

Electrical Conductivity of $[\text{Co}(\text{en})_2\text{Cl}_2] \text{Cl}$ in Mixed Solvents (Alcohol + Water) at 298K

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ABSTRACT

The electrical conductivity of trans – dichloro bis(ethylenediamine) cobalt (III) chloride $[\text{Co}(\text{en})_2\text{Cl}_2] \text{Cl}$ has been measured in (methanol + water), (ethanol + water) and (isoproponal + water) at different percentages and at 298K. The conductivity parameter, Λ_o (equivalent conductivity at infinite dilution, K_a ; (association constant) and R the (distance parameter) between the ions were calculated by using Lee – Wheaton equation for symmetrical electrolytes.

The Walden product ($\Lambda \circ \eta$) found to be not constant for the three media which describe as to the change of ion-solvation and of the solvent structure.

(III)

(+)

$[\text{Co}(\text{en})_2\text{Cl}_2] \text{Cl}$

298

$[\text{Co}(\text{en})_2\text{Cl}_2] \text{Cl}$

298 (+) (+) (+)

) K_a () Λ_o

- () R (

($\Lambda \circ \eta$)

INTRODUCTION

As in other fields of inorganic chemistry some research on coordination compounds has been carried out in non aqueous media in the case in which the coordination compounds reacts with water, the use of non aqueous solvent may be advantageous. A familiar example is the determination of the number of ions in a compound by

measuring the conductivity in nitromethane or other highly polar solvent (Geselowitz and Taube, 1982). A conductance measurements of $[\text{Cu}(\text{en})_2]\text{So}_4$ (2:2) in formamide at 25°C were performed to see whether a high dielectric constant would control ion associated to any extent. The data were analyzed by using the Fuoss – Onsager equation for unassociated electrolytes (Johari and Tewari, 1965).

Copper (II) complexes of secondary and tertiary N – substituted ethylenediamines were prepared by Meek and Ehrhardt (Meek and Ehrhardt, 1965) and their conductivity were measured and found to be increased with time in nitromethane.

Evidences suggested that the substitution of (en) for ammonia in the coordination sphere increase the degree of association. The osmotic and activity coefficients are reported (Masterton, 1967) for the compounds $\text{trans} - [\text{Co}(\text{en})_2 \text{NCS. CL}]\text{X}$, where $x = \bar{I}, \bar{Br}, \bar{Cl}$ or $\bar{\text{NO}}_3$. The results are lower than those of most simple 1:1 salts.

The conductances and solubilities of oxalato bis (ethylenediamine) cobalt (III) chloride, bromide, iodide, and perchlorate were determined in water at 25°C by Kubota (Kubota, 1971) and the data were analyzed on the basis of the Fuoss Onsager Skinner theory. Kaneko and Wada (Kaneko and Wada, 1978) studied the conductance behavior of some tris (ethylenediamine) cobalt (III) complexes in dilute aqueous solutions at 25°C to investigate the ion – pair formation.

Solvation and ionic association of bi (β -diketonato) (diamine)cobalt (III) perchlorates in alcohols and ketones have been measured by (Ito et al., 1988).

Their conductances were measured in methanol, ethanol, 2-propanol, methyl ketone at 25°C. $[\text{Co}(\text{acac})_2 (\text{en})]\text{ClO}_4$ in alcohols was found to be strongly disassociated and this can be explained as being due to the strong solvation of $[\text{Co}(\text{acac})_2 (\text{en})]^+$. Tatehata and Asaba (Tatehata and Asaba, 1988), determined by a conductance method at 25°C the diastereometric ion – pair formation constants of $\text{C}_1\text{-Cis (N)-} [\text{Co}(\text{OX})(\text{gly})_2]$ and $\beta\text{-Cis}[\text{Co}(\text{OX})(\text{edda})]$ with $[\text{Co}(\text{en})]^{3+}$ in solution [where (edda) is ethylenediamine N, N – diacetate dianion, (gly) is glycinate anion].

Yokoyama et al. (Yokoyama et al., 1992), measured the conductivities for aqueous solutions of the chloride, bromide, Iodide, nitrate, and perchlorate of the tris (ethylenediamine) cobalt (III) complex, $[\text{Co}(\text{en})_3]^{+3}$ with varying temperatures from 0 to 50°C.

Singh et al. (Singh et al., 1995), have prepared the complex heterobimetallic salts of the formula $[\text{M}(\text{L-L}) (3)] [\bar{\text{M}} (\text{DED})(2)]$ [$\text{M}=\text{Zn}(\text{II}), \text{Ni}(\text{II}), \text{Co}(\text{II}), \text{Cd}(\text{II}); \bar{\text{M}}=\text{Ni}(\text{II}), \text{Zn}(\text{II}), \text{Cd}(\text{II}); \text{L-L} = 2,2\text{-bipyridyl (bipy)}; 1,10\text{-phenanthroline or ethylenediamine (en)}$]. The measured conductivities are inconsistent with those expected for 2:2 electrolytes suggesting that extensive ion pairing is occurring between the highly charged cationic and anionic species in nitrobenzene.

