

Study of Free Energy Transfer in 10, 20 and 30% Dioxane in Dioxane-Water System for Deprotonation of Glycine and Related Thermodynamic Quantities At 298.15 K by E.M.F Measurement

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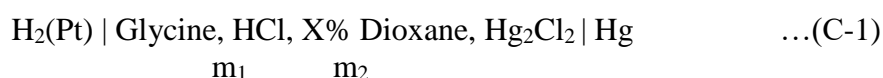
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ABSTRACT

The deprotonation constant of glycine in 10%, 20% and 30% dioxane in dioxane-water system at 298.15 K was determined using the cell:



and e.m.f. of the cell was given by

$$E = E^0 - (2.303RT/F) (\log m_{\text{H}^+} m_{\text{Cl}^-} + \log \gamma_{\text{H}^+} \gamma_{\text{Cl}^-}) \quad \dots(1)$$

where $\log \gamma_{\text{H}^+} \gamma_{\text{Cl}^-} = - (2A' \sqrt{\mu} / (1 + \sqrt{\mu})) + \beta_1 \mu$ (Modified Davies Equation) $\dots(2)$

which has been given by B Prasad in our laboratory called modified Davies equation for calculating activity coefficient in the system and hence deprotonation constant of glycine can be calculated by calculating m_{H^+} given by the formula:

$$\log m_{\text{H}^+} = (E^0 - E)/K - \log m_{\text{Cl}^-} + (2A' \sqrt{\mu} / (1 + \sqrt{\mu})) - \beta_1 \mu \quad \dots(3)$$

Therefore Free energy transfer can be calculated for 10, 20 and 30% dioxane in dioxane-water system during determination of deprotonation constant of glycine at 298.15 K.

Keywords : Deprotonation Constant, Dioxane-Water, Modified Davies Equation, Solvent Effect, Free Energy Transfer.

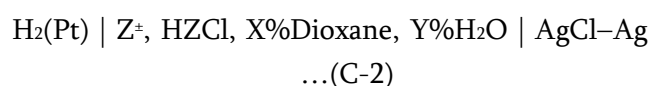
I. INTRODUCTION

Physico-chemical studies of electrolytic solution have been topic of great interest and challenging one since the development of chemical science. Electrolytes show varying properties in different solvents

depending upon their nature either singly or in mixed one and aqueous solution¹⁻² have been extensively studied in comparison to aquo organic system. Although water is good and exceptional solvent for large number of solutes but need of the hour compelled workers to change attention from aqueous

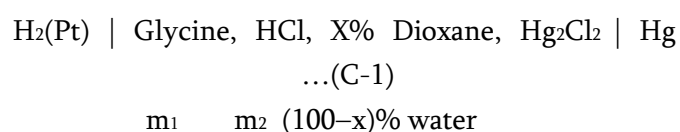
system to aquo-organic system because many chemical reactions which could not be carried out in aqueous medium would be possible in non-aqueous or in aquo-organic system and in this process the course, mechanism and kinetics of the reactions changes drastically and there are so many instances that if a reaction is not possible in aqueous solution but the same is possible in organic medium and when medium is changed from water to non aqueous^{3,4}, visible changes have been observed in the reactivity of the ions like solubility⁵⁻⁶, oxidation-reduction reaction and acid-base behavior.⁷

The dissociation constant of monoprotic and diprotic acid have been determined in water and dioxane water system by different workers⁸⁻¹⁰ using cells without liquid junction potential besides deprotonation constant of amino acids¹¹⁻¹⁵ in water have been determined using cells of the same type. Apart from these experiments, the dissociation constant of other types of electrolytes have been determined in aqueous solution potentiometrically using cell without liquid junction potential by different workers.¹⁶⁻²² The deprotonation constant of glycine has been determined by Birdsall²³ in 20, 45 and 70% dioxane content of dioxane-water system using cell of the following type:



Therefore, it is well established that e.m.f measurement with a cell having no liquid junction potential gives the most accurate result. Deprotonation constant indicates the nature of the substance to donate hydrogen ions. Furthermore, deprotonation constant is also important for a chemical sciences, biochemistry and pharmaceutical industry and by studying deprotonation constant we can get information about fraction of biomolecules to get absorbed and about ionization behavior and so pK_a is of great importance. Further pK_a measurement

enables us determination of proportion of the molecules in the ionized or deionized state. Apparent pK_a is an experimentally determined value of molecules or bio-molecules, the value is the pH at which the number of ionized and deionized are equal. Knowing the importance of pK₁ value, curiosity arose to determine the pK₁ value of glycine in 10, 20 and 30% dioxane in dioxane-water system at 298.15 K by setting up the following cell.



