

Ionic Conductivity in Binary Solvent Mixtures. 4. Dimethyl Sulfoxide + Water at 25 °C

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The conductance behavior of L-ascorbic acid has been studied in dimethyl sulfoxide and dimethyl sulfoxide + water mixtures in 0, 20, 40, 60, 80, and 100 mass % DMSO at 25 °C and is explained on the basis of simple dissociation ($\text{HX} \rightleftharpoons \text{H}^+ + \text{X}^-$), homoconjugation ($\text{HX} + \text{X}^- \rightleftharpoons \text{HX}_2^-$), and dimerization equilibria ($2\text{HX} \rightleftharpoons (\text{HX})_2$). To obtain the ionic conductivity of ascorbate ion, the conductance behavior of sodium ascorbate has been studied in all the above mixtures.

1. Introduction

Dimethyl sulfoxide (DMSO) is one of the most widely used members of the class of dipolar aprotic solvent, which is liquid over a wide temperature range (18 °C to 189 °C). It has a fairly high relative permittivity (46.68, 25 °C), a moderate viscosity (1.996 mPa s, 25 °C), and an extensive dissolving power. DMSO is a highly associated liquid, as is shown by the high values of its enthalpy and entropy of vaporization (12.64 and 23.16, respectively).

Interesting changes in the structure of DMSO + water mixtures are shown by the presence of maxima in the viscosity as well as the thermodynamic properties at a particular solvent composition (Parker, 1965). Aqueous DMSO is used in biological studies (Ashwood-Smith, 1966) and has also been proposed as a pharmaceutical or pharmacological substrate (Fox and Whittingham, 1975).

L-Ascorbic acid [3-oxo-L-gulofuranolactone (enol form)], also known as Vitamin C, is a nutritional therapeutic agent and an antiscorvy factor. The purpose of the present work was to study the conductance behavior of L-ascorbic acid in DMSO and its water mixtures and to correlate the observed behavior with the solvent properties. Also, from the algebraic sum of the limiting molar conductivities of tetrabutylammonium iodide, sodium tetraphenylboride, and sodium iodide, the value of the limiting molar conductivity of the tetrabutylammonium tetraphenylboride reference electrolyte is calculated for the determination of the limiting ionic conductivities (λ_0) of the systems studied in 80 mass % DMSO.

2. Experimental Section

2.1. Chemicals. **2.1.1. Solvents.** The purification and storage of DMSO and water have been described elsewhere (Srivastava and Jagasia, 1995). Known masses of DMSO and water were mixed in the desired proportions just prior to use.

2.1.2. Reagents. L-Ascorbic acid (S.d.fine) was recrystallized from a mixture of methanol–ethyl ether–petroleum ether, dried, and kept over anhydrous CaCl_2 in a desiccator. The purity ascertained iodometrically was >99%.

Sodium ascorbate was prepared by neutralizing ascorbic acid with sodium bicarbonate in aqueous solution at ≤ 40 °C and precipitating it from the solution with ethanol.

Purity was ascertained iodometrically and was found to be >99% pure.

Tetrabutylammonium iodide (SRL, polarographic grade) was a high-purity material and was used without any further treatment.

Sodium tetraphenylboride (SRL GR) was recrystallized thrice with pure methanol and dried in a vacuum oven at 90 °C.

Sodium iodide (SRL) was recrystallized thrice from aqueous ethanol and dried at 100–110 °C prior to use.

Sodium chloride (Merck, GR) was recrystallized thrice with an ethanol–water mixture.

Anhydrous hydrochloric acid solution in pure DMSO was prepared by bubbling through the solvent, gaseous anhydrous HCl gas, generated from H_2SO_4 and NaCl using a glass assembly. Its concentration was determined by Volhard's method.

Tetraethylammonium perchlorate was prepared by neutralizing tetraethylammonium hydroxide (Merck, GR) with perchloric acid (Merck GR). The solution was evaporated with care, and the solid obtained was recrystallized from hot water and dried in a vacuum.

2.2. Conductance Measurements. All conductances were measured at (25 ± 0.05) °C using a dip type cell with cell constant (1.080 ± 0.001) cm^{-1} with lightly platinized electrodes, as described elsewhere (Srivastava and Shankar, 1998). The reported molar conductivities have been corrected for the solvent conductivity. All conductivity measurements were repeated at least twice to get concordant results.

3. Results and Discussions

The physical properties (density ρ , relative permittivity ϵ , and viscosity η) of DMSO + water mixtures (Khoo, 1971; Le Bel and Goring, 1962) are given in Table 1. The molar conductivity Λ of L-ascorbic acid as a function of concentration C in 0, 20, 40, 60, 80, and 100 mass % DMSO at 25 °C is given in Table 2. The molar conductivity of L-ascorbic acid in DMSO + water mixtures does not vary linearly with the square root of the acid concentration. In contrast, it indicates that these systems involve considerable ion association. An acid HX in DMSO and in DMSO + water mixtures may be considered to undergo the following equilibria to account for its observed behavior.

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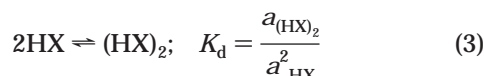
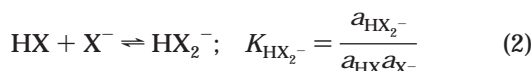
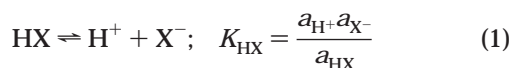
Table 1. Relative Permittivity ϵ , Density σ , and Viscosity η for DMSO + Water Mixtures at 25 °C

DMSO mass %	ϵ^a	$\sigma^b/\text{g}\cdot\text{cm}^{-3}$	$\eta^b/\text{mPa}\cdot\text{s}$
00	78.3	0.9971	0.89
20	77.9	1.0242	1.34
40	76.4	1.0535	2.22
60	73.3	1.0823	3.42
80	64.7	1.0960	3.45
100	46.4	1.0956	1.99

^a Khoo, 1971. ^b Le Bel and Goring, 1962.