Akrawi (Akrawi et al., 2002) have measured the electrical conductivity of a mixture of both symmetrical and unsymmetrical complexes. $\{[\text{Ni}(\text{en})_3](\text{NO}_3)_2\} + \{[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}\}$ in methanol at 298K. The results were analyzed using Lee – Wheaton equation. The equivalent conductivities have been compared with the experimental value at the best – fit values of (R) and they are found to be in a good agreement. Besides it have been

calculated the ionic consternation, activities coefficient, transport number for each ion present in the solution.

The present paper describes the electrical conductivity of Trans-dichloro bis (ethylenediamine) cobalt chloride in methanol+ H_2O , ethanol+ H_2O and isopropanol+ H_2O at different percentages and at 298K.

EXPERIMENTAL

Materials:

Methanol, ethanol and isopropanol (Fluka) were of high purity and used directly. Conductivity water was prepared and used for conductivity measurements.

Trans dichloro bis ethylenediamine cobalt(III) chloride $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ (Fernelius, 1946) was prepared by adding 12g. of 10% solution of ethylenediamine (BDH, $\geq 99.5\%$), with stirring, to a solution of (3.5g. 0.020 mole) cobalt chloride hexa hydrate (Fluka, $\geq 99\%$) in 10cm^3 of water in a 100cm^3 beaker. A vigorous stream of air is passed through the solution for (2 hrs). Then 7cm^3 of concentrated hydrochloric acid is added, and the solution is evaporated on steam bath until a crust forms over the surface (15cm^3). This solution is allowed to cool and stand overnight before the bright – green square plates of the hydrochloride of the trans form are filtered. These crystal then washed with alcohol and ether, then dride at 110°C . At this temperature the hydrogen chloride is lost and the crystals crumble to a dull – green poder. This yield is 1.5g. (60% based on the ethylenediamine). The physical properties and C.H.N analysis of $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ are: Colour green, M.P/ $^\circ\text{C}$ 241, M. wt 285.43

Found			Calculated		
C%	H%	N%	C%	H%	N%
16.52	5.45	19.92	16.81	5.60	19.61

Apparatus:

Conductivity measurements were made using Digital Conductivity Meter (Jenway) with a frequency range of $50\text{Hz} - 3\text{kHz}$ and sensitivity between 10^{-1} and 10^{-9} Siemens. The temperature of the cell and its consents was kept constant at $25 \pm 0.1^\circ\text{C}$ using water – circulating thermostate type VHS β radiometer. The design of the conductance cell and the nitrogen was the same as that previously used (Akrawi, 1981), the cell constant was checked regularly and found to be 0.94cm^{-1} .

General conductivity measurements:

The following method has been used for measuring the conductivity of each electrolyte: In a clean, dry and weighted empty conductivity cell kept at constant temperature ($25 \pm 0.1^\circ\text{C}$), 100 ml of conductivity water was added and the conductance of the solvent was measured, and the cell was reweighted again.

Into the cell, a certain amount of the concentrated $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ (10^{-3}m) aqueous solution was injected into the cell from a plastic syringe which was weighted before and after each addition, and the conductivity of the solution was measured. This process was repeated generally for about 12 additions and after all the additions have been made, the cell was re weighted to find the change over the whole run which was found to be not more than 0.025%.

The above procedure was repeated for measuring the conductivity in different percentages of: (methanol + water), (ethanol + water) and (isopropanol + water) each at 298K.

The input data to the computer program (SYM₄) are: solvent data (Temperature T, Dielectric constant D and Viscosity η); association constant K_A and limiting equivalent conductivity Λ_0 ; and R in the form Rmin., Rmax., ΔR , together with the solution moralities and the corresponding equivalent conductances.

Tables (1-3), shows the solution moralities (mole L⁻¹) and the equivalent conductivities (S mol⁻¹ cm²) of [Co(en)₂Cl₂]Cl in different percentages of (methanol + water), (ethanol+water) and (isopropanol+water) respectively.

Figures (1-3), illustrate the relation between the molar concentration and the equivalent conductance of the [Co(en)₂Cl₂]Cl at different ratios of each (alcohol+water).

RESULTS AND DISCUSSION

It is well known that oppositely charged ions tend to form ion pairs in a medium of low dielectric constant. At constant temperature, free ions and ion pairs are at their equilibrium concentration αC and $C(1-\alpha)$ respectively, (where C = concentration, α = degree of dissociation).

This state described by the thermodynamic equilibrium constant for the ion – association reaction (K) since ion pair is a non – conducting entity, where α is the ratio of the molar conductivity of electrolyte (Λ) to that of free ions (Λ_0). The distance parameter R must be the least distance that two free ions can approach before they merge into an ion pair. Or, in other words, R is the further distance of separation of two paired ions.

