Table IV. Solubilities and Densities of Magnesium Sulfate Aqueous Solution at Temperatures (θ) of 10, 15, 20, 25, 30, 35, and 40 °C[a]

	solubility					density	
θ, °C	this study	ref 16	ref 17	ref 18	ref 19	this study	ref 16
10		93.56	80.07	91.58	82.00		1260
15	98.32	104.52*	92.17	101.00*	93.02*	1274.12	1275.8*
20		115.73	105.53	111.47	106.65		1290.0
25	119.14	130.08*	120.22	124.66*	120.66	1302.14	1307.8*
30		145.9	136.29	139.32	134.43		1330.0
35	151.83	160.76*	153.76	154.20*	150.04	1335.45	1340.0*
40		178.02	172.60	170.97	170.84		1350.0

[a] Units: solubility, kg of hydrate/100 kg of free water; density, kg/m^3. Values marked with an asterisk are interpolated data.

w_{eq}, solubility, kg of hydrate/100 kg of free water

x, ethanol concentration, kg of ethanol/kg of free water

ρ, density of solution, kg/m^3

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Transference Numbers of Lithium Chloride and Tetraalkylammonium Halides in 1-Butanol at Various Temperatures

Jagtar S. Banait* and **Gurpreet S. Bhatti**

Department of Chemistry, Punjabi University, Patiala 147 002, India

Transference numbers of lithium chloride and some tetraalkylammonium halides have been measured in 1-butanol at 298.15, 308.15, and 318.15 K up to concentrations of 11.84 × 10^{-2} mol dm^{-3}. The transference numbers of the ions show much less variation with concentration and are in accordance with the Debye–Hückel limiting law. Transference numbers of the ions at infinite dilution have been evaluated from the linear plots of the transference number versus the square root of the concentration. Variation of limiting transference numbers of the ions with temperature agrees with the theoretical predictions. Relative solvation numbers of the ions evaluated at all the temperatures by the method suggested by Carman show some anomalies. Solvation numbers of the ions have been evaluated at 298.15 K by combining the present transference number data with reported conductance data. The solvation numbers of the ions reveal that the anions in 1-butanol are heavily solvated as compared to the cations of comparable sizes. This is in line with the protic nature of this solvent.

Conductance data of some electrolytes in 1-butanol have been reported in the literature (*1–4*). However, limiting equivalent ionic conductances have been evaluated on the basis of the Walden product of large ions and the Kohlrausch law of independent ion migration. Recently, we reported (*5*) the transference numbers of tetrabutylammonium bromide in this solvent only at one temperature (298.15 K). In this paper, transference number measurements by Hittorf's method of some electrolytes at 298.15, 308.15, and 318.15 K are reported. The data at various temperatures have been used to evaluate relative solvation numbers of the ions by using Carman's equation (*6*), and the data at 298.15 K have been used to evaluate solvation numbers of the ions in this solvent.

Experimental Section

1-Butanol was purified essentially by the method reported earlier (*5*, *7*). Freshly distilled solvent was used in each experiment, and the purity (greater than 99.9 mol %) of the solvent was checked by its conductivity and density measurements.

All the electrolytes (BDH AnalaR or Reidal Pure, with stated purity levels of 99 mol % or better) used in these studies were recrystallized from conductivity water and dried under reduced pressure at an elevated temperature (373.15 K) before use.

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* To whom correspondence should be addressed.

Table I. Cation Transference Numbers (t_+) of Various Electrolytes in 1-Butanol as a Function of Concentration and Temperature