The main problem in determination of ionization constant of an electrolyte is value of activity coefficient of the species involved in the medium under study so that ion solvent interaction must be taken into consideration resulting it would be helpful in understanding the structure of the medium. In this regard, a modified Davies equation has been found more suitable, given by Prasad²⁴ in our laboratory.

$$\log \gamma_i = -A' z_i^2 \sqrt{\mu} / 1 + \sqrt{\mu} + \beta_1 \mu \quad \dots(4)$$

and for our experiment upto 30% dioxane content activity coefficient of HCl is given by the following equation.

$$\log \gamma_{\text{H}^+} \gamma_{\text{Cl}^-} = -2A' \sqrt{\mu} / 1 + \sqrt{\mu} + \beta_1 \mu \quad \dots(5)$$

where A' is Debye Huckel constant and β_1 is additive parameter.

II. Experimental Detail

The acidic dissociation constant of Glycine for 10, 20 and 30% dioxane (m/m) in dioxane-water system was determined by the EMF measurement for the cell (C-1) at 298.15 K. The Glass cell was set-up in duplicate. The electrodes were fitted in

interchangeable cones and could be put in and test from the cell as and when needed. All chemicals used for the purpose are of AR or GR quality. Buffer solution of glycine was prepared by mixing one molar solution of glycine and half molar of hydrochloric acid. After attaining equilibrium the EMF value have been noted for 10, 20 and 30% dioxane in dioxane-water system at 298.15 K and the values are mean of two reading of the two cell after correction for barometric pressure, vapour pressure and bubbler depth.

III. Result and Discussion

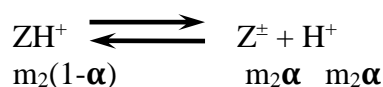
It is assumed that the following equilibria take place during deprotonation of protonated Glycine as.

population, increase in the agricultural productivity is urgent to meet rising food needs [3,4]. Chemical pesticides are considered the main component in protecting agricultural products in the field and store to maintain crop yield and quality [5,6]. Fruits and vegetables are important nutritional components in different societies. They are recommended to be eaten fresh, unpeeled, and unprocessed for their high nutritional value and content of minerals, vitamins, fibers, and antioxidants [7,8].

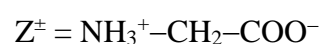
On the other hand, food (especially fruits and vegetables) is one of the main ways through which humans are exposed to pesticides, at a rate five times higher than other methods such as air and water [9,10]. Accordingly, efforts to ensure a sustainable use of chemical pesticides to avoid the increase of pesticide levels in the environment and food commodities are necessary. Studies have demonstrated that exposure to pesticides has dose-related chronic and acute toxicity in humans through different mechanisms including deregulation of transporters or enzymes involved in xenobiotic metabolism [11,12]. District Singrauli also affected with variable environmental issues in Madhya Pradesh State of

India, due their established industries. Local fruits and vegetable grower allowed selling these growing crops in local market after needed pesticides treatments [13,14]. These crops of fruits and vegetables consume through the local population and directly indirectly exposed to them. Vegetables frequently ensure good financial return per area unit, specially because consumers often prefer products with good aspect as if that would guarantee their health and quality [15]. The required rates of application of pesticides may vary, under different agricultural and climatic conditions, from country to country, and between regions of the same country [16].

Hence, the aim of this study was to determine pesticide residues in vegetable and fruit samples in local markets in Singrauli region and to show the differences and frequencies in two kinds of pesticides detection. The most common two pesticides and the type of crops their findings are also shown. This study will help understanding of the most applied these taken two pesticides on vegetables and fruits.



where $\text{ZH}^+ = \text{NH}_3^+-\text{CH}_2-\text{COOH}$



and $\text{Z} = \text{NH}_2-\text{CH}_2-\text{COOH} = \text{glycine}$

If α be the degree of dissociation of ZH^+ in the buffer of Glycine and hydrochloric acid

$$m_z^\pm = m_1 + m_2\alpha$$

$$m_{\text{H}^+} = m_2\alpha$$

$$m_{\text{ZH}^+} = m_2(1-\alpha)$$

$$\mu = \frac{1}{2} m_{\text{H}^+} + \frac{1}{2} m_{\text{ZH}^+} + \frac{1}{2} m_{\text{Cl}^-}$$

$$= \frac{1}{2} m_2 \alpha + \frac{1}{2} m_2 (1-\alpha) + \frac{1}{2} m_2$$

$$\mu = m_2$$

In the cell C-1 and arbitrary value is assigned to μ equation (3):

and taking E^0 , β and A' , m_{H^+} is calculated from

$$\log m_{H^+} = (E^0 - E)/K - \log m_{Cl^-} + (2A' \sqrt{\mu}/(1 + \sqrt{\mu})) - \beta_1\mu$$

and when this m_{H^+} value is put in $m_{H^+} = m_2\alpha$ and $\mu = m_2$, a new value of μ is obtained. This value of μ is again put in the equation (3).

