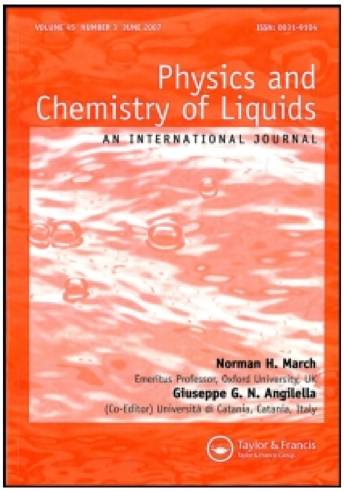

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Electrical conductances of tetrabutylammonium bromide and tetrapentylammonium bromide in 2-ethoxyethanol + water mixtures at 308.15, 313.15, 318.15 and 323.15 K

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The electrical conductances of the solutions of tetrabutylammonium bromide (Bu_4NBr), and tetrapentylammonium bromide (Pen_4NBr) in 2-ethoxyethanol (**1**) + water (**2**) mixed solvent media containing 0.25, 50 and 0.75 mass fractions of 2-ethoxyethanol (w_1) have been reported at 308.15, 313.15, 318.15 and 323.15 K. The conductance data have been analyzed by the 1978 Fuoss conductance–concentration equation in terms of the limiting molar conductance (Λ^0), the association constant (K_A) and the association diameter (R). These two electrolytes are found to exist essentially as free ions in the solvent mixtures with $w_1 = 0.25$ and 0.50 over the entire temperature range; however, slight ionic association was observed in the mixed solvent medium richest in 2-ethoxyethanol. The electrostatic ion–solvent interaction is found to be very weak for the tetraalkylammonium ions in the aqueous 2-ethoxyethanol mixtures investigated.

Keywords: Electrical conductance; Tetrabutylammonium bromide; Tetrapentylammonium bromide; 2-Ethoxyethanol + water mixed solvent media; Limiting molar conductance; Association constant

1. Introduction

Studies on the transport properties of electrolytes in different solvent media are of great importance to obtain information regarding the solvation and association behavior of ions in solutions. Earlier, we have investigated [1–8] these properties for a wide variety of electrolytes in different solvents in great detail. The solvent properties like the viscosity and the relative permittivity have also been taken into account in determining the extent of ionic association and the solute–solvent interactions which enabled many to interpret the unique structure of the solvent. Recently, we have reported [9] the results of conductance measurements of two symmetrical tetraalkylammonium bromides, namely, tetraethylammonium bromide (Et_4NBr), and tetrapropylammonium bromide (Pr_4NBr) in 2-ethoxyethanol (**1**) + water (**2**) mixtures at 308.15, 313.15, 318.15 and 323.15 K. We have now extended this work to study the conductometric behavior of the next two higher homologues, tetrabutylammonium bromide (Bu_4NBr) and

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tetrapentylammonium bromide (Pen_4NBr) in the same solvent systems at 308.15, 313.15, 318.15 and 323.15 K to obtain a comprehensive view of the ion–ion and ion–solvent interactions in these media.

2. Experimental

2.1. *Materials*

2-Ethoxyethanol (G.R.E. Merck) was dried with potassium carbonate and distilled twice in an all glass distillation set immediately before use and the middle fraction was collected. The purified solvent had a density (ρ_0) of 0.92497 $g\,cm^{-3}$ and a viscosity (η_0) of 1.8277 mPa s at 298.15 K; these values are found to be in good agreement with the literature values [10,11]. Triply distilled water with a specific conductance of $<10^{-6}\,S\,cm^{-1}$ at 308.15 K was used for the preparation of the mixed solvents by mass. The physical properties of 2-ethoxyethanol (**1**) + water (**2**) mixed solvents used in this study at 308.15, 313.15, 318.15 and 323.15 K are reported in table 1. The relative permittivities of 2-ethoxyethanol (**1**) + water (**2**) mixtures at the experimental temperatures were obtained with the equations as described in the literature [12] using the literature density and relative permittivity data of the pure solvents [7,13] and the densities of the mixed solvents given in table 1.