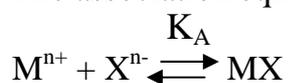
For the specific case of solutions containing only a single symmetrical electrolyte this equation has the following general form:-

$$\Lambda_{equiv.} = f(\Lambda_0, R, K_A)$$

Where Λ_0 is equivalent conductance at infinite dilution, K_A the pairwise ion association constant. And R the distance parameter which is defined as the distance between anion and cation at which short-range forces become strong enough to impede further approach of the ions, allowing the formation of CIP or SSIP (Akrawi, 1981).

Lee and Wheaton (Lee and Wheaton, 1979) consider a symmetrical electrolyte MX; and they assumed the solute to be completely dissociated into the species M^{n+} and X^{n-} .

The association equilibrium :



Will exist between the two species, and if K_A is the pairwise association constant, then :

$$K_A = \frac{[MX]}{[M^{n+}][X^{n-}]_{f_{\pm}^2}}$$

Where f_{\pm} is the activity coefficient.

The model used in this work is that founded by Lee – Wheaton:

$$\Lambda = \Lambda_o \left[1 + C_1 \beta_k + C_2 (\beta_k)^2 + C_3 (\beta_k)^3 \right] - \frac{\rho k}{1 + kR} \left[1 + C_4 \beta_k + C_5 (\beta_k)^2 + \frac{kR}{12} \right]$$

where Λ_o is the molar conductivity at infinite dilution, $\beta = 2q, k^2 = 1600\pi N_A q C \alpha$, $q = \frac{e^2}{8\pi E_o E_1 kT}$,

$C_1 \rightarrow C_5$ and other symbols are either defined in the cited literature (Akrawi, 1981) or have their usual significant.

The model was considered as non – linear function of three parameters $\Lambda = \Lambda(\Lambda_o, k_A, R)$, and was resolved by an iterative procedures.

A broader range of R was scanned by a series of successive steps of definite size for each step, the parameters Λ_o and k_A are obtained at the end of a sequence of alternating linearizations and least – square optimizations of the model, when Λ_o satisfied the criterion for convergence. In a row of three parameter sets, the (best) is the one by which the model is best fitted to experimental data, i.e the minimal standard deviation ($\sigma\Lambda$) is attend. The step size is then lowered by an order of magnitude, and the search is continued in the region around the (best) R.

This procedure is repeated until the final (best triplet) of parameters (Λ_o, k_A, R) is established when the step size equals to 0.1 pm.

Values of the limiting molar conductivity Λ_o , ion – association constant (k_A), distance parameter (R) and standard deviation of experimental Λ from the model $\sigma(\Lambda)$, obtained by using LW equation are shown in Table (4). This analysis has done to understand the solute – solvent interactions characteristics in different mixed solvents.

It is natural that the Λ_o value depends on the medium in each mixture, it decreases with increasing the percentage of any alcohol. Also it is found that the Λ_o 's decrease in the order = [methanol + water] > (ethanol + water) > (isopropanol + water)]. This is due to the increase of the viscosity of the medium in the same order (Doe et al., 1990).

Table 3: The plot of equivalent conductance Λ (Siemens equiv.⁻¹ cm²) against the square root of molar concentration \sqrt{C} (mole. L⁻¹) for [Co(en)₂Cl₂]Cl in a mixture of (Isoproponal + water) at 298K.

100% Isoproponal		80% Isoproponal		60% Isoproponal		100% (H ₂ O)	
\sqrt{C}	Λ	\sqrt{C}	Λ	\sqrt{C}	Λ	\sqrt{C}	Λ
0.02641	4.0424	0.04285	25.5893	0.05091	42.0597	0.04285	194.9910
0.03859	3.7870	0.04472	25.3800	0.09345	41.7836	0.04472	194.5800
0.04285	3.5825	0.04644	25.2781	0.05461	41.9192	0.04644	193.9447
0.04644	3.4866	0.04803	25.2556	0.05570	41.7993	0.04803	193.4910
0.04952	3.4491	0.05091	25.0183	0.05674	41.7414	0.04952	193.1506
0.05091	3.2632	0.05222	24.8166	0.05773	41.7364	0.05091	192.1697
0.05345	3.2900	0.05345	24.6753	0.05956	41.5917	0.05222	191.9844
0.05461	3.1518	0.05674	24.5194	0.06123	41.6106	0.05345	191.4808
0.05570	3.3318	0.05773	24.5342	0.06201	41.5485	0.05461	191.3157
0.05674	3.2108	0.05956	24.3722	0.06276	41.5200	0.05570	190.8229
0.05773	3.1020	0.06042	24.4610	0.06348	41.5206	0.05674	190.6095
0.05956	3.1789	0.06123	24.3146	0.06416	41.5484	0.05773	190.0699
0.06042	3.0898	0.06201	24.1959	0.06546	41.4541	0.05867	189.7693
0.06123	3.0080	0.06276	24.1007	0.06607	41.3366	0.05956	189.1497
0.06348	3.0324	0.06546	23.9074	0.06666	41.4544	0.06042	188.9938
0.06416	2.9677	0.06607	23.8977	0.06778	41.3276		
0.0666	2.9610	0.06666	23.8997	0.06831	41.2934		
		0.06723	23.9132	0.06882	41.2768		
				0.06931	41.2769		
				0.07026	41.3190		
				0.07114	41.2272		