$$\log m_{H^+} = (E^0 - E)/K - \log m_{Cl^-} + (2A' \sqrt{\mu}/(1 + \sqrt{\mu})) - \beta_1\mu$$

till constant value of m_{H^+} is obtained upto 6th places of decimal and from this value of m_{H^+} , $m_{Z^{\pm}}$ and m_{ZH^+} were calculated.

From dissociation of protonation glycine we have,

$$K_1 = (m_{H^+} m_{Z^{\pm}}/m_{ZH^+}) \cdot (Y_{H^+} Y_{Z^{\pm}}/ Y_{ZH^+}) \quad \dots(6)$$

or $\log K_1 = \log K_1(A) + \log (Y_{H^+} Y_{Z^{\pm}}/ Y_{ZH^+}) \quad \dots(7)$

$K_1(A)$ = Apparent Dissociation Constant

K_1 = Real Dissociation Constant

$$\log K_1 = \log K_1(A) - ((2A'\sqrt{\mu}) / (1 + \sqrt{\mu})) + \beta_1\mu \quad \dots(8)$$

$$\log K_1(A) = m_{H^+} m_{Z^{\pm}} / m_{ZH^+} = m_{H^+} \cdot (m_1 + m_{H^+}) / m_2 - m_{H^+} \quad \dots(9)$$

where $m_{Z^{\pm}} = m_1 + m_2\alpha$

$$m_{H^+} = m_2\alpha$$

$$m_{ZH^+} = m_2 (1 - \alpha)$$

$$\beta_1 = \beta_{H^+} + \beta_{ZH^+} + \beta_{Z^{\pm}}$$

$$\log K_1(A) = \log K_1 + (2A'\sqrt{\mu}) / (1 + \sqrt{\mu}) - \beta_1\mu \quad \dots(10)$$

or $\log K_1 - \beta_1\mu = \log K_1(A) - (2A'\sqrt{\mu}) / (1 + \sqrt{\mu}) \quad \dots(11)$

$$\log K_1 - \beta_1\mu = q$$

where $q = \log K_1(A) - (2A'\sqrt{\mu}) / (1 + \sqrt{\mu})$

or $q = \log K_1 - \beta_1\mu$

or $-q = pK_1 + \beta_1\mu \quad \dots(12)$

Now, $-q$ is plotted against μ for 10, 20 and 30% dioxane-water system at 298.15 K.

Table 1: Plot of $-q$ ($-\log K_1(A) + (2A^1\sqrt{\mu}) / (1 + \sqrt{\mu})$) against μ for 10% dioxane in dioxane-water system at 298.15 K for cell C-1.

μ	$-q$
0.0112205	2.41
0.018249	2.37
0.028735	2.32
0.039457	2.26
0.053289	2.19
0.067815	2.133

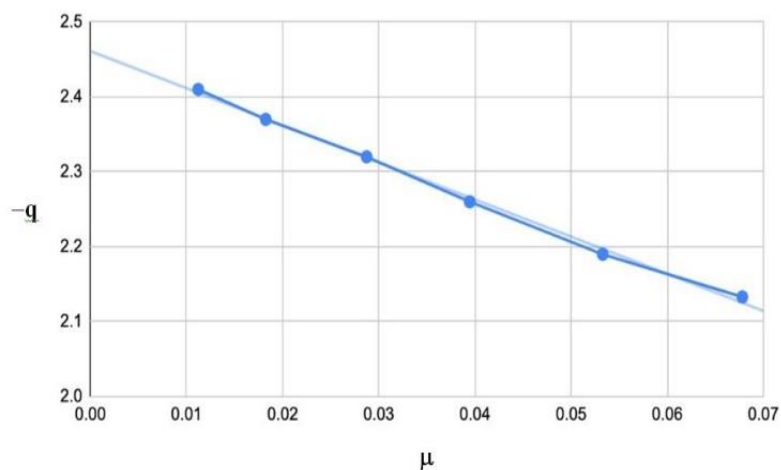


Fig. 1: The plot of $-q$ against μ for the dissociation constant of Glycine in 10% dioxane at 298.15 K.

