

Electrical Conductances of Tetrabutylammonium Bromide, Sodium Tetraphenylborate, and Sodium Bromide in *N,N*-Dimethylformamide at (308.15, 313.15, 318.15, and 323.15) K

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Precise conductance measurements of solutions of tetrabutylammonium bromide (Bu_4NBr), sodium tetraphenylborate (NaBPh_4), and sodium bromide (NaBr) in *N,N*-dimethylformamide 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). The limiting ionic conductances have been estimated from an appropriate division of the limiting molar conductivity of the “reference electrolyte” Bu_4NPh_4 . Slight association was found for all these salts in this solvent medium. The limiting molar conductances of electrolytes as well as single-ion conductivity values increase appreciably with temperature.

Introduction

Knowledge of the state of association of electrolytes in solution and of their interaction with solvent molecules is essential for a proper understanding of their behavior in solution. The study of electrolyte solutions in various solvents like acetonitrile,^{1–4} methanol,^{5–8} 2-methoxyethanol,^{9–12} 1,2-dimethoxyethane,^{13,14} 2-ethoxyethanol,^{15–17} etc. have been done extensively. However, systematic investigation of the transport properties of electrolytes in *N,N*-dimethylformamide has not been done to the same extent.^{18–20} Although Safonova et al.¹⁸ have reported temperature-dependent limiting conductivities on different ions in DMF, they have analyzed their data by means of the Lee Wheaton conductance equation. However, in the present study, conductance data have been analyzed by the Fuoss 78 conductance–concentration equation^{21,22} based on a more realistic physical picture than the primitive model.

The measurement of the electrical conductance of a non-aqueous electrolyte solution provides valuable information about its transport properties. Values of the equivalent or molar conductance of an electrolyte at infinite dilution (Λ^0), ionic association constant (K_A), and the association diameter (R) can be obtained from conductance data as a function of concentration by using the appropriate equation. Several equations and approaches are available for the analysis of conductance data, depending on the precision required. The selection of an equation for the determination of Λ^0 , K_A , and R is open to debate.^{18,23} In this paper, an attempt has been made to reveal the nature of various types of interactions prevailing in the solution of tetrabutylammonium bromide (Bu_4NBr), sodium tetraphenylborate (NaBPh_4), and sodium bromide (NaBr) in *N,N*-dimethylformamide by using precise conductivity measurements at (308.15, 313.15, 318.15, and 323.15) K.

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Table 1. Physical Properties of *N,N*-Dimethylformamide at (308.15, 313.15, 318.15, and 323.15) K

<i>T</i> /K	$\rho/\text{g}\cdot\text{cm}^{-3}$		$\eta/\text{mPa}\cdot\text{s}$		ϵ^c
	this work	lit. ^a	this work	lit. ^b	
308.15	0.93492	0.93571	0.71025	0.7063	35.78
313.15	0.93085	0.93073	0.66907	0.6683	34.94
318.15	0.92612	0.92598	0.64015	0.6330	34.16
323.15	0.92204	0.92114	0.59989	0.6004	33.36

^a From ref 24. ^b From ref 25. ^c From ref 26.

Experimental Section

N,N-Dimethylformamide (Merck, India, 99 % pure) was dried over molecular sieves and distilled under reduced pressure fractionally. The middle fraction was collected and redistilled. The physical properties of the purified solvent were found to be in good agreement with the literature values.^{24,25} Table 1 shows the comparison of experimental values of density and viscosity of *N,N*-dimethylformamide with literature data.^{24,25} The relative permittivities of *N,N*-dimethylformamide at different temperatures were obtained from the literature²⁶ and are included in Table 1.

All the salts were of Fluka purum or puriss grade. Tetrabutylammonium bromide (Bu_4NBr) was purified by recrystallization from acetone, and recrystallized salt was dried in vacuo at 333.15 K for 48 h. Sodium tetraphenylborate (NaBPh_4) was recrystallized three times from acetone and then dried under vacuum at 353.15 K for 72 h. Sodium bromide (NaBr) was dried in vacuo for 72 h immediately prior to use and was used without further purification.

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 having an uncertainty of 0.1 %. The cell was calibrated by the method of Lind et al.²⁷ 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.^{28,29} 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 cm³ 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. Kinematic viscosities were measured using a suspended level Ubbelohde-type viscometer.

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. The experimental uncertainties in density, viscosity, and conductivity were always within 0.01 %, 0.01 %, and 0.10 %, respectively.

