Ionic Conductivity in Binary Solvent Mixtures. 2. Ethylene Carbonate + Water at 25 °C

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The conductance behavior of potassium iodide, potassium perchlorate, silver perchlorate, silver picrate, sodium iodide, sodium tetraphenylboride, and tetrabutylammonium iodide has been studied in 20, 40, 60, and 80 mass % ethylene carbonate + water mixtures at 25 °C. The conductance data are treated on the basis of the Fuoss equation, and the limiting molar conductivity (Λ_o), association constant (K_A), and cosphere diameter (R) are derived from the treatment. From the algebraic sum of the limiting molar conductivities of tetrabutylammonium iodide, sodium tetraphenylboride, and sodium iodide, the value of limiting molar conductivity of tetrabutylammonium tetraphenylboride reference electrolyte is calculated for the determination of the limiting ionic conductivity (λ_o) of the systems studied.

Introduction

Ethylene carbonate (1,3-dioxolan-2-one) is one of the more inert of the dipolar aprotic solvents with respect to its acid−base properties as well as other types of chemical attack. Ion−dipole type solute−solvent interactions are expectedly much favored in ethylene carbonate, owing to its high dipole moment (4.87) (*8*). Ethylene carbonate is a good solvent for electrochemical studies, as it has a high relative permittivity (89.78 at 40 °C) (*8*), but relatively less attention has been directed toward it as a solvent (*10, 13*), probably because of its high freezing point (37 °C). Ethylene carbonate + water mixtures are more convenient for practical utilization, as they are miscible at various proportions (viz. 20, 40, 60, and 80 mass % ethylene carbonate + water) at 25 °C with relative permittivity higher than that of water.

The ionic conductivities of certain ions in a 20 mass % propylene carbonate + ethylene carbonate mixture have been reported in our previous work (*12*). The purpose of the present work is to study the behavior of certain univalent electrolytes in different ethylene carbonate + water mixtures through conductivity measurements and to determine their ionic mobilities.

Experimental Section

Solvents. Commercially available (99% pure, Fluka) ethylene carbonate was distilled thrice under reduced pressure. In each distillation only the 80% middle distilled product was collected. The specific conductivity of the purified solvent varied between (1×10^{-7} and 3×10^{-7}) $S{\cdot}cm^{-1}$ at 40 °C.

Demineralized water distilled twice from a Pyrex glass still was used to prepare ethylene carbonate + water mixtures. The specific conductivity of water varied between (7×10^{-7} and 9×10^{-7}) $S{\cdot}cm^{-1}$ at 25 °C.

Both ethylene carbonate and water were stored in sealed containers to prevent contamination from the atmosphere. Known masses of each, ethylene carbonate and water, were mixed to obtain the desired compositions with an accuracy of ±0.01%. All solutions were prepared using a single pan balance (K. Roy, India, K-15 Super) with ±0.01 mg precision.

Table 1. Properties of Ethylene Carbonate (1) + Water (2) at 25 °C

$100W_1$	ϵ^a	$\rho/g{\cdot}cm^{-3\ a}$	$\eta/mPa{\cdot}s^b$
00^c	78.54	0.9971	0.89
20	80.00	1.0551	1.05
40	81.55	1.1178	1.18
60	83.24	1.1842	1.36
80	86.25	1.2595	1.60
100^d	89.78	1.321	1.85

[a] Reference 9. [b] Data obtained from the present work. [c] Reference 3. [d] At 40 °C, ref 8.

Chemicals. Tetrabutylammonium iodide (Sisco, India) and sodium tetraphenylboride (Merck) were of analytical grade and were used without further purification.

Potassium iodide (Loba, India) and sodium iodide (BDH) were recrystallized twice from aqueous ethanol and dried at (100−110) °C before use.

Potassium perchlorate was prepared by dissolving potassium hydroxide in a slight excess of perchloric acid. On evaporation of this solution, crystals of hydrated potassium perchlorate were separated out, recrystallized twice from water and finally from aqueous ethanol, and dried in a vacuum oven at 150 °C. The purity of the salt was checked by the flame photometric method (*14*).