	298.15 ± 0.01 K		308.15 ± 0.01 K		318.15 ± 0.01 K	
electrolyte	C, mol dm^{-3}	t_+ (±0.0001)	C, mol dm^{-3}	t_+ (±0.0001)	C, mol dm^{-3}	t_+ (±0.0001)
LiCl	0.0514	0.5428	0.0508	0.5369	0.0542	0.5310
	0.0642	0.5441	0.0681	0.5382	0.0665	0.5320
	0.0805	0.5454	0.0791	0.5391	0.0721	0.5328
	0.0958	0.5466	0.0924	0.5443	0.0884	0.5338
	0.1070	0.5482	0.1112	0.5518	0.1080	0.5352
Me_4NCl	0.0524	0.5741	0.0516	0.5689	0.0500	0.5649
	0.0704	0.5744	0.0634	0.5690	0.0642	0.5650
	0.0881	0.5743	0.0768	0.5690	0.0738	0.5651
	0.0966	0.5739	0.0848	0.5691	0.0889	0.5653
	0.1122	0.5742	0.0998	0.5694	0.1028	0.5655
Et_4NCl	0.0646	0.5958	0.0557	0.5915	0.0583	0.5859
	0.0825	0.5964	0.0695	0.5919	0.0715	0.5864
	0.0911	0.5971	0.0845	0.5923	0.0895	0.5871
	0.1046	0.5976	0.0974	0.5931	0.1032	0.5879
	0.1184	0.5981	0.1121	0.5937	0.1156	0.5883
Bu_4NCl	0.0589	0.5292	0.0554	0.5251	0.0568	0.5201
	0.0731	0.5301	0.0706	0.5263	0.0727	0.5214
	0.0904	0.5313	0.0837	0.5270	0.0850	0.5221
	0.1049	0.5323	0.0992	0.5283	0.0976	0.5230
	0.1161	0.5330	0.1115	0.5294	0.1081	0.5235
Bu_4NBr[a]	0.0579	0.5015	0.0562	0.4965	0.0532	0.4926
	0.0866	0.5016	0.0694	0.4965	0.0691	0.4923
	0.1188	0.5018	0.0845	0.4969	0.0825	0.4921
	0.1286	0.5022	0.1001	0.4964	0.0994	0.4918
			0.1138	0.4963	0.1135	0.4913
Bu_4NI	0.0681	0.4365	0.0645	0.4381	0.0454	0.4304
	0.0771	0.4347	0.0771	0.4356	0.0632	0.4256
	0.0843	0.4340	0.0930	0.4342	0.0819	0.4222
	0.0988	0.4301	0.1080	0.4312	0.0982	0.4193
	0.1140	0.4270	0.1180	0.4299	0.1080	0.4177

[a] Experimental values at 298.15 K are from ref 5.

A modified Hittorf's transference number cell with three compartments separated with well-greased stopcocks was used. It was filled with the solution and immersed into an oil thermostat controlled to ±0.01 K. After equilibration, a current of 5–10 mA was employed, and the electrolysis was carried out for 12 to 24 h. Two sets of experiments were performed at each concentration. The procedure and the other details were essentially the same as reported by Paul et al. (*8*). The equation for the calculation of transference numbers is the one suggested by Wear and co-workers (*9*).

The concentrations of the solutions before and after electrolysis were obtained from measurement of the halide ion concentration using Volahdr's method (*10*). Four estimations were performed for each solution, and the results agreed within ±0.0002 g/100 g. All materials as far as possible were handled and transferred in a drybox.

Results and Discussion

Transference numbers of lithium, tetramethylammonium, tetraethylammonium, and tetrabutylammonium chlorides and tetrabutylammonium bromide and tetrabutylammonium iodide have been measured up to concentrations of 11.84×10^{-2} mol dm^{-3} in 1-butanol at 298.15, 308.15, and 318.15 K. The measurements were made at moderately high concentrations because of the very high resistance offered by the cell, as well as the difficulties in accurate estimations of the change in concentrations. All the relevant data are summarized in Table I.

The variation of transference numbers with concentration conforms with the Kay and Dye equation (*11*):

$$\left(\frac{dt_\pm}{dC^{1/2}}\right)_{C\to 0} = \frac{(t_\pm{}^\circ - 0.5)B}{\Lambda^\circ}$$

where $t_\pm$ is the ionic transference number at concentration C, $t_\pm{}^\circ$ is the limiting ionic transference number, and B is the electrophoretic parameter.

Limiting transference numbers of the cations have been evaluated by the extrapolation of the linear plots of the cation transference number versus the square root of the concentration.

