are taken in two separate reagent bottles and kept in a thermostat for half an hour. When the solutions attain lab temperature, KI solution was added to K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> solution, mixed well. Immediately 5ml of the reaction mixture was pipetted out into clean conical flask containing ice and two drops of starch indicator. Then it is titrated against 0.005M hypo solution from the burette till color changes from blue to colorless. Reading was taken at regular intervals of 5, 10, 15, 20, 25 and 30 min. To determine the V<sub>∞</sub> value, the mixture is kept at higher temperature for half an hour and then titration was carried out. The graph was plotted taking log(V<sub>∞</sub> - V<sub>t</sub>) v/s time and log [(V<sub>∞</sub> - V<sub>0</sub>) / (V<sub>∞</sub> - V<sub>t</sub>)] v/s time.

First order reaction between 0.2M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.005M Hypo at 35°C:

The above experiment was repeated taking 25ml of 0.2M KI solution and 25ml of 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> solution kept at thermostat at 35°C. Activation energy of the solution was calculated.

Second order reaction between 0.04M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.0025M Hypo at lab temperature:

For the second order reaction, 0.04M KI solution and 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> solution of each about 25ml are taken in different reagent bottles and kept at lab temperature. After attaining the constant temperature they were mixed and immediately 5ml of the mixture was pipetted out and added few crushed ice and titrated against std. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution using starch as indicator. The titrations were carried out at different intervals of time as before. The final titre value V<sub>∞</sub> also carried out. The rate constant was calculated.

**Result:**

- 1) Rate constants for 1<sup>st</sup> order reaction of 0.2M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.005M Hypo at lab temperature:-----
  - a) By calculation = ----- min<sup>-1</sup>
  - b) By graph of log (v<sub>∞</sub>-v<sub>t</sub>) v/s time = ----- min<sup>-1</sup>
  - c) By graph of  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = ----- min<sup>-1</sup>
- 2) Rate constants for 1<sup>st</sup> order reaction of 0.2M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.005M Hypo at 35°C
  - a) By calculation = ----- min<sup>-1</sup>
  - b) By graph of log (v<sub>∞</sub>-v<sub>t</sub>) v/s time = ----- min<sup>-1</sup>
  - c) By graph of  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = ----- min<sup>-1</sup>
- 3) Activation energy for the reaction KI+K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>
  - a) By calculation = ----- kJ/mol
  - b) By graph of log (v<sub>∞</sub>-v<sub>t</sub>) v/s time = ----- kJ/mol
  - c) By graph of  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time = ----- kJ/mol
- 4) Rate constant of second order reaction of KI + K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> at lab temperature
  - a) From calculation = -----dm<sup>3</sup>/mol/min
  - b) From graph = ----- dm<sup>3</sup>/mol/min

### Observations and Calculations:

1) 1<sup>st</sup> order reaction between 0.2M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.005M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> at lab temperature:

Time (min)	Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> added in cm <sup>3</sup>	(V <sub>∞</sub> -V <sub>t</sub> )	log (V <sub>∞</sub> -V <sub>t</sub> )	$\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$	$k_1 = \frac{2.303}{t} \log \left[ \frac{V_{\infty}-V_0}{V_{\infty}-V_t} \right] \text{ (min}^{-1}\text{)}$
0					
5					
10					
15					
20					
25					
30					

V<sub>∞</sub> = ---- cm<sup>3</sup>

Mean k<sub>1</sub> =

From graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time, k<sub>2</sub> = ----- min<sup>-1</sup>

From graph log (V<sub>∞</sub>-V<sub>t</sub>) v/s time, k<sub>2</sub> = ----- min<sup>-1</sup>

2) 1<sup>st</sup> order reaction between 0.2M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.005M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> at 35°C:

Time (min)	Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> added in cm <sup>3</sup>	(V <sub>∞</sub> -V <sub>t</sub> )	log (V <sub>∞</sub> -V <sub>t</sub> )	$\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$	$k_2 = \frac{2.303}{t} \log \left[ \frac{V_{\infty}-V_0}{V_{\infty}-V_t} \right] \text{ (min}^{-1}\text{)}$
0					
5					
10					
15					
20					
25					
30					

V<sub>∞</sub> = ---- cm<sup>3</sup>

Mean k<sub>2</sub> =

From graph  $\log \left[ \frac{(V_{\infty}-V_0)}{(V_{\infty}-V_t)} \right]$  v/s time, k<sub>2</sub> = ----- min<sup>-1</sup>