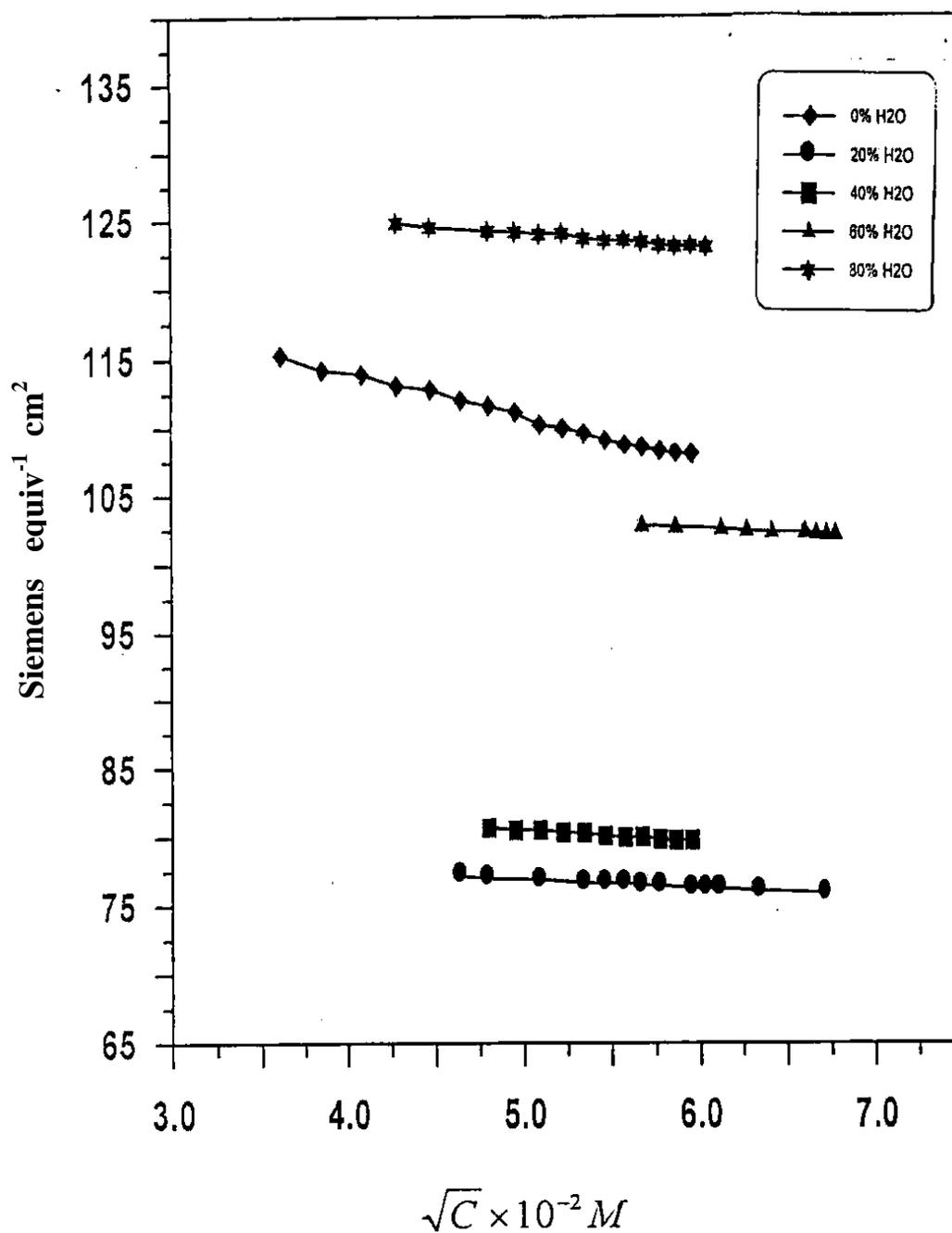


Figure 1: The plot of equivalent conductivity against the square root of molar concentration of $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ in (Methanol + Water) at 298K^o