Silver perchlorate was prepared by dissolving freshly prepared silver oxide in a slight excess of perchloric acid. The solution was carefully evaporated, and the obtained solid was allowed to dry in vacuum at ~60 °C. The required silver oxide was freshly prepared by adding an excess of sodium hydroxide solution to a solution of silver nitrate and washing the precipitated oxide with water until the filtrate gave only a very faint pink coloration to phenolphthalein indicator.

Silver picrate was prepared by the reaction of picric acid and an excess of silver nitrate; the product obtained was recrystallized twice from ethanol and air dried. The purity of the silver salts was tested by Volhard's method (*14*).

Conductivity Measurements. All conductivity measurements were made at (25 ± 0.05) °C using a dip-type cell (cell constant 1.073 cm^{-1}) with a lightly platinized electrode, as described earlier (*11*). The conductivity meter was regularly standardized using standard potassium chloride solutions. All molar conductivities reported here have been calculated after correcting for the solvent

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Table 2. Conductance Data in Ethylene Carbonate (1) + Water (2) at 25 °C

$100W_1 = 20$		$100W_1 = 40$		$100W_1 = 60$		$100W_1 = 80$	
10^4c/mol·dm^{-3}	Λ/S·cm·mol^{-1}	10^4c/mol·dm^{-3}	Λ/S·cm^2·mol^{-1}	10^4c/mol·dm^{-3}	Λ/S·cm^2·mol^{-1}	10^4c/mol·dm^{-3}	Λ/S·cm^2·mol^{-1}
Bu_4NI							
394.6	59.60	265.4	53.12	210.7	47.96	189.5	38.17
315.2	61.86	225.9	53.90	154.0	50.25	136.7	39.22
218.0	64.44	170.6	55.74	109.8	51.61	85.9	40.87
137.3	68.19	123.2	57.10	85.3	52.19	55.3	42.35
88.2	72.07	79.2	59.51	61.7	54.12	35.6	44.08
63.8	73.32	54.8	61.51	45.1	54.33	23.2	44.56
46.6	75.85	41.9	63.36	32.1	55.89	14.6	45.55
34.7	78.67	31.2	63.98	21.7	56.80	8.9	47.47
24.0	79.41	21.5	64.59	14.1	57.33	3.0	48.06
16.2	79.64	14.5	65.04	9.4	58.13		
				6.3	59.13		
$NaPh_4B$							
279.0	59.79	250.8	51.26	237.0	38.55	278.7	29.86
205.7	60.41	181.2	51.79	173.6	40.88	221.4	30.45
140.1	61.33	141.8	52.10	125.4	41.36	168.7	31.13
83.3	62.63	107.7	52.54	92.4	42.23	124.3	31.86
50.8	63.70	80.5	53.17	62.1	43.43	83.6	32.75
37.0	64.09	61.5	53.65	42.6	44.48	57.3	33.45
27.0	64.39	43.8	54.19	28.9	45.39	40.8	33.98
19.7	64.75	29.6	54.80	17.6	45.94	27.7	34.47
15.2	64.87	19.2	55.18	10.4	47.03	17.9	34.90
12.2	65.21	12.9	55.49	6.0	48.01	12.0	35.16
9.2	65.39	8.6	55.79			8.0	35.41
						4.0	35.93
NaI							
233.0	96.89	171.8	78.71	263.4	55.11	320.2	50.26
184.8	97.91	135.4	79.73	216.0	56.58	253.5	51.23
147.6	99.12	102.1	81.54	172.4	58.43	202.8	51.99
116.3	100.73	73.3	83.71	135.9	60.45	159.9	52.95
87.7	102.94	50.2	85.78	102.5	63.26	120.6	54.01
62.9	106.00	35.8	87.07	73.6	65.31	86.4	55.75
43.1	108.82	24.2	88.58	50.4	67.75	28.5	57.99
30.7	110.27	15.7	89.55	35.9	69.27	18.5	54.05
20.8	111.19	10.5	89.66	24.3	70.69	12.4	58.44
13.5	111.90	7.0	89.99	7.1	74.55	8.3	58.67
9.0	112.73						
6.1	113.62						
KI							
192.5	98.73	173.4	82.83	225.9	64.11	176.8	42.45
155.8	102.44	120.9	85.13	194.8	64.95	144.1	44.68
118.6	104.25	84.9	87.60	138.4	66.85	108.8	45.91
84.3	106.47	58.7	90.39	94.3	68.72	71.9	47.53
57.4	109.65	44.9	91.70	63.4	71.01	42.9	50.02
38.6	112.86	31.9	94.20	43.4	72.49	26.7	51.81
18.8	116.51	21.6	95.78	30.9	73.78	19.0	52.83
12.7	117.29	14.0	98.31	20.9	74.38	12.8	54.09
8.3	118.13	9.4	99.02	13.6	75.34	8.3	55.78
		3.2	100.58	9.1	76.57	1.9	57.42
$KClO_4$							
221.4	90.51	190.7	81.01	169.3	67.92	220.3	54.76
168.7	92.31	154.7	82.70	132.0	68.61	156.9	56.19
124.3	94.18	119.3	84.50	97.5	69.67	125.9	56.97
83.6	96.52	86.7	86.43	68.5	71.34	103.7	57.52
57.3	98.31	59.1	88.40	47.4	72.23	80.1	58.35
40.8	99.64	39.0	89.59	36.2	73.43	61.5	59.01
27.6	100.51	28.4	91.27	25.8	74.29	42.0	59.88
17.9	101.79	19.6	92.46	17.4	74.96	21.5	60.97
12.0	102.58	14.0	93.01	11.3	75.41	15.9	61.30
8.0	103.31	9.4	93.72	7.6	75.93	11.6	61.78
4.0	104.29	7.1	94.39	5.1	76.51	8.7	62.08
		3.5	95.51	2.5	77.40	5.8	62.47
$AgClO_4$							
229.8	93.63	172.8	74.16	180.8	58.46	305.8	49.48
205.6	94.52	140.8	74.87	129.8	59.71	275.6	50.19
178.1	95.55	103.6	76.02	112.6	59.96	241.2	50.59
146.3	97.13	59.6	77.71	92.8	60.58	201.7	50.97
109.4	98.73	29.3	79.43	69.8	61.38	145.9	51.61
92.9	99.91	23.9	79.69	59.5	61.76	90.3	53.38
75.3	101.03	20.6	79.71	48.4	61.98	65.3	54.24
56.3	102.52	17.2	80.14	36.6	62.59	38.3	55.37
36.0	104.12	13.9	80.79	23.9	63.12	24.0	55.93
25.2	105.36	10.5	81.41	17.2	63.52	16.7	56.76
19.7	106.06			13.7	63.69		
14.0	106.34			10.2	63.81		

