

A study on the transport behaviour of tetraphenylarsonium, tetraphenylphosphonium and some common ions in propylene carbonate from viscosity and conductance data

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Viscosity measurements for solutions of tetraphenylarsonium chloride (Ph_4AsCl), tetraphenylphosphonium bromide (Ph_4PBr) and sodium tetraphenylborate (NaBPh_4) in propylene carbonate (PC) are reported at 25, 35 and 45°C. Electrical conductances of these salts and that of tetrabutylammonium tetraphenylborate (Bu_4NBPh_4) have also been measured at 25°C. Viscosity data have been analysed by means of Jones-Dole equation and B coefficients evaluated. Conductance data have been analysed by 1978 Fuoss conductance equation in terms of equivalent conductance (Λ_0), association constant (K_A) and association distance (R). Limiting ionic values have been determined using the 'reference electrolytes' Bu_4NBBu_4 and Bu_4NBPh_4 . Stokes radii have also been evaluated and the study indicates that Na^+ and Cl^- are substantially solvated while other ions are scarcely solvated and remain as free ions in this medium.

Viscosity and conductance data provide us valuable information on the ion-solvent interactions of electrolytes in aqueous, non-aqueous and mixed solvents^{1,2}. The potentiality of propylene carbonate (PC) as a solvent medium for a variety of applications of technological importance are also well known^{3,4}. We report here the viscosity and electrical conductance of the solutions of Ph_4PBr , NaBPh_4 and Bu_4NBPh_4 in propylene carbonate. The results have been discussed to understand the nature of ion-solvent interactions.

Materials and Methods

Propylene carbonate (Merck, Germany; >99% pure) was purified as described earlier³. The purified solvent had a density of 1.1988 g cm⁻³ and viscosity of 2.4711 cP at 25°C and these values agree well with the corresponding literature values^{5,6}: 1.1993 g cm⁻³ and 2.480 cP respectively.

Tetraphenylarsonium chloride (Fluka) was recrystallised from absolute ethanol by adding dry ether and dried in vacuum⁷. Tetraphenylphosphonium bromide (Fluka, puriss) was dissolved in absolute ethanol, reprecipitated by the addition of dry ether and vacuum dried at 100°C⁷. Sodium tetraphenylborate (Fluka, puriss) was recrystallised three times from acetone and dried in vacuum at 80°C for 72 hours⁸. Tetrabutylammonium tetraphenylborate was prepared by mixing equi-

molar quantities of NaBPh_4 and Bu_4NBr . The precipitate was washed 5 times by water and dried. The bulky white precipitate was then recrystallised four times from 1:3 water-acetone and finally dried for several days at 40°C under vacuum⁹.

For viscometric study, the concentration of the salt solutions varied in the range of 0.01-0.08 mol dm⁻³ and the solutions were prepared by weight dilution of the stock solution (ca. 0.1 mol kg⁻¹). The conversion of molality into molarity was done using the density values. Kinematic viscosities were measured at the desired temperature (accurate to $\pm 0.01^\circ\text{C}$) using a suspended Ubbelohde type viscometer. Densities were measured by using an Ostwald-Sprengel type pycnometer having a bulb volume of 25 cm³. The precisions of density and viscosity measurements were $\pm 3 \times 10^{-5}$ g cm⁻³ and 0.05% respectively. All measurements were done in a water thermostat having an accuracy of $\pm 0.01^\circ\text{C}$.

For conductometric study, ten to thirteen solutions of different concentrations ranging from 0.05 to 0.005 mol dm⁻³ were prepared for each salt. The electrical conductances were measured at 25°C with a Pye Unicam PW 9509 conductivity meter at a frequency of 2000 Hz. Details of experimental procedure have been described previously^{3,10}.

Results and Discussion

The density (ρ) and viscosity (η) data for the electrolytes in the molarity range 0.01-0.08 mol dm⁻³ at 25, 35 and 45°C are given in Table 1.

The relative viscosity (η_r) data of the electrolytes in solution have been analysed with the Jones-Dole equation

$$(\eta/\eta_0 - 1)/\sqrt{c} = A - B\sqrt{c} \quad \dots (1)$$

where η and η_0 are the viscosities of the solution and solvent, respectively and c is the molar concentration.

