

Electrolytic Conductances of Tetrabutylammonium Tetrphenylborate in Propylene Carbonate + Acetonitrile Mixtures at (25, 30, and 35) °C

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The electrolytic conductances of tetrabutylammonium tetrphenylborate in a number of propylene carbonate + acetonitrile mixtures at (25, 30, and 35) °C are reported. The limiting molar conductances, association constants, and distance of closest approach of ions are evaluated by means of the Lee–Wheaton equation. Gilkerson's solvent–solute interaction energy term is calculated. Values of the Walden product are also reported.

Introduction

Conductometric studies on electrolyte solutions have diverse applications to electrochemical power sources (Salomon, 1990). An important solvent used for nonaqueous electrolytes in high-energy lithium batteries is propylene carbonate (Gabano, 1983; Ue, 1994), which is a colorless liquid of wide liquid range (mp –40 °C, bp 242 °C) (Boschke, 1972) with a high dielectric constant (65.1 at 25 °C) (Moumouzias et al., 1991).

As solvent composition plays an important role in the conductivity of an electrolyte, there is an interest in mixtures of propylene carbonate with other aprotic solvents (Matsuda et al., 1983; Tobishima and Yamaji, 1984).

Tetrabutylammonium tetrphenylborate is a useful reference electrolyte because both ions have about the same volume and half the limiting conductance is assumed to give the single ion conductances of Bu_4N^+ and BPh_4^- , whereby the single conductances of other cations and anions can be obtained (Brown and Fuoss, 1960). In this work, the electrolytic conductance behavior of Bu_4NBPh_4 in propylene carbonate + acetonitrile at (25, 30, and 35) °C is studied.

Experimental Section

Propylene carbonate (Merck, >99%) and acetonitrile (Merck, min 99.5%) were stored in flasks with molecular sieves (5 Å for propylene carbonate and 3 Å for acetonitrile) for 24 h and then were treated by fractional distillation twice. The final product of both had a specific conductance lower than $10^{-7} \text{ S cm}^{-1}$.

As proposed by Barthel et al. (1980), KCl (Merck, min 99.5%) was recrystallized from conductivity water, dried at about 80 °C under vacuum, ground, and dried once more for two days at about 290 °C in the presence of P_2O_5 . Bu_4NBPh_4 (Fluka, >99%) was recrystallized from an acetone + water mixture, dried, ground, and dried once more under vacuum.

The cells for the conductance measurements were similar to those discussed by Dagget et al. (1951). Their electrodes were platinized to minimize polarization effects (Johnes and Christian, 1935; Johnes and Bollinger, 1935). In order to determine the constants of the cells, the equation of Lind–Zwolenik–Fuoss (Lind et al., 1959) was used on various concentrations of aqueous potassium chloride solutions at 25 °C. A Beckman (model RC–18A) conductivity bridge with accuracy $\pm 0.05\%$ was used to measure the

Table 1. Physical Properties of Propylene Carbonate (1) + Acetonitrile (2) at (25, 30, and 35) °C

100 w_1	$d/(\text{g cm}^{-3})$	$\eta/(\text{mPa s})$	ϵ
25 °C			
100	1.1995	2.512	65.16
90	1.1401	1.675	60.67
70	1.0387	0.968	53.03
50	0.9503	0.642	47.05
30	0.8736	0.479	42.05
0	0.7759	0.344	35.99
30 °C			
100	1.1926	2.272	64.13
90	1.1355	1.535	59.54
70	1.0333	0.906	52.12
50	0.9448	0.605	46.13
30	0.8681	0.453	41.23
0	0.7717	0.328	35.24
35 °C			
100	1.1887	2.080	63.07
90	1.1301	1.420	58.48
70	1.0280	0.846	51.32
50	0.9396	0.575	45.34
30	0.8630	0.432	40.45
0	0.7662	0.314	34.54

resistance of the solutions. All the solutions and the cells were maintained in a thermostated oil bath (Leeds and Northrup, model 4956, with a precision of ± 0.002 °C).