Table 2. (Continued)

$100W_1 = 20$		$100W_1 = 40$		$100W_1 = 60$		$100W_1 = 80$	
10^4c/mol·dm^{-3}	Λ/S·cm·mol^{-1}	10^4c/mol·dm^{-3}	Λ/S·cm^2·mol^{-1}	10^4c/mol·dm^{-3}	Λ/S·cm^2·mol^{-1}	10^4c/mol·dm^{-3}	Λ/S·cm^2·mol^{-1}
			Ag-Pic				
322.8	80.36	310.7	66.77	227.4	49.61	184.4	40.61
285.7	81.36	298.5	67.15	201.3	49.97	121.9	41.68
238.9	82.60	276.3	67.22	169.7	50.48	85.6	42.03
188.9	83.81	251.3	67.66	136.1	51.35	59.2	42.38
137.0	85.85	223.0	68.45	34.4	54.11	45.2	42.48
85.4	88.22	190.6	69.27	16.4	55.06	26.3	43.25
62.1	89.43	136.7	70.31	11.7	55.40	18.7	43.60
23.7	92.24	90.0	71.82	6.9	55.80	11.0	43.73
16.9	92.96	57.9	73.07	4.0	55.99	6.3	44.09
10.0	93.95	34.5	73.93			3.2	44.39
		15.8	75.12				

Table 3. Derived Conductivity Parameters in Ethylene Carbonate (1) + Water (2) at 25 °C

