Effect of Physical Properties on Conductivity Measurements of Acetyl acetonylbinylidine 4-aminosalycilic Acid in Different Percentages of Ethanol/ Water Mixture

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ABSTRACT

The effect of physical properties on solute solvent interaction were studied by conductivity measurements of acetylacetonylbinylidine 4-aminosalycilic acid in different percentages of ethanol-water (5,10, 15, 20, 25% of ethanol) at 298.15K using Lee-Wheaton equation of conductivity. The results show that the association constant (K_A) increase with increasing ethanol percentages and viscosity and decreasing dielectric constant of the solvent and the distance of ion in solution (R). On the other hand, the equivalent conductance at infinite dilution (Λ_o) increases with increasing ethanol percentages due to the decreasing of the distance between ions in solution (R), which means that ion pairs convert from solvent separated ion (SSIP) to contact ion pairs (CIP) at best fit value of $\sigma\Lambda$.

Keywords: acetylacetonylbinylidine 4-aminosalycilic acid, effect of physical properties on conductivity measurements.

تأثير الصفات الفيزيائية على قياسات التوصيلية لمركب اسيتايل اسيتنوايلينلدين–4–امينو حامض السالسيلك في نسب مختلفة من مزيج الايثانول والماء

الملخص

أجريت الدراسة على الصفات ألفيزيائية جراء تداخل المذيب والمذاب للمركب اسيتال اسيتنوايلينلدين – 4 – امينو حامض السالسيلك في نسب مختلفة من الايثانول والماء (5,10,15,20,25%) بواسطة قياسات التوصيلية عند درجة حرارة298.15 مطلقة باستخدام معادلة لي و ويثون للتوصيلية للمحاليل المتجانسة. واوضحت النتائج ان ثابت التجمع الايوني يزداد بزيادة نسبة الايثانول ولزوجة المذيب مع قلة ثابت العازل والمسافة بين الايونات في ألمحلول، ومن ناحية أخرى فان التوصيل المكافئ عند التخفيف اللانهائي (Λ_o) يزداد بزيادة نسبة الايثانول، ويعود السبب في ذلك الى قلة المسافة بين الايونات في المحلول (R). والذي يعني أن المزدوج الايوني يتحول من مزدوج مفصول بجزيئات مذيب (SSIP) إلى مزدوج ايوني غير مفصول بجزيئات مذيب (CIP) عند أحسن قيم للانحراف القياسي $\sigma\Lambda$.

الكلمات الدالة: اسيتايل اسيتنوايلينلدين–4–امينو حامض السالسيلك، تأثير الصفات ألفيزياوية على قياسات التوصيلية.

INTRODUCTION

The discovery of the electrical conductivity of material prepared from tetrathiofulavtene and tetracyanoquinodimethene (Frranis *et al*., 1973) initiated on intense searches for conductance materials which is still in progress. A number of other materials of similar type, with heterocyclic systems, have been found to play a major role in providing electron donor molecules. The effect of

hydrogen bonding, relative permittivity and temperature on the transport properties and the association behavior was studied through the measurements of the specific conductance of sodium acetate, sodium propionate and sodium salicylate in 10%, 20% and 30% (w/w) methanol-water (MeOH-H_2O), ethanol-water (EtOH-H_2O) and glycerol-water (GlyOH-H_2O) mixtures at different temperatures (293, 298, 303 and 308 ^{o}K). The conductivity data have been analyzed using Lee–Wheaton conductivity equation. The molar conductance (Λ), limiting molar conductance (Λ_o), association constants (K_A), Walden product ($\Lambda_o\eta_o$), hydrodynamic radii (R_H), fluidity ratio(R), the activation energy of the transport process (E_a) and standard thermodynamic parameters of association (ΔG_A, ΔH_A and ΔS_A) were calculated and discussed (El-Dossoki, 2010). The conductivity of several alkali metal thiocyanates in water-methanol mixtures was measured at 25 ^{o}C. The data were analyzed using the Lee-Wheaton theory for symmetrical electrolytes to obtain ion association constant (K_A) limiting molar conductivity (Λ_o) and limiting ionic conductivity in all the solvent systems (Kubota and Horimoto, 1999). Measurements of the electrical conductivity of the compounds 2-(2,4-dichlorophenoxy methyl-5-(3-chlorophenyl)-1,3,4-oxadiazole and 2,5-di(2,4-dichlorophenoxy methyl)-1,3,4-oxadiazole in mixture of methanol water as a solvent at different percentages were analyzed using Lee-Wheaton equation the values of Λ_o, K_A and R are calculated (Akrawi, 2002) (Al-Sattar, 2002). Correlation of the molecular structure and thermal study of the electrical conductivity of benzaldazine and its nitro derivatives were carried out, the values of activation energies obtained from the measurements, as well as, the energy gaps calculated from UV- visible spectra indicated that all compounds behave like a semiconducting material (El-Sayed *et al.*, 1991). The electrical conductivities of 5-(P-substituted) phenylazo barbituric acid compounds and their complexes were measured, the results illustrate faint semi conductivity behavior for these system, the conductivities were found to depend on the structure of the compounds. The metal ion forms a bridge between the ligands to facilitate the transfer of current carriers with some degree of delocalization in the excited state (Masoud and Khalil, 1986).

