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Solvent Effect of Polyhydric Alcohol on the Biochemical Potential of Iso-amyl Methanoate

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Abstract: ***The biochemical potential of Iso-amyl methanoate is due to formation of methanoic acid as its solvolysis product. It is best highlighted by studying the kinetics of acid catalysed solvolysis of Iso-amyl methanoate in aquo-Glycerol reaction media having different concentration of Glycerol (trihydric alcohol) from 20-80% (v v) and at different temperatures ranging from 20 to 40°C. The depletion and enhancement observed respectively in iso-composition and iso-dielectric activation energies reveal that the transition state is solvated and initial state is desolvated with addition of Glycerol in reaction media, Almost unity value of the slope of the plots of log k values against log [H^+] values shows that the reaction follows A_{AC}^2 mechanism. From the values of iso-kinetic temperature, which comes to be 283.0, it may be concluded that in water-Glycerol reaction media, the reaction follows Barclay-Butler rule and there is weak but acceptable interaction between solvent and solute.***

Keywords: Protic Solvent, Substituted methanoate, Iso-dielectric, Iso-Kinetic, Specific solvation, Leffler's rule, Desolvation. Solvent-solute Interaction, Mechanistic pathways

I. INTRODUCTION

Various kineticists have reported in their works on the solvent effect of dipolar aprotic solvents like DMSO, DMF, Dioxan on the on the acid catalysed hydrolysis of aliphatic methanoate, but the studies on the solvent effect on the solvent effect of polyhyodric alcohol on the biochemical potential of Iso-amyl methanoate have not been communicated as yet.

Hence it is thought essential to study the solvent effect of a polyhidric alcohol Glycerol (trihydric alcohol) on the acid catalysed hydrolysis of Iso-amyl methanoate, as its biochemical use as flavouring agent seems to be very useful in the food technology and also in manufacturing of soft drinks.

II. EXPERIMENTAL & CALCULATION

Export quality of Iso-amyl methanoate of Fluka grade packed in Switzerland and Merck grade of Glycerol of high purity were used. The kinetics of the reaction was studied by adding 0.70 ml of ester through a German make graduated syringe pipette into 50 ml of 0.5 M solution of HCL. The reaction was found to obey the first order kinetic equation and the evaluated values of specific rate constants have been recorded in Table-1. The variation of log k values with mol% of Glycerol have been tabulated in Table – II and variation of log k with log [H_2O] of the reaction media are recorded in Table-III. From the slope of the plots of log k versus log [H_2O], the number of water molecules associated with the transition state of the reaction have been evaluated and are placed in Table -IV. The values of both the iso-composition and iso-dielectric activation energies have been enlisted in Table-V and Table-VI respectively. The numerical values of thermodynamic activation parameters were calculated using Wynne-Jones and Eyring equation and are synchronised in Table-VII. Effect of change in [H^+] ion concentration of the reaction media on the specific rate constants of the reaction has been shown in Table-VIII.

III. RESULTS AND DISCUSSION

Table-I shows that the rate constant values of the reaction decrease with increasing proportion of Glycerol in the reaction media. On plotting log k values against mol% of Glycerol (from recorded values in Table - II) as shown in Fig.-1, it is obvious that up to 17.80 mol% of Glycerol in the reaction media, the rate of the reaction falls rapidly but beyond (above) 17.80 mol% of Glycerol, the depletion in the rate follows the slow path. From Fig. 1, it is also apparant that with increase in temperature of the reaction, the rate of reaction decreases more sharply. The decreasing trend in the values of the specific rate constants needs to be discussed in the light of Hughes and Ingold[5] predictions according to which an increase in the dielectric constant values of the reaction media causes an increase in the rate when there is concentration or constructions of charges on the transition stage and causes a decrease in the rate when there is diffusion or destruction of charges on the transition stage.

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The values of dielectric constants of the reaction media go on decreasing with gradual addition of Glycerol. So the findings are fully in accordance with the qualitative prediction of Hughes and Ingold[5]. However, these findings are in agreement with the qualitative prediction of Laidler and Landskroener[6] and also with the earlier views of Singh & Sweety et al[9]., Kumari & Singh et al[8]. and also with the recent reports of Akanksha & Singh et al[9]. and Dubey & Singh et al.[10], who predicted that the rate of ion-dipolar reaction decreases with decrease in the dielectric constant values of the reaction media.

A. Evaluation Of Number Of Water Molecules Involved In The Formation Of Activated Complex Of The Reaction And Mechanism Of The Reaction

Robertson[11] has derived an equation,which is as:

$\log k = \log k' + n \log [H_2O]$

where 'n' is the solvation number i.e. the number of water molecules associated with the transition state of the reaction and is evaluated from the slopes of the plots of log k versus log $[H_2O]$.

Robertson et al[12]. suggested that value of 'n' for unimolecular reactions is fairly high while that of bimolecular reactions, it will be low.

From the recorded values of log k and log $[H_2O]$ in Table - III, the log k values were plotted against log $[H_2O]$ as shown in Fig.-2 and the evaluated values of the slopes of the straight lines have been enlisted in Table-IV.

From Fig.-2 it is clear that at each temperature two intersecting straight lines are obtained at log $[H_2O]$ value 1.475 which corresponds to 53.70% of water concentration (v/v) in aquo-Glycerol reaction media.