From graph log (V<sub>∞</sub>-V<sub>t</sub>) v/s time, k<sub>2</sub> = ----- min<sup>-1</sup>

Therefore activation energy,  $E_a = \frac{2.303 R \log (k_1/k_2) T_1 T_2}{T_2 - T_1}$   
= ----- kJ/mol

3) 2<sup>nd</sup> order reaction between 0.04M KI + 0.02M K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> v/s 0.0025M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> at lab temperature:

Time (min)	Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> added in cm <sup>3</sup>	(v <sub>∞</sub> -v <sub>t</sub> )	$\frac{1}{(v_{\infty}-v_t)}$	(v <sub>t</sub> -v <sub>0</sub> )	$k_2 = \frac{10(v_t-v_0)}{t \times M \times (v_{\infty}-v_0)(v_{\infty}-v_t)}$ (dm <sup>3</sup> /mol/min)
0					
5					
10					
15					
20					
25					
30					

V<sub>∞</sub> = ---- cm<sup>3</sup>

Mean k =

From the graph  $\frac{1}{(v_{\infty}-v_t)}$  v/s time, slope = -----

$$\therefore k = \frac{\text{Slope} \times 10}{M}$$

$$= \text{dm}^3/\text{mol}/\text{min}$$

## DETERMINATION OF HEAT OF NEUTRALISATION AND HEAT OF DILUTION

---

**Aim:** To determine the strength of HCl by thermometric titration, heat of neutralization of NaOH and HCl & NaOH and CH<sub>3</sub>COOH and heat of dilution of HCl.

**Principle:** Thermometric titration involves the measurement of the change of temperature of a solution. So during titration as a function of volume of the titrant added, the change in temperature occurs due to the heat evolved or absorbed during the reaction. The titrations are carried out in an adiabatic condition to minimize the heat transfer between the system and the surrounding. Thermometric titration can be applied to neutralization. The heat change of the reaction is given by,  $\Delta H^\circ = T\Delta S^\circ + \Delta G^\circ = T\Delta S - RT \ln k$

A known volume of HCl of exactly known concentration is allowed to react completely with a strong alkali in a dilute solution and rise in temperature was determined by knowing the heat capacity of calorimeter. The thermometer was immersed in the acid solution and alkali. By knowing their specific heat the amount of heat liberated can be obtained with known volume of HCl. The heat of neutralization can be calculated as; Heat of neutralisation =  $\frac{1000Q}{VC}$

Where Q = amount of heat evolved, C = concentration in eq / litre.

### Procedure:

25 cm<sup>3</sup> of 0.1N HCl was taken in a calorimeter which was fitted with stirrer and a thermometer. It was allowed to attain constant temperature, which is noted. A solution of 1N NaOH is taken in a burette. At every one minute interval of time, 0.5ml of NaOH was added to the HCl solution and the solution was stirred well. The temperature was noted during every addition. A graph was plotted by taking temperature along y – axis and volume of NaOH along x- axis.

A dry empty calorimeter and stirrer were weighed. 25 ml of 1N HCl was added into the calorimeter. The temperature was noted at every minute. At the end of the 5<sup>th</sup> minute 25 ml of 1N NaOH was added and stirred well. The temperature was noted at every minute. The Calorimeter containing NaOH and HCl was weighed. A graph of temperature v/s time was plotted. The same procedure was operated for acetic acid and NaOH of 1N each. A graph of temperature v/s time was plotted, from which  $\Delta T$  can be calculated.

Heat of dilution: About 25 ml of 1N HCl was taken in a calorimeter with a stirrer and thermometer. The temperature was noted at every minute. At the end of the fifth minute, 25 ml of distilled water was added and temperature was noted ay every minute. The calorimeter with mixture was weighed. A graph was plotted with temperature v/s time from which  $\Delta T$  can be calculated. Using these values, heat of dilution of HCl was determined.