EXPERIMENTAL

Preparation of compound:

The compound (Acetyl acetonyl binylidene 4-amino salicylic acid) was prepared by mixing 0.08mole (8.0 g) of acetyl acetone with 0.16 mole (24.48 g) of 4-aminosalicylic acid (Vogel, 1967). Added to the mixture 150 ml of ethanol, the mixture was refluxed for 6 hours, the product was cooled in ice path, followed by washing the product with cold ethanol and dried.

The UV apparatus absorption spectra were measured using UV-Visible 1650 Shimadzu spectrophotometer in conjunction with a computerized recorder. The IR spectra for solid imines were measured by computerized Bruker Tensor 27 using KBr disc method. Electrothermal melting point apparatus was used for measurement of melting point of solid samples (370 $^{\circ}$C). The UV absorption in ethanol (λnm) and molar extinction coefficient ($\epsilon_{max.}$) show three bands 317.17 (20000), 284(13520), 240(16470) respectively. The IR spectra (cm^{-1}) for OH, CO, CN, CH are 3388.16, 1670, 1616, 1597.88 respectively (Azzouz *et al.*, 2010). The reactions were as follows:
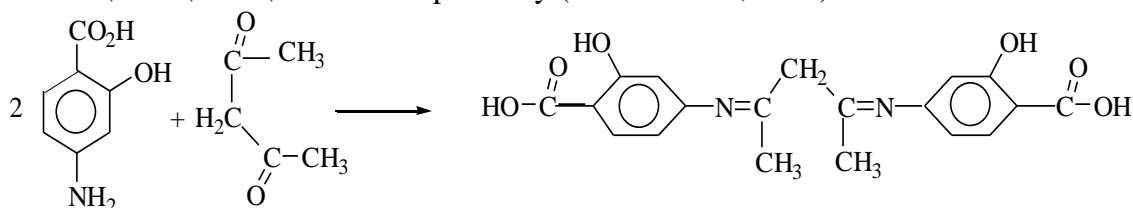
General procedure

Triply distilled water (Daniels *et al.*, 1970) was used in conductivity measurements. A general method was used for measuring the conductance of the electrolytes. The instrument used for the

measurements was Jenway conductivity meter with sensitivity 10^{-6} micro semens. The conductivity cell was weighed and kept at constant temperature then purified with nitrogen gas passed through the cell: 100 ml of purified solvent as added and nitrogen gas was passed for further (10-15) minutes, where upon the cell plus its content were weighed. A certain amount of complex solution was injected in to the cell from a plastic syringe (which was weighed before and after each addition), nitrogen gas was passed for several minutes and the conductivity of the solution was measured. After all the addition have been made (generally 15), the cell was reweighted to find the weight change over the hall run which was found not more than (0.02%).