From the recorded values of the slopes of the plots of log k versus log $[H_2O]$ in Table -III, it is clear that below or before log $[H_2O]$ value 1.475, which corresponds to 53.70% of water in the reaction media, the number of water molecules associated with the activated complex increases from 0.290 to 0.842 with increase in temperature of the reaction from 20 to 40°C. Similarly, for above 53.70% water concentration in the reaction media, the number of water molecules involved in the formation of the activated complex increase from 0.450 to 1.565 with rise in temperature from 20 to 40°C

Overall, it is concluded that number of water molecules associated with the activated complex increase from 0.290 to 1.565 with rise in temperature from 20 to 40°C and from this trend, in the light of the guidelines of Robertson et al[12]. it is inferred that the mechanistic pathway of the reaction is changed from bimolecular to unimolecular with increase in water concentration or with decrease in Glycerol content of the reaction media and also with increase in the temperature of the reaction.

From the enhancing trend of number of water molecules involved in the formation of the activated complex, it is also inferred that on addition of Glycerol in the reaction media, the equilibrium of water is shifted from its dense form to bulky form.

$$(H_2O)_d \rightleftharpoons (H_2O)_b$$

Similar observations and inferences. have been earlier reported by Priyanka & Singh et al[13]. and Pathak & Singh et al[14]. and recently also by Kumar & Singh et al[15].s and Kaushalendra & Singh et al[16].

B. Solvent effect on Iso-composition Activation energy (E_C) of the Reaction

From the slopes of Arrehenius plots of log k versus $10^3/T$ as shown in Fig. - 3, the values of iso-composition activation energy (E_C) of the reaction have been evaluated and are tabulated in Table - V. From Table-V, it is obvious that E_C values go on decreasing from 101.43 to 62.85 kJ/mol with increasing the concentration of Glycerol from 20 to 80% (v/v) in reaction media. This trend is probably due to solvation changes taking place either at initial state level or at the transition state level or at the level of both the initial and transition states as reported earlier by Elsemongy et al[17]. in this field. Considering the extent of solvation to be a dominant factor, the following three factors seem to be responsible for decrease in E_C values with gradual addition of

C. Glycerol in the Reaction Media

The transition state is solvated and the initial state is desolvated,

(i) The transition state is desolvated less than the initial state, and (m) The transition state is solvated more than the initial state. The transition state being large cation (Ester H^+ is available more for solvation by Glycerol molecule than the initial state, so the first factor seems to be operative in this case and it is also supported by the decrease in entropy of activation (ΔS^*) of the reaction as shown in Table - VII. So situation first is the more plausible explanation for decrease in E_C values of the reaction as recorded in Table - V.

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It is, therefore, inferred that E_C values of the reaction go on decreasing due to solvation of the transition state and desolvation of initial state. Similar interretations for depletion in E_C value have also been reported earlier by Singh & Hafizee et al[18]. and recently by Kumar & Singh et al[19].

D. Effect of Solvent on the Iso-dielectric Activation Energy (E_D) of the Reaction

With a view to minimise the dielectric effect, the iso-dielectric activation energy was evaluated from the slopes of the Arrhenius plots of log K_D values (Obtained from interpolation of the plots of log k values against D values of the reaction media) against 1/T. The value thus obtained have been tabulated in Table-VI. From the Table-VI, it is found that E_D values go on increasing from 75.03 to 122.51 kJ/mol with increase in D values from D=50 to D=75. This trend of increase in E_D values is quite in agreement with changes (decrease) in E_C values of this reaction and also with the findings of Wolford[20] and with the earlier findings of Monalisa & Singh et al[21]., However, Singh & Singh et al[22]. and Pathak & Singh et al[23]. have also reported similar interpretations for the changes in Iso-dielectric Activation energy of the solvolysis reactions.

E. Solvent Effect on Thermodynamic Activation Parameters of the Reaction

The three thermodynamic parameters namely enthalpy of activation (ΔH*), free energy of activation (ΔG*) and entropy of activation (ΔS*) of the reaction were evaluated using Wynne-Jones and Eyring equation[4] and have been mentioned in Table -VII. From the values enlisted in Table-VII, it is clear that ΔG* values of the reaction go on increasing with simultaneous decrease in both the ΔH* and ΔS* values of the reaction.

In order to study the variation in these thermodynamic parameters more clearly, these were plotted against the mol% of Glycerol which have been shown in Fig. 4, 5 and 6 for ΔH*, ΔG* and ΔS* respectively. From Fig. - 4, 5 and 6, it is clear that ΔH*, ΔG* and ΔS* vary non-linearly to the considerable extent with the increasing concentration (mol%) of Glycerol and this is the indication of specific solvation taking place in the reaction media according to Saville and Hudson[24].

The ΔH* and ΔS* are complimentary to each other as the resulting net property of ΔG* in Table-VII is a well behaved function. From the values of ΔG* in Table-VII and also from the Fig.-5, it is clear that ΔG* is being little affected by the solvent composition (mol%). However, there is considerable enhancement (from 82.73 to 85.10 kJ/mol at 30°C) in ΔG* values.

From Table-VII, it is also clear that ΔG* values are found to increase simultaneously with depletion in both the ΔH* and ΔS* values.

From the thermodynamic relation-$\Delta G^* = \Delta H^* - T\Delta S^*$

it is apparant that enhancement in ΔG* values with simultaneous decrease in both of ΔH* and ΔS* values of the reaction is only possible when the extent of depletion in ΔS* values is greater than in ΔH* values. From these findings, it is concluded that the acid catalysed hydrolysis of Iso-amyl methanoate in aquo-Glycerol media is entropy controlled but enthalpy dominating reaction.