Plots of $(\eta_r - 1)/\sqrt{c}$ against \sqrt{c} for the electrolytes have been found to be linear and the experimental A values have been calculated using the least squares method. The A coefficients were also calculated at 25°C from the physical properties

Table 1—Concentration (c), density (ρ) and relative viscosity (η_r) of Ph₄AsCl, Ph₄PBr and NaBPh₄ in PC at 25, 35 and 45°C

| c (mol dm ⁻³) | ρ (g cm ⁻³) | η_r | c (mol dm ⁻³) | ρ (g cm ⁻³) | η_r |
|--------------------------------|---------------------------------|----------|--------------------------------|---------------------------------|----------|
| Ph ₄ AsCl, 25°C | | | Ph ₄ PBr, 25°C | | |
| 0.0137 | 1.1995 | 1.017 | 0.0151 | 1.1997 | 1.020 |
| 0.0276 | 1.2002 | 1.035 | 0.0300 | 1.2005 | 1.037 |
| 0.0326 | 1.2005 | 1.040 | 0.0399 | 1.2010 | 1.049 |
| 0.0375 | 1.2007 | 1.047 | 0.0551 | 1.2019 | 1.067 |
| 0.0425 | 1.2010 | 1.052 | 0.0699 | 1.2027 | 1.084 |
| 0.0526 | 1.2015 | 1.064 | | | |
| NaBPh ₄ , 25°C | | | Ph ₄ AsCl, 35°C | | |
| 0.0100 | 1.1990 | 1.013 | 0.0136 | 1.1902 | 1.017 |
| 0.0251 | 1.1992 | 1.034 | 0.0274 | 1.1907 | 1.033 |
| 0.0401 | 1.1994 | 1.051 | 0.0323 | 1.1909 | 1.039 |
| 0.0501 | 1.1995 | 1.063 | 0.0372 | 1.1911 | 1.045 |
| 0.0598 | 1.1966 | 1.074 | 0.0421 | 1.1912 | 1.051 |
| 0.0802 | 1.1998 | 1.099 | 0.0522 | 1.1916 | 1.063 |
| Ph ₄ PBr, 35°C | | | NaBPh ₄ , 35°C | | |
| 0.0150 | 1.1904 | 1.018 | 0.0104 | 1.1900 | 1.010 |
| 0.0298 | 1.1911 | 1.035 | 0.0283 | 1.1904 | 1.031 |
| 0.0396 | 1.1916 | 1.046 | 0.0454 | 1.1908 | 1.049 |
| 0.0546 | 1.1923 | 1.062 | 0.0570 | 1.1911 | 1.061 |
| 0.0694 | 1.1930 | 1.079 | 0.0679 | 1.1914 | 1.072 |
| | | | 0.0913 | 1.1919 | 1.096 |
| Ph ₄ AsCl, 45°C | | | Ph ₄ PBr, 45°C | | |
| 0.0135 | 1.1783 | 1.016 | 0.0149 | 1.1786 | 1.017 |
| 0.0271 | 1.1785 | 1.033 | 0.0295 | 1.1792 | 1.033 |
| 0.0320 | 1.1787 | 1.038 | 0.0392 | 1.1796 | 1.043 |
| 0.0368 | 1.1788 | 1.044 | 0.0541 | 1.1802 | 1.059 |
| 0.0417 | 1.1789 | 1.050 | 0.0687 | 1.1808 | 1.075 |
| 0.0516 | 1.1791 | 1.061 | | | |
| NaBPh ₄ , 45°C | | | | | |
| 0.0099 | 1.1783 | 1.012 | 0.0493 | 1.1798 | 1.058 |
| 0.0246 | 1.1789 | 1.030 | 0.0589 | 1.1801 | 1.070 |
| 0.0394 | 1.1794 | 1.047 | 0.0789 | 1.1808 | 1.093 |