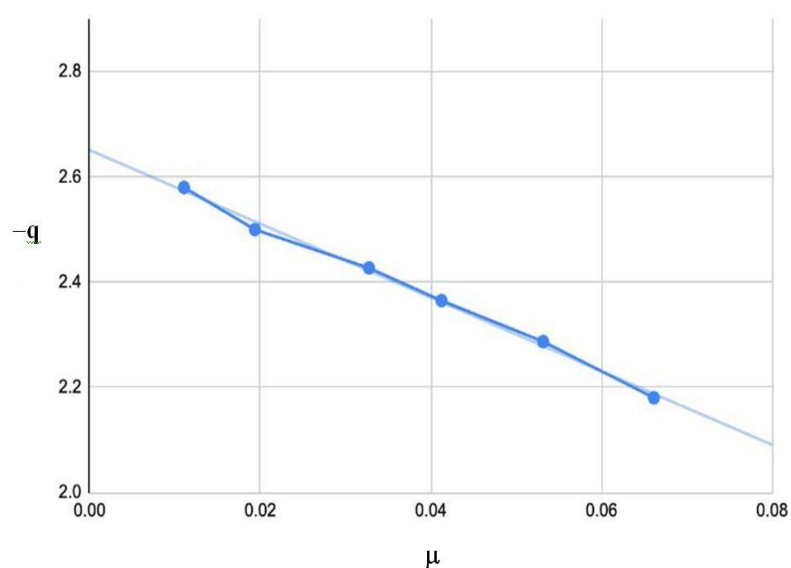


Fig.: 2 The plot of $-q$ against μ for the dissociation constant of Glycine in 20% dioxane at 298.15 K.

Table 2: Plot of $-q$ ($-\log K_1(A) + (2A^1\sqrt{\mu}) / (1 + \sqrt{\mu})$) against μ for 20% dioxane in dioxane-water system at 298.15 K for cell C-2.

μ	$-q$
0.01106	2.58
0.019367	2.5
0.03275	2.427
0.04124	2.365
0.053156	2.287
0.06613	2.18

Table 3: Plot of $-q$ ($-\log K_1(A) + (2A\sqrt{\mu}) / (1 + \sqrt{\mu})$) against μ for 30% dioxane in dioxane-water system at 298.15 K for cell C-2.

μ	$-q$
0.01236	2.654
0.022567	2.572
0.030251	2.521
0.045296	2.418
0.057355	2.312
0.070215	2.23

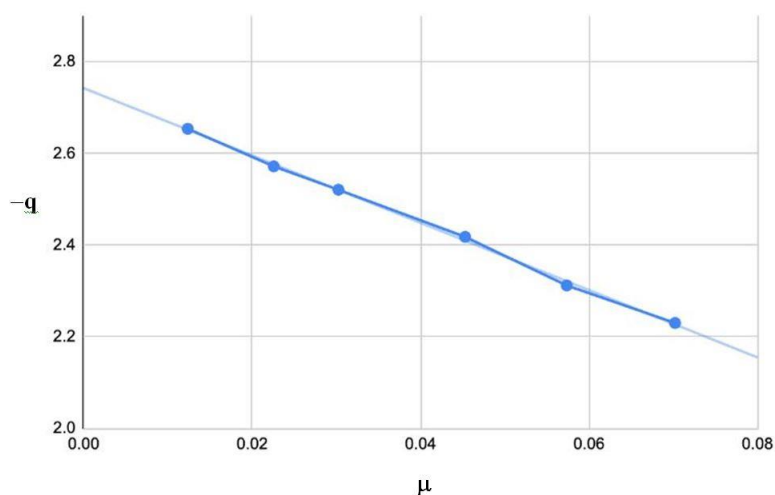


Fig. 3: The plot of $-q$ against μ for the dissociation constant of Glycine in 30% dioxane at 298.15 degree K.

In Fig. 1, 2 and 3 the plots have been extrapolated at $\mu = 0$ and we get the value of pK_1 in 10, 20 and 30% dioxane in dioxane-water system at 298.15 K and the related thermodynamic quantities ΔG^0 , ΔH^0 and ΔS^0 were calculated at 298.15 K and recorded in Table 4.

Table 4: The values of pK_1 , ΔG^0 , ΔS^0 and ΔH^0 in 10, 20 and 30% dioxane in dioxane-water system at 298.15 K for the cell C-1.