Similar findings and inferences have also been reported earlier by Kumar & Singh et al[25] and recently by R. T. Singh[26].

F. Solvent Effect on Solvent-Solute Interaction in the aquo-Glycerol Reaction Media

For highlighting solvent-solute interaction for a solvolysis reaction, Barclay and Butler[27] have correlated the enthalpy of activation (ΔH*) and the entropy of activation (ΔS*) by means of the relationship-

$$\delta m\ (\Delta H^*) = \beta\ \delta m\ (\Delta S^*)$$

where ß is a constant called iso-kinetic temperature and it is evaluated from the slope of plots of ΔH* values against ΔS* value.

From the recorded values of ΔH* and ΔS* in Table VII, ΔH* values were plotted against ΔS* (at 30°C) which is shown in Fig.-7. The plot consists of a straight line whose slope value has been evaluated to be 282.59 ≈ 283.0 which is less than 300. On the guidelines of Lefller, it is concluded that there is weak but considerable solvent-solute interaction for acid catalysed hydrolysis of Iso-amyl methanoate in aquo-Glycerol reaction media.

Such interpretations for solvent-solute interaction have also been reported earlier by Singh & Kumari et al[29] and recently by Singh & Hafizee et al[30].

G. Effect of change of [H^+] in Concentration of the Reaction Media

In order to investigate the effect of change in acid concentration of relation media (H^+ in concentration) on the acid catalysed hydrolysis of Iso-amyl methanoate in aquo-Glycerol media, experiments were performed to study the kinetics at various concentrations of HCl (from 0.1M to0.8 M), keeping the temperature, solvent composition and ionic strength of the reaction media constant.

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The reactions were carried out at 25°C in the reaction media having 20% (v/v) concentration of Glycerol and the evaluated values of specific rate constants have been tabulated in Table - VIII. From the tabulated values of log k and log $[H^+]$ in Table - VIII, log k values were plotted against log $[H^+]$ and has been shown in Fig.-8. From Fig. -8, it is clear that the plot is an excellent straight line showing linear dependence of rate of reaction on $[H^+]$ ion concentration. The slope of the log k versus log $[H^+]$ plot is evaluated to be 0.998 which is almost equal to unity. From this value of slope (unity), it may be inferred on the basis of the hypothesis of Zucker and Hammett[31] that acid catalysed hydrolysis of Iso-armyl methanoate in aquo-Glycerol media follows $A_{AC}{}^2$ mechanism.
Similar conclusions have also been reported earlier by Singh & Jha et al[3] and recently by Singh & Navendu et al[32] and Kumar, N[33].

Table - I

Specific rate constant values of Acid catalysed hydrolysis of Iso-amyl methanoate in water-Glycerol media
k x 10^3 in min^{-1}

Temp in °C	% of Glycerol (v/v)						
	20%	30%	40%	50%	60%	70%	80%
20° C	53.55	50.65	48.36	45.43	41.68	38.89	34.59
25° C	106.56	96.23	87.82	79.41	70.50	62.16	52.80
30° C	207.87	183.32	159.44	138.74	118.20	100.72	81.21
35° C	390.03	331.28	276.44	231.05	191.69	158.03	120.98
40° C	737.73	602.28	478.85	386.99	311.03	242.94	179.80

Table - II

Variation of log k values of the reaction at different temperatures with mol % Iso-amyl methanoate in water-Glycerol media.

% of Glycerol (v/v)	Mol % of Glycerol	3 + log k values				
		20°C	25°C	30°C	35°C	40°C
20%	5.81	1.7288	2.0276	2.3178	2.5911	2.8679
30%	9.56	1.7046	1.9833	2.2632	2.5202	2.7798
40%	14.12	1.6845	1.9436	2.2026	2.4416	2.6802
50%	19.79	1.6573	1.8999	2.1422	2.3637	2.5877
60%	27.01	1.6199	1.8482	2.0726	2.2826	2.4928
70%	36.54	1.5898	1.7935	2.0031	2.1987	2.3855
80%	49.67	1.5390	1.7226	1.9096	20827	2.2548

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Table - III

Variation of log k values of the reaction with log [H_2O] values of water-Glycerol system (media) at different temperatures.

% of Glycerol	% of H_2O	log [H_2O]	3 + log k values				
			20°C	25°C	30°C	35°C	40°C
20%	80%	1.6478	1.7288	2.0276	2.3178	2.5911	2.8679
30%	70%	1.5898	1.7046	1.9833	2.2632	2.5202	2.7798
40%	60%	1.5229	1.6845	1.9436	2.2026	2.4416	2.6802
50%	50%	1.4437	1.6573	1.8999	2.1422	2.3637	2.5877
60%	40%	1.3468	1.6199	1.8482	2.0726	2.2826	2.4928
70%	30%	1.2218	1.5898	1.7935	2.0031	2.1987	2.3855
80%	20%	1.0458	1.5390	1.7226	1.9096	2.0827	2.2548

Table - IV

Values of the slopes of the plots of log k versus log [H_2O] at different temperatures

Temperature in °C	Slope - I when log[H_2O] value is below 1.475	Slope - II when log[H_2O] value is above 1.475
20°C	0.290	0.450
25°C	0.437	0.778
30°C	0.595	0.966
35°C	0.730	1.251
40°C	0.842	1.565

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Table - V

Evaluated values of Iso-composition Activation Energy (E_c or E_{exp}) of the reaction in water-Glycerol media

% of EG	20%	30%	40%	50%	60%	70%	80%
E_c value in kJ/mol	101.43	95.59	89.08	82.84	76.90	71.04	62.85

Table - VI

Evaluated values of Iso-Dielectric Activation Energy (E_D) of the reaction at different desired 'D' values of the water-Glycerol media.