$100W_1$	$\Lambda_o = \Delta\Lambda_o$/S·cm^2·mol^{-1}	K_A	K_R	K_S	R/°A	σ/%	$\Lambda_o\eta$/S·cm^2·mol^{-1}·Pa·s
			Bu_4NI				
20	84.38 ± 0.46	50.40	19.83	1.54	17.4	0.73	0.886
40	67.93 ± 0.29	43.47	19.14	1.27	17.2	0.59	0.802
60	59.84 ± 0.22	56.10	29.40	0.91	20.3	0.61	0.814
80	49.03 ± 0.23	136.82	80.70	0.69	29.5	0.68	0.784
			$NaPh_4B$				
20	66.19 ± 0.05	37.79	28.59	0.32	20.0	0.15	0.695
40	56.71 ± 0.05	83.57	68.03	0.23	27.6	0.14	0.669
60	48.59 ± 0.25	69.53	40.17	0.73	22.8	0.80	0.661
80	36.12 ± 0.04	43.35	25.03	0.73	19.2	0.20	0.578
			NaI				
20	115.59 ± 0.36	47.49	27.09	0.75	19.6	0.56	1.214
40	91.78 ± 0.20	27.66	11.72	1.36	14.2	0.36	1.083
60	76.21 ± 0.21	45.51	13.56	2.36	15.1	0.36	1.036
80	59.57 ± 0.15	17.96	7.80	1.30	12.2	0.44	0.953
			KI				
20	121.01 ± 0.39	43.75	21.04	1.08	17.8	0.47	1.271
40	102.41 ± 0.21	88.94	50.76	0.75	24.8	0.32	1.208
60	78.27 ± 0.19	63.93	38.29	0.67	22.4	0.36	1.064
80	58.18 ± 0.27	132.00	66.43	0.99	27.5	0.70	0.931
			$KClO_4$				
20	104.97 ± 0.10	39.00	23.94	0.63	18.7	0.18	1.102
40	96.06 ± 0.14	48.56	28.08	0.73	19.9	0.28	1.134
60	77.68 ± 0.12	75.95	53.30	0.42	25.3	0.29	1.056
80	63.00 ± 0.06	37.11	23.34	0.59	18.7	0.17	1.008
			$AgClO_4$				
20	108.88 ± 0.12	24.37	12.22	0.99	14.4	0.11	1.143
40	82.77 ± 0.21	78.42	60.21	0.30	26.4	0.25	0.977
60	64.48 ± 0.07	22.94	14.13	0.62	16.9	0.11	0.877
80	58.10 ± 0.19	55.51	38.58	0.44	24.0	0.33	0.930
			Ag-Pic				
20	95.25 ± 0.08	27.67	15.71	0.76	15.9	0.08	1.000
40	76.43 ± 0.13	16.84	9.05	0.86	12.8	0.17	0.902
60	56.42 ± 0.09	34.84	22.65	0.54	18.4	0.16	0.767
80	44.41 ± 0.13	49.92	38.92	0.28	24.3	0.30	0.711

conductivity. All conductivity measurements were repeated to get concordant results.

Viscosity Measurements. The viscosity of the ethylene carbonate + water mixtures was determined using a Brookfield LVT DVII viscometer with a UL adapter.

Results and Discussion

The properties, relative permittivity (ϵ) (*9*), density (ρ) (*9*), and viscosity (η), of ethylene carbonate + water mixtures are given in Table 1. The molar conductivity (Λ) as a function of concentration (c) in 20, 40, 60, and 80 mass % ethylene carbonate + water mixtures at 25 °C is given in Table 2. The conductivity results for tetrabutylammonium iodide, sodium tetraphenylboride, sodium iodide, potassium iodide, potassium perchlorate, silver perchlorate, and silver picrate were analyzed by the Fuoss conductance concentration equation (*5*). The computer program "SCAN ON RHO", was used as described in the previous paper (*12*) to determine the best fit conductivity parameters such as limiting molar conductivity (Λ_o), association constant (K), and cosphere diameter (R) (Table 3) for the systems studied.