% Dioxane-water	pK_1	$-\beta_1$ KJ mol ⁻¹	ΔG^0 KJ mol ⁻¹	$-\Delta S^0 \times 10^3$ KJK ⁻¹ mol ⁻¹	ΔH^0 KJ mol ⁻¹
10	2.45	5.84	13.998	12.497	10.272
20	2.62	6.59	14.945	39.0673	3.295
30	2.74	7.29	15.680	36.168	5.493

By the method of least square²⁷, the values of deprotonation constant are given by the equation:

$$\log K_1 = -A^*/T + D^* - C^*T \quad \dots(13)$$

and for 10, 20 and 30% (m/m) dioxane in dioxane-water system are given by the following equations:

For 10% (m/m) dioxane

$$\log K_1 = -4179.255/T + 23.723225 - 0.0409778T \quad \dots(14)$$

For 20% (m/m) dioxane

$$\log K_1 = -678.3820/T + 1.4152361 - 0.0057964T \quad \dots(15)$$

For 30% (m/m) dioxane

$$\log K_1 = -2117.1090/T + 10.4921639 - 0.0205886T \quad \dots(16)$$

And value of A*, D* and C* have been recorded in Table (5) for 10, 20 and 30% (m/m) dioxane in dioxane-water content.

The values pK₁ calculated from these equations are in good agreement with the experimental values as shown in Table (4). The values of pK₁ increases with the increase in dioxane content of dioxane-water system as evident from Table (4). These results are similar to the results of other workers^{20,21,23,25,26,28} in dioxane-water system and it is crystal clear an effect due to lowering of dielectric constant of the medium under consideration.

Table 5: The value of A*, D* and C* calculated by least square²⁰ method.

mass %Dioxane	A*	D*	C*
10	4179.2550	23.7823222	0.0409778
20	687.3820	1.4152361	0.0057964
30	2117.1090	10.4921639	0.0205886

Table 6: Comparison of values of pK₁ for general equation and experimental value at 298.15 K in 10, 20 and 30% dioxane in dioxane-water system.

mass %Dioxane	Experimental value	General equation value
10	2.45	2.45
20	2.62	2.61
30	2.74	2.74

Thermodynamic quantities for deprotonation of glycine in 10, 20 and 30% dioxane in dioxane-water system can be calculated by the following formula:

$$\Delta G^0 = -19.1438 (-A^* + D^*T - C^*T^2) \quad \dots(17)$$

$$\Delta H^0 = -19.1438 (-A^* - C^*T^2) \quad \dots(18)$$

$$\Delta S^0 = -19.1438 (D^* - 2C^*T) \quad \dots(19)$$

Study of Free Energy Transfer in the system

Free energy transfer from water to aquo-organic system is measure of ion-solvent interaction. The purpose of calculating free energy transfer is mainly study of effect of the aquo-organic solvent upon structure of zwitter ion hence may be used for purification and separation of amino acids. The free energy transfer of cation and anion from water to aquo-organic system usually negative and positive respectively.²⁹⁻³¹

To study free energy transfer, it is necessary to understand structural arrangement of zwitter ion with the dioxane-water system. Water has quasi-crytalline structure at ordinary temperature and a dynamic

equilibrium probably exists between 3-dimensional hydrogen bonded clusters and the denser monomer whereas dioxane has more basic character than water due to its having two methylene groups. Glycine exists in zwitter ion form and there is possibility of interaction between glycine zwitter ion and dioxane and there may be large number of dioxane molecules may be involved forming layered structure with glycine and hence no further decrease in dielectric constant. It is expected that addition of small amount of dioxane may give rise to two effect, the first dioxane can be accommodate with solvent structure, it may strengthen the water structure; since dioxane is better proton acceptor; in case it is not accommodated because of its large size it may cause a breakdown in 3-dimensional structure but it is evident from viscometric study of dioxane water mixture that dioxane is not accommodated in the solvent structure and so it breakdown the 3-dimensional structure of water. Therefore, it is very difficult to ascertain exact nature of detailed molecular structure in different of glycine mass percent content of the organic solvent.