D values	D = 50	D = 55	D = 60	D = 65	D = 70	D = 75
E_D values in kJ/mol	75.03	84.52	90.71	101.48	112.52	122.51

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Table - VII

Consolidated Values of Thermodynamic Activation parameters (ΔH*, ΔG* and ΔS*) of the reaction in water-Glycerol system at different temperatures.

ΔH* and ΔG* in kJ/mol, ΔS* in J/K/mol

% of Glycerol (v/v)	Mol % of Glycerol	ΔH* in kJ/mol	20°C		25°C		30°C		35°C		40°C	
			ΔG*	ΔS*	ΔG*	ΔS*	ΔG*	ΔS*	ΔG*	ΔS*	ΔG*	ΔS*
20%	5.81	97.42	83.22	48.44	82.98	48.45	82.73	48.47	82.53	48.34	82.25	48.46
30%	9.56	92.46	83.36	31.08	83.23	30.98	83.05	31.08	82.82.94	31.91	82.78	30.95
40%	14.12	86.12	83.47	9.06	3.46	8.94	83.39	9.01	83.41	8.82	83.37	8.91
50%	19.79	80.07	83.62	- 12.12	83.71	- 12.20	83.75	- 12.14	83.87	- 12.33	83.93	- 12.39
60%	27.01	74.73	83.83	- 31.06	84.00	- 31.11	84.15	- 31.09	84.35	- 31.22	84.50	- 31.34
70%	31.54	68.22	84.00	- 53.87	84.31	- 54.01	84.56	- 53.92	84.84	- 53.96	85.14	- 54.06
80%	49.67	61.27	84.29	- 78.56	84.72	- 78.70	85.10	- 78.65	84.52	- 78.76	85.92	- 78.77

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Table - VIII

Effect of $[H^+]$ on the Specific rate constant values of Acid catalysed hydrolysis of Iso-amyl methanoate in water-Glycerol media at constant Ionic strength (μ = 0.9)

Concentration of Glycerol = 20% (v/v) Temp. - 25°C

$[H^+]$	[KCl]	μ	k x 10^3 in min^{-1}	2 + log$[H^+]$	3 + log k	value of the slope of the plot of log k vesus log $[H^+]$
0.10	0.80	0.90	21.24	1.0000	1.3271	
0.15	0.75	0.90	31.64	1.1761	1.5003	
0.20	0.70	0.90	42.38	1.3010	1.6272	
0.25	0.65	0.90	53.47	1.3979	1.7281	
0.30	0.60	0.90	63.43	1.4771	1.8023	0.998
0.40	0.50	0.90	84.10	1.6021	1.9248	
0.50	0.40	0.90	106.56	1.6990	2.0276	
0.60	0.30	0.90	128.03	1.7782	2.1073	
0.70	0.20	0.90	148.46	1.8451	2.1716	
0.80	0.10	0.90	171.83	1.9030	2.2351	

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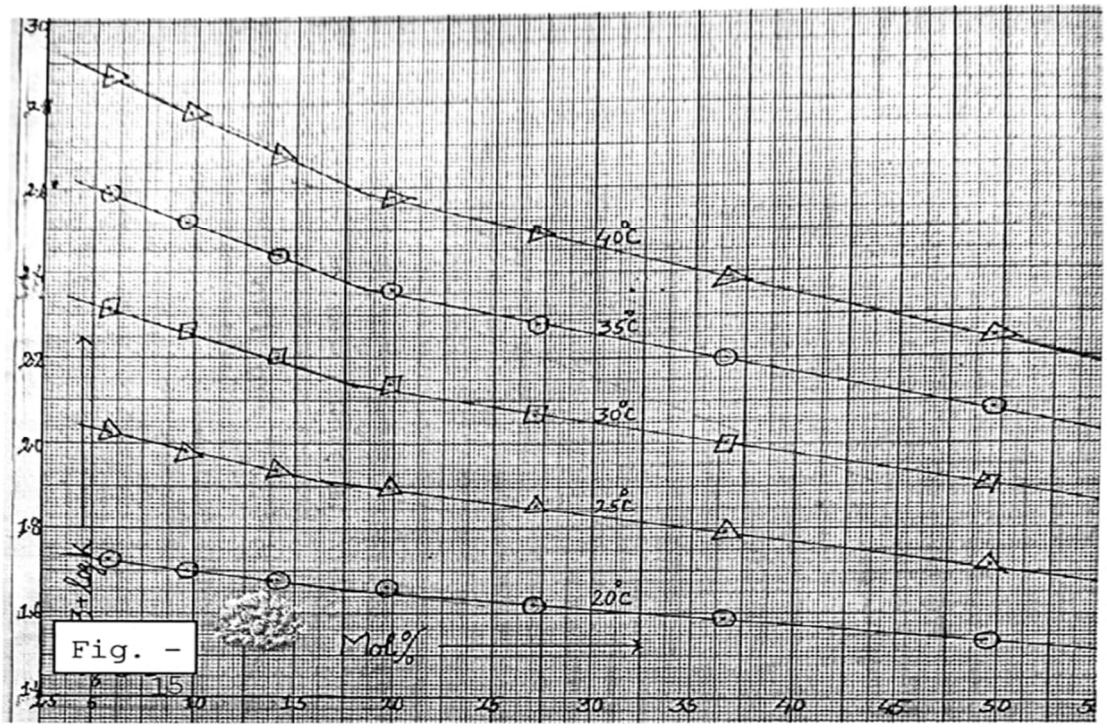