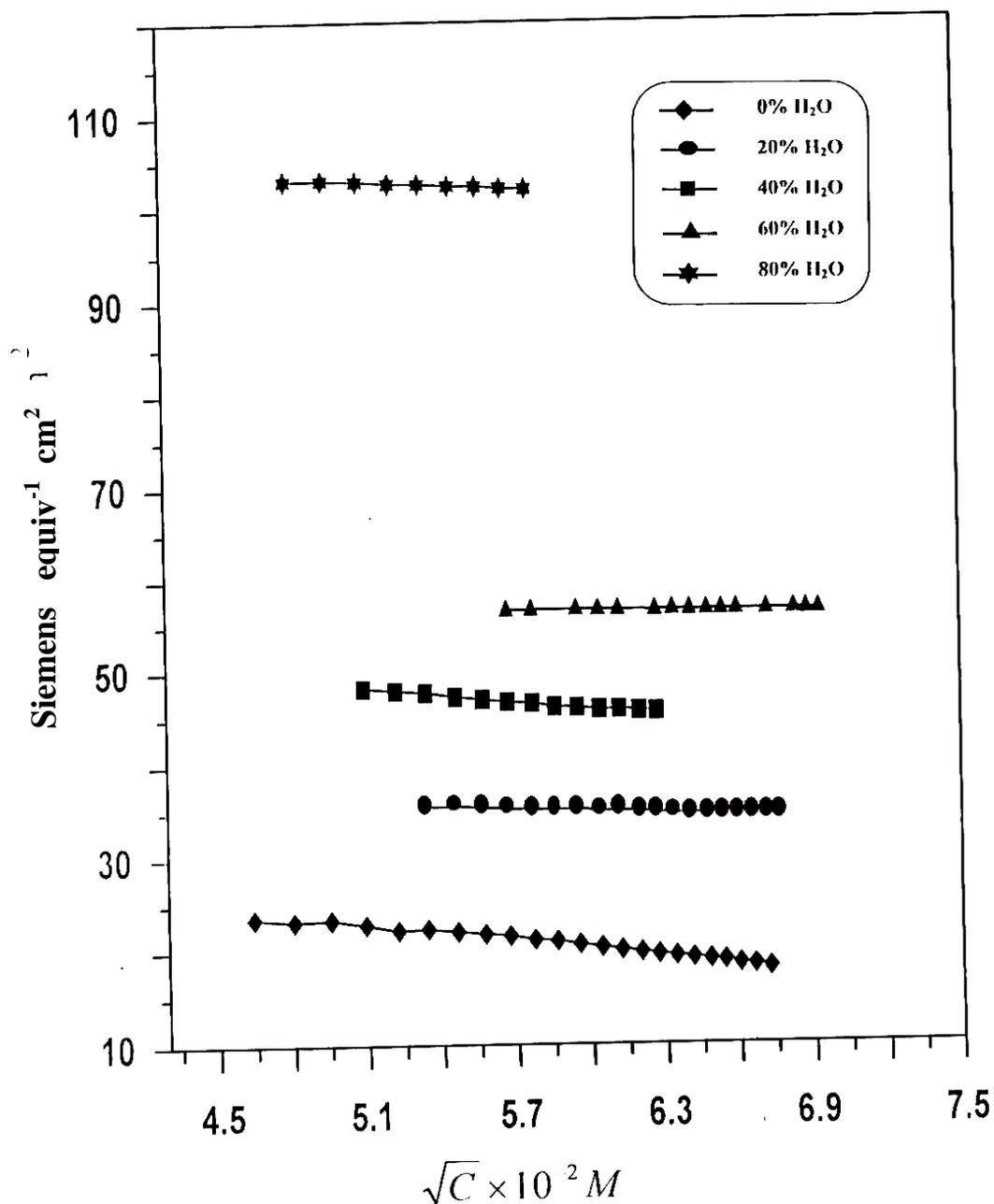


Figure 2: The plot of equivalent conductivity against the square root of molar concentration of $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ in (Ethanol + Water) at 298K^o