According to Frank and Evan³², when a neutral molecule dissociates, it forms a charged ions which immobilizes solvent molecules causing decrease in entropy. We are studying dioxane-water system in which dissociation of glycine takes place and we know that dioxane is more basic than water and it enhances the acidity of water molecules to which it is bonded³³; this is because of inductive effect of two methylene groups in each side of oxygen atoms of dioxane molecules. Further, dioxane molecules form hydrogen bonding with water molecules³⁴, this causes dioxane molecule becomes more basic and less acidic than water. It is evident that a cation will interact more strongly with the oxygen water system moiety in dioxane-water system while anion will interact more strongly with other hydrogen atom of methylene group of dioxane. This type of ion solvent interaction will cause a primary solvation sheath around the ions. This is the region "A" of Frank and Wen.³⁵ In the region "A" around the ion, water molecules are in a sort of frozen state while a bit away there is a normal 3-dimensional water structure called region "C". In between region "A" and region "C", there is region "B" where the water molecules are disorganized but in case of aquo-organic solvent Frank and Wen simple model needs to be modified because of structural change of water molecule. Due to addition of the organic solvent structural change of water may also depend upon mass % of organic component added. But it is very difficult to ascertain the exact nature of detailed molecular structure in different mass % of organic content and how it changes with change of mass % of organic content. Therefore, it becomes a tedious job in these circumstances to correlate quantitatively the variation of ΔG^0 , ΔH^0 and ΔS^0 both change in concentration of organic components and with change in temperature. But in the case of study of deprotonation of glycine there is an increase in pK_1 value with increase in mass % of dioxane. Therefore, with an increase in mass % of dioxane dissociation of glycine is not favored.

From Born equation³³ or modified Born equation^{34,35} the role of solvent on the ionisation weak acid and weak base is not clearly understood. In such circumstances, the problem is extremely complicated in the case of zwitter ion upon addition of 10-30% dioxane content in dioxane water system at 298.15 K.

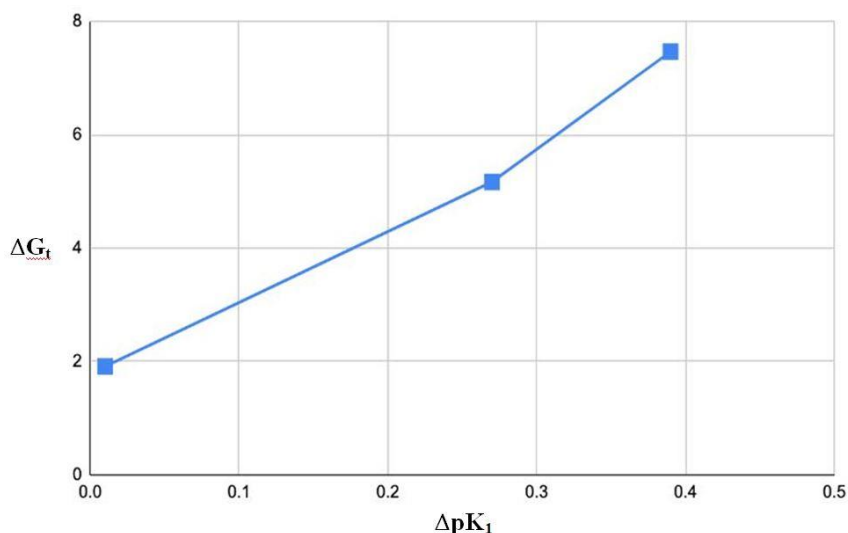
The free energy transfer (ΔG_t) of glycine from water to 10, 20 and 30% (m/m) dioxane in dioxane-water system at a given temperature have been calculated by the following thermodynamic relation:

$$\Delta G_t = 2.30259 RT\Delta pK_1 \quad \dots(20)$$

where, $\Delta pK_1 = pK_s - pK_w$ in which the subscript s stand for mixed solvent and w stand for water and the value of ΔG_t and ΔpK_1 have been recorded in table 7.

Table: 7

% Dioxane (m/m)	ΔpK_1	ΔG_t
10	0.01	1.914
20	0.27	5.169
30	0.39	7.466

Fig. 4: Free energy transfer (ΔG_t) for glycine in 10, 20 and 30% dioxane in dioxane-water system at 298.15 K.

$$\Delta p = pK_w - pK_s$$

From table 4 it is evident that pK_1 value increases with increase in dioxane content of dioxane-water system. These results are similar to result of other workers^{21,22,25-27,29} and it is clear that it is due to lowering of dielectric constant of the medium. From Born equation³³ or modified Born equation^{34,35} the role of solvent on the ionisation weak acid and weak base is not clearly understood. In such circumstances, the problem is extremely complicated in the case of zwitter ion upon addition of 10-30% dioxane content in dioxane water system.

So it can be concluded that dominating role is played by dielectric constant because with increase in dioxane-content there is increase in pK_1 values which indicates that there is lesser dissociation of glycine.

In our study the variation in thermodynamic quantities like ΔG^0 , ΔH^0 and ΔS^0 have been studied by the graph. It is evident from fig. 4 that there is an increase in standard free energy change with increase in pK_1 value which indicates that deprotonation of glycine is not favorable with increase in dioxane content at 298.15 K.