The compound is very stable in the solvent used as shown below:

Compound conc.$*10^{-4}$	t.minute	Conductivity.μ.S
1.6	5	$4*10^{-6}$
1.6	10	$4*10^{-6}$
1.6	15	$4*10^{-6}$
1.6	20	$4*10^{-6}$
1.6	25	$4*10^{-6}$

RESULTS AND DISCUSSION

At constant temperature, the concentration of free ions ($C\alpha$) and ion-pairs [$C(1-\alpha)$] are considered to exist at equilibrium. This state was described by the thermodynamic equilibrium (K) for the ion-association reaction. Since, the ion pairs are non-conducting entities, and the degree of their dissociation (α) can be given by the ratio between the molar conductivity of the electrolyte (Λ) and the free ion ($\Lambda_{-C\alpha}$). The combination of the expressions for K and α can then be given as:

$$\Lambda = \frac{(\Lambda - C\alpha)}{1 + K(C/C^{\circ})\alpha y^{2\pm}} \qquad \ldots\ldots\ldots\ldots (1)$$

Where $C^{\circ} = 1$ mole dm^{-3}, $y^{\pm}$ is the mean activity coefficient which can be obtained from equation 2:

$$y^{\pm} = \exp(\frac{Kq}{1 + KR}) \qquad \ldots\ldots\ldots\ldots (2)$$

which refer to the dissociated part of the electrolyte, R is the distance parameter which must be the least distance that two free ions can approach before they merge into an ion-pair, or in other words, it is the farest distance that separates two ions. The parameters K and q can be obtained from equation 3 and 4 respectively:

$$K^2 = 16000 \, \pi \, N_A \, q \, C\alpha \qquad \ldots\ldots\ldots\ldots (3)$$

$$q = \frac{e^2}{8\pi\varepsilon^{\circ}\varepsilon_r KT} \qquad \ldots\ldots\ldots\ldots (4)$$

If it is assumed that the dissociated part of the weak electrolyte acts like a completely hypothetical dissociated strong electrolyte of the same type, then $\Lambda_{C\alpha}$ can be replaced by any theoretical equation for conductivity in order to construct a conductivity model, which was found by Lee-Wheaton (Lee and Wheaton, 1978), equation (5) will become:

$$\Lambda_{c\alpha} = \Lambda_o [1 + C_1(KR)(\varepsilon K) + C_2(KR)(\varepsilon K)^2 + C_3(KR)(\varepsilon K)^3] - (\rho K)/$$

$$(1 + KR)[1 + C_4(KR)(\varepsilon K) + C_5(KR)(\varepsilon K)^2 + (KR)/12] \ldots\ldots\ldots (5)$$

Where the concentration dependent terms are both the plasma parameter (εK) and (KR), while the concentration coefficients ε, K and ρ can be obtained from equations 6-8 respectively.

$$\varepsilon = |Zi|^2 e^2 / DKT \qquad \text{........... (6)}$$

$$K^2 = 8\pi\ Ne^2\ |Zi|^2 C/1000\ DKT \qquad \text{........... (7)}$$

$$\rho = F\zeta e|Z| / 3\pi\eta \qquad \text{........... (8)}$$

Where F is the Faraday constant (9.6486×10^4), ζ is the conversion factor (volt to e.s.u.) = 1/299.7925, and all other terms have previously been defined in details (Lee and Wheaton, 1978). Conductivity measurement of mixed solvent (ethanol-water) solution of acetylacetonyl binylidene 4-amino salicylic acid) in different percentages of ethanol - water (5,10,15,20,25% of ethanol) at 298.15K was carried out. The values of the molar conductance (Λ) of the solutions were given in Tables (1) (A-E). The correlations between the concentration of the aqueous solution of the compound above and their equivalent conductance are shown in Fig. (1 A-E) which shows a non linear relation, that means they are a weak electrolytes. The conductance data obtained were first analyzed in accordance with Lee-Wheaton equation.