Both Bu_4NBr and Pen_4NBr were of Fluka purum grade and were purified by recrystallization from acetone and the recrystallized salts were dried *in vacuo* at 333.15 K for 48 h.

2.2. *Measurements*

Conductance measurements were carried out on a Pye-Unicam PW 9509 conductivity meter at a frequency of 2000 Hz using a dip-type cell of cell constant 1.15 cm^{-1} and

Table 1. Properties of 2-ethoxyethanol (**1**) + water (**2**) mixtures with $w_1 = 0.25$, 0.50, and 0.75 at 308.15, 313.15, 318.15 and 323.15 K.

T (K)	ρ_0 ($g\,cm^{-3}$)	η_0 (mPa s)	ε
$w_1 = 0.25$			
308.15	1.00354	1.8430	60.13
313.15	1.00021	1.5293	58.70
318.15	0.99781	1.2738	57.37
323.15	0.99582	1.0923	56.11
$w_1 = 0.50$			
308.15	0.99361	1.9234	44.30
313.15	0.98514	1.7195	43.03
318.15	0.98004	1.4552	41.95
323.15	0.97610	1.2762	40.96
$w_1 = 0.75$			
308.15	0.95451	1.7002	27.93
313.15	0.95147	1.5293	27.29
318.15	0.94873	1.3498	26.68
323.15	0.94625	1.1901	26.10

having a precision of 0.10%. The cell was calibrated by the method of Lind and co-workers [14] using aqueous potassium chloride solutions. The measurements were made in a water bath maintained within ±0.005 K of the desired temperature. The details of the experimental procedure have been described earlier [15,16]. Solutions were prepared by mass for the conductance runs, the molalities being converted to molarities by the use of densities measured with an Ostwald–Sprengel type pycnometer of about 25 cm^3 capacity. Several independent solutions were prepared and runs were performed to ensure the reproducibility of the results. Due correction was made for the specific conductance of the solvent by subtracting the specific conductance of the relevant solvent medium from those of the salt solutions.

The kinematic viscosities were measured using a suspended level Ubbelohde-type viscometer.

In order to avoid moisture pickup, all solutions were prepared in a dehumidified room with utmost care. In all cases, the experiments were performed at least in five replicates for each solution and at each temperature and the results were averaged (repeatabilities were always within $\pm 0.10\,\text{S cm}^2\,\text{mol}^{-1}$). The experimental uncertainties in density, viscosity and conductivity were always within 0.01, 0.08 and 0.12%, respectively.

3. Results and discussion

The measured molar conductances (Λ) of electrolyte solutions as functions of molar concentration (c) in 2-ethoxyethanol (**1**) + water (**2**) mixtures with $w_1 = 0.25$, 0.50 and 0.75 at 308.15, 313.15, 318.15 and 323.15 K are given in table 2.

The conductance data have been analyzed by the 1978 Fuoss conductance–concentration equation [17,18]. For a given set of conductivity values (c_j, Λ_j; $j = 1, \ldots, n$), three adjustable parameters – the limiting molar conductivity (Λ^0), association constant (K_A) and the association diameter (R), are derived from the following set of equations:

$$\Lambda = p[\Lambda^0(1 + RX + EL)] \tag{1}$$

$$p = 1 - \alpha(1 - \gamma) \tag{2}$$

$$\gamma = 1 - K_A c \gamma^2 f^2 \tag{3}$$

$$-\ln f = \frac{\beta k}{2(1 + kR)} \tag{4}$$

$$\beta = \frac{e^2}{\varepsilon k_B T} \tag{5}$$

$$K_A = K_R(1 + K_S) \tag{6}$$

where RX is the relaxation field effect, EL is the electrophoretic countercurrent, γ is the fraction of unpaired ions, and α is the fraction of contact-pairs, K_A is the overall pairing

Table 2. Equivalent conductances and corresponding molarities of electrolytes in 2-ethoxyethanol (**1**) + water (**2**) mixtures with $w_1 = 0.25$, 0.50, and 0.75 at 308.15, 313.15, 318.15, and 323.15 K.