Fig. – 15

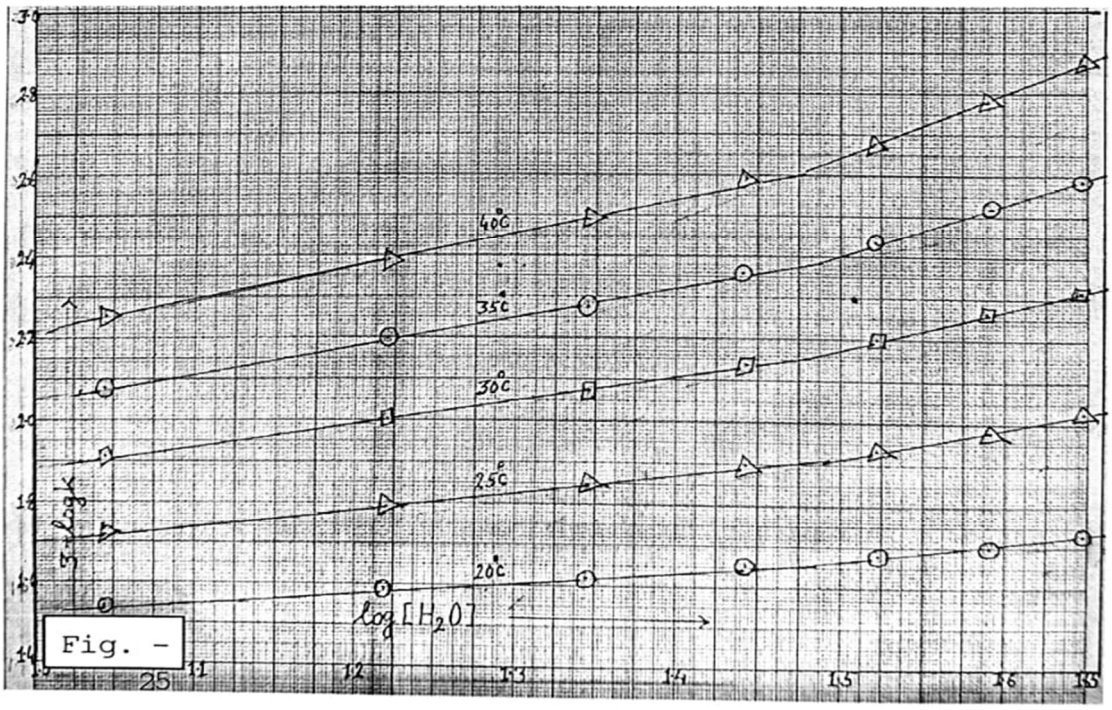


Fig. – 25

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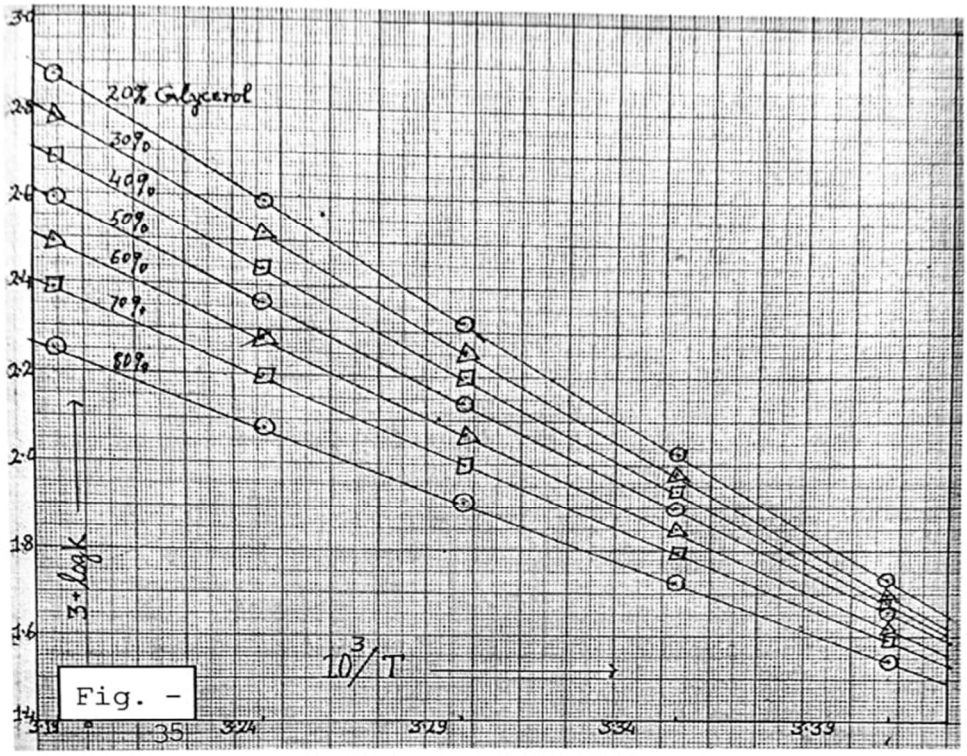