For cleaning cells and glass apparatus, an aqueous solution of DECON, 5%, and distilled water were used. The preparation of the solution was made in a glovebox, under dry nitrogen. After measuring the conductance of the pure solvent or solvent mixture in the cell, amounts of the stock solution were dispensed into the cell from a weight buret. Stock solutions were prepared by mass. If we suppose that W_1 is the mass of the salt in the stock solution, W_2 is the mass of the stock solution, W_3 is the mass of the stock solution which is added each time, W_4 is the initial mass of the solvent into the cell, and R_1 and R_2 are correction factors correcting for the weight in air compared to that in a vacuum for the salt and the solvent, respectively, then the molality m of the solution is

$$m = \frac{W_3 W_1 R_1 1000 / W_2}{[W_4 + W_3(1 - W_1/W_2)] R_2 M} \quad (1)$$

The molarity of the solution c would be given by

$$c = md/(1 + 0.001mM) \quad (2)$$

Table 2. Molar Conductances for Bu₄NBPh₄ in Propylene Carbonate (1) + Acetonitrile (2) at 25 °C

$w_1 = 1.0$		$w_1 = 0.9$		$w_1 = 0.7$	
$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$
1.1148	17.090	3.1042	25.447	3.1727	42.890
4.1947	16.815	5.1400	25.215	3.6130	42.732
5.3296	16.755	6.8049	25.114	4.4103	42.583
7.9670	16.640	8.7441	24.963	5.0478	42.481
10.0810	16.519	10.1306	24.876	6.1771	42.308
12.2545	16.443	12.1998	24.739	7.2672	42.150
16.9410	16.301	14.0489	24.629	9.2004	41.901
20.0340	16.215	15.0131	24.581	11.1039	41.689
25.1710	16.086	17.4207	24.460	13.9899	41.388
33.4028	15.905	19.5028	24.383	17.8151	41.045
39.5528	15.783	20.3552	24.356	25.6712	40.460
46.3726	15.680				
56.7218	15.500				

$w_1 = 0.5$		$w_1 = 0.3$		$w_1 = 0.0$	
$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$
0.6099	64.220	1.8450	84.489	2.0146	115.366
2.6393	63.150	3.8415	83.385	4.8527	113.253
3.7422	62.710	6.2169	82.445	4.8968	113.123
4.1226	62.610	7.2696	82.063	7.7096	111.542
5.6551	62.130	9.4614	81.396	10.5421	110.236
7.2802	61.728	11.5820	80.848	12.7523	109.356
9.3509	61.308	13.4830	80.365	17.0236	107.922
11.6928	60.879	15.8179	79.844	20.6814	106.800
13.9697	60.503	22.1197	78.629	25.0865	105.700
16.7712	60.090	26.9166	77.880	34.8358	103.525
20.4176	59.580	35.1764	76.750		

Table 3. Molar conductances for Bu₄NBPh₄ in Propylene Carbonate (1) + Acetonitrile (2) at 30 °C

$w_1 = 1.0$		$w_1 = 0.9$		$w_1 = 0.7$	
$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$
1.6826	18.875	2.0810	27.865	1.1737	47.100
3.4733	18.730	3.5326	27.680	2.2649	46.780
4.1413	18.680	4.7171	27.550	4.8813	46.200
5.1045	18.610	7.0585	27.302	10.0839	45.430
6.1094	18.551	8.6902	27.184	12.1071	45.158
7.6057	18.473	11.1024	27.040	14.5470	44.900
9.5623	18.384	12.5431	26.953	22.2700	44.220
11.8063	18.286	14.6224	26.833	27.4880	43.890
14.6888	18.180	17.0821	26.685		
18.2306	18.060				
19.8101	18.022				
22.9490	17.953				

$w_1 = 0.5$		$w_1 = 0.3$		$w_1 = 0.0$	
$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$	$10^4 c / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{ mol}^{-1})$
1.4693	68.030	0.4981	90.950	2.2775	121.576
2.8632	67.382	2.4147	89.250	4.8178	119.433
4.1040	66.930	3.9901	88.365	8.7547	117.324
5.6744	66.440	5.3970	87.745	11.906	116.050
8.0813	65.850	6.7240	87.262	15.101	114.800
9.8750	65.477	8.2550	86.744	17.529	113.960
11.378	65.171	11.776	85.710	20.686	112.960
14.399	64.628	12.896	85.416	24.633	111.900
19.442	63.854	14.647	85.050	27.257	111.250
25.932	63.100	17.147	84.510	32.548	110.110
				36.791	109.210
				40.788	108.520

where d is the density of the solution and M the molecular weight of the salt.