$w_1 = 0.25$		$w_1 = 0.50$		$w_1 = 0.75$	
c (mol dm^{-3})	Λ (S cm^2 mol^{-1})	c (mol dm^{-3})	Λ (S cm^2 mol^{-1})	c (mol dm^{-3})	Λ (S cm^2 mol^{-1})
$T = 308.15$ K					
Bu_4NBr					
0.005012	61.25	0.002013	54.14	0.000503	43.37
0.007518	59.72	0.003020	53.31	0.000754	42.97
0.010024	58.26	0.004026	52.90	0.001005	42.57
0.012530	57.06	0.005033	52.45	0.001257	42.26
0.015036	55.93	0.006040	51.99	0.001508	41.98
0.017542	55.01	0.007046	51.66	0.001759	41.72
0.020048	54.12	0.008053	51.16	0.002010	41.43
0.025060	52.23	0.010066	50.57	0.002513	40.98
Pen_4NBr					
0.005008	58.91	0.002103	50.89	0.000499	40.14
0.007312	57.35	0.003154	50.10	0.000748	39.57
0.010016	55.91	0.004205	49.46	0.000997	39.10
0.012520	54.87	0.005256	48.91	0.001247	38.66
0.015024	53.78	0.006308	48.35	0.001496	38.30
0.017528	52.77	0.007359	47.97	0.001745	37.93
0.020032	51.92	0.008410	47.44	0.001995	37.60
0.025040	50.20	0.010513	46.70	0.002493	37.06
$T = 313.15$ K					
Bu_4NBr					
0.004990	66.34	0.002004	58.89	0.000500	48.57
0.007484	64.67	0.003006	58.22	0.000750	48.11
0.009980	63.23	0.004008	57.64	0.001001	47.67
0.012475	61.96	0.005010	57.09	0.001251	47.33
0.014970	60.92	0.006012	56.55	0.001501	47.04
0.017466	59.83	0.007014	56.17	0.001751	46.77
0.019961	58.96	0.008016	55.76	0.002001	46.47
0.024953	57.19	0.010020	55.09	0.002501	46.01
Pen_4NBr					
0.004986	63.99	0.002093	55.91	0.000496	45.93
0.007478	62.31	0.003139	54.79	0.000745	45.26
0.009971	60.97	0.004186	54.23	0.000993	44.93
0.012464	59.77	0.005232	53.71	0.001241	44.57
0.014958	58.77	0.006279	53.20	0.001489	44.26
0.017451	57.76	0.007325	52.69	0.001737	43.98
0.019944	56.81	0.008372	52.32	0.001985	43.72
0.024932	55.15	0.010465	51.50	0.002482	43.19
$T = 318.15$ K					
Bu_4NBr					
0.004970	71.43	0.001993	63.71	0.000498	54.47
0.007455	69.75	0.002990	63.20	0.000746	54.00
0.009941	68.20	0.003987	62.45	0.000995	53.67
0.012426	67.04	0.004984	62.00	0.001244	53.31
0.014912	65.92	0.005981	61.53	0.001493	53.00
0.017398	64.84	0.006978	61.05	0.001741	52.72
0.019884	63.92	0.007975	60.57	0.001990	52.41
0.024857	62.12	0.009968	59.89	0.002488	52.02

(Continued)

Table 2. Continued.