Table 2—Theoretical A values, experimental A coefficients (A_{obs}) and viscosity B values at 25°, 35° and 45°C

| Salts | A _{theo} (dm ^{3/2} mol ^{-1/2}) | A _{obs} (dm ^{3/2} mol ^{-1/2}) | | | B ^v (dm ³ mol ⁻¹) | | |
|--|---|--|--------|--------|--|-------------------|-------------------|
| | | 25° | 35° | 45°C | 25° | 35° | 45°C |
| Ph ₄ AsCl | 0.0112 | 0.0111 | 0.0066 | 0.0066 | 1.176 (±0.007) | 1.166 (±0.003) | 1.146 (±0.003) |
| Ph ₄ PBr | 0.0106 | 0.0207 | 0.0149 | 0.0126 | 1.150 (±0.003) | 1.087 (±0.001) | 1.049 (±0.001) |
| NaBPh ₄ | 0.0154 | 0.0166 | 0.0134 | 0.0081 | 1.175 (±0.001) | 1.158 (±0.004) | 1.130 (±0.002) |
| Bu ₄ NBPh ₄ ^a | 0.0176 | 0.0135 | 0.0198 | 0.0237 | 1.302 (±0.061) | 1.283 (±0.013) | 1.256 (±0.002) |

^aData taken from ref. (15)

Table 3—Ionic B coefficients (B) in propylene carbonate at 25, 35 and 45°C

| Salts | B (dm ³ mol ⁻¹) | | |
|---------------------------------|---|-------|-------|
| | 25° | 35° | 45° |
| Ph ₄ As ⁺ | 0.924 | — | — |
| Ph ₄ P ⁺ | 0.692 | 0.659 | 0.638 |
| Bu ₄ N ⁺ | 0.585 | 0.577 | 0.564 |
| Na ⁺ | 0.458 | 0.452 | 0.438 |
| Cl ⁻ | 0.252 | — | — |
| Br ⁻ | 0.458 | 0.428 | 0.411 |
| BPh ₄ ⁻ | 0.717 | 0.706 | 0.692 |

 Table 4—Ionic radii (R_±) and solvation number (n_s) of ions in PC at 25°C

| Ion | r _c (Å) | R _± (Å) | n _s |
|---------------------------------|-----------------------|-----------------------|----------------|
| Ph ₄ As ⁺ | 6.40 ^a | 5.27 | -8.55 |
| Ph ₄ P ⁺ | 6.30 ^a | 4.79 | -9.05 |
| Bu ₄ N ⁺ | 4.94 ^b | 4.53 | -3.18 |
| Na ⁺ | 1.17 ^b | 4.18 | 2.12 |
| Cl ⁻ | 1.64 ^b | 3.42 | 0.89 |
| Br ⁻ | 1.80 ^b | 4.17 | 1.78 |
| BPh ₄ ⁻ | 4.80 ^b | 4.84 | -0.28 |

^ataken from ref. (4); ^btaken from ref. (24)

of the solvent by using Falkenhagen-Vernon equation

$$A_{\text{theo}} = \frac{0.2577 \Lambda_0}{\eta_0 (\epsilon T)^{0.5} \lambda_0^+ \lambda_0^-} \left[1 - 0.6863 \left(\frac{\lambda_0^+ - \lambda_0^-}{\Lambda_0} \right)^2 \right] \quad \dots (2)$$

The conductance data required in these calculations have been taken from the present work. A-coefficients (A_{theo}) thus calculated from Eq. (2) are recorded in Table 2. These A_{theo} values have been used for the analysis of viscosity data. In view of the weak temperature dependence of A coefficients, the A_{theo} values at 25°C were utilised at the other two temperatures also. Viscosity B coefficients thus calculated by using the least squares method are also presented in Table 2.

Ionic B values were calculated by_{11,12} using Bu₄NBPh₄ as the 'reference electrolyte' and the values for Bu₄N⁺ and Ph₄B⁻ have been taken from our earlier work¹³. Ionic B coefficients have been recorded in Table 3.

Ionic B values have also been analysed on the

basis of Einstein's equation and the corresponding ionic radii evaluated. The number of solvent molecules (n_s) bound to an ion in the primary region of solvation have also been calculated^{13,14}. Table 4 deals with the ionic radii (R_±) and solvation number of ions at 25°C.