Table 1 A: Values of concentration (mole litter^{-1})and equivalent conductance (equiv^{-1}.s^{-1}.cm^2) in a mixture of (5 % ethanol + 95% water) at 298.16 º K

C * 10^{-4}	Λ	$\sqrt{C}$
0.740	59.810810	0,0086023
1.071	46.283846	0,0103489
1.379	39.572153	0,011743
1.666	34.897959	0,0129073
1.935	33.152454	0,0139104
2.187	32.181069	0,0147885
2.424	29.707095	0,0155692
2.647	29.206649	0,0162696
2.857	27.525373	0,0169026
3.055	27.626841	0,0174785
3.243	29.879740	0,0180083
3.421	29.219526	0,0184959
3.589	29.130677	0,0189446
3.755	28.956058	0,0193649
3.902	28.700666	0,0197534
4.047	28.354336	0,0201171
4.186	28.143812	0,0204597
4.318	27.508105	0,0207797
4.444	27.542754	0,0210807
4.565	27.437020	0,0213658
4.680	27.068376	0,0216333
4.791	26.973491	0,021883
4.897	26.557075	0,0221291
5.000	26.336000	0,0223606

Table 1 B: Values of concentration (mole litter^{-1})and equivalent conductance (equiv^{-1}.s^{-1}.cm^2) in a mixture of (10 % ethanol + 90% water) at 298.16 º K

C * 10^{-4}	Λ	√ C
0.740	66.713513	0,0086023
1.071	54.571428	0,0103489
1.379	50.001450	0,011743
1.666	43.408163	0,0129073
1.935	38.902325	0,0139104
2.187	36.471879	0,0147885
2.424	34.210396	0,0155692
2.647	32.985266	0,0162696
2.857	30.917745	0,0169026
3.055	30.850409	0,0174785
3.243	29.691026	0,0180083
3.421	28.921368	0,0184959
3.589	27.794928	0,0189446
3.755	26.892143	0,0193649
3.902	26.401845	0,0197534
4.047	26.161601	0,0201171
4.186	25.585284	0,0204597
4.318	25.228346	0,0207797
4.444	24.972097	0,0210807
4.565	24.801752	0,0213658
4.680	24.780769	0,0216333
4.791	24.675015	0,021883
4.897	24.648083	0,0221291
5.000	24.337200	0,0223606

Table 1C: Values of concentration (mole litter^{-1})and equivalent conductance (equiv^{-1}.s^{-1}.cm^2) in a mixture of (15 % ethanol + 85% water) at 298.16 º K

C * 10^{-4}	Λ	√ C
0.740	81.324324	0,0086023
1.071	58.095238	0,0103489
1.379	48.152284	0,011743
1.666	41.632653	0,0129073
1.935	36.635658	0,0139104
2.187	33.207133	0,0147885
2.424	30.759900	0,0155692
2.647	29.863996	0,0162696
2.857	27.918795	0,0169026
3.055	27.077577	0,0174785
3.243	25.853839	0,0180083
3.421	24.955860	0,0184959
3.589	24.555029	0,0189446
3.755	23.958455	0,0193649
3.902	23.447975	0,0197534
4.047	22.784284	0,0201171
4.186	22.393215	0,0204597
4.318	22.110236	0,0207797
4.444	21.575157	0,0210807
4.565	21.494852	0,0213658

4.680	21.402564	0,0216333
4.791	21.396368	0,021883
4.897	21.386759	0,0221291
5.000	21.256800	0,0223606

Table 1 D: Values of concentration (mole litter^{-1})and equivalent conductance   (equiv^{-1}.s^{-1}.cm^2)   in a mixture of (20 % ethanol + 80% water) at 298.16 º K

C * 10^{-4}	Λ	√ C
0.384	73.843750	0,0061967
0.740	43.281081	0,0086023
1.071	32.212885	0,0103489
1.379	26.110224	0,011743
1.666	23.204081	0,0129073
1.935	20.927131	0,0139104
2.187	19.728395	0,0147885
2.424	18.430693	0,0155692
2.647	17.648658	0,0162696
2.857	17.101155	0,0169026
3.055	16.291325	0,0174785
3.243	16.072155	0,0180083
3.421	15.742765	0,0184959
3.589	15.375313	0,0189446
3.755	15.048735	0,0193649
3.902	14.821629	0,0197534
4.047	14.693847	0,0201171
4.186	14.400860	0,0204597
4.318	14.314960	0,0207797
4.444	14.253375	0,0210807
4.565	14.210733	0,0213658
4.680	14.232051	0,0216333
4.791	14.221665	0,021883
4.897	14.205431	0,0221291
5.000	14.157600	0,0223606