Results and Discussion

Propylene carbonate + acetonitrile solvent mixture properties were measured in a previous work (Moumouzias et al., 1991) and are summarized in Table 1. Molar conductances Λ at different concentrations c are listed in Tables 2–4.

Experimental results were treated by the Lee–Wheaton equation (Lee and Wheaton, 1978a,b). The set of equations

were in the form

$$\Lambda = \gamma \Lambda_i \quad (3a)$$

$$K_A = (1 - \gamma) / (\gamma^2 c f^2) \quad (3b)$$

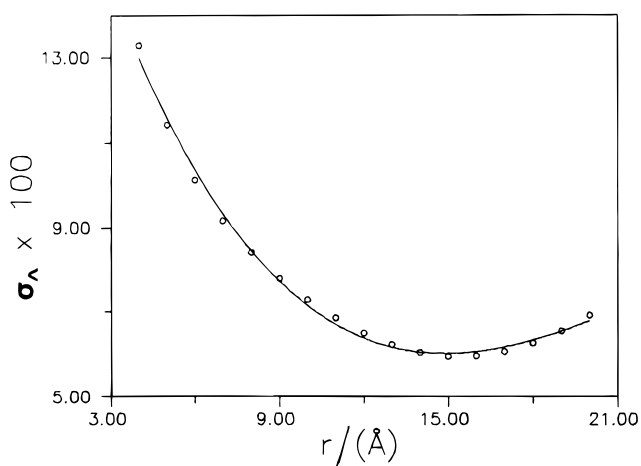
$$-\ln f = \frac{(2\pi N e^6 / 1000 (\epsilon k T))^{1/2} (c \gamma)^{1/2}}{1 + (8\pi N e^2 / 1000 \epsilon k T)^{1/2} r(c \gamma)^{1/2}} \quad (3c)$$

where Λ_i is the theoretical molar conductance that results from the equation, γ the degree of dissociation, f the activity

Table 4. Molar Conductances for Bu₄NBPh₄ in Propylene Carbonate (1) + Acetonitrile (2) at 35 °C

$w_1 = 1.0$		$w_1 = 0.9$		$w_1 = 0.7$	
$10^4 d / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{mol}^{-1})$	$10^4 d / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{mol}^{-1})$	$10^4 d / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{mol}^{-1})$
1.4457	20.793	1.8330	30.668	2.0536	50.870
4.2853	20.470	5.5375	30.182	4.3547	50.350
7.5053	20.325	8.4868	29.920	5.9529	50.005
10.8216	20.162	10.6473	29.760	9.6037	49.476
15.4478	19.991	14.8149	29.498	12.5727	49.107
19.3047	19.858			15.5330	48.774
23.0603	19.740			18.5220	48.467
27.0793	19.625			23.4243	48.026
32.4309	19.485			25.6629	47.880
36.6939	19.400			29.6206	47.600
43.7299	19.300			41.0018	46.900

$w_1 = 0.5$		$w_1 = 0.3$		$w_1 = 0.0$	
$10^4 d / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{mol}^{-1})$	$10^4 d / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{mol}^{-1})$	$10^4 d / (\text{mol dm}^{-3})$	$\Lambda / (\text{S cm}^2 \text{mol}^{-1})$
1.2436	72.415	1.8234	94.700	4.0563	126.82
2.6832	71.660	3.0044	93.850	9.8079	123.67
5.2053	70.780	4.8311	92.920	12.2276	122.56
7.4188	70.180	7.5106	91.870	15.5439	121.22
9.6124	69.698	9.7727	91.210	18.0735	120.32
12.0302	69.198	12.5682	90.395	19.8412	119.73
14.2523	68.778	15.9503	89.534	22.7114	118.93
18.4447	68.082	19.7399	88.800	25.3607	118.25
20.9450	67.714	26.5933	87.560	27.3528	117.75
23.9849	67.310	30.8073	86.900	30.6545	117.12
27.3726	66.910				
33.0327	66.400				

**Figure 1.** Dependence of the deviation σ_Λ (eq 4) on the distance of closest approach r for Bu₄NBPh₄ in acetonitrile at 30 °C.

coefficient, N Avogadro's number, e the electron charge, k Boltzmann's constant, T the absolute temperature, and r the distance of closest approach of ions.