The measured equivalent conductances and the corresponding molarities for Ph₄AsCl, Ph₄PBr, NaBPh₄ and Bu₄NBPh₄ in PC are recorded in Table 5. Conductance data have been analysed by the Fuoss 1978 conductance equation^{15,16}:

$$\Lambda = p[\Lambda_0(1 + R_X) + E_L] \quad \dots (3)$$

$$p = 1 - \alpha(1 - \gamma) \quad \dots (4)$$

$$\gamma = 1 - K_\Lambda c \gamma^2 f^2 \quad \dots (5)$$

$$-\ln f = \beta x / 2(1 + xR) \quad \dots (6)$$

$$\beta = e^2 / \epsilon k_B T \quad \dots (7)$$

$$K_\Lambda = K_R / (1 - \alpha) = K_R (1 + K_S) \quad \dots (8)$$

where R_X and E_L are relaxation and hydrodynamic terms, respectively, and the other terms have their usual significance. The parameters Λ₀, K_Λ and R were obtained by solving the above equations. Initial Λ₀ values for the iteration proce-

Table 5—Equivalent conductances (Λ) and corresponding molarities (c) of the salts in PC at 25°C

| 10^4c (mol dm ⁻³) | Λ (S cm ² mol ⁻¹) | 10^4c (mol dm ⁻³) | Λ (S cm ² mol ⁻¹) |
|------------------------------------|---|------------------------------------|---|
| Ph ₄ AsCl | | Ph ₄ PBr | |
| 526.010 | 20.95 | 503.161 | 22.01 |
| 424.854 | 21.54 | 425.378 | 22.56 |
| 375.326 | 21.85 | 390.417 | 22.81 |
| 325.538 | 22.21 | 352.436 | 23.11 |
| 276.169 | 22.63 | 300.803 | 23.49 |
| 226.221 | 22.98 | 271.034 | 23.74 |
| 175.895 | 23.42 | 240.035 | 24.06 |
| 125.065 | 23.98 | 210.937 | 24.30 |
| 90.716 | 24.46 | 191.420 | 24.53 |
| 51.498 | 25.03 | 152.793 | 24.97 |
| | | 127.613 | 25.27 |
| NaBPh ₄ | | 80.867 | 26.03 |
| 495.579 | 13.66 | 51.653 | 26.63 |
| 426.918 | 14.08 | | |
| 380.376 | 14.36 | Bu ₄ NBPh ₄ | |
| 351.022 | 14.58 | 217.489 | 14.52 |
| 298.843 | 14.92 | 187.250 | 14.79 |
| 271.135 | 15.16 | 158.174 | 15.10 |
| 239.549 | 15.41 | 138.402 | 15.31 |
| 210.876 | 15.65 | 118.630 | 15.50 |
| 189.741 | 15.83 | 98.859 | 15.73 |
| 155.893 | 16.21 | 79.087 | 16.00 |
| 125.704 | 16.51 | 59.315 | 16.28 |
| 90.385 | 16.94 | 39.543 | 16.65 |
| 52.179 | 17.53 | | |

ture were obtained from Shedlovsky extrapolation of the data. Details of the data analysis have been mentioned earlier³.

Values of the limiting equivalent conductances (Λ_0), association constants (K_A) and association distance parameters (R) have been recorded in Table 6.

The single ion conductances (λ_0^\pm) were evaluated from the division of Λ_0 values of 'reference electrolytes' viz. Bu₄NBPh₄^{12,17} and Bu₄NBBu₄¹⁷ using the following relationships:

$$\frac{\lambda_0(\text{Bu}_4\text{N}^+)}{\lambda_0(\text{Ph}_4\text{B}^-)} = \frac{r_{\text{Ph}_4\text{B}^-}^3}{r_{\text{Bu}_4\text{N}^+}^3} = \frac{5.35}{5.00} = 1.07 \quad \dots (9)$$

$$\lambda_0(\text{Bu}_4\text{N}^+) = \lambda_0(\text{Bu}_4\text{B}^-) \quad \dots (10)$$

Bu₄NBBu₄ has been used here in order to have a comparative study of the ion conductances based on these two reference electrolytes. The Λ_0 value of Bu₄NBBu₄ has been taken from our previous

work³. The limiting ionic conductances thus calculated have been recorded in Table 7.