$w_1 = 0.25$		$w_1 = 0.50$		$w_1 = 0.75$	
c (mol dm^{-3})	Λ (S cm^2 mol^{-1})	c (mol dm^{-3})	Λ (S cm^2 mol^{-1})	c (mol dm^{-3})	Λ (S cm^2 mol^{-1})
Pen_4NBr					
0.004966	69.07	0.002082	60.52	0.000494	51.26
0.007449	67.52	0.003123	59.88	0.000740	50.78
0.009932	66.15	0.004164	59.08	0.000987	50.34
0.012416	65.00	0.005205	58.60	0.001234	49.92
0.014900	63.89	0.006246	57.95	0.001481	49.57
0.017384	62.87	0.007287	57.50	0.001728	49.20
0.019868	61.96	0.008329	57.03	0.001974	48.87
0.024836	60.31	0.010411	56.19	0.002468	48.30
$T = 323.15$ K					
Bu_4NBr					
0.004951	76.14	0.001982	69.13	0.000495	60.12
0.007427	74.18	0.002973	68.29	0.000742	59.50
0.009904	72.60	0.003964	67.61	0.000990	59.11
0.012380	71.32	0.004955	67.20	0.001237	58.90
0.014857	70.07	0.005946	66.77	0.001485	58.47
0.017334	69.05	0.006937	66.16	0.001732	58.17
0.019812	67.89	0.007928	65.84	0.001980	57.90
0.024767	66.06	0.009911	64.98	0.002475	57.43
Pen_4NBr					
0.004947	74.58	0.002070	66.19	0.000491	57.23
0.007422	72.90	0.003105	65.38	0.000737	56.62
0.009896	71.44	0.004140	64.49	0.000982	56.21
0.012370	70.33	0.005175	63.96	0.001228	55.72
0.014845	69.25	0.006210	63.44	0.001473	55.39
0.017320	68.30	0.007245	63.07	0.001719	54.99
0.019795	67.34	0.008281	62.68	0.001964	54.65
0.024746	65.67	0.010351	61.92	0.002455	54.09

constant evaluated from the association constants of contact-pairs, K_S, of solvent-separated pairs, K_R, ε is the relative permittivity of the solvent, e is the electronic charge, k_B is the Boltzmann constant, k^{-1} is the radius of the ion atmosphere, c is the molarity of the solution, f is the activity coefficient, T is the temperature in absolute scale and β is twice the Bjerrum distance. The computations were performed on a computer using the program as suggested by Fuoss. The initial Λ^0 values for the iteration procedure were obtained from Shedlovsky extrapolation [19] of the data. Input for the program is the set (c_j, Λ_j; $j = 1, \ldots, n$), n, ε, η, T, initial value of Λ^0, and an instruction to cover a preselected range of R values.

In practice, calculations are made by finding the values of Λ^0 and α which minimize the standard deviation, σ,

$$o = \left[\sum \frac{[\Lambda_j(\text{Calcd}) - \Lambda_j(\text{Obsd})]^2}{n-2}\right]^{1/2} \tag{7}$$

for a sequence of R values and then plotting against R; the best-fit R corresponds to the minimum in σ *versus* R curve. However, for these two electrolytes investigated, since a preliminary scan using a unit increment of R values from 4 to 20 produced no significant minima in the σ *versus* R curves, the R value was assumed to be $R = a + d$, where a is the sum of the ionic crystallographic radii and d is given by [18]

$$d = 1.183\left(\frac{M}{\rho_0}\right)^{1/3} \tag{8}$$

where M is the molecular weight of the solvent and ρ_0 its density.

The values of Λ^0, K_A, and R obtained by this procedure are reported in table 3.

Table 3 and figure 1 show that the equivalent conductivity values (Λ^0) of the two tetraalkylammonium salts increase as the temperature increases in all 2-ethoxyethanol (**1**) + water (**2**) mixtures. The Λ^0 values have been fitted to the following polynomial in T:

$$\Lambda^0\ (\mathrm{S\,cm^2\,mol^{-1}}) = a_0 + a_1[308.15 - T(K)] + a_2[308.15 - T(K)]^2 \tag{9}$$

and the coefficients of these fits along with the standard deviations (σ) are given in table 4.

The limiting molar conductivity values (Λ^0) of Bu_4NBr are always found to be higher than those of Pen_4NBr. Earlier, it has also been observed [9] that the Λ^0 values of tetraethylammonium bromide were always higher than those of tetrapropylammonium bromide. This means that the mobilities of the tetraalkylammonium ions decrease in the order: $Et_4N^+ > Pr_4N^+ > Bu_4N^+ > Pen_4N^+$ (anion being common) in all of the mixed solvent media over the entire temperature range investigated. Now, a comparison of this trend in mobility with the crystallographic sizes of these ions, which is in the order [20] $Et_4N^+ < Pr_4N^+ < Bu_4N^+ < Pen_4N^+$, shows that the larger the size of the bare ion, the smaller is its ionic mobility. This indicates that the relative actual sizes of these ions as they exist in solutions follow the order: $Et_4N^+ < Pr_4N^+ < Bu_4N^+ < Pen_4N^+$. This observation, thus, clearly demonstrates that the electrostatic ion–solvent interaction is very weak for these ions in aqueous 2-ethoxyethanol solutions which is to be expected because of their large crystallographic radii and hence low surface charge density. Had these interactions been very strong in this medium, the limiting molar conductivity