Three parameters can be determined from the conductance data. The value of conductance at infinite dilution Λ_0 , the value of the association constant K_A , and the distance of closest approach of ions r that gives the best fit to the conductance equation. The minimum in the σ_Λ against r plot is the criterion of "fit". σ_Λ is given by

$$\sigma_\Lambda = \left[\frac{\sum_j (\Lambda_{j(\text{cal})} - \Lambda_{j(\text{obs})})^2}{(n - 2)} \right]^{1/2} \quad (4)$$

where n is the number of observations. An example of this plot for acetonitrile at 30 °C is given in Figure 1. For propylene carbonate at (30 and 35) °C and propylene carbonate (90 mass %) + acetonitrile (10 mass %) at (25 and 30) °C cases, there were flat instead of sharp minima in the plots. For these cases, the r values were calculated from the equation

$$r = a + [M(Nd)]^{1/3} \quad (5)$$

where a is the sum of the crystallographic radii of the ions, d the density of the solvent, and M its molecular weight (Fuoss, 1978). For mixed solvents M is replaced by the mole fraction average molecular weight:

$$M = x_1 M_1 + x_2 M_2 \quad (6)$$

The values of molar conductances at infinite dilution Λ_0 , association constants K_A , distances of closest approach r , and deviations σ_Λ calculated by means of the Lee–Wheaton equation are listed in Table 5.

No literature data were found for the mixtures. As can be seen from Table 6, for the pure solvents, the literature results are in good agreement with our values.

At all temperatures, on decreasing the concentration of propylene carbonate, an increase of the Λ_0 values was observed. This is attributed to the decrease of the viscosity of the mixtures. As expected, on increasing the temperature, an increase in Λ_0 values was observed.

For the pure propylene carbonate and the mixtures, no literature data were found on association constants. The reported association constants at 25 °C for pure acetonitrile were 28.9 dm³ mol⁻¹ (Barthel et al., 1990), 8.2 dm³ mol⁻¹ (Brown and Fuoss, 1960), 6 dm³ mol⁻¹ (Berns and Fuoss, 1960), and 5 dm³ mol⁻¹ (Gill and Nording, 1983). We found 13.6 dm³ mol⁻¹. The different values are due to the different equations and methods of analysis which were used by the authors. On reducing the propylene carbonate concentration, the association constant increases. It seems that, as the addition of acetonitrile reduces the dielectric constant of the solvent medium, stronger attractive Coulombic forces between the ions are developed and stable ion pairs are formed (Prabhu et al., 1993; Kraus, 1954; Papadopoulos and Ritzoulis, 1987). On increasing the temperature, a light decrease in the association constant is observed. This behavior has also been reported by others (Barthel et al., 1983; Barthel et al., 1986a; Barthel et al., 1986b; Acevedo et al., 1989).

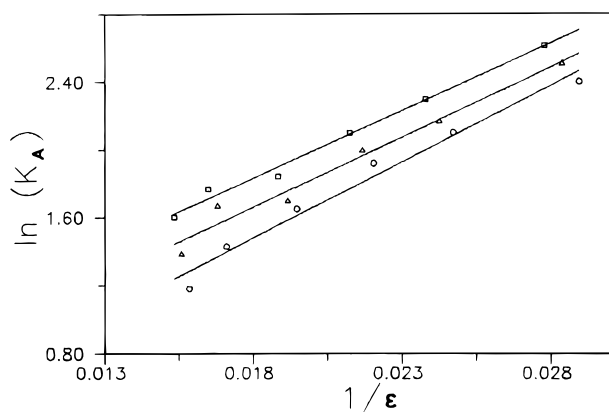
Table 5. Conductance Parameters for Bu₄NBPh₄ in Propylene Carbonate (1) + Acetonitrile (2) at (25, 30, and 35) °C