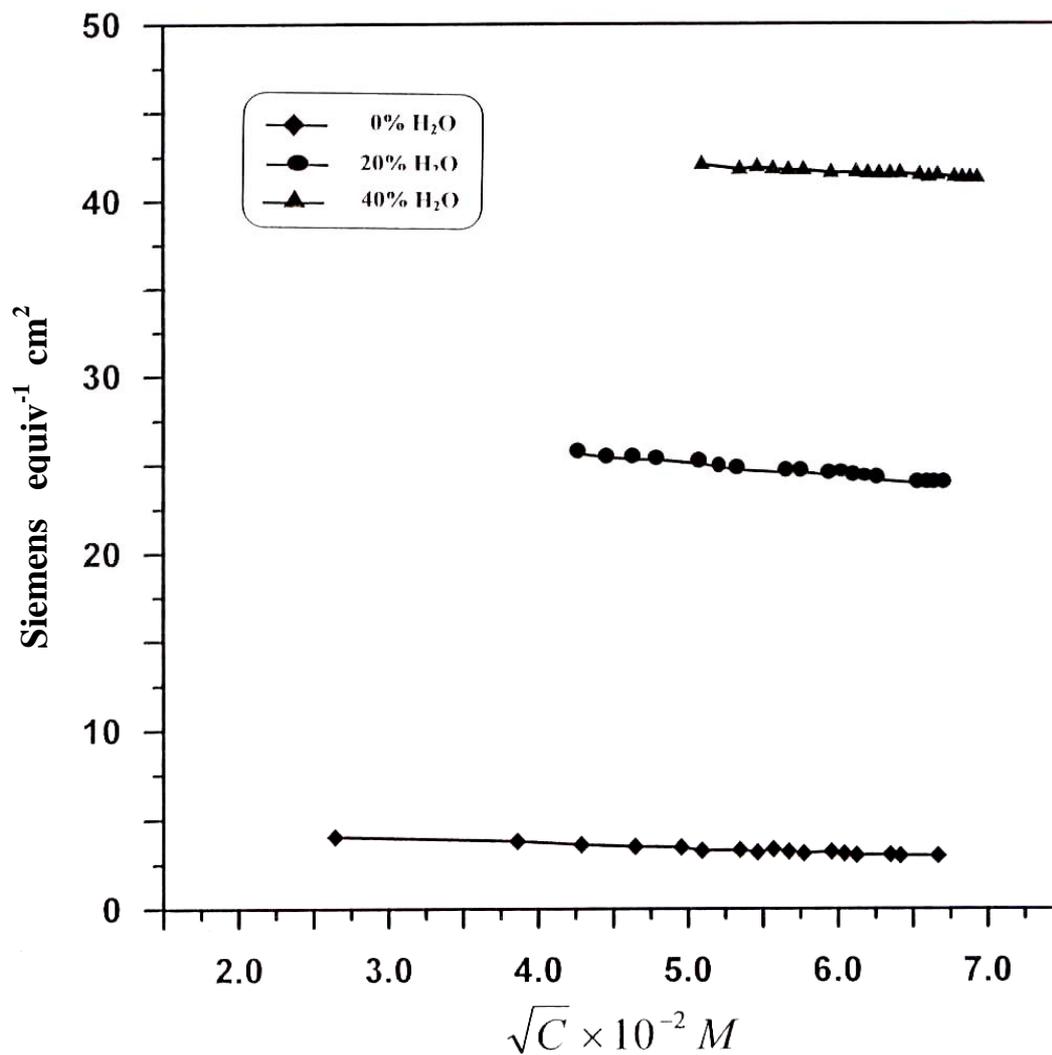


Figure 3: The plot of equivalent conductivity against the square root of molar concentration of $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ in (Isopropanol + Water) at 298K^o

Table 4: Λ_o , k_A , R and $\sigma(\Lambda)$ for $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ in different mixed (alcohol + water) at 298K.

Methanol + Water				
Percentage of alcohol	$\Lambda_o/S \text{ mole}^{-1} \text{ cm}^2$	K_A	R/A°	$\sigma(\Lambda)$
-	204.797 (207.0)*	19.232	28	0.140
20	129.183 (129.0)*	4.898	28	0.079
40	107.353 (106.5)*	1.179	28	0.033
60	85.688 (85.0)*	4.028	28	0.049
80	82.461 (81.5)*	2.966	20	0.036
100	127.241 (127.0)*	32.235	10	0.191
Ethanol + Water				
Percentage of alcohol	$\Lambda_o/S \text{ mole}^{-1} \text{ cm}^2$	K_A	R/A°	$\sigma(\Lambda)$
-	204.797 (207.0)*	19.232	28	0.140
20	108.258 (108.2)*	7.567	28	0.067
40	59.626 (59.6)*	6.199	28	0.035
60	59.146 (59.1)*	64.819	12	0.112
80	42.138 (42.1)*	4.438	8	0.062
100	44.695 (44.6)*	817.471	22	0.032
Isopropanol + Water				
Percentage of alcohol	$\Lambda_o/S \text{ mole}^{-1} \text{ cm}^2$	K_A	R/A°	$\sigma(\Lambda)$
-	204.797(207.0)*	19.232	28	0.140
20	-	-	-	-
40	-	-	-	-
60	44.882 (44.8)*	1.178	20	0.048
80	29.092 (29.09)*	23.485	10	0.062
100	6.916 (5.0)*	1040.705	2	0.0113

* The values of Λ_o between two brackets are obtained by extrapolation of the straight line.

The ion – association constant k_a values are in the order (methanol +water) < (ethanol + water) < (isopropanol + water). This order is expected that such an increase in k_A is partly due to a decrease in the dielectric constant (Hawlicka and Grabowski, 1991) and partly can be explained in terms of diminution in dielectric constant in the vicinity of an ion – pair (Evans and Gardam, 1968). Besides the values of k_A 's generally increase with increasing the percentage of alcohol in each mixture. This is due to the decreasing of the dielectric constant by increasing the percentage of any alcohol.

The distance parameter R is almost constant, this can be explained that it is more characteristic of the salt (Nilson, 1973) which is the same in the three mixtures, but at higher percentage of alcohol, R usually decreases and the solvent effect becomes more effective. The values of R indicates the formation of solvent – separated ion – pairs in each mixture.

The values of $\sigma(\Lambda)$ are small indicating the good applicability of the conductivity equation used (LW equation).

A plot of Λ_0 against the composition of the solvent mixture is shown in figure (4). In the three mixed solvent composition, the limiting equivalent conductance Λ_0 decrease by increasing the wt % of any alcohol since the dielectric constant of the medium decreasing. Beside, the curve of the plot is higher for (methanol + water) due to the higher mobility as compared to that in the lower dielectric medium. In case of methanol and ethanol mixture, a minimum is obtained at 80% of alcohol and then Λ_0 increase when the solvent is only alcohol, this is because the characteristic of pure alcohol is predominate (Islam and Ansari, 1989).