- Result:**
1. Strength of HCl solution = -----N
  2. Heat of neutralization of NaOH + HCl = ----- kJ/mol
  3. Heat of neutralization of CH<sub>3</sub>COOH + NaOH = -----kJ/mol
  4. Heat of dilution of HCl = ----- kJ/mol



**Observation and Calculations:**

a) Thermometric titration: 25 cm<sup>3</sup> of 0.1N HCl v/s 1N NaOH:

Volume of NaOH (cm <sup>3</sup> )	Temperature (°C)	Volume of NaOH (cm <sup>3</sup> )	Temperature (°C)
0		3.5	
0.5		4	
1		4.5	
1.5		5	
2		5.5	
2.5		6	
3		6.5	

From the graph;

Volume of NaOH corresponding to the end point = ----cm<sup>3</sup>

$$\text{Strength of HCl } N_1 = \frac{(N \times V)_{\text{NaOH}}}{V_{\text{HCl}}} \\ = \text{----- } N$$

b) Heat of Neutralization:

Specific heat of copper, S<sub>1</sub> = 385 J/kg/K

Specific heat of water, S<sub>2</sub> = 4190 J/kg/K

1) 25ml of 1N HCl v/s 25ml of 1N NaOH:

Weight of empty calorimeter + stirrer W<sub>1</sub> = -----g

Weight of mixture + calorimeter + stirrer W<sub>2</sub> = -----g

Weight of mixture = W = W<sub>2</sub> – W<sub>1</sub> = -----g

Time (min)	Temperature (°C)	Time (min)	Temperature (°C)
0		6	
1		7	
2		8	
3		9	
4		10	
5		11	

$$\text{Heat of neutralisation, } \Delta H = \frac{W_1 S_2 + W S_1}{\frac{25 \times M}{1000}} \Delta T \\ = \text{-----kJ/mol}$$

2) 25 ml of CH<sub>3</sub>COOH + 25 ml of 1N NaOH:

Time (min)	Temperature (°C )	Time (min)	Temperature( °C )
0		6	
1		7	
2		8	
3		9	
4		10	
5		11	

Weight of empty calorimeter + stirrer W<sub>1</sub> = -----g

Weight of mixture + calorimeter W<sub>2</sub> = ----- g

Weight of mixture W = W<sub>2</sub>- W<sub>1</sub> = -----g

$$\text{Heat of neutralisation, } \Delta H = \frac{W_1 S_2 + W S_1}{\frac{25 \times M}{1000}} \Delta T$$

$$= \text{-----kJ/mol}$$

c) Heat of Dilution:

25 ml of 1N HCl + 25 ml of H<sub>2</sub>O:

Time (min)	Temperature (°C )	Time (min)	Temperature( °C )
0		6	
1		7	
2		8	
3		9	
4		10	
5		11	

Weight of stirrer + empty calorimeter W<sub>1</sub> = -----g

Weight of mixture + calorimeter W<sub>2</sub> = ----- g

Weight of mixture W = W<sub>2</sub>- W<sub>1</sub> = -----g

$$\Delta H = \frac{W_1 S_2 + W S_1}{\frac{25 \times M}{1000}} \Delta T$$

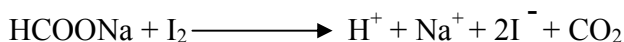
$$= \text{-----kJ/mol}$$

## KINETICS OF IODINE AND SODIUM FORMATE REACTION

**Aim:** To determine the rate constant and energy of activation of the reaction between iodine and sodium formate.

### Principle:

The reaction between sodium formate and iodine is as follows:



The kinetics of reaction can be considered under pseudo – unimolecular reaction. It can be studied by titration of the mixture against sodium thiosulphate using starch as an indicator. The titre value gives the value of (a – x). A graph of log (a – x) v/s time gives a straight line with a slope from which rate constant (k) can be calculated using the relation  $k = 2.303 \times \text{slope}$ . By knowing k at two different temperatures,  $E_a$  (energy of activation) can be calculated.

### Procedure:

25ml of 0.005 M  $\text{I}_2$  and 0.1M sodium formate solution was taken in two different reagent bottles and kept in thermostat at room temperature. After attaining same temperature, the two solutions were mixed and suddenly 5 ml of this was titrated against 0.0025M sodium thiosulphate using starch indicator. After every five minutes, similar titrations were carried out using ice, which arrest further reaction. This gives accurately the reaction of unreacted iodine and sodium thiosulphate.

The same experiment was repeated at high temperature.

The similar procedure was carried out for 0.0025M  $\text{I}_2$  and 0.1M HCOONa against 0.00125M sodium thiosulphate at lab temperature.

The graph of log (a – x) v/s t and  $\log\left(\frac{a}{a-x}\right)$  v/s t was plotted. The activation energy can be calculated by knowing rate constants at different temperatures.