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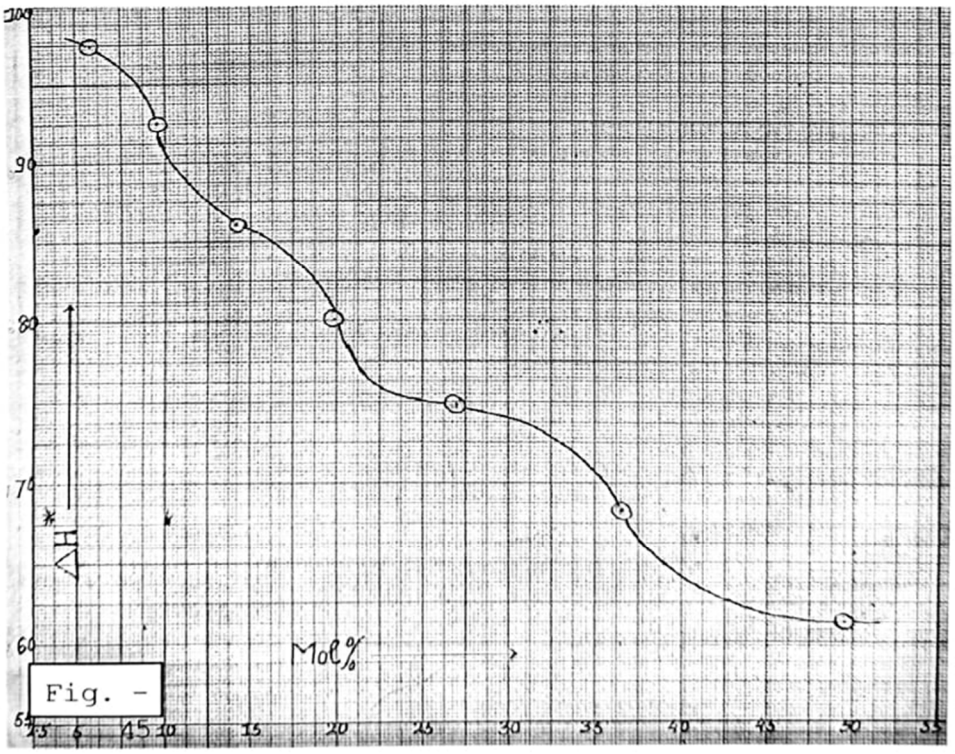
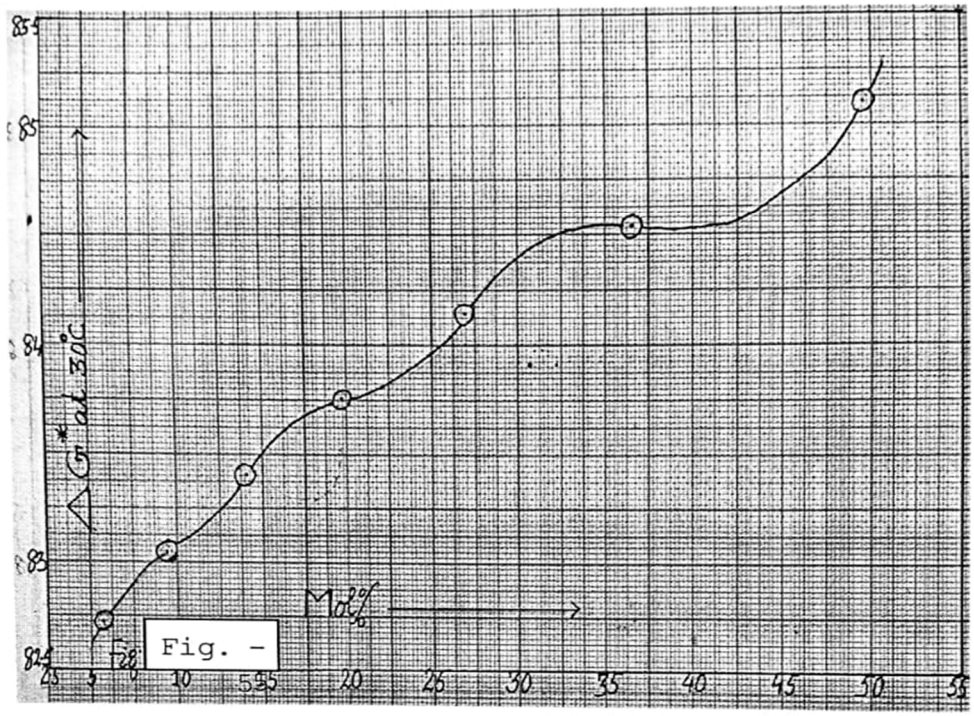
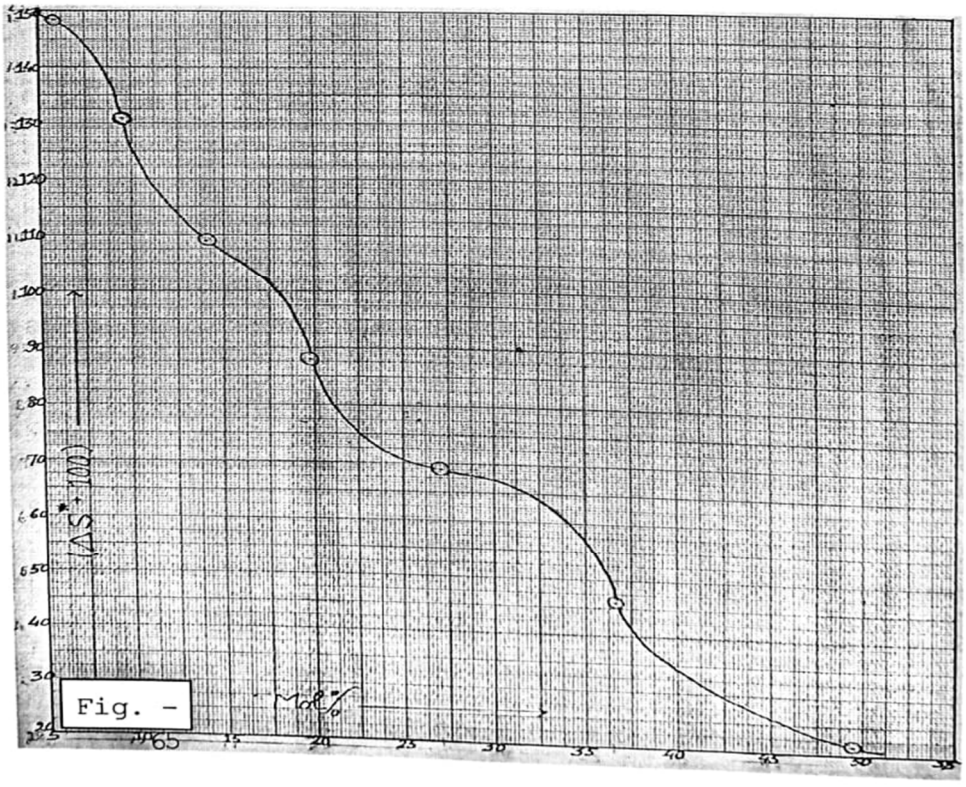