In order to analyse the structural changes of the solution when varying the solvent composition, figure (5), the Walden product ($\Lambda_0\eta$) for the three media represented as a function of the reciprocal dielectric constant $\frac{1}{D}$. From this figure it is clear that the Walden product is not constant. This can be described as to the change of ion solvation and of the solvent structure (Domench and Miro, 1988). Besides, there is a maxima at this may be due to the various degree of solvation of the cations with increasing amount of alcohol in any mixture (Hikmat, 2002).

The height of these maxima are different and so at different percentage depending on the structure breaking power and dehydration of the ions upon addition of the second solvent (Broadwater and kay, 1970).

Comparing our results with the only available data from the literature:

To compare the conductivity and the association of our results for $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ with $[\text{Co}(\text{en})_3]\text{Cl}_2$ (Al-Tamer, 1999) both in water and methanol and at 298K by LW equation, it is found that :

In water:

Compounds	K_A	Λ_0	R/A^0
$[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$	19.23	204.79	28.0
$[\text{Co}(\text{en})_3]\text{Cl}_2$	36.0	145.75	10.0

In methanol:

Compounds	K_A	Λ_0	R/A^0
$[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$	32.2	127.24	10.0
$[\text{Co}(\text{en})_3]\text{Cl}_2$	872.0	118.35	8.3

It is concluded that:

K_A for (2:1) > K_A for (1:1) both in water and in methanol

K_A for (2:1) and } in water < K_A for (2:1) and } in methanol
for (1:1) } for (1:1) }

While

Λ_0 for (2:1) < Λ_0 (1:1) both in water and in methanol

Λ_0 for (2:1) and } in water > Λ_0 for (2:1) and } in methanol
for (1:1) } for (1:1) }