The limiting transference numbers of the cations at all the temperatures are given in Table II. It has been observed that the limiting cation transference numbers in 1-butanol decrease with an increase in the temperature for all the ions. Various workers (*12–14*) have predicted the theoretical variation of the limiting transference numbers of the ions with temperature. In general (*13*), it is believed that the limiting transference numbers of the cation and the anion of an electrolyte tend to become more nearly equal with an increase in temperature; i.e., the difference between the limiting transference numbers of the cation and the anion of an electrolyte decreases with an increase in temperature. The results indicate that the variation of limiting cation transference numbers in 1-butanol is in accord with this general rule except that there is a slight variation in the case of tetrabutylammonium bromide and iodide. However, such exceptions are also reported in literature; e.g., the difference in the transference numbers of the ions of potassium chloride in water (*12*) increases with the increase in temperature. In the present case, the anomalous behavior of tetrabutylammonium bromide and iodide may be due to a large difference in the sizes and the trend of solvation of the cations and anions of these electrolytes.

The transference numbers of the ions of an electrolyte depend upon the relative sizes of its solvated cation and anion in solution. Generally, the crystallographic radii of the cation and anion of an electrolyte do not differ much, and both types of ions are solvated in the solution. As the solvation of the ions

Table II. Limiting Transference Number of Cations ($t_+°$) at Various Temperatures

	$t_+°$		
electrolyte	298.15 K	308.15 K	318.15 K
LiCl	0.5310 ± 0.0005	0.5275 ± 0.0007	0.5217 ± 0.0008
Me_4NCl	0.5745 ± 0.0002	0.5705 ± 0.0004	0.5674 ± 0.0004
Et_4NCl	0.5883 ± 0.0006	0.5856 ± 0.0006	0.5797 ± 0.0004
Bu_4NCl	0.5208 ± 0.0005	0.5156 ± 0.0004	0.5124 ± 0.0003
Bu_4NBr	0.5010 ± 0.0005	0.4966 ± 0.0007	0.4931 ± 0.0004
Bu_4NI	0.4680 ± 0.0007	0.4607 ± 0.0003	0.4516 ± 0.0005

generally decreases with the rise in temperature and the highly solvated ions are affected more, therefore, the difference between the transference numbers of the cation and anion of an electrolyte tends to decrease with the rise in temperature. However, in the case of tetrabutylammonium bromide and tetrabutylammonium iodide, cations have no solvent molecule in their primary solvation sphere, whereas anions are heavily solvated in 1-butanol. Thus, the size (in solution) of only anions decreases with the increase in temperature, which results in the increase in the transference numbers of the anions. The transference numbers of the cations of all the tetraalkylammonium salts decrease with the increase in temperature, irrespective of the values of the limiting transference numbers of these ions.

Venkatasetty and Brown (*2*, *3*) have reported the conductance data of lithium, ammonium, and tetrabutylammonium iodides at 323.15 K in various solvents, including 1-butanol. Using the value of the limiting equivalent conductance of tetrabutylammonium iodide at 323.15 K and the Walden product of tetrabutylammonium ion in 1-butanol, we estimated the limiting transference number of tetrabutylammonium ion. The estimated value of the transference number of tetrabutylammonium ion at 323.15 K is much less than that at 298.15 K, showing thereby that the cation transference number of tetrabutylammonium iodide decreases with the increase in temperature and thus supports the present studies.

Assuming ions move with the solvent molecules in their primary solvation sphere under the influence of the applied electric field, Carman (*6*) suggested a method for the evaluation of relative solvation numbers of the ions from the transference number data alone. It has been observed that the relative solvation numbers of the ions in 1-butanol evaluated by Carman's method (*6*) show some anomalies; e.g., the relative solvation numbers of the cations are more than those of anions of smaller size, and the relative solvation numbers of the same anion are different in different electrolytes. These, however, do not seem true especially in the case of tetraalkylammonium ions, which have been shown to have no solvent molecule in their primary solvation sphere because of their very large size. A survey of the literature (*15*) reveals that the relative solvation numbers evaluated by this method in other solvents also show such anomalous behavior. It is, therefore, considered worthwhile to compute the solvation numbers of the ions at 298.15 K by using the present transference number data and the available conductance data (*1*–*4*).