100w ₁	Λ_{∞} / (S cm ² mol ⁻¹)	K_{Λ} / (dm ³ mol ⁻¹)	$r/\text{\AA}$	100 σ_{Λ} / (s cm ² mol ⁻¹)
25 °C				
100	17.38	4.96	15.6	0.87
90	26.22	5.87	15.4	1.31
70	44.31	6.32	15.3	1.77
50	65.32	8.16	14.6	1.93
30	87.21	9.95	15.3	2.17
0	119.88	13.61	14.6	5.22
30 °C				
100	19.28	4.00	15.6	0.78
90	28.56	5.32	15.4	1.33
70	48.10	5.15	13.7	2.46
50	69.81	7.39	14.2	1.79
30	92.47	8.75	15.0	1.75
0	126.50	12.29	14.8	5.96
35 °C				
100	21.18	3.21	15.6	2.06
90	31.37	4.17	14.5	0.01
70	52.27	5.60	15.3	2.30
50	74.15	7.76	15.8	3.92
30	97.64	8.39	15.4	3.03
0	133.66	11.92	15.1	9.41

Table 6. Literature Values of Λ_{∞} (S cm² mol⁻¹) in Propylene Carbonate (1) and Acetonitrile (2)

propylene carbonate (1)	acetonitrile (2)
17.14 ^a	119.80 ^c
17.38 ^b	119.85 ^d
	119.40 ^e
	120.00 ^f
	119.85 ^b

^a Fuoss and Hirsch (1960). ^b This work. ^c Barthel et al. (1990). ^d Brown and Fuoss (1960). ^e Berns and Fuoss (1960). ^f Gill and Nording (1983).

**Figure 2.** Dependence of the logarithm of the association constant on the reciprocal of the dielectric constant for propylene carbonate (1) + acetonitrile (2): □, 25 °C; △, 30 °C; ○, 35 °C.

The mean value of the minimum for the three temperatures in σ_{Λ} against r plots was observed at $(15 \pm 0.8) \text{\AA}$. This value is similar to that produced through eq 5. Consequently, we may admit that the ion pairs are separated by one solvent molecule.

Plots of the logarithm of the association constant against the reciprocal of the dielectric constant are given in Figure 2. If we use the following Fuoss equation containing Gilkerson's factor $\exp(-E_S/RT)$ (Fuoss, 1958; Gilkerson, 1956)

$$K_{\Lambda} = (4\pi N\alpha^3/3000) \exp(-E_S/RT) \exp[e^2/(\alpha kT\epsilon)] \quad (7)$$

we get from the slopes of the plots the value $(6.4 \pm 0.7) \text{\AA}$

Table 7. Values of the Walden Product (W) for Bu₄NBPh₄ in Propylene Carbonate (1) + Acetonitrile (2) at (25, 30, and 35) °C

100w ₁	W (S cm ² mol ⁻¹ mPa s)		
	$t = 25 \text{ °C}$	$t = 30 \text{ °C}$	$t = 35 \text{ °C}$
100	43.65	43.77	44.05
90	43.93	43.84	44.53
70	42.87	43.60	44.24
50	41.90	42.22	42.61
30	41.80	41.96	42.15
0	41.23	41.49	41.94

for α , where α is the center-to-center distance of the ions in a pair. This value is lower than the value $(15 \pm 0.8) \text{\AA}$ obtained for r from the σ_{Λ} against r plots. This is attributed to the different model on which eq 7 was based. Using for α the values obtained from the slopes, we can evaluate from the intercept of the plots the values $-1295.1 \text{ J mol}^{-1}$ for 25 °C, $-1168.6 \text{ J mol}^{-1}$ for 30 °C, and $-1445.7 \text{ J mol}^{-1}$ for 35 °C for the solvent-solute interaction factors. The negative sign indicates that the ion pair-solvent interaction term is greater than the ion-solvent interaction terms (Gilkerson, 1956).

The Walden product is given in Table 7. It seems to be nearly constant with temperature, but it changes with concentration. Changes in the Walden product with concentration are common (Berns and Fuoss, 1960; Kraus, 1954; Ramana-Murti and Yadav, 1972; Evans et al., 1971; Lind and Fuoss, 1961a,b, 1962; Spiney and Shedlovsky, 1967; Goffredi and Shedlovsky, 1967), and they can be attributed to changes in ion solvation and ion-solvent interactions. Obviously the sphere in a continuum fails as a hydrodynamic model for real ions in real solvents (James and Fuoss, 1975).

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