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Partial Molar Volumes and Viscosity *B*-Coefficient of *N*-Phenylbenzohydroxamic Acid in Dimethylsulfoxide at Different Temperatures

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The densities and viscosities of *N*-phenylbenzohydroxamic acid (PBHA) have been determined as a function of concentration in dimethylsulfoxide (DMSO) at various temperatures, (298.15, 303.15, 308.15, 313.15, and 318.15) K. The density data have been analyzed using Masson's equation, and partial molar volume (V_ϕ^0) at infinite dilution and the slope S_v^* of Masson's equation are computed. The limited partial molar expansibilities (ϕ_E^0) have also been calculated from the temperature dependence of limiting partial molar volume (V_ϕ^0). The viscosity data have been analyzed using the Jones−Dole equation, and the viscosity *B*-coefficients are calculated. The results have been discussed in terms of solute−solvent interaction, and it was found that PBHA acts as a structure maker in the present system.

Introduction

Dimethylsulfoxide (DMSO), a typical aprotic solvent having both polar and nonpolar groups, is an important solvent in chemistry, biotechnology, and medicine for the dissolution of various substances and as an antidepressing agent of living cells. The volumetric behavior of electrolyte and nonelectrolyte solutions can provide useful information regarding solute−solute and solute−solvent interaction. The apparent molar volumes and expansibilities at infinite dilution and viscosities are important tools to study solute−solvent interaction.[1–11] Hydroxamic acids are a group of weak organic acids having the general formula RC (=O) N (R′) OH. They are weaker proton donors than structurally related carboxylic acids, RC (=O) OH. These acids are found in the tissues of plants and in metabolites of bacteria and fungi. Hydroxamic acids have been recognized as compounds of pharmacological, toxicological, and pathological importance.[12–18] Hydroxamic acids are strong chelates, and the study of their complexation with a metal ion is of particular importance.[19–22] *N*-Phenylbenzohydroxamic acid trivially named as PBHA ($C_6H_5NHO{\cdot}C_6H_5C{=}O$) is a versatile metal extractant and behaves as a nonelectrolyte. The purpose of the present study is to determine the densities, viscosities, apparent molar volumes, apparent molar expansibilities at infinite dilution, and viscosity *B*-coefficient of PBHA in DMSO in order to understand the solution behavior of PBHA.

Experimental Section

Materials and Methods. *N*-Phenylbenzohydroxamic acid was prepared in the laboratory by the procedure reported in the literature.[23] The solute was purified by recrystallizing thrice with benzene and dried over phosphorus pentaoxide in a vaccum desiccator for several hours. The melting point was determined on a Tempo apparatus and is uncorrected. IR spectra were recorded with an FTIR 8400 Series Shimazdu (Japan) using KBr pellets. Elemental analysis was determined with a Vario-EL analysis apparatus. PBHA: observed mp of 121 °C and reported mp of 122 °C in the literature; IR, cm^{-1}, 3100, 1640, 1340, and 1018. Anal. ($C_{13}H_{12}NO_2$) Calcd: C, 73.23; N, 6.57; H, 5.20. Found: C, 74.68; N, 6.41; H, 5.16. DMSO was purchased from Merck. A stock solution of 0.4694 M was prepared by dissolving PBHA in 100 mL of DMSO. Solutions of varying concentration from (0.4694 to 0.0469) M were then prepared from the stock solution by a mass dilution technique. Uncertainties in solution concentrations were estimated to be ± 0.001 units.

Measurement of Density, ρ. Densities of dimethylsulfoxide and hydroxamic acid solutions were determined using a 10^3 cm^3 double-armed pycnometer at temperatures (298.15, 303.15, 308.15, 313.15, and 318.15) K. The pycnometer was calibrated at the desired temperatures with freshly prepared triple distilled water. The estimated precision of the density measurement of solutions was ± 0.03 $\text{kg}{\cdot}\text{m}^{-3}$. The reproducibility of the density measurements was ± 0.04 $\text{kg}{\cdot}\text{m}^{-3}$.