Results and Discussion

The measured molar conductances (Λ) of electrolyte solutions as functions of molar concentration (c) in *N,N*-dimethylformamide 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.^{21,22} For a given set of conductivity values (c), (c_j , Λ_j ; $j = 1, \dots, n$), 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

$$\Lambda = p[\Lambda^0(1 + RX) + EL] \quad (1)$$

$$p = 1 - \alpha(1 - \gamma) \quad (2)$$

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

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

$$\beta = \frac{e^2}{\epsilon k_B T} \quad (5)$$

$$K_A = K_R(1 + K_S) \quad (6)$$

where RX is the relaxation field effect; EL is the electrophoretic countercurrent; γ is the fraction of unpaired ions; α is the fraction of contact pairs; K_A is the overall pairing constant evaluated from the association constant 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 molar concentration 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 extrapolation³⁰ of the data. Input for the program is the set (c_j , Λ_j ; $j = 1, \dots, n$), n , ϵ , η , T , initial value of Λ^0 , and an instruction to cover a preselected range of R values.

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

$$\sigma = \left[\sum [\Lambda_j(\text{calcd})^2 - \Lambda_j(\text{obsd})]/(n - 2) \right]^{1/2} \quad (7)$$

for a sequence of R values and then plotting σ against R . The best-fit R corresponds to the minimum in $\sigma(\%)$ vs R curve. However, for all of these electrolytes investigated, since a preliminary scan using a unit increment of R values from 4 to 20 produced no significant minima in the $\sigma(\%)$ vs R curves, the R value was assumed to be $R = a + d$, where

Table 2. Equivalent Conductances and Corresponding Molarities of Electrolytes in *N,N*-Dimethylformamide at (308.15, 313.15, 318.15, and 323.15) K

$T = 308.15$ K		$T = 313.15$ K		$T = 318.15$ K		$T = 323.15$ K	
c	Λ	c	Λ	c	Λ	c	Λ
mol·dm ⁻³	S·cm ² ·mol ⁻¹	mol·dm ⁻³	S·cm ² ·mol ⁻¹	mol·dm ⁻³	S·cm ² ·mol ⁻¹	mol·dm ⁻³	S·cm ² ·mol ⁻¹
Bu ₄ NBr							
0.00199	83.660	0.00198	87.081	0.00197	91.344	0.00196	95.028
0.00299	81.486	0.00298	84.571	0.00296	88.887	0.00295	92.747
0.00406	79.237	0.00404	82.591	0.00402	86.312	0.00400	90.087
0.00505	76.821	0.00502	80.378	0.00499	84.059	0.00497	87.461
0.00606	74.420	0.00603	78.158	0.00599	81.519	0.00597	85.222
0.00706	72.548	0.00702	76.110	0.00698	79.152	0.00695	82.895
0.00806	70.121	0.00801	73.562	0.00797	76.856	0.00793	80.058
0.00906	68.147	0.00901	71.765	0.00896	74.358	0.00891	77.723
NaBr							
0.00196	86.729	0.00195	91.348	0.00194	97.044	0.00194	102.648
0.00314	84.252	0.00312	89.295	0.00310	95.173	0.00309	100.854
0.00404	82.226	0.00403	87.679	0.00400	93.637	0.00399	99.371
0.00503	80.127	0.00501	85.902	0.00498	92.054	0.00496	98.004
0.00612	77.807	0.00610	83.811	0.00606	90.174	0.00604	96.008
0.00711	75.858	0.00709	82.089	0.00704	88.322	0.00702	94.597
0.00810	73.852	0.00807	80.116	0.00803	86.660	0.00799	92.712
0.00919	71.553	0.00916	78.340	0.00911	84.728	0.00907	90.999
NaBPh ₄							
0.00200	57.503	0.00199	60.809	0.00198	64.199	0.00197	67.606
0.00304	56.344	0.00302	59.545	0.00300	62.882	0.00299	66.357
0.00407	55.065	0.00405	58.314	0.00403	61.611	0.00401	64.972
0.00504	53.904	0.00502	57.197	0.00499	60.482	0.00497	63.775
0.00593	52.982	0.00590	56.075	0.00587	59.531	0.00585	62.749
0.00711	51.570	0.00708	54.701	0.00704	58.035	0.00701	61.447
0.00815	50.374	0.00811	53.558	0.00806	56.802	0.00803	60.110
0.00914	49.311	0.00910	52.368	0.00905	55.569	0.00901	58.934

Table 3. Derived Conductivity Parameters of Electrolytes in *N,N*-Dimethylformamide at (308.15, 313.15, 318.15, and 323.15) K