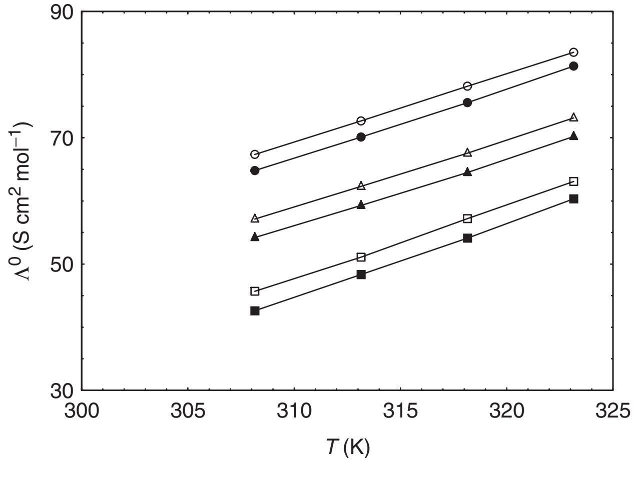

Figure 1. Temperature dependence of the limiting molar conductances of tetrabutylammonium bromide (○, $w_1 = 0.25$; △, $w_1 = 0.50$; □, $w_1 = 0.75$) and tetrapentylammonium bromide (●, $w_1 = 0.25$; ▲, $w_1 = 0.50$; ■, $w_1 = 0.75$) in 2-ethoxyethanol (**1**) + water (**2**) mixtures.

Table 3. Derived conductivity parameters of electrolytes in 2-ethoxyethanol (**1**) + water (**2**) mixtures with $w_1 = 0.25$, 0.50, and 0.75 at 308.15, 313.15, 318.15, and 323.15 K.

T (K)	Λ^0 (S cm^2 mol^{-1})	K_A (dm^3 mol^{-1})	$\Lambda^0\eta_0$ (S cm^2)	R (Å)	100σ (Λ^0 mol^{-1} Pa s)
$w_1 = 0.25$					
Bu$_4$NBr					
308.15	67.35 ± 0.11	13.31 ± 0.23	0.1241	10.29	0.12
313.15	72.67 ± 0.06	11.74 ± 0.11	0.1111	10.31	0.06
318.15	78.15 ± 0.06	10.46 ± 0.09	0.0996	10.31	0.06
323.15	83.52 ± 0.08	10.54 ± 0.13	0.0912	10.30	0.07
Pen$_4$NBr					
308.15	64.80 ± 0.08	13.41 ± 0.18	0.1194	10.58	0.09
313.15	70.11 ± 0.05	11.72 ± 0.10	0.1072	10.60	0.16
318.15	75.55 ± 0.04	10.02 ± 0.07	0.0963	10.60	0.04
323.15	81.33 ± 0.05	8.73 ± 0.08	0.0888	10.59	0.05
$w_1 = 0.50$					
Bu$_4$NBr					
308.15	57.13 ± 0.07	7.96 ± 0.34	0.1099	10.63	0.12
313.15	62.31 ± 0.04	8.07 ± 0.17	0.1071	10.65	0.06
318.15	67.54 ± 0.10	6.79 ± 0.38	0.0983	10.65	0.13
323.15	73.14 ± 0.09	5.96 ± 0.11	0.0933	10.66	0.11
Pen$_4$NBr					
308.15	54.20 ± 0.04	11.83 ± 0.18	0.1042	10.92	0.06
313.15	59.29 ± 0.11	10.35 ± 0.49	0.1019	10.94	0.17
318.15	64.49 ± 0.08	9.04 ± 0.32	0.0938	10.94	0.11
323.15	70.21 ± 0.09	6.92 ± 0.33	0.0896	10.95	0.12
$w_1 = 0.75$					
Bu$_4$NBr					
308.15	45.68 ± 0.04	17.85 ± 0.93	0.0777	11.23	0.08
313.15	51.09 ± 0.03	15.60 ± 0.58	0.0781	11.23	0.05
318.15	57.19±0.09	8.82 ± 0.93	0.0772	11.23	0.09
323.15	63.07 ± 0.07	7.01 ± 1.16	0.0750	11.23	0.19
Pen$_4$NBr					
308.15	42.61 ± 0.03	38.73 ± 0.69	0.0894	11.52	0.05
313.15	48.35 ± 0.05	20.14 ± 1.10	0.0802	11.52	0.10
318.15	54.11 ± 0.06	19.94 ± 1021	0.0791	11.52	0.11
323.15	60.34 ± 0.05	16.96 ± 0.96	0.0780	11.53	0.08