Measurement of Viscosity, η. The viscosity was measured by means of a suspended level Ubbelohde viscometer. The viscometer was submerged in a thermostat bath (Maharana, instrument manufacturing company, Ajmer, India) at temperatures (298.15, 303.15, 308.15, 313.15, and 318.15) K with a resolution of ± 0.01 K.

The viscosity values were determined using the relation

$$\eta = \rho(Kt - L/t) \qquad (1)$$

where η is the viscosity; ρ is the density of the liquid; t is the flow time. K and L are constants for a given viscometer. The flow time was measured with an electronic stopwatch capable of measuring time within ± 0.01 s. At least three repetitions of each data set reproducible to 0.05 s were obtained. The values of constant K and L were obtained by measuring the flow time of triply distilled water at temperatures (298.15, 303.15, 308.15, 313.15, and 318.15) K using the density and viscosity values of water from the literature.[24] Linear regression analysis of a plot of $\eta t/\rho$ against t^2 for triply distilled water at five temperatures provides estimates of $K = 2.45{\cdot}10^{-9}$ $\text{m}{\cdot}\text{s}^{-2}$ and $L = -0.10986{\cdot}10^{-4}$ m^2 the as slope and intercept of the plot, respectively, with a correlation coefficient of 0.9981. The estimated precision of experimental viscosities was 0.001 $\text{mPa}{\cdot}\text{s}$. The experimental values of densities, ρ_0, and viscosities, η_0, of DMSO at (298.15, 303.15, 308.15, 313.15, and 318.15) K are given in Table 1.

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Table 1. Properties of DMSO

	$\rho_o \cdot 10^3$/kg·m^{-3}		η_o/mPa·s	
T/K	this work	lit.	this work	lit.
298.15	1.09475	1.0955[a]	1.9932	1.9960[c]
303.15	1.09076	1.0896[b]	1.8006	1.7979[d]
308.15	1.08606	1.0855[a] 1.0847[b]	1.6520	1.6540[c] 1.6092[d]
313.15	1.08045	1.0797[b]	1.5160	1.4492[d]
318.15	1.07526	1.0757[a] 1.0748[b]	1.3980	1.3960[c] 1.3173[d]

[a] ρ_o.[25] [b] ρ_o.[26] [c] η_o.[26] [d] η_o.[26]

Table 2. Densities, ρ, of PBHA in DMSO from T = (298.15 to 318.15) K

	$\rho \cdot 10^3$/kg·m^{-3}				
C/mol·dm^{-3}	T/K = 298.15	T/K = 303.15	T/K = 308.15	T/K = 313.15	T/K = 318.15
0.0469	1.09723	1.09369	1.08907	1.08472	1.07880
0.0938	1.09839	1.09433	1.09008	1.08626	1.08058
0.1408	1.09859	1.09495	1.09161	1.08693	1.08155
0.1877	1.09936	1.09603	1.09183	1.08756	1.08282
0.2347	1.10014	1.09727	1.09314	1.08834	1.08333
0.2816	1.10075	1.09829	1.09408	1.08959	1.08433
0.3286	1.10146	1.09891	1.09564	1.09074	1.08580
0.3755	1.10202	1.09988	1.09652	1.09197	1.08686
0.4225	1.10275	1.10055	1.09767	1.09311	1.08792
0.4694	1.10280	1.10096	1.09881	1.09424	1.08902

Table 3. Viscosities, η, of PBHA in DMSO from T = (298.15 to 318.15) K

	η/mPa·s				
C/mol·dm^{-3}	T/K = 298.15	T/K = 303.15	T/K = 308.15	T/K = 313.15	T/K = 318.15
0.0469	2.4910	2.1026	1.8667	1.6653	1.2957
0.0938	2.5739	2.1314	1.9207	1.7068	1.3124
0.1408	2.7135	2.2940	2.0651	1.7989	1.3777
0.1877	2.7519	2.3247	2.0711	1.8260	1.4186
0.2347	2.7752	2.4027	2.0879	1.8380	1.4293
0.2816	2.8596	2.4166	2.1468	1.8906	1.4712
0.3286	2.9875	2.4780	2.2280	1.9580	1.5200
0.3755	3.0235	2.5097	2.2478	1.9974	1.5425
0.4225	3.0857	2.5372	2.2754	2.0043	1.5497
0.4694	3.1369	2.5820	2.3327	2.0149	1.5652

Results and Discussions

The measured values of densities and viscosities of PBHA in DMSO as a function of concentration at (298.15, 303.15, 308.15, 313.15, and 318.15) K are listed in Table 2 and Table 3, respectively. The densities and viscosities of PBHA in DMSO increase linearly at a particular temperature (Figures 1 and 2).