### Result:

- 1) Rate constant for reaction between 0.005M  $\text{I}_2$  and 0.1M Sodium formate at -----°C
  - a) By calculation = -----min<sup>-1</sup>
  - b) From graph log(a-x) v/s time = -----min<sup>-1</sup>
  - c) From graph log(a/a-x) v/s time = -----min<sup>-1</sup>
- 2) Rate constant for reaction between 0.005M  $\text{I}_2$  and 0.1M Sodium formate at 35°C
  - a) By calculation = -----min<sup>-1</sup>
  - b) From graph log(a-x) v/s time = -----min<sup>-1</sup>
  - c) From graph log(a/a-x) v/s time = -----min<sup>-1</sup>
- 3) Energy of activation
  - a) By calculation = -----kJ/mol
  - b) From graph log(a-x) v/s time = ----- kJ/mol
  - c) From graph log(a/a-x) v/s time = ----- kJ/mol
- 4) Rate constant for reaction between 0.0025M  $\text{I}_2$  and 0.1M Sodium formate at 35°C
  - a) By calculation = -----min<sup>-1</sup>
  - b) From graph log(a-x) v/s time = -----min<sup>-1</sup>
  - c) From graph log(a/a-x) v/s time = -----min<sup>-1</sup>

**Observations and calculations:**

Reaction between 0.005M I<sub>2</sub> and 0.1M HCOONa v/s 0.0025M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution at lab temperature: ---  
-----°C

Time (min)	Volume of hypo (a - x) cm <sup>3</sup>	log (a - x)	$\log \left( \frac{a}{a-x} \right)$	$k = \frac{2.303}{t} \log \left( \frac{a}{a-x} \right) \text{ min}^{-1}$

Mean k = ----- min<sup>-1</sup>

Reaction between 0.005M I<sub>2</sub> and 0.1M HCOONa v/s 0.0025 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution at 35°C:

Time (min)	Volume of hypo (a - x) cm <sup>3</sup>	log (a - x)	$\log \left( \frac{a}{a-x} \right)$	$k = \frac{2.303}{t} \log \left( \frac{a}{a-x} \right) \text{ min}^{-1}$

Mean k = ----- min<sup>-1</sup>

Reaction between 0.0025M I<sub>2</sub> and 0.1M HCOONa v/s 0.00125 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution at lab temperature: ---  
-----°C

Time (min)	Volume of hypo (a - x) cm <sup>3</sup>	log (a - x)	$\log \left( \frac{a}{a-x} \right)$	$k = \frac{2.303}{t} \log \left( \frac{a}{a-x} \right) \text{ min}^{-1}$

Mean k = ----- min<sup>-1</sup>

## HEAT OF SOLUTION BY SOLUBILITY METHOD

---

**Aim:** To determine heat of solution of benzoic acid by solubility method.

### Principle:

The dissociation of solid into a liquid is normally accompanied by either absorption or evolution of heat during the reaction. The heat of solution is the heat evolved or absorbed when one mole of a substrate is dissolved in a solution, which is practically saturated. It differs from the heat of the solution at infinite dilution by an amount equal to the heat of dilution from saturated to infinite dilution.

The variation of solubility  $S$  with temperature is given by the equation,  $\frac{d(\ln S)}{dt} = \frac{\Delta H}{RT^2}$ , Where  $S$  is solubility in 1000g of solvent,  $\Delta H$  is the heat of solution,  $T$  is absolute temperature.

On integration we get,  $\log S = \frac{\Delta H}{2.303RT} + C$ ; where  $C$  is constant.

$\Delta H$  is assumed to be independent of temperature. A plot of  $\log S$  v/s  $1/T$  will be a straight line.  $\Delta H$  can be calculated from graph. Integrating above equation between  $T_1$  and  $T_2$ , we get,

$$\log S_2 - \log S_1 = \frac{\Delta H}{2.303R} \left[ \frac{1}{T_1} - \frac{1}{T_2} \right]$$

### Procedure:

Standardisation of NaOH : About 2.042g of potassium biphthalate crystals were weighed and dissolved in 100ml of standard flask. This solution was titrated against NaOH using phenolphthalein to find the strength of NaOH.

About 200 ml of distilled water was placed in a beaker and warmed to 50 – 60°C. The benzoic acid was added in the increasing amounts to the beaker with efficient stirring until small amounts of benzoic acid remains undissolved. Allowed to cool the solution and made four equal volumes of parts. Four reagent bottles containing saturated solution were placed at different temperature. When the solution attains constant temperature of the bath, 10ml of the solution was pipetted out. To prevent sucking of small crystals into the pipette, along with the solution, a small piece of cotton was tied to the tip of the pipette. After pipetting out the solution cotton was removed and solution was transferred to already weighed dry conical flask. The conical flask along with solution was reweighed.