Table 1 E: Values of concentration (mole litter^{-1})and equivalent conductance (equiv^{-1}.s^{-1}.cm^2) in a mixture of (25 % ethanol + 75% water) at 298.16 º K

C * 10^{-4}	Λ	√ C
0.384	98.281250	0,0061967
0.740	66.162162	0,0086023
1.071	51.333333	0,0103489
1.379	40.385786	0,011743
1.666	35.081632	0,0129073
1.935	31.469767	0,0139104
2.187	29.242798	0,0147885
2.424	28.066831	0,0155692
2.647	26.511522	0,0162696
2.857	25.312265	0,0169026
3.055	24.373158	0,0174785
3.243	23.589269	0,0180083
3.421	22.809120	0,0184959
3.589	22.395096	0,0189446
3.755	21.921171	0,0193649

3.902	21.827268	0,0197534
4.047	21.574499	0,0201171
4.186	21.223602	0,0204597
4.318	21.023622	0,0207797
4.444	20.702970	0,0210807
4.565	20.377656	0,0213658
4.680	20.160256	0,0216333
4.791	19.969943	0,021883
4.897	19.933428	0,0221291
5.000	19.624800	0,0223606
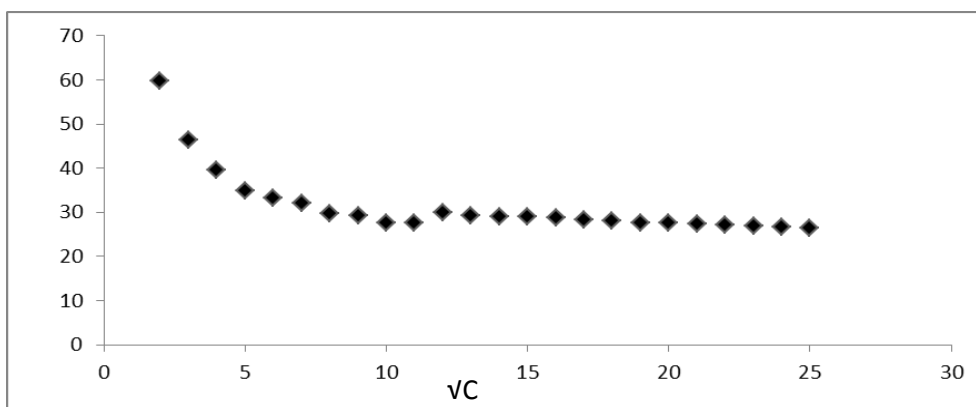
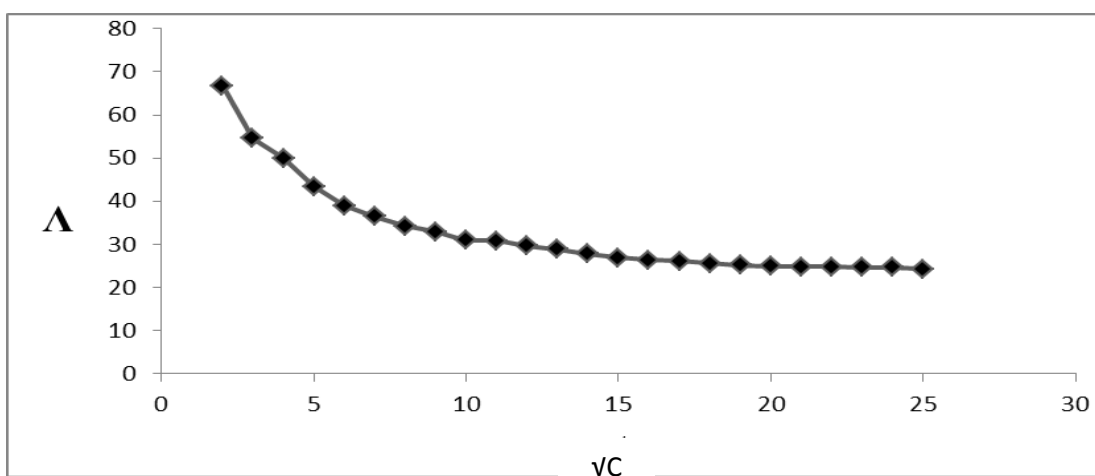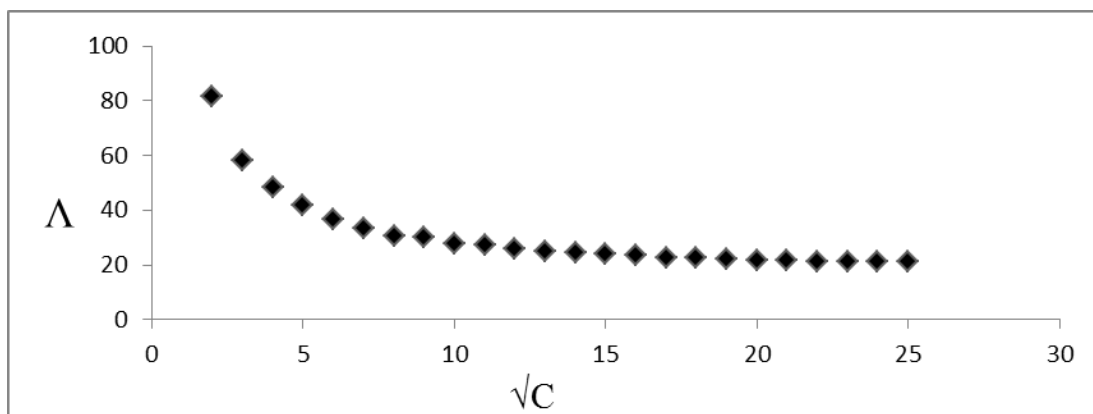**Fig. 1 A: Relation between $\Lambda_{equiv.}$ and square root of concentration) in a mixture of (5 % ethanol + 95% water) at 298.16 º K**
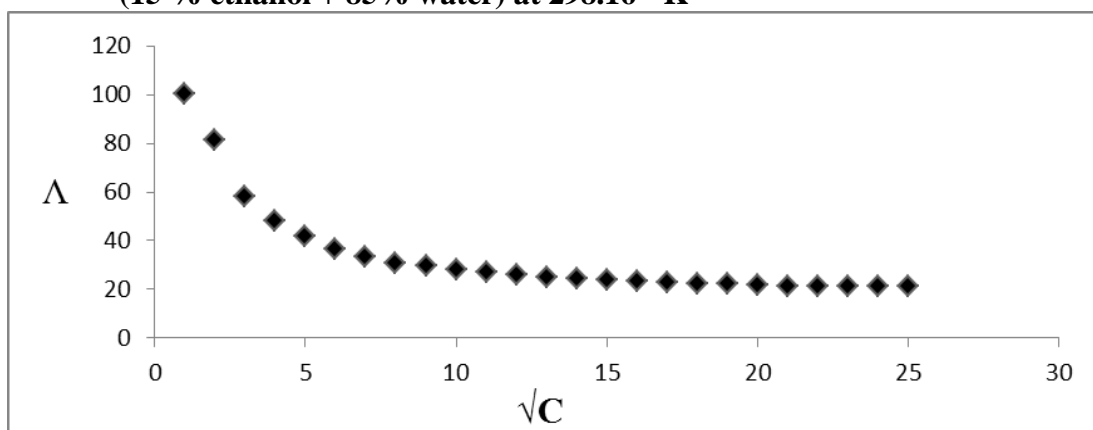

Fig. 1 B : Relation between $\Lambda_{equiv.}$ and square root of concentration) in a mixture of (10% ethanol + 90% water) at 298.16 º K