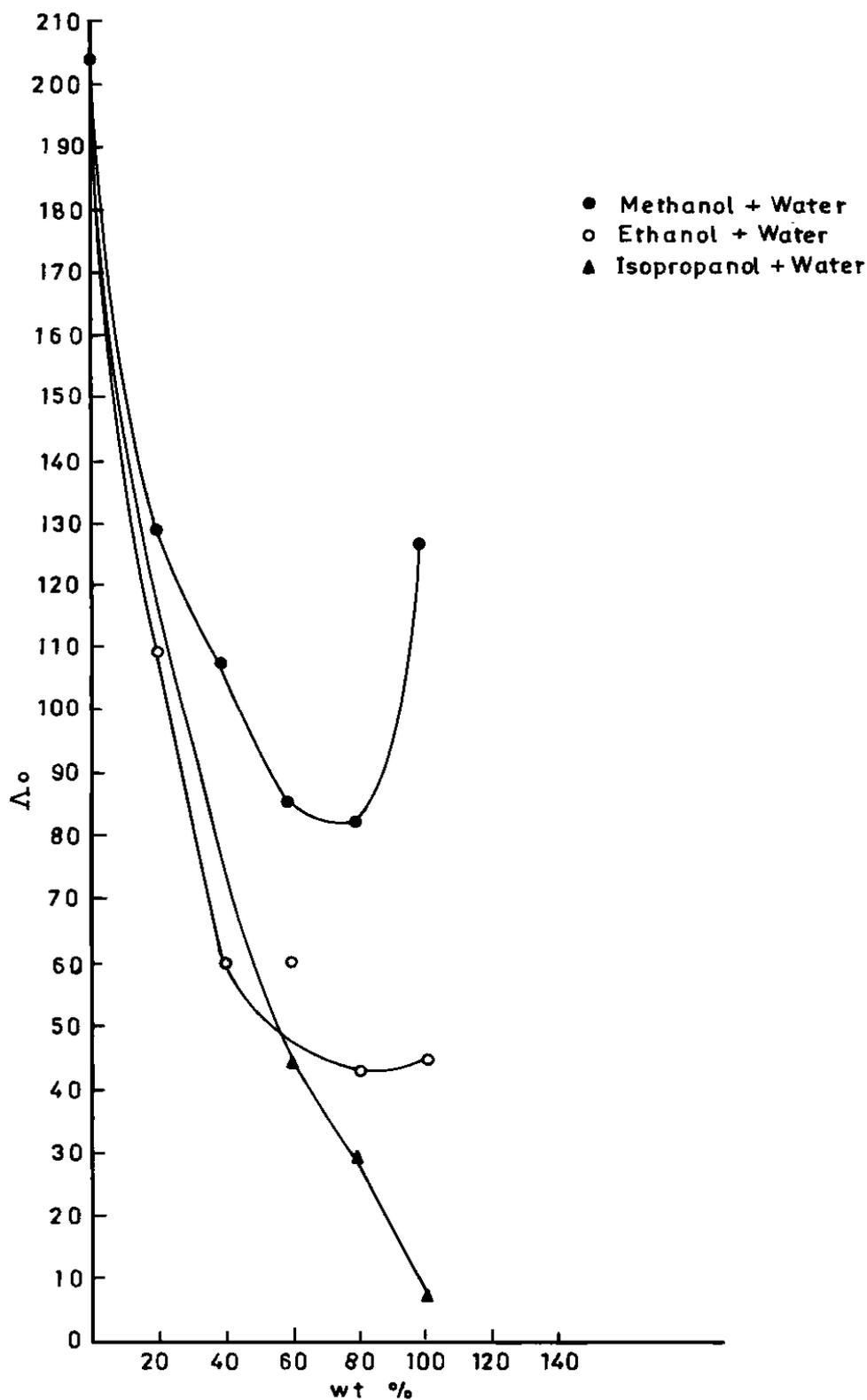


Figure (4) : The plot of limiting equivalent conductance against the wt% of $[\text{CO}(\text{en})_2\text{Cl}_2]\text{Cl}$ in a mixture of (alcohol + water) at 298K.

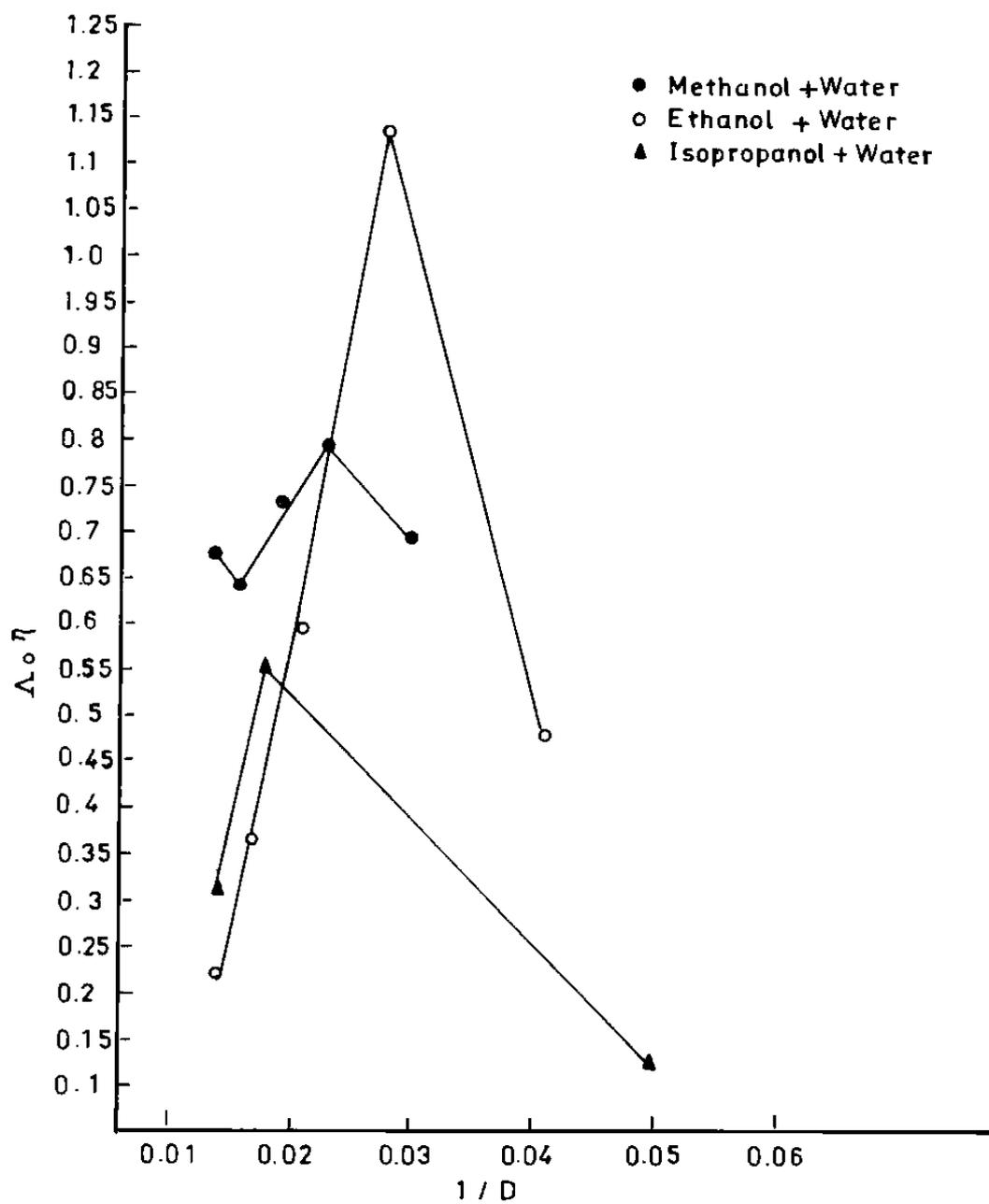


Figure (5) : The plot of the walden product against $1/D$ for $[\text{Co}(\text{en})_2\text{Cl}_2]\text{Cl}$ in a mixture of (alcohol + water) at 298K.

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