Table: 8

% Dioxane (m/m)	ΔG^0	pK_1
10	13.998	2.45
20	14.945	2.62
30	15.68	2.74

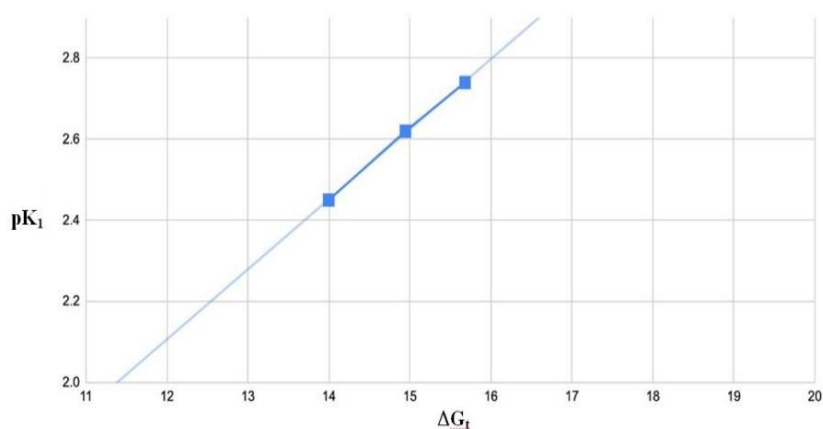
Fig. 5: Plot of pK_1 against ΔG^0 at 298.15 K for 10, 20 and 30%

Table: 9

Mass % dioxane (m/m)	pK ₁	-ΔS° KJ K ⁻¹ mol ⁻¹
10	2.45	-12.497
20	2.62	-39.0763
30	2.74	-36.168

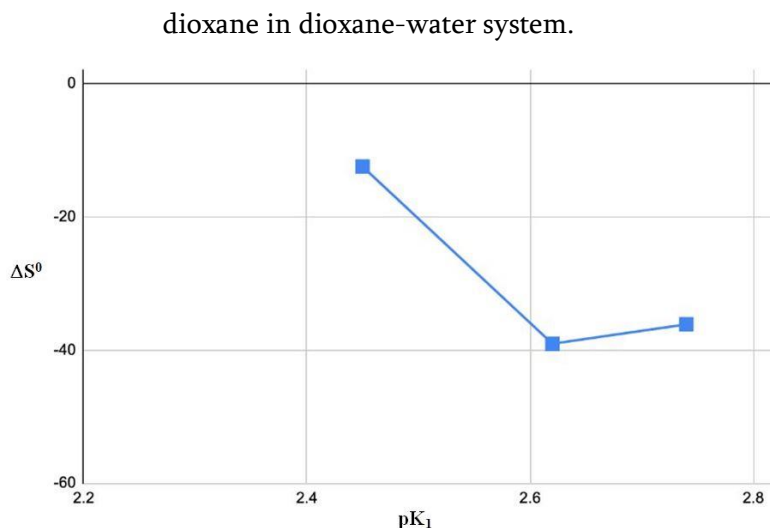


Fig 6 : Plot of pK₁ against -ΔS° at 298.15 K for 10, 20 and 30% dioxane in dioxane-water system.

From table 9, it is evident that the value of standard enthalpy change is positive so the reaction is endothermic throughout the dissociation process. In 10% dioxane content the value of standard enthalpy change is maximum, indicating maximum endothermic reaction in the process of deprotonation of glycine. In 20% dioxane-content the value of standard enthalpy change is minimum so the deprotonation is least endothermic. As the percent of dioxane increases to 30, standard enthalpy change is increased but less than in 10% dioxane-content so in 30% dioxane content the reaction is not as much endothermic as in 10% dioxane content.

Table:10

Mass % dioxane (m/m)	pK ₁	ΔH° KJ mol ⁻¹
10	2.45	10.272
20	2.62	3.295
30	2.74	5.493