Volumetric Studies. The experimental values of densities were used to calculate the apparent molar volumes of PBHA in DMSO using the following expression[27]

$$V_\phi = 1000(\rho_0 - \rho)/C\rho\rho_0 + M_2/\rho \tag{2}$$

where V_ϕ is the apparent molar volume; C is the molarity of PBHA in solution; M_2 is the molar mass of PBHA; and ρ_o and ρ are the densities of the DMSO and solution, respectively. The resulting values of V_ϕ of solute are reported in Table 4. The positive values of V_ϕ of PBHA in DMSO indicate strong solute−solvent interactions. These interactions are strengthened with concentration at a particular temperature and are weakened with a rise in temperature at a constant concentration of solute.

The apparent molar volume at infinite dilution of solute was calculated using a least-squares treatment of the plot of V_ϕ versus $C^{1/2}$ using Masson's expression.[28]

$$V_\phi = V_\phi^0 + S_v^* C^{1/2} \tag{3}$$

where V_ϕ^0 is the apparent molar volume at infinite dilution and S_v^* is the experimental slope. The calculated values of V_ϕ^0 and S_v^* are along with their standard error listed in Table 5. V_ϕ^0 is also regarded for solute−solvent interaction as it is the apparent molar volume at infinite dilution, by definition, and is independent of solute−solute interaction. The positive values indicate the presence of strong solute−solvent interactions. These interactions are weakened with a rise in temperature.

The variation of V_ϕ^0 with temperature can be expressed as

$$V_\phi^0 = a + bT + cT^2 \tag{4}$$

The temperature T is expressed in Kelvin. The coefficients a, b, and c have been estimated by the least-squares fitting of the apparent molar volume at infinite dilution data in eq 4, and the following equation is obtained.

$$V_\phi^0 = -1704.160(\pm 1056.454) + 12.963T(\pm 6.859) - 0.023T^2(\pm 0.011) \tag{5}$$

The limiting apparent molar expansibilities can be obtained by differentiating eq 4 with respect to temperature.

$$\phi_E^0 = (\delta V_\phi^0/\delta T)_P = b + 2cT \tag{6}$$

where ϕ_E^0 is apparent molar expansibility at infinite dilution and p is the pressure. The values of ϕ_E^0 are listed in Table 5. Negative expansibility (i.e., decreasing volume with increasing temperature) is a characteristic property of aqueous solutions of hydrophobic solutes.[29] Although the solvent in the present system is not water, it is still a slightly polar solvent due to the S=O group. On the other hand, the PBHA studies have a hydrophobic/hydrophilic balance. So, one can say the negative ϕ_E^0 values originate from the highly hydrophobic characters of the PBHA.

Studies on Viscous Flow. The solute−solvent interaction can be discussed through the change of a dynamic property such as viscosity. The variation of relative viscosity for PBHA in DMSO can be represented by the Jones−Dole equation.[30]

$$\eta_r = \eta/\eta_0 = 1 + AC^{1/2} + BC \tag{7}$$

where η_r is the relative viscosity; η and η_0 are the viscosities of solution and solvent; and C is the concentration. The data of

Table 4. Apparent Molar Volume, V_ϕ, of PBHA in DMSO from T = (298.15 to 318.15) K

	$V_\phi \cdot 10^{-6}$ /m^3·mol^{-1}				
C/mol·dm^{-3}	T/K = 298.15	T/K = 303.15	T/K = 308.15	T/K = 313.15	T/K = 318.15
0.0469	150.1460	141.2160	140.2600	117.8180	131.3259
0.0938	162.3300	161.9617	158.6750	142.8613	147.7585
0.1408	171.2200	169.2690	161.5117	156.4297	160.7366
0.1877	173.3418	170.5510	168.8830	163.3787	161.8344
0.2347	174.5440	171.5439	169.2234	166.9352	166.8793
0.2816	175.8000	171.4266	170.5359	167.7722	168.6333
0.3286	176.4400	172.9653	169.7459	168.5509	168.2858
0.3755	177.2320	173.2577	170.7118	168.9489	169.4050
0.4225	177.4648	174.1195	170.8444	169.3859	169.9538
0.4694	178.9400	175.2600	170.9989	169.7274	170.4437