<i>T</i> K	Λ^0 S·cm ² ·mol ⁻¹	K_A dm ³ ·mol ⁻¹	R A ⁰	σ % ^a
		Bu ₄ NBr		
308.15	92 ± 0.96	49 ± 3.0	11.86	0.06
313.15	94 ± 0.91	45 ± 3.0	11.86	0.04
318.15	100 ± 1.0	49 ± 4.0	11.87	0.06
323.15	104 ± 1.0	48 ± 3.0	11.88	0.05
		NaBr		
308.15	94 ± 0.75	43 ± 2.0	7.80	0.07
313.15	97 ± 0.58	31 ± 2.0	7.81	0.05
318.15	102 ± 0.54	26 ± 1.0	7.82	0.05
323.15	107 ± 0.47	23 ± 1.0	7.83	0.04
		NaBPh ₄		
308.15	61 ± 0.31	32 ± 1.0	11.36	0.02
313.15	65 ± 0.31	30 ± 1.0	11.36	0.02
318.15	68 ± 0.33	29 ± 1.0	11.37	0.01
323.15	71 ± 0.27	27 ± 1.0	11.38	0.03

^a $\sigma/\% = 100\sigma/\Lambda^0$.

a is the sum of the ionic crystallographic radii and *d* is given by²²

$$d = 1.183(M/\rho_0)^{1/3} \quad (8)$$

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

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

To investigate the specific behavior of the individual ions comprising these electrolytes, it is necessary to split the limiting molar electrolyte conductances into their ionic components.

In the absence of accurate transport number data for the system in the temperature range investigated here, we have used the "reference electrolyte" method for the division of Λ^0 into their ionic components. Tetrabutylammonium tetraphenylborate (Bu₄NBPh₄) has been used as the "reference electrolyte".³¹ This electrolyte was used as the "reference electrolyte" also by Fuoss and Hirsh³² to evaluate the limiting ionic conductances in several organic solvents. We have divided the Λ^0 values of Bu₄NBPh₄ into ionic components using the following relationships.³³

$$\Lambda^0(\text{Bu}_4\text{NBPh}_4) = \lambda^0(\text{Bu}_4\text{N}^+) + \lambda^0(\text{Ph}_4\text{B}^-) \quad (9)$$

$$\frac{\lambda^0(\text{Bu}_4\text{N}^+)}{\lambda^0(\text{Ph}_4\text{B}^-)} = \frac{r(\text{Ph}_4\text{B}^-)}{r(\text{Bu}_4\text{N}^+)} = \frac{5.35}{5.00} \quad (10)$$

$$\lambda^0(\text{Bu}_4\text{N}^+) = 0.517\Lambda^0(\text{Bu}_4\text{NBPh}_4) \quad (11)$$

The ionic radii (*r*) values have been taken from the literature.^{34,35} The limiting molar conductivity (Λ^0) of the "reference electrolyte" Bu₄NBPh₄ was obtained by considering the Kohlrausch rule which allows the calculation of the Λ^0 value for a given electrolyte by the appropriate combination of others. The Λ^0 values of Bu₄NBr, NaBPh₄, and NaBr obtained in this medium have been used to obtain the Λ^0 value of Bu₄NBPh₄ through the following equation

$$\Lambda^0(\text{Bu}_4\text{NBPh}_4) = \Lambda^0(\text{Bu}_4\text{NBr}) + \Lambda^0(\text{NaBPh}_4) - \Lambda^0(\text{NaBr}) \quad (12)$$

The limiting ion conductances calculated from the above equations are recorded in Table 4 and agreed well with the literature.¹⁸

The association constants (K_A) listed in Table 3 indicate that these salts are slightly associated in *N,N*-dimethylformamide.

Table 4. Limiting Ionic Conductances in *N,N*-Dimethylformamide at (308.15, 313.15, 318.15, and 323.15) K

<i>T</i> /K	$\lambda_{\pm}^0/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$			
	Na ⁺	Bu ₄ N ⁺	Br ⁻	Ph ₄ B ⁻
308.15	32.73	30.59	61.53	28.59
313.15	34.58	32.19	62.74	30.07
318.15	36.15	34.11	66.26	31.86
323.15	38.46	35.26	69.05	32.94

This implies that a preponderant portion of each salt remains dissociated in this solvent medium. This is quite expected because of the moderate relative permittivity of the solvent medium.

The limiting ionic equivalent conductances (λ^0) decrease in the order $\lambda_{\text{Br}^-}^0 > \lambda_{\text{Na}^+}^0 > \lambda_{\text{Bu}_4\text{N}^+}^0 > \lambda_{\text{Ph}_4\text{B}^-}^0$ in this solvent medium within the temperature range of (308.15 to 323.15) K.

The limiting equivalent conductances of the electrolytes as well as the single-ion conductivity values increase appreciably with temperature. The effect of temperature on $\lambda_{\text{Na}^+}^0$ and $\lambda_{\text{Br}^-}^0$ ions is more pronounced as compared to that of $\lambda_{\text{Bu}_4\text{N}^+}^0$ and $\lambda_{\text{Ph}_4\text{B}^-}^0$ in the present solvent medium.

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