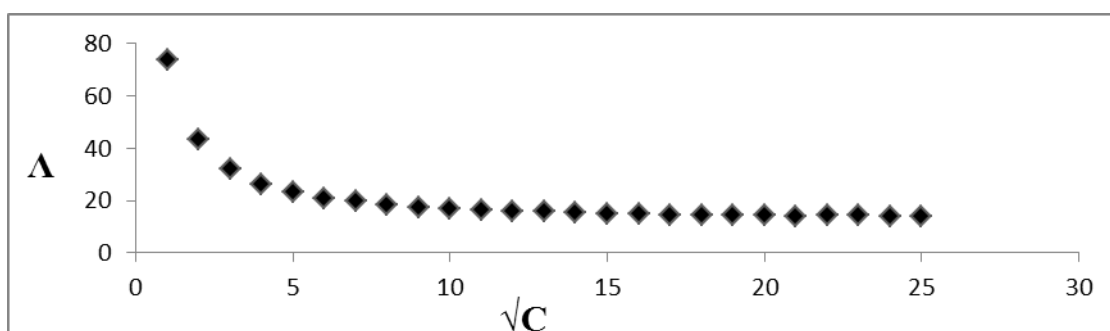**Fig. 1 C : Relation between $\Lambda_{equiv.}$ and square root of concentration) in a mixture of (15 % ethanol + 85% water) at 298.16 º K**

Fig. 1 D: Relation between $\Lambda_{equiv.}$ and square root of concentration) in a mixture of (20% ethanol + 80% water) at 298.16 º K

Fig. 1 E : Relation between $\Lambda_{equiv.}$ and square root of concentration) in a mixture of (25 % ethanol + 75% water) at 298.16 º K

The values of analysis (K_A, Λ_o, R, $\sigma\Lambda$) of the mixed solvent solution of acetyl acetonylbinylidene 4-amino salicylic acid) are shown in Table 2

Table 2: Values of K_A, $\Lambda_\circ$, Rand σ (A) for acetyl acetonylbinyledene 4- amino salicylic acid in ethanol + water mixtures at 298.16ºK

%	K_A	$\Lambda_\circ$	$R(A^\circ)*10^{-8}$	σ (A)

5	13390	71.581	11	0.003
10	20770	91.9	10	0.004
15	32820	95.27	5	0.002
20	35369	100	4	0.00
25	46389	104.188	3	0.00

From (Table 2), the values of K_A increase with increasing ethanol percentages, due to increasing viscocity and density with decreasing dielectric constants of the mixed solvents as shown in (Table 3). On the other hand the values of Λ_o also incease, this because of the ion pairs convert from solvent separated ion (SSIP) to contact ion pair (CIP), this can be illustrated by decreasing the values of R (the main distance between ions in solution) due to decreasing the water content of the solvent which is responsible of hydrogen bonding. The values of $\sigma \Lambda$ are very low which indicate the fitness of the equation to determine the constants (K_A, Λ_o and R).

Table 3: Density, Viscosity and Dielectric constant of ethanol: water mixture at 298.16ºK

%	Density	Viscosity	Dielectric constant
5	0.0005	0.00900	74.1
10	0.0010	0.00910	72.1
15	0.0016	0.00921	69.3
20	0.0022	0.00932	67.2
25	0.0027	0.00937	64.5

If Stokes law were obeyed in a system, the value of the Walden product ($\eta_o \Lambda_o$) would become constant only if the effective radius of the ion remains constant in the different media. It was suggested that the major deviations in the Walden product was due to the variation of the electrochemical equilibrium between ions and the solvent molecules, depending on the composition of the mixed polar solvents (Hemes, 1974). This is the case in the behavior of the present system as indicated in Fig. (2), where the ions suffer various degrees of solvation with different mixtures of ethanol and water as described by (Hameed *et al*., 2008). Table (3) shows some physical constants of ethanol-water mixtures and the Walden product ($\eta \Lambda_o$) for this electrolyte, This behavior is similar to that obtained (Hawlicka and Grabowski, 1990) for NaI and tetraethylammonium iodide in methanol-water mixture, which refered the increase of the equivalent conductivity to the effect of the viscosity.