The solution was titrated against standard NaOH (0.1N) using phenolphthalein as indicator. The procedure was repeated for other solutions which were at different temperatures. Finally the heat of solution was calculated using the equation,  $\Delta H = \frac{2.303RT_1T_2}{T_2-T_1} \log \left( \frac{S_2}{S_1} \right)$  kJ/mol.

A graph was drawn by plotting ( $-\log S$ ) against ( $1/T$ ) for different temperature from which  $\Delta H$  can be calculated.

### Result:

The heat of solution of benzoic acid; 1) From direct calculation = ---- kJ/mol  
2) From graphical method = ---- kJ/mol

**Observation and calculation:****Standardization of NaOH:**

Weight of potassium biphthalate taken = w = -----g

Therefore strength of potassium biphthalate =  $\frac{w \times 10}{\text{Mol.wt}}$

Trial No.	I	II	III
Final reading			
Initial reading			
Volume of NaOH consumed (cm <sup>3</sup> )			

Volume of NaOH solution consumed ----- cm<sup>3</sup>

Strength of NaOH solution  $N_1 = \frac{(V \times N)_{\text{pot.biphthalate}}}{V_{\text{NaOH}}}$

To determine solubility (S);

Temp	Wt. of empty conical flask x (g)	Wt. of flask + 10 ml of benzoic acid y (g)	Wt. of solution a = y - x (g)	Volume of NaOH V <sub>1</sub> (cm <sup>3</sup> )	Strength of acid solution $N_2 = \frac{(V_1 \times N_1)}{10}$	Wt. of benzoic acid, $b = \frac{N_2 \times E \times 10}{1000}$ (g)	Wt. of solvent in solution (a-b) g	Solubility $S = \frac{b \times 100}{(a-b)}$ g/dm <sup>3</sup>

Equivalent weight of benzoic acid = 122

Temp K	1/T x 10 <sup>-3</sup>	Solubility S	log S	1+log S	$\Delta H = \frac{2.303RT_1T_2}{T_2-T_1} \log \left( \frac{S_2}{S_1} \right)$ (kJ/mol)

Mean  $\Delta H$  = ----- kJ/mol

From Graph of (---+logS) v/s 1/T ; slope = -----

$\therefore \Delta H = 2.303 \times R \times \text{Slope}$

= ----- kJ/mol

## DETERMINATION OF MOLECULAR WEIGHT BY CRYOSCOPIC METHOD

---

**Aim:** To determine the molecular weight of the given unknown solute by measuring the depression of freezing point of a solid produced by the solute.

### Principle:

According to theory of dilute solutions the depression of freezing point produced by dissolving a definite mass of solute in a given mass of solvent so as to get a dilute solution is given by the equation;

$$\Delta T_f = \frac{1000 \times K_f \times W_2}{MW_1}$$

$W_1$  is the weight of solvent;  $W_2$  is that of solute;  $K_f$  is cryoscopic constant;  $M$  is the molecular weight of solute.

Thus  $M$  calculated as; 
$$M = \frac{1000 \times K_f \times W_2}{\Delta T_f \times W_1}$$

### Procedure:

#### 1) Determination of $K_f$ of solvent:

About 2.5g of Biphenyl was weighed accurately and transferred to the boiling tube fitted with thermometer and stirred. The boiling tube was clamped vertically with the help of a stand and kept in a hot water contained in beaker.

Biphenyl was melted continuously to obtain the clear solution. The tube was removed from water bath and was placed in an air jacket. The temperature was noted for every 30 seconds. The graph was plotted with temperature on y – axis and time on x- axis. The freezing point was then determined by the above plot.

About 0.3g of solute, whose molecular weight was known, was weighed accurately and placed into the above boiling tube. The mixture was heated over the bath so as to obtain a homogeneous solution. The freezing point of the solution was determined in a similar manner as above. Knowing the mass of solute and solvent, the molecular weight of the solute and cryoscopic constant was calculated.

Further a 0.2g of solute was added to the above solution and the freezing point was determined and  $K_f$  was calculated.

#### 2) Determination of molecular weight of unknown solute:

About 2.5g of solvent (biphenyl) was weighed accurately and transferred into the boiling tube. To this 0.3g of unknown solute was added. This mixture was heated to get a homogeneous solution. The freezing point of solution was determined in exactly similar manner explained above. Further a 0.2g of unknown solute was added to the above solution and the depression of freezing point was determined.