Table 5. Apparent Molar Volume at Infinite Dilution V_ϕ^0, Experimental Slope S_v^*, and Apparent Molar Expansibilities at Infinite Dilution ϕ_E^0, of PBHA in DMSO from T = (298.15 to 318.15) K

T	$V_\phi^0 \cdot 10^{-6}$	$S_v^* \cdot 10^{-6}$	$\phi_E^0 \cdot 10^{-6}$
K	m^3·mol^{-1}	m^3·mol$^{-3/2}$·dm$^{-3/2}$	m^3·mol^{-1}
298.15	146.181 (±4.518)	52.514 (±8.890)	−0.7519
303.15	141.252 (±6.537)	55.266 (±12.865)	−0.9819
308.15	138.708 (±5.889)	54.291 (±11.589)	−1.2119
313.15	132.443 (±6.840)	60.646 (±12.888)	−1.4419
318.15	125.403 (±6.437)	74.201 (±12.668)	−1.6719

Table 6. Relative Viscosities η_r of PBHA in DMSO from T = (298.15 to 318.15) K

	η_r/mPa·s				
C/mol·dm^{-3}	T/K = 298.15	T/K = 303.15	T/K = 308.15	T/K = 313.15	T/K = 318.15
0.0469	1.2498	1.1677	1.1300	1.0984	0.9268
0.0938	1.2913	1.1837	1.1627	1.1259	0.9388
0.1408	1.3614	1.2740	1.1469	1.1866	0.9855
0.1877	1.3806	1.2910	1.2537	1.2045	1.0147
0.2347	1.3923	1.3344	1.2639	1.2124	1.0224
0.2816	1.4347	1.3421	1.2995	1.2471	1.0524
0.3286	1.4989	1.3762	1.3487	1.2916	1.0873
0.3755	1.5169	1.3938	1.3606	1.3175	1.0103
0.4225	1.5481	1.4091	1.3774	1.3219	1.1085
0.4694	1.5738	1.4340	1.4120	1.3291	1.1196

Table 7. Value of the B-Coefficient of PBHA in DMSO from T = (298.15 to 318.15) K

T/K	viscosity B-coefficient·10^{-6}/m^3·mol^{-1}	dB/dT
298.15	0.7601 (±0.0360)	
303.15	0.6244 (±0.0529)	
308.15	0.7010 (±0.0517)	
313.15	0.5679 (±0.0393)	
318.15	0.4254 (±0.0673)	−0.0167

η_r are presented in Table 6, where A and B are the constants, characteristic of ion−ion and ion−solvent interaction, respectively. In the case of nonelectrolytes, A = 0, and this reduces the [7]Jones−Dole equation to

$$\eta_r = \eta/\eta_1 = 1 + BC \tag{8}$$

where B is the Jones−Dole B-coefficient and is obtained by plotting η_r against C, where slopes of the straight line yield the B-coefficient.

Einstein[30] proposed an equation, which describes the concentration dependence of the relative viscosity of the solution of nonelectrolytes.

$$\eta = \eta_1(1 + 2.5\nu) \tag{9}$$

In this equation, ν is the aggregate volume of the particles in a unit volume (1 cm^3) of this solution, and the coefficient of η is 2.5. If the term 2.5ν is taken to be valid for nonelectrolytes, then it is equivalent to the product BC in the Jones−Dole expression.[30] The values of BC and 2.5ν obtained are nearly of the same magnitude in the present system[31,32] and are reported in Table 8. The B-coefficient measures size and shape effects as well as the structural effect induced by solute−solvent interaction.[33] A large and positive B-coefficient for PBHA in DMSO indicates a structure-making action (hydrophobic and hydrogen bonding actions) of solute on solvents.[34] The sign of dB/dT values gives important information regarding the structure-making and structure-breaking roles of solute in the solvent media.[35,36] As evident from Table 7, a negative dB/dT trend was observed. This is an indication of the structure-making action of PBHA by hydrogen bonding.[37]