The A coefficients reported in Table 2, are small and positive for all the electrolytes. The experimental A coefficients of Ph₄AsCl and NaBPh₄ at 25°C agree well with the corresponding theoretical values though in other cases, the variation is comparatively high. It has already been established that a very small fractional percentage of experimental uncertainty can lead to large variations between theoretical and experimental A values⁹.

Table 2 further shows that the viscosity B coefficients are large and positive and the B values of Ph₄AsCl is greater than that of Ph₄PBr at all the three temperatures. Similar results have also been obtained in aqueous system¹⁸ though the values in water (1.08 and 0.98 for Ph₄AsCl and 1.05 and 0.95 for Ph₄PBr at 25° and 35°C respectively) are less than the corresponding values in PC. B coefficients of Ph₄PBr in PC at 25 and 35°C are smaller than the corresponding values in DMSO (1.356 and 1.270 at 25 and 35°C respectively¹⁷). On the contrary, the values of NaBPh₄ in PC are larger than the respective B values in DMSO viz. 1.14, 1.07 and 1.01 at 25, 35 and 45°C respectively¹⁹. Table 2 also shows that the viscosity B coefficients decrease with the rise of temperature but its temperature coefficients are quite small.

From Table 3, we see that the ionic B values decrease in the order Ph₄As⁺ > Ph₄P⁺ > Bu₄N⁺ > Na⁺ and Ph₄B⁻ > Br⁻ > Cl⁻. Ionic B value of Cl⁻ ion in PC at 25°C was calculated by taking the B value of Et₄N⁺ ion ($B_{\text{Et}_4\text{N}^+} = 0.258$) from our earlier work¹³ and that of Et₄NCl ($B = 0.51$) from the literature²⁰ assuming that the rule of additivity holds true in this case.

From Table 4, we see that the R_+ values of the cations are in the order: Ph₄As⁺ > Ph₄P⁺ > Bu₄N⁺ > Na⁺. Comparison with their ionic radii shows that the tetraphenyl ions are scarcely solvated in propylene carbonate. However, R_+ value for Na⁺ ion is about four times of its crystallographic radius indicating that this ion is substantially solvated in this medium. Amongst the anions, although the ionic radius of Ph₄B⁻ is comparable to its R_- value but the R_- values of Cl⁻ and Br⁻ are much greater than their corresponding crystallographic radii suggesting that these ions are likely to be solvated in this solvent. Similar behaviour of solvation of Br⁻ ion in PC has been reported by Yeager *et al.*²¹ from spectroscopic investigation. On the other hand, our conductance data for some bromide salts in PC³ predicts poor solvation of this ion and similar result has also been ob-

Table 6—Conductance parameters of the electrolytes in PC at 25°C

| Salts | Λ_0 (S cm ² mol ⁻¹) | K_Λ (dm ³ mol ⁻¹) | Walden product | R (Å) | σ |
|-----------------------------------|---|---|-------------------|----------|----------|
| Ph ₄ AsCl | 27.21 ± 0.02 | 4.57 ± 0.07 | 0.672 | 11.20 | 0.10 |
| Ph ₄ PBr | 29.01 ± 0.02 | 5.52 ± 0.05 | 0.717 | 9.30 | 0.07 |
| NaBPh ₄ | 19.58 ± 0.02 | 7.75 ± 0.08 | 0.484 | 14.00 | 0.09 |
| Bu ₄ NBPh ₄ | 18.30 ± 0.02 | 8.16 ± 0.12 | 0.452 | 18.20 | 0.08 |

Table 7—Limiting ionic conductances (λ_0^\pm), Walden products ($\lambda_0^\pm \eta_0$) and Stokes radii (r_s) of the ions in PC at 25°C

| Ion | λ_0^{+a} | λ_0^{+b} | $\lambda_0^+ \eta_0^a$ | $\lambda_0^+ \eta_0^b$ | r_s^a | r_s^b |
|---------------------------------|--------------------|------------------|------------------------|------------------------|---------|---------|
| Ph ₄ As ⁺ | 11.31 | — | 0.279 | — | 2.93 | — |
| Ph ₄ P ⁺ | 10.77 | 10.79 | 0.266 | 0.267 | 3.08 | 3.06 |
| Bu ₄ N ⁺ | 9.44 | 9.46 | 0.234 | 0.235 | 3.49 | 3.48 |
| Na ⁺ | 10.72 | 10.74 | 0.265 | 0.265 | 3.09 | 3.09 |
| Cl ⁻ | 15.90 ^c | — | 0.393 | — | 2.08 | — |
| Br ⁻ | 18.24 | 18.22 | 0.451 | 0.450 | 1.81 | 1.82 |
| Ph ₄ B ⁻ | 8.86 | 8.84 | 0.219 | 0.218 | 3.74 | 3.75 |