Similar observations were also noted for some electrolytes in other mixed solvents (Jauher *et al.*, 1988). This may be attributed to the selective solvation of ions besides the solvodynamic viscous force.
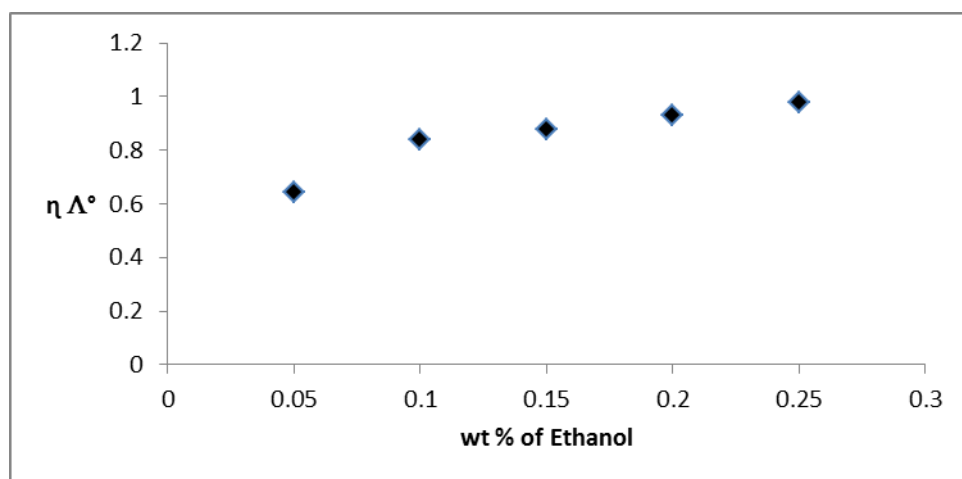
Fig. 2: Relation between Walden product and the percentages of ethanol – water mixture

The ethanol-water molecule is probably poorly ordered which leads to a conclusion that its structure has little effect on the movement of the ions in the solution leading to larger conduction in solution (Al Dabbagh *et al.,* 1996). Eyring's enthalpy of activation of the charge transport ($\Delta H^{\ddagger}$) (Vesna *et al*., 2008), can be obtained from the temperature dependence of Λ_o by the equation

Ln Λ_o + 2⁄3 Ln d $_o$ = - $\Delta H/RT$ + C

where d_o is the density of solvent. The values of enthalpy of activation at 298.15 K are given in Table 4 which show the variation of ($\Delta H^{\ddagger}$) between the positive and the negative values due to the solvation effect in the low percentages of ethanol (5% and 10%) which may be attributed to the low values of density and viscosity and high dielectric constant of the mixture. The other percentages (15% , 20% and 25%) have a negative values of ($\Delta H^{\ddagger}$) due to the effect of low values of dielectric constant of the mixed solvent. $\Delta G°$ is negative which indicates that the reaction is spontaneous as shown in Fig. 3, and $\Delta S°$ positive due to the low orientation of the molecules.

Table 4: Vartation of Thermodynamic Parameters ($\Delta H°$, $\Delta G°$, $\Delta S°$)

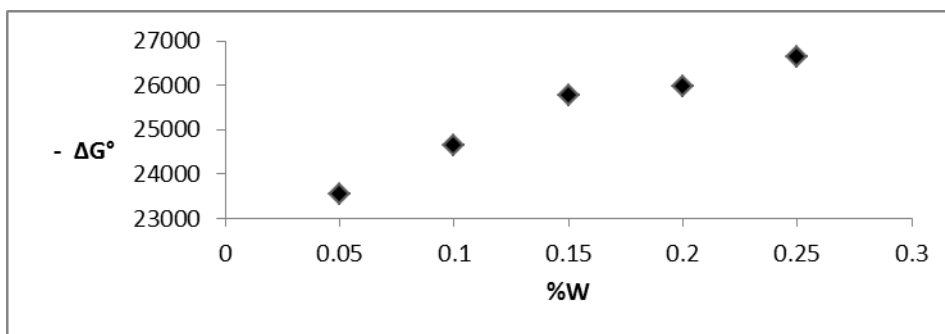| $\Delta H^{\ddagger}$ J mol^{-1}	$\Delta G°$ J mol^{-1}-	$\Delta S°$ J K^{-1} mol^{-1}	%
1974.293	23555.182	85.623	5
209.3960	24643.423	83.353	10
-656.61	25777.592	84.253	15
-1306.721	25963.008	82.694	20
-1743.150	26635.245	83.485	25

Fig. 3 : Relation between Gibbs free energy and the weigh percent of ethanol – water mixture

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