### Result:

- 1)  $K_f$  of the solvent ----- g/mol K  
Molecular weight of unknown solute ---- g/mol
- 2)  $K_f$  of the solvent ----- g/mol K  
Molecular weight of unknown solute ---- g/mol

**Observations and Calculations:**

Time (sec)	Temperature (°C)				
	2.5g of biphenyl	2.5g of biphenyl + 0.3g of naphthalene	2.5g of biphenyl + 0.5g of naphthalene	2.5g of biphenyl + 0.3g of unknown solute	2.5g of biphenyl + 0.5g of unknown solute

Depression of freezing point,  $\Delta T_f = \text{-----}$

Molecular weight of naphthalene = 128.017g

Weight of biphenyl  $W_1 = \text{-----g}$

Weight of naphthalene,  $W_2 = \text{-----g}$

$$K_f = \frac{MW_1 \times \Delta T_f}{1000 \times W_2} \text{ g/mol K}$$

$$M = \frac{1000 \times K_f \times W_2}{\Delta T_f \times W_1} \text{ g/mol}$$



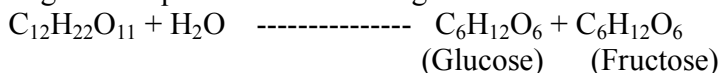
## KINETICS OF INVERSION OF CANESUGAR

---

**Aim:** To determine the rate constant of inversion of cane sugar in the presence of an acid using a polarimeter.

### Principle:

Cane sugar is optically active and dextro rotatory. In the presence of an acid it undergoes hydrolysis to give an equimolar mixture of glucose and fructose. The reaction is called inversion of cane sugar.



Glucose is dextro rotatory and fructose is leavo rotatory. Leavo rotatory of fructose is more than dextro rotation of glucose. Hence the mixture of glucose and fructose becomes leavo rotatory. The rate of reaction can be followed by measuring the angle of rotation of a plane polarized light using a polarimeter.

The rate of inversion depends only on the concentration of the sugar since concentration of the acid and water which is in large excess remains constant. Hence the reaction follows first order kinetics and obeys;  $k = \frac{2.303}{t} \log(a/a-x)$

Let  $r_o$ ,  $r_\infty$  and  $r_t$  be the angles of rotation by the plane polarized light by the solution, initially, at the end of reaction and after sometime 't' respectively. Then the initial concentration of cane sugar 'a' is proportional to the total roatation ( $r_o - r_\infty$ ) and concentration of sugar (a-x) at time 't' is proportional to the angle of rotatio0n ( $r_t - r_\infty$ ). Hence we can write  $k = \frac{2.303}{t} \log(r_o - r_\infty / r_t - r_\infty)$

### Procedure:

10g of sucrose was dissolved in 100ml of distilled water. Then 25ml of this 10% sucrose solution and 25ml each of 1N  $\text{H}_2\text{SO}_4$  and 1N HCl were taken in three separate dry reagent bottles. The three solutions were kept in thermostat, at lab temperature. The polarimeter tube was filled with distilled water and plane polarized light was set. Then 10% sucrose solution and 1N HCl were mixed. Polarimeter tube was rinsed with this solution and then filled with the same solution. The initial reading at zero minute was noted. Then subsequent readings after 5, 10, 15, 20, 25 and 30 minutes were taken. The remaining solution heated at 70°C for half an hour and cooled to the lab temperature to obtain  $r_\infty$  value.

The above experiment was repeated by taking 1N  $\text{H}_2\text{SO}_4$  and 10% sucrose solution. The graph,  $\log(r_o - r_\infty / r_t - r_\infty)$  v/s time and  $\log(r_t - r_\infty)$  v/s time were plotted for the two experiments and from the graph, rate constant of inversion of sucrose was calculated.

### Result:

1. the rate constant for the inversion of sugar catalyzed by  $\text{H}^+$  of the acid using 1N HCl acid;
  - a) By calculation -----  $\text{min}^{-1}$
  - b) By graph = -----  $\text{min}^{-1}$
2. the rate constant for the inversion of sugar catalyzed by  $\text{H}^+$  of the acid using 1N  $\text{H}_2\text{SO}_4$ ;
  - a) By calculation -----  $\text{min}^{-1}$
  - b) By graph = -----  $\text{min}^{-1}$

**Observations and calculations:**

1) 1N HCl + sucrose solution in polarimeter:

Zero reading for water =  $r_w$  =-----

Time (min)	Scale reading $r_s$	Angle of rotation $r_t = r_w - r_s$	$r_t - r_\infty$	$\log(r_t - r_\infty)$	$X = \log \frac{(r_o - r_\infty)}{(r_t - r_\infty)}$	$k = \frac{2.303 X}{t}$

Mean  $k$  = -----  $\text{min}^{-1}$ 2) 1N  $\text{H}_2\text{SO}_4$  + sucrose solution in polarimeter:Zero reading for water =  $r_w$  =-----

Time (min)	Scale reading $r_s$	Angle of rotation $r_t = r_w - r_s$	$r_t - r_\infty$	$\log(r_t - r_\infty)$	$X = \log \frac{(r_o - r_\infty)}{(r_t - r_\infty)}$	$k = \frac{2.303 X}{t}$

Mean  $k$  = -----  $\text{min}^{-1}$

## VERIFICATION OF FREUNDLICH AND LANGMUIR ADSORPTION ISOTHERM

---

**Aim:** To verify Freundlich and Langmuir adsorption isotherm using aqueous solution of acetic acid by charcoal.

### Principle:

The Freundlich Isotherm may be written as,  $\log (x/m) = \log K + 1/n \log C$ . Thus a plot of  $\log x/m$  v/s  $\log C$  will be a straight line.

The Langmuir adsorption isotherm can be expressed as  $c / (x/m) = 1/ab + c/b$ . Thus a plot of  $c / (x/m)$  v/s  $C$  will be a straight line from which the constants 'a' and 'b' can be found.

### Procedure:

Five well cleaned and dried stoppered reagent bottles were taken and labeled from 1 to 5. About 1g of powdered activated charcoal was weighed and added to each of bottles. Then exactly 5, 10, 15, 20 and 25ml of 0.1N acetic acid solution and 45, 40, 35, 30, and 25ml of distilled water were added to the reagent bottles numbered from 1 to 5 respectively, by means of burette. The stoppered bottles were shaken vigorously for about 20 minutes and kept aside for about 1 hour with frequent shaking.

The supernatant liquid of each of the bottle was filtered through a dry filter paper. The 10ml of filtrate from each bottle were taken and titrated against 0.1N NaOH solution using phenolphthalein as an indicator. Then equilibrium concentration of the acid in each case was calculated. A plot of  $[2 + \log x/m]$  v/s  $[3 + \log C_e]$  and  $[C_e / (x/m)]$  v/s  $C_e$  helps to verify the Freundlich and Langmuir adsorption isotherm.

### Result:

Verification of Freundlich adsorption isotherm;

i)  $n = \text{-----}$

ii)  $K = \text{----- min}^{-1}$

Verification of Langmuir adsorption isotherm;

i)  $a = \text{-----}$

ii)  $b = \text{-----}$

**Observations and Calculations:**

$$x = \frac{(C_o - C_e) \times V \times \text{Eq. wt of acid}}{1000}$$

x = amount of acid adsorbed.

$C_o$  = initial concentration of an acid

$C_e$  = equilibrium concentration of acid

V = total volume of solution in each bottle.

Equivalent weight of acetic acid = 63

Bottle no	Amount of charcoal in g (m)	Initial concentration of acetic acid ( $C_o$ )	Volume of filtrate	Volume of 0.1N NaOH $\text{cm}^3$	Equilibrium concentration of acid ( $C_e$ ) ( $N \times V$ )/10	Acetic acid adsorbed in g (x)
1	1	0.1	10			
2	1	0.2	10			
3	1	0.3	10			
4	1	0.4	10			
5	1	0.5	10			

$C_e / (x/m)$	$\log(x/m)$	$2 + \log(x/m)$	$\log C_e$	$3 + \log C_e$

Verification of Freundlich adsorption isotherm:

From graph;

- i) Slope =  $1/n$  = ----- thus  $n$  = -----
- ii) Intercept =  $\log K$  = ----- thus  $K$  = ---- $\text{min}^{-1}$

Verification of Langmuir adsorption isotherm:

- i) Slope =  $1/b$  = ----- thus  $b$  = -----
- ii) Intercept =  $1/(ab)$  = ----- thus  $a$  = -----

## PHASE DIAGRAM OF TWO COMPONENT SYSTEMS

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**Aim:** To determine the phase diagram of naphthalene – biphenyl system and determination of eutectic temperature.

**Principle:**

Solid and liquid can exist in an equilibrium at the freezing point. When a homogeneous binary mixture is cooled, a temperature is reached when a solid phase separated out. The composition of the two phases depends upon the nature of the two components. The method used here to obtain the freezing point diagram of binary mixture is thermal analysis.