Table 8. Values of BC (2.5ν) of PBHA in DMSO from T = (298.15 to 318.15) K

C/mol·dm^{-3}	T/K = 298.15	T/K = 303.15	T/K = 308.15	T/K = 313.15	T/K = 318.15
0.0469	0.2498	0.1677	0.1300	0.0984	0.0732
0.0938	0.2913	0.1837	0.1627	0.1259	0.0612
0.1408	0.3614	0.2740	0.1469	0.1866	0.0145
0.1877	0.3806	0.2910	0.2537	0.2045	0.0147
0.2347	0.3923	0.3344	0.2639	0.2124	0.0224
0.2816	0.4347	0.3421	0.2995	0.2471	0.0524
0.3286	0.4989	0.3762	0.3487	0.2916	0.0873
0.3755	0.5169	0.3938	0.3606	0.3175	0.0103
0.4225	0.5481	0.4091	0.3774	0.3219	0.1085
0.4694	0.5738	0.4340	0.4120	0.3291	0.1196

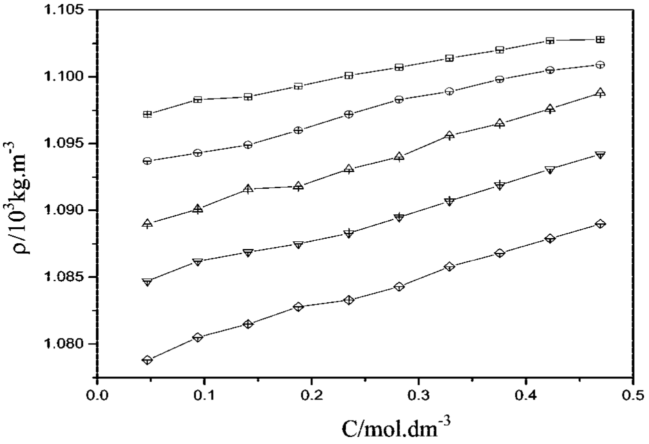

Figure 1. Plot of density vs concentraction of PBHA in DMSO at —□—, 298.15 K; —○—, 303.15 K; —Δ—, 308.15 K; —▽—, 313.15 K; —◇—, 318.15 K.

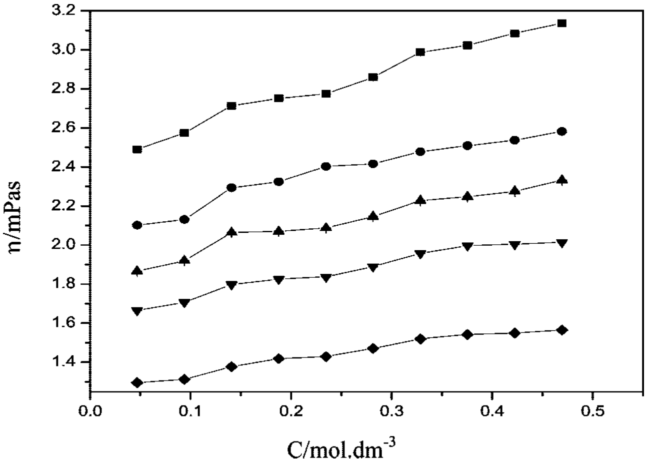

Figure 2. Plot of viscosity vs concentraction of PBHA in DMSO at —■—, 298.15 K; —●—, 303.15 K; —▲—, 308.15 K; —▼—, 313.15 K; —◆—, 318.15 K.

Conclusion

The hydroxamic acid functional group, −NOH·C=O, shows a wide range of biological activity, and viscosity is an important parameter that affects the permeation through biological membranes. Using density and viscosity data, partial molar volume and apparent molar expansibilities at infinite dilution and the Jones−Dole B-coefficient have been computed. The behavior of this parameter suggests strong solute−solvent interactions. The negative values dB/dT suggest that PBHA acts as structure maker in DMSO through hydrogen bonding.

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