Using the values of limiting molar conductivity obtained from computation of the conductivity data and considering that the limiting molar conductivity is an algebraic sum of ionic conductivities of all ions present in the molecule, we calculated the value of the limiting molar conductivity for tetrabutylammonium tetraphenylboride by the following relation:

$$\Lambda_o(Bu_4NBPh_4) = \Lambda_o(Bu_4NI) + \Lambda_o(NaBPh_4) - \Lambda_o(NaI) \quad (1)$$

Tetrabutylammonium tetraphenylboride was used as a reference electrolyte (*2, 7*) for the determination of the limiting ionic conductivities. According to the assumption of Fuoss et al. (*6*), the limiting transference number of the Bu_4N^+ ion in Bu_4NBPh_4 is 0.519 and independent of the

Table 4. Limiting Ionic Conductivity, λ_o, and Ionic Walden Product, $\lambda_o\eta$, in Ethylene Carbonate (1) + Water (2) at 25 °C

$100W_1$	Bu_4N^+	Na^+	K^+	Ag^+	Ph_4B^-	I^-	ClO_4^-	Pic^-
			Limiting Ionic Conductivity, λ_o/S·cm^2·mol^{-1}					
0[a]	19.5	50.08	73.48	61.9	18.1	76.8	67.3	30.37
20	18.15	49.36	54.78	58.69	16.83	66.23	50.19	36.56
40	17.05	40.90	51.53	38.24	15.81	50.88	44.53	38.19
60	16.72	33.09	35.15	21.95	15.50	43.12	42.53	34.47
80	13.28	23.82	22.43	17.53	12.30	35.75	40.57	26.88
			Ionic Walden Product, $\lambda_o\eta$/S·cm^2·mol^{-1}·Pa·s					
0	0.174	0.446	0.654	0.551	0.161	0.683	0.599	0.270
20	0.191	0.518	0.575	0.616	0.177	0.695	0.527	0.384
40	0.201	0.483	0.608	0.451	0.187	0.600	0.525	0.451
60	0.227	0.450	0.478	0.298	0.211	0.586	0.578	0.469
80	0.212	0.381	0.359	0.280	0.197	0.572	0.649	0.430

[a] Reference 3.

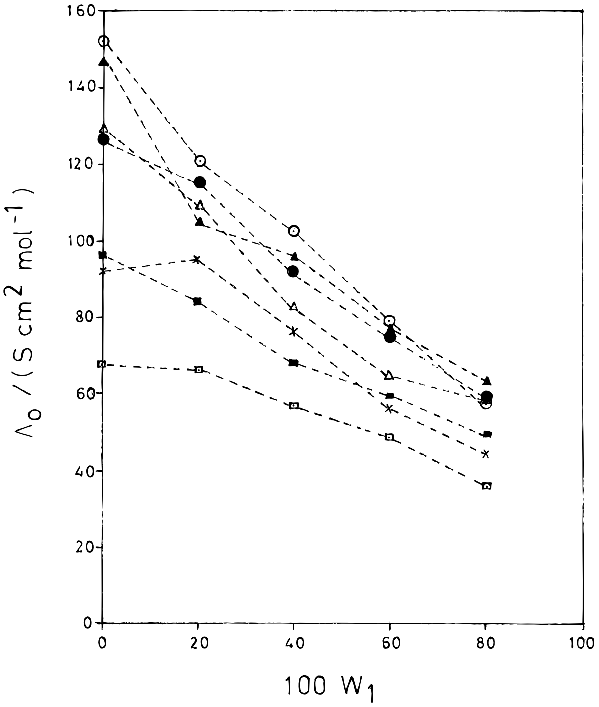

Figure 1. Variation of Λ_o with mass % ethylene carbonate (W_1): Bu_4NI (■); $NaPh_4B$ (⊡); NaI (●); KI (⊙); $KClO_4$ (▲); $AgClO_4$ (△); Ag-Pic (×).

kind of solvent used, so the limiting ionic conductivity was calculated on the basis of the following relation:

$$\lambda_o(Bu_4N^+) = 0.519\Lambda_o(Bu_4NBPh_4) \tag{2}$$

The values of the limiting ionic conductivity (λ_o) in 20, 40, 60, and 80 mass % ethylene carbonate + water mixtures calculated using the above equation are given in Table 4. The ionic mobility decreases with the increasing amount of ethylene carbonate in the mixture in all cases except the picrate ion, as expected because of the gradual increase in viscosity of the solvent at the ethylene carbonate rich region. The unusual behavior of the picrate ion has also been observed in propylene carbonate and propylene carbonate + ethylene carbonate mixtures (*12*) and also in sulfolane + water mixtures (*11*) earlier.