The method consists of cooling of molten mixtures of the components and a graph was drawn with temperature v/s time. The graph was the cooling curve. This curve was a continuous curve as long as there is no phase change during cooling. When the solid separates, the rate of cooling decreases. In consequence the cooling curve exhibits a break at the freezing point.

**Procedure:**

2.5g of naphthalene was weighed accurately and placed in a boiling tube with a thermometer and stirrer. The boiling tube was clamped vertically with the help of stand kept in hot water contained in a beaker. Naphthalene was melted continuously to obtain clear solution. The tube was removed from the bath and placed in an air jacket. The stopwatch was started and the temperature was noted for every 30 seconds. The temperature at which the crystals are formed from the molten mass was noted. It is continued to get a constant temperature. The experiment was repeated by adding 0.5g, 1.0g and 1.5g of Biphenyl.

The test tube was washed with water, dried and 2.5g of biphenyl was taken and melted. The temperatures were recorded as above. Then 0.5g of Naphthalene was added and experiment was repeated. The process was also repeated for 1g of naphthalene. The cooling curves were plotted with temperature versus time for each mixture.

A graph of constant temperature along y-axis and percentage composition along x-axis was drawn. From this graph, the eutectic temperature of system and composition of Naphthalene and biphenyl at the temperature was determined.

**Report:**

1. Eutectic temperature of the Biphenyl-Naphthalene system =
2. Eutectic composition of the system
  - a. Biphenyl=
  - b. Naphthalene=

**Observations and Calculations:****Biphenyl - Naphthalene system:**

Time (sec)	Temperature (°C)			
	2.5g of biphenyl	2.5g of biphenyl + 0.5g of naphthalene	2.5g of biphenyl + 1.0g of naphthalene	2.5g of biphenyl + 1.5g of naphthalene

**Naphthalene - Biphenyl system:**

Time (sec)	Temperature (°C)			
	2.5g of biphenyl	2.5g of biphenyl + 0.5g of naphthalene	2.5g of biphenyl + 1.0g of naphthalene	2.5g of biphenyl + 1.5g of naphthalene

Weight of Naphthalene (g)	Weight of Biphenyl (g)	% composition of Naphthalene	% composition of Biphenyl	Freezing point(°C)

## CLEANSING POWER OF DETERGENTS

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**Aim:** To compare the cleansing power of two samples of detergents.

**Principle:** Detergent action or cleansing property of soaps and detergents on mainly upon the extent of reduction in the surface tension of water which they course. The greater the reduction, the more will the cleansing power of the detergents. Thus cleansing power of the detergents can simply be compared by measuring the surface tensions of their solutions containing equal weights in a fixed volume of water.

**Procedure:** About 0.5g of two samples of soaps were weighed and separately added to preheated 100ml of water in a beaker. The contents are stirred very gently to make the solution, taking care that no foam was formed. The solutions were allowed to stand so that insoluble impurities settled down and then filtered. The number of drops formed by a fixed volume of two solutions and water was determined using a stalagmometer filled with solution by very gentle suction, so that no foam is formed.

Suppose  $n$ ,  $n_1$ ,  $n_2$  are the number of drops for fixed volume of water, solution 1 and solution 2 respectively. Let  $d$ ,  $d_1$ ,  $d_2$  be their respective densities, then  $v_1/v = nd_1 / n_1d$  or  $v_1 = nd_1v / n_1d$   
Similarly,  $v_2 = nd_2v / n_2d$ , where  $v$ ,  $v_2$ ,  $v_3$  are surface tensions of water, solutions 1 and solutions 2 respectively. Since the solutions are very dilute, their densities will almost be same as that of water  
Hence,

$$v_1 = nv_1/n_1 \text{ or } v-v_1 = (1-n/n_1) v, \quad v_2 = nv_2/n_1 \text{ or } v-v_2 = (1-n/n_2) v$$

The ratio of cleansing power,  $v-v_1 / v-v_2 = (1-n/n_1) / (1-n/n_2)$

Result:

- 1) Surface tension of solution 1 =
- 2) Surface tension of solution 2=
- 3) Ratio of cleansing power =

**Observations and Calculations:**

Trial No	Number of drops of water	Number of drops for soap 1	Number of drops for soap 2

Room temperature =

Density of water at ----°C =

Surface tension of water at ----°C=

Weight of empty density bottle=

Weight of empty density bottle + water =

Therefore weight of water =