Fig. –

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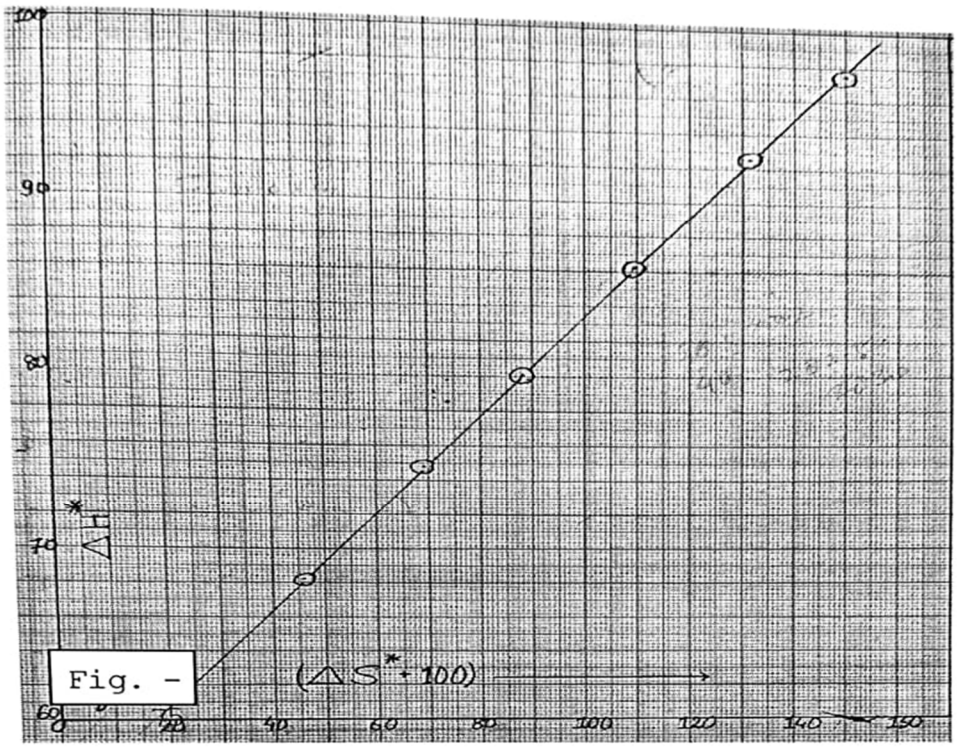

100
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80
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ΔH^*
$(\Delta S^* + 100)$
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Fig. –