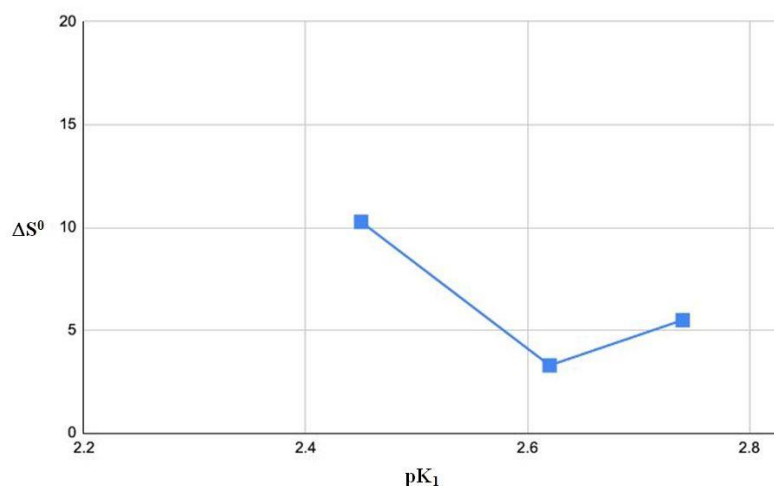


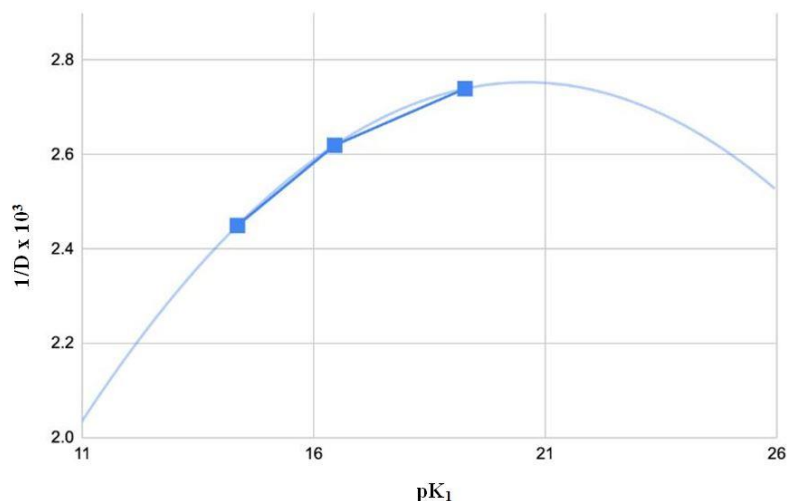
Fig. 7: Plot of pK₁ against ΔH° at 298.15 K for 10, 20 and 30% dioxane in dioxane-water system.

From fig. 10, it is evident that with increase in dioxane content the value of pK₁ which indicates that in higher content of dioxane in dioxane-water system, the deprotonation is unfavourable.^{36,37}

Variation of pK₁ against 1/D x 10³ at 10, 20 and 30% dioxane in dioxane-water system at 298.15 K. It is evident from the plot that with increase in dioxane-content, there is curvature in the plot and not straight line. It indicates that there is more interaction between dioxane and glycine molecule existing in zwitter ion.

Table 11

Mass % dioxane (m/m)	pK ₁	1/D x 10 ³
10	2.45	14.351
20	2.62	16.45
30	2.74	19.268

Fig. 8: Plot of pK₁ against 1/D x 10³ at 298.15⁰ K.

Some physical properties³⁸⁻⁴⁰ of dioxane-water systems have been given in table 12-14.

Table 12: For 10% (m/m) dioxane.

Temp/K	A'/Kg ^{-1/2} mol ^{-1/2}	P _{soln} /mm	D	do/gm cm ⁻³	k
	R ₁	R ₂	R ₃	R ₄	R ₅
288.15	0.5989	14.7	73.22	1.0118	0.057172
298.15	0.611	27.6	69.68	1.0059	0.059156
308.15	0.6250	48.6	66.32	1.0016	0.061140
318.15	0.6410	82.0	63.12	0.9994	0.063124

Table 13: For 20% (m/m) dioxane.

Temp/K	A'/Kg ^{-1/2} mol ^{-1/2}	P _{soln} /mm	D	do/gm cm ⁻³	k
	R ₁	R ₂	R ₃	R ₄	R ₅
288.15	0.7356	17.0	64.00	1.0193	0.057172
298.15	0.7531	32.4	60.79	1.0142	0.059156
308.15	0.7725	55.6	57.73	1.0091	0.061140
318.15	0.7934	105.0	54.83	1.0038	0.063124

Table 14: For 30% (m/m) dioxane.

Temp/K	$A'/Kg^{-1/2} mol^{-1/2}$	P_{soln}/mm	D	$do/gm\ cm^{-3}$	k
	R ₁	R ₂	R ₃	R ₄	R ₅
288.15	0.9339	19.3	54.76	1.0293	0.057172
298.15	0.9581	35.2	51.90	1.0216	0.059156
308.15	0.9854	62.2	49.19	1.0158	0.061140
318.15	1.0161	102.2	46.62	1.0119	0.063124

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