The variation of the limiting molar conductivity with mass % ethylene carbonate is shown in Figure 1. A steady decrease in Λ_o for each electrolyte with a corresponding increase in viscosity of the solvent mixture has been observed.

The variation of salt and ionic Walden product for each system with mass % ethylene carbonate is presented in Figures 2 and 3, respectively.

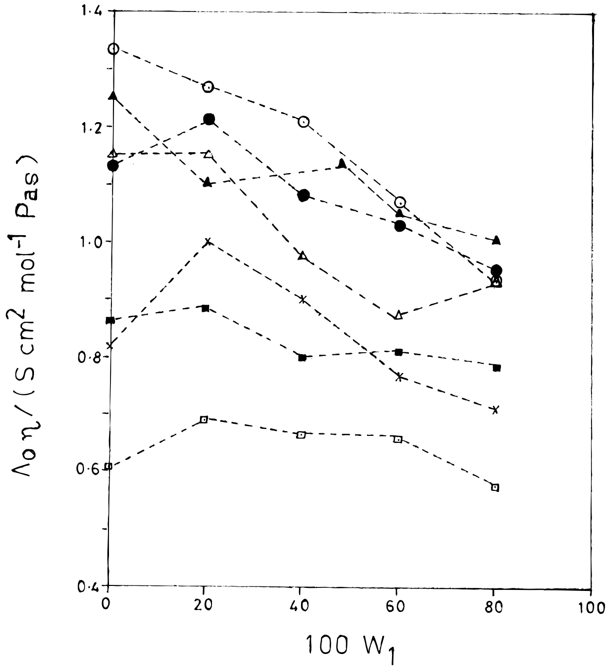

Figure 2. Variation of salt Walden product $\Lambda_o\eta$ with mass % ethylene carbonate (W_1): Bu_4NI (■); $NaPh_4B$ (⊡); NaI (●); KI (⊙); $KClO_4$ (▲); $AgClO_4$ (△); Ag-Pic (×).

It is observed from Figure 2 that the sodium salts ($NaPh_4B$ and NaI), potassium iodide, and silver picrate show a steady decrease in Walden product while in the case of other electrolytes various minima and maxima were observed. However, it is necessary to know ionic rather than salt Walden products in order to avoid erroneous conclusions about ion–solvent interactions. Various reasonings (*4, 16*) including the effect of dielectric loss due to the ion–solvent dipole interaction on the mobility of ions (*1, 15*) are given for variation of the ionic Walden product in different compositions of solvents. From Figure 3 it is observed that the bulkier ions, tetrabutylammonium and tetraphenylboride, nearly obey the Walden rule while the other ions show deviation from it. The Walden product values for Ag^+, Na^+, and I^- are found to increase from 0 to 20 mass % ethylene carbonate + water mixtures and thereafter to decrease up to the 80 mass % ethylene carbonate + water mixture. The increase in the Walden product from 0 to 20 mass % ethylene carbonate + water mixtures in the case of Ag^+, Na^+, I^-, and Pic^- could be rationalized due to the less relative decrease in their conductivities as compared to the increase in the viscosity of the solvent mixtures. Above 20 mass % ethylene carbonate + water mixture the decrease in the Walden product in the case of Ag^+, Na^+, and I^- is due to the large decrease in ionic conductivities with the increasing amount