^acalculations based on Bu₄NBBu₄, ^bcalculations based on Bu₄NBPh₄, ^cfrom ref. (22).

tained by Takeda and his coworkers²² from their conductivity measurements. Though differences are noteworthy we should not expect a close correlation between the derived values based on viscosity B coefficients and ionic conductances, since the transition state for viscous flow is somewhat different from that for the movement of an ion in an electric field¹⁷. Further, we have derived R_+ values of the ions from Einstein's equation¹³ where the ion has been assumed to be rigid sphere moving in a continuum and the equation in practice, may be expressed as $B_+ = 6.307 \times 10^{21} \times R_+$. Later on, Krungalz²³ made an elaborate study on some tetraalkylammonium ions in some non-aqueous solvents and proposed a modified equation: $B_{R,N} = k \times R_{R,N}^3$, where k is very much solvent dependent. Thus, the R_+ values calculated by these two methods may be different and this may also be one of the reasons for the observed differences between the ionic values based on viscometric and conductometric data.

Table 6 shows that the limiting equivalent conductances (Λ_0) of the tetraphenyl salt decrease in the order: Ph₄PBr > Ph₄AsCl > NaBPh₄ > Bu₄NBPh₄. Since the ionic radii of Ph₄P⁺ and Ph₄As⁺ are close to each other and both the ions are large enough, it appears that the higher mobility of Br⁻ ion compared to Cl⁻ ion is primarily responsible for the larger Λ_0 value of Ph₄PBr.

From Table 7, we see that the limiting ion conductances (λ_0) obtained on the basis of two reference electrolytes viz. Bu₄NBBu₄ and Bu₄NBPh₄ are in very close agreement with each other and it

suggests that either of the above two reference electrolytes can be used for the division of Λ_0 values into its ionic components. Due to low solubility of Ph₄AsBPh₄ in PC, the Λ_0 value could not be measured, so the λ_0 for Ph₄As⁺ ion was calculated by taking λ_0 of Cl⁻ ion ($\lambda_0 = 15.90$) from the literature²². The λ_0 values for Na⁺ and Br⁻ ions obtained by us (10.72 and 18.24 respectively) are in good agreement with the values ($\lambda(\text{Na}^+) = 10.45$ and $\lambda(\text{Cl}^-) = 18.70$) reported by Takeda *et al.*²² Results indicate that the mobility of Br⁻ ion is greater than that of Cl⁻ ion and is most likely due to substantial solvation of Cl⁻ ion in this medium.

The Stokes radii of tetraphenyl ions (Table 7) are less than the corresponding crystallographic radii (r_c)^{4,24} suggesting that these ions are almost unsolvated in this system. However, for Na⁺ ion the Stoke radius (3.09 Å) is much greater than the crystallographic radius (0.95 Å) and this indicates its substantial solvation in this medium. This is also consistent with the results obtained from the present viscometric study. Moreover, the Stokes radius obtained by us (3.09 Å) is in excellent agreement with the literature value (3.10 Å) (ref. 22) Table 7 further shows that the Br⁻ ion has the highest mobility among the anions. The lower mobility of Cl⁻ ion than Br⁻ ion is due to the enhanced ion-solvent interactions between Cl⁻ ion and solvent molecules which in its turn increases the size of Cl⁻ ion in solution. Larger Stokes radius of Cl⁻ ion compared to that of Br⁻ ion also supports the above viewpoint. On the other hand,

it appears that larger sizes of tetraphenyl ions, their low charge density values and the moderately high relative permittivity of PC render these ions to be free, unassociated and almost unsolvated in this solvent.

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