Limiting equivalent ionic conductances of various ions have been computed by combining the conductance and transference number data (*16*). The limiting equivalent conductances of tetrabutylammonium ion evaluated from the limiting cation transference number and conductance data of tetrabutylammonium chloride, tetrabutylammonium bromide, and tetrabutylammonium iodide have been found to be (8.10 ± 0.04) × 10^{-4}, (8.05 ± 0.02) × 10^{-4}, and (8.03 ± 0.04) × 10^{-4} Ω^{-1} m^2 mol^{-1}, respectively. All these values agree within the experimental error. The ionic conductance of tetrabutylammonium ion ((8.06 ± 0.04) × 10^{-4} Ω^{-1} m^2 mol^{-1}) used for the evaluation of the ionic conductance of the other ions with use of the Kohlrausch law of independent ion migration is the mean of the above three values. All the relevant data are recorded in Table III.

Table III. Limiting Equivalent Ionic Conductances ($\lambda_\pm°$) and Solvation Numbers (*n*) of the Ions in 1-Butanol at 298.15 K

ion	$\lambda_\pm° \times 10^4$, Ω^{-1} m^2 mol^{-1}	*n*	ion	$\lambda_\pm° \times 10^4$, Ω^{-1} m^2 mol^{-1}	*n*
Li^+	8.41	3.0	$(i\text{-}Am)_3BuN^+$	8.01	0.0
NH_4^+	6.99	4.5	$Hept_4N^+$	6.59	0.0
Me_4N^+	10.11	0.9	Cl^-	7.38	3.9
Et_4N^+	10.63	0.0	Br^-	7.99	3.1
Pr_4N^+	9.06	0.0	I^-	8.99	2.3
Bu_4N^+	8.06	0.0	ClO_4^-	10.95	1.3

Solvation numbers of the ions have been evaluated from the above limiting equivalent ionic conductances, essentially by the same method reported by Paul et al. (*8*, *17*), and are recorded in Table III.

The solvation number of ammonium ion is greater than that of lithium ion. This may be due to steric factors (*18*, *19*). Various workers (*20*–*22*) have attributed this anomalous behavior to the tetrahedral structure of ammonium ion, as compared to the spherically symmetrical structures of alkali metal ions.

The solvation numbers of the anions decrease with the increase in the size of the anions. The crystallographic radii of bromide and chloride ions are more than three times larger than that of lithium ions, yet the solvation numbers of these anions are more than that of lithium ion. This indicates that the anions are heavily solvated as compared to the cations in 1-butanol.

Solvation numbers of anions in 1-butanol, in comparison with those in *N*,*N*-dimethylformamide, *N*-methylformamide, dimethyl sulfoxide, acetonitrile, nitromethane, etc., have been found to be higher (*23*). The protic solvents have been reported to solvate the anions more in comparison to aprotic solvents. The reason is the tendency of the molecules of protic solvents to form hydrogen bonds with anions, so the solvation process is not only due to the anion–dipole interactions but also due to the hydrogen bonds between the anion and solvent molecules. The higher values of solvation numbers of the anions in this solvent show the protic nature of 1-butanol.

Registry No. LiCl, 7447-41-8; BuOH, 71-36-3; Me_4NCl, 75-57-0; Et_4NCl, 56-34-8; Bu_4NCl, 1112-67-0; Bu_4NBr, 1643-19-2; Bu_4NI, 311-28-4; Li, 7439-93-2; NH_4^+, 14798-03-9; Me_4N^+, 51-92-3; Et_4N^+, 66-40-0; Pr_4N^+, 13010-31-6; Bu_4N^+, 10549-76-5; $(i\text{-}Am)_3BuN^+$, 21570-53-6; $Hept_4N^+$, 35414-25-6; Cl^-, 16887-00-6; Br^-, 24959-67-9; I^-, 20461-54-5; ClO_4^-, 14797-73-0.

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Mutual Diffusion Coefficients of Some Binary Liquid Systems: Benzene–*n*-Alkyl Alcohol

Guggilam Ramprasad, Ashok K. Mukherjee, and Tarun R. Das*

Department of Chemical Engineering, Indian Institute of Science, Bangalore 560012, India

The mutual diffusion coefficients for binary liquid systems of benzene–*n*-alkyl alcohol at various compositions have been determined by the diaphragm cell method at 28 °C. The alcohols used were the members of *n*-paraffinic alcohols ranging from C_1 to C_8. The maximum possible experimental error is 14%. The data were fitted with a generalized correlation, giving the deviation from the experimental data to within 2.75%, on average.