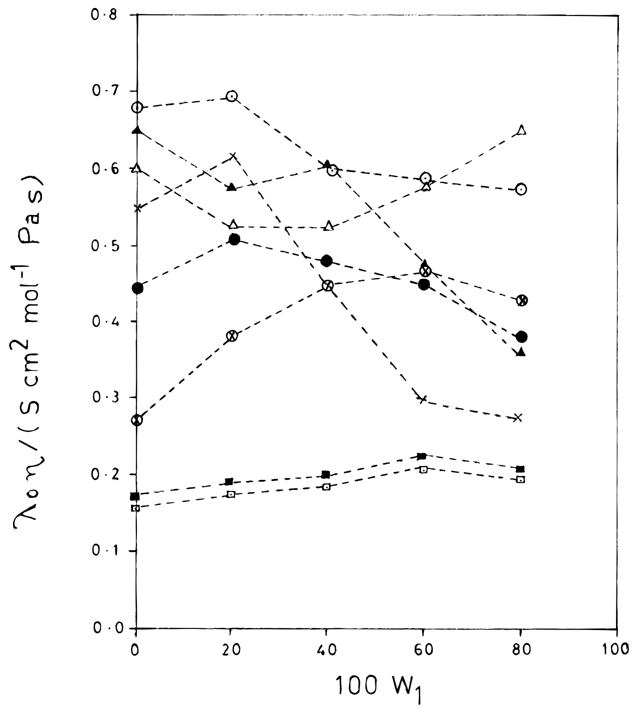

Figure 3. Variation of ionic Walden product $\lambda_o\eta$ with mass % ethylene carbonate (W_1): Bu_4N^+ (■); Na^+ (●); K^+ (▲); Ag^+ (×); Ph_4B^- (⊡); I^- (⊙); ClO_4^- (△); Pic^- (⊗).

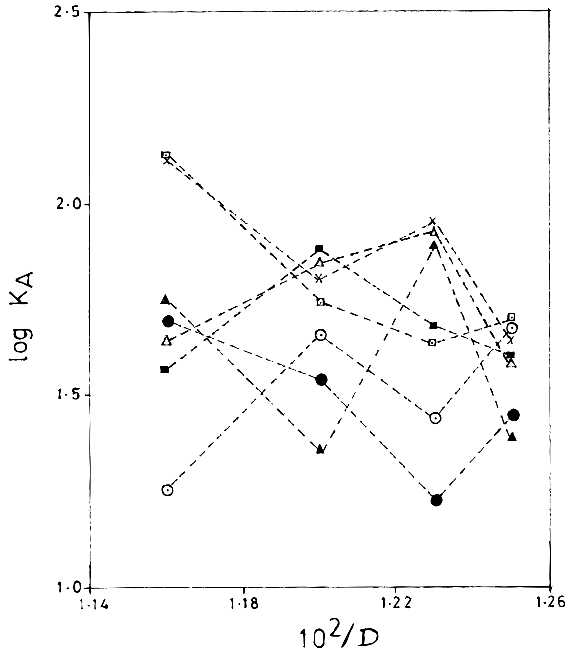

Figure 4. Plots of log K_A vs $1/D$: Bu_4NI (⊡); $NaPh_4B$ (△); NaI (⊙); KI (×); $KClO_4$ (■); $AgClO_4$ (▲); AgPic (●).

of ethylene carbonate in the solvent mixtures. The Walden product in the case of K^+ and ClO_4^- remains almost constant up to the 40 mass % ethylene carbonate + water mixture. Beyond this composition, there is a sharp increase in the case of ClO_4^- and a sharp decrease in the case of K^+. This opposite trend for the two ions could be understood on the basis of the relative solvation effect.

The association constant or the conductivity pairing constant (K_A) for all the systems is found to be generally appreciable (>15), which reflects a strong association in ethylene carbonate + water mixtures. The association constant is the product of two terms (5), one (K_R) of which depends explicitly on the relative permittivity of the solvent while the other (K_S) is a system specific constant depending on short-range ion–ion and ion–solvent interactions. The relationship is given as

$$K_A = K_R(1 + K_S) \tag{3}$$

The conventional log K_A vs $1/D$ plots given in Figure 4 never showed any linear variation for all the salts studied, indicating that the ion association may not be solely dependent on the relative permittivity of the medium; however, the other factors like shape, size, dipole moment, and possibly other details of solvent molecules could also be taken into account for the rationalization of association constant values.

The cosphere diameter (R) values for all the salts under study in ethylene carbonate + water mixtures were also reported in Table 3. The R values were found to have a somewhat irregular trend in the solvents studied. As R is a measure of the extent of influence of ionic charge in the solvent and consequently the ionic association, R and K_A values are expected to decrease with the increase in the relative permittivity of the solvents, but no systematic trend in R values for all the salts studied has been observed in the studied solvent mixtures. Since the best fit conductivity parameters can be reproduced equally well over a wide range of arbitrarily chosen R values, a comprehensive correlation of the cosphere diameter of the respective systems could not be made in the present instance.

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