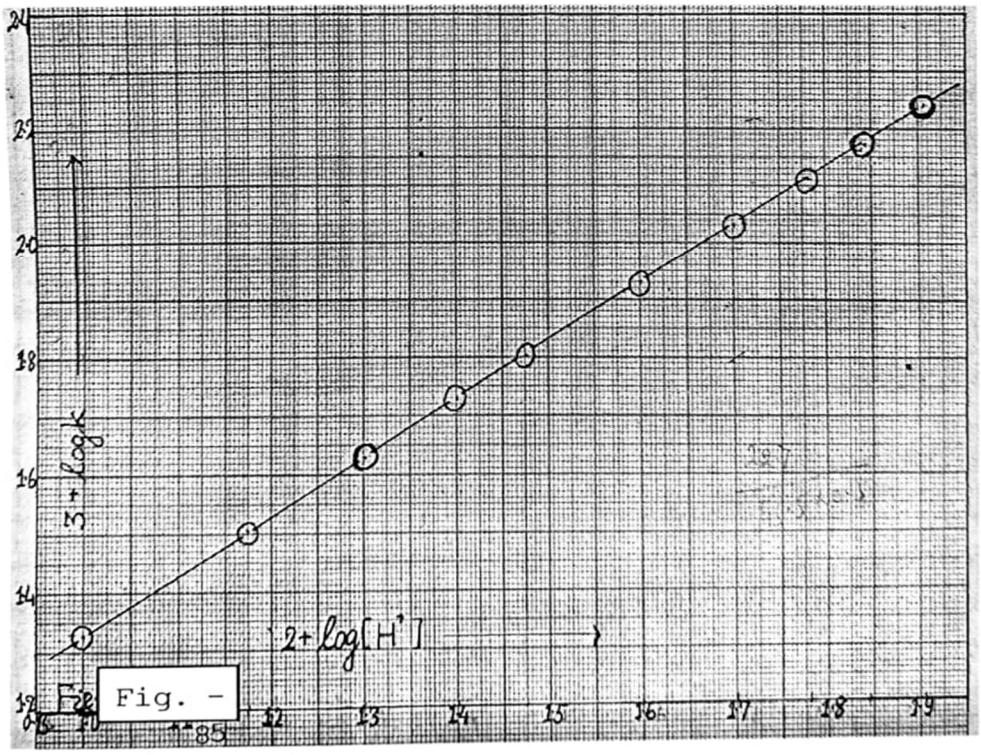
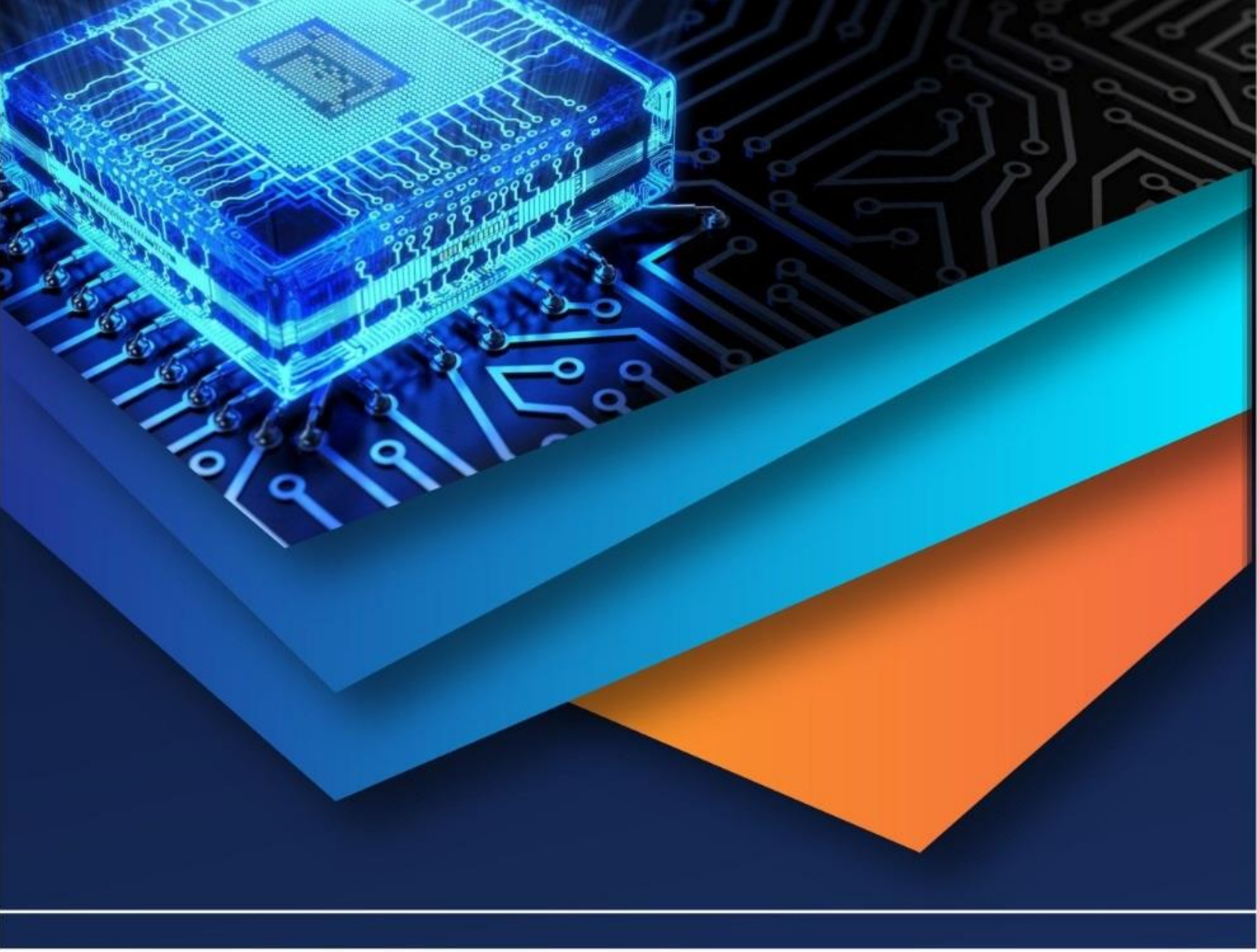

24
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12
$\log k + 5$
$2 + \log[H^+]$
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19

Fig. –

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