Table 4. Coefficients of equation (9) and the standard deviations (σ).

w_1	Electrolyte	a_0 (S cm^2 mol^{-1})	$-a_1$ (S cm^2 mol^{-1} K^{-1})	a_2 (S cm^2 mol^{-1} K^{-2})	σ (S cm^2 mol^{-1})
0.25	Bu$_4$NBr	72.93 ± 0.13	1.0528 ± 0.0406	0.0068 ± 0.0026	0.13
	Pen$_4$NBr	69.93 ± 0.01	1.0423 ± 0.0021	0.0029 ± 0.0001	0.01
0.50	Bu$_4$NBr	62.07 ± 0.25	1.0065 ± 0.0805	0.0051 ± 0.0041	0.26
	Pen$_4$NBr	59.19 ± 0.08	0.9948 ± 0.0266	0.0052 ± 0.0017	0.08
0.75	Bu$_4$NBr	51.34 ± 0.17	0.8822 ± 0.0546	0.0154 ± 0.0035	0.17
	Pen$_4$NBr	49.50 ± 0.55	0.4548 ± 0.1764	0.0416 ± 0.0113	0.56

values should have been in the reverse order: $Et_4NBr < Pr_4NBr < Bu_4NBr < Pen_4NBr$, because a smaller ion with greater surface charge density is expected to associate more solvent molecules thus resulting in a bigger solvodynamic entity – which is obviously not the case here. A similar trend in the Walden products, $\Lambda^0\eta_0$ (table 3) also points to the same conclusion.

The Walden product values ($\Lambda^0\eta_0$) for the electrolytes studied here, in general, show pronounced variations with increasing temperature (table 3). Therefore, the Stokes law cannot be applied in 2-ethoxyethanol (**1**) + water (**2**) because the $\Lambda^0\eta_0$ values, according to this law, would be expected to be independent of temperature [21]. Since the ions are often far from being spherical and since they are of the same order of magnitude as the solvent molecules, it is questionable whether the retarding effect of the latter can be accurately described by the macroscopic viscosity as has been done in the derivation of the Stokes law. Hence, the Stokes law cannot be considered quantitatively reliable. Such failure of this law has also been observed earlier in other solvent media [22, 23].

The association constants (K_A) listed in table 3 for these two electrolytes are practically negligible (i.e. the K_A values are either very close to or less than 10) in the mixed solvent media with $w_1 = 0.25$ and 0.50 over the entire temperature range. So, the numerical values of K_A should not be taken seriously [24]. One can only conclude that these two electrolytes exist essentially as free ions in both the solvent mixtures in the temperature range 308.15–323.15 K. This is expected because the relative permittivities of the solvent mixtures are fairly high ($40.96 \le \varepsilon \le 60.13$). In the solvent mixture with $w_1 = 0.75$ with comparatively lower relative permittivity, these salts are found to be slightly associated.

Thus, it can be concluded that two tetraalkylammonium bromides (Bu_4NBr and Pen_4NBr) investigated here exist essentially in the form of free ions aqueous 2-ethoxyethanol solutions with $w_1 = 0.25$ and 0.50 over the entire temperature range 308.15–323.15 K. Slight ionic association was observed in the mixed solvent medium with $w_1 = 0.75$. The electrostatic ion–solvent interaction is found to be very weak for the tetraalkylammonium ions in the aqueous 2-ethoxyethanol mixtures investigated.

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