The present article reports the experimentally obtained diffusion coefficients for the binary systems of benzene–alcohols. The alcohols used were the first eight members of the homologous series of normal paraffinic alcohols.

Experimental Section

The diaphragm cell method (*1*) was used to measure the integral diffusion coefficients, as it is the only successful method based on Fick's first law and combines experimental simplicity with accuracy. A G-4 grade diaphragm was used in the present work. The description of the apparatus is given in Figure 1. The volume of each compartment is 70 mL only, differing by not more than 1.0 mL. The liquid layers adjacent to the surfaces of the diaphragm were stirred by magnetic stirrers that were operated by rotating permanent bar magnets fitted outside the cell. In each rotation of the magnets, the diaphragm is wiped twice. The rate of rotation of the stirrers was kept constant at 40 rotations/min.

The diaphragm cell was calibrated at 25 °C by diffusing 0.1 N potassium chloride solution (kept in the bottom compartment) into double-distilled water. The equation used to calculate the integral diffusion coefficients is

$$D_{AB} = \frac{1}{\beta t} \ln \left(\frac{C_{i,B} - C_{i,T}}{C_{f,B} - C_{f,T}} \right) \tag{1}$$

For the 0.1 N KCl–water system, the integral diffusion coefficient D_{AB}, to be used in eq 1, is obtained from

$$D_{AB} = \frac{C_B D_{C_B} - C_T D_{C_T}}{C_B - C_T} \tag{2}$$

where

$$C_B = \tfrac{1}{2}(C_{i,B} + C_{f,B}) \tag{3}$$

and

$$C_T = \tfrac{1}{2}(C_{i,T} + C_{f,T}) \tag{4}$$

The parameter D_{C_B} is an integral diffusion coefficient that would be found in a run of vanishingly short duration with the initial concentrations C_B and zero on two sides of the diaphragm, respectively. In a similar way D_{C_T} is defined for initial concentrations C_T and zero. The values of D_{C_B} and D_{C_T} were obtained from the work of Tilley and Woolf (*2*).

The cell constant, β, was measured after every 240 h of cell usage. The value of β after a period of 960 h of cell usage was found to have increased by about 2.5% of the initial value of 1917.9 m^{-2}.

In the diffusion runs, each of which was of about 24-h duration, the bottom compartment was always filled with higher density solutions. The temperature of the water bath was maintained at 28 ± 0.5 °C.

All the chemicals used in the present work were analytical reagent grade. All of them, except ethanol, were used without further purification, while the latter has been purified as described by Vogel (*3*). Potassium chloride solutions were analyzed by freshly prepared silver nitrate solution.

Compositions of the benzene–alcohol solutions were analyzed by an Abbe refractometer with a resolution of 0.0001 refractive index (RI) units. The calibration of the refractive index data was done by measuring the refractive index at various known compositions of the mixture at 28 °C.

Results

Table I lists the densities, refractive indices, and viscosities of the pure components used in this study, as well as those given in the literature.

The experimental diffusion coefficients for the benzene–*n*-paraffinic alcohol systems at various compositions are given in Table II. Each datum is an average of three runs, the variations in diffusion coefficients obtained from individual runs being within 2% from the average. The mole fraction of benzene given in Table II, column 3, indicates the initial composition of the respective benzene–alcohol mixture in the top compartment, into which benzene from the bottom compartment (containing pure benzene) diffuses and from which the alcohol diffuses into the bottom compartment. The maximum error in each of the parameters affecting the diffusion coefficient (eq 1) is as follows: for each of the composition parameters $C_{i,B}$, $C_{i,T}$, $C_{f,B}$, and $C_{f,T}$, the maximum error is 0.5×10^2 mol m^{-3}; for the cell constant, β, it is 5.0 m^{-2}. The maximum error in the determination of time, *t*, is several orders of magnitude less than that for the other parameters. The resultant maximum possible error in the diffusion coefficient, D_{AB}, could then be as high as 14%.

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