Table 2. Molar Conductance Λ at Various Concentrations c of L-Ascorbic Acid in DMSO + Water Mixtures at 25 °C

$10^4 c/\text{mol}\cdot\text{dm}^{-3}$	$\Lambda/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$	$10^4 c/\text{mol}\cdot\text{dm}^{-3}$	$\Lambda/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$
0 mass % DMSO		20 mass % DMSO	
101.38	24.86	60.30	16.28
76.14	28.24	39.85	19.13
47.26	34.22	23.75	23.14
31.19	40.66	14.79	27.17
10.26	59.28	10.05	29.34
9.35	60.81	8.10	31.33
8.43	63.75	6.12	33.62
7.51	66.25	4.11	40.08
6.58	71.02	2.58	44.02
5.65	72.97	1.55	51.34
4.72	79.55		
2.84	91.76		
40 mass % DMSO		60 mass % DMSO	
80.84	5.85	125.43	1.02
64.28	6.45	97.64	1.12
45.43	7.54	81.16	1.20
29.49	9.05	62.50	1.33
17.85	11.08	41.19	1.58
11.70	12.86	29.36	1.80
9.17	14.01	16.62	2.26
7.89	14.73	9.88	2.80
5.30	17.14	8.50	2.93
1.34	27.44	6.41	3.27
0.67	32.29		
80 mass % DMSO		100 mass % DMSO	
118.24	0.16	112.64	0.06
92.54	0.18	84.89	0.07
81.09	0.18	52.23	0.10
68.86	0.20	37.94	0.14
48.88	0.24	28.30	0.17
34.33	0.29	21.61	0.21
18.67	0.40	13.22	0.32
14.57	0.45	8.90	0.43
10.38	0.54		
6.68	0.58		
6.97	0.64		
5.25	0.72		



A trial and error procedure (Srivastava and Mukherjee, 1983) is followed to determine the thermodynamic values for dissociation (K_{HX}), homoconjugation ($K_{\text{HX}_2^-}$), and dimerization (K_{d}) constants representative of an acid system.

Assuming that the activity coefficients of all uncharged species are unity and that those of the ions H^+ , X^- , and HX_2^- are equal, eqs 1–3 can be expressed as

$$K_{\text{HX}} = \frac{[\text{H}^+][\text{X}^-]f_i^2}{[\text{HX}]} \quad (4)$$

$$K_{\text{HX}_2^-} = \frac{[\text{HX}_2^-]}{[\text{HX}][\text{X}^-]} \quad (5)$$

$$K_{\text{d}} = \frac{[(\text{HX})_2]}{[\text{HX}]^2} \quad (6)$$

where f_i denotes the activity coefficient of the ions. Accordingly, the ratio $K_{\text{HX}_2^-}/K_{\text{HX}}$ can be related as follows:

$$\frac{K_{\text{HX}_2^-}}{K_{\text{HX}}} = \frac{[\text{HX}_2^-]}{[\text{H}^+][\text{X}^-]^2 f_i^2} \quad (7)$$

From the charge-neutrality rule

$$[\text{H}^+] = [\text{X}^-] + [\text{HX}_2^-] \quad (8)$$

Thus, substituting for $[\text{HX}_2^-]$ in eq 7, one gets

$$\frac{K_{\text{HX}_2^-}}{K_{\text{HX}}} = \frac{[\text{H}^+] - [\text{X}^-]}{[\text{H}^+][\text{X}^-]^2 f_i^2} \quad (9)$$

Equation 9 can now be rearranged to give

$$(K_{\text{HX}_2^-})[\text{H}^+]f_i^2[\text{X}^-]^2 + K_{\text{HX}}[\text{X}^-] - K_{\text{HX}}[\text{H}^+] = 0 \quad (10)$$

If values of K_{HX} and $K_{\text{HX}_2^-}$ are available, the equilibrium concentration of X^- can be obtained by eq 10 for a particular value of $[\text{H}^+]$, which in the present situation also determines the ionic strength I and can, therefore, be used to obtain f_i from any standard equation such as the Debye–Hückel limiting law ($-\log f_i = AZ_1^2\sqrt{I}$). Substituting the values of $[\text{X}^-]$ and f_i and those of the constants K_{HX} , $K_{\text{HX}_2^-}$, and K_{d} according to the relationships given by eqs 4–6, the total concentration C_{HX} of acid can be expressed as

$$C_{\text{HX}} = \frac{[\text{H}^+][\text{X}^-]f_i^2}{K_{\text{HX}}} + [\text{X}^-] + \frac{2K_{\text{HX}_2^-}[\text{X}^-][\text{H}^+]f_i^2}{K_{\text{HX}}} + \frac{2K_{\text{d}}[\text{H}^+]^2[\text{X}^-]^2 f_i^4}{K_{\text{HX}}^2} \quad (11)$$

For a given set of values of K_{HX} , $K_{\text{HX}_2^-}$, and K_{d} the total acid concentration C_{HX} can be calculated for an assumed value of $[\text{H}^+]$, which also yields the equilibrium concentration of the ions X^- and HX_2^- . Thus, one can obtain the molar conductance, $\Lambda_{\text{C}_{\text{HX}}}$, making use of the definition

$$\Lambda_{\text{C}_{\text{HX}}} = \frac{\sum C_i \lambda_i}{C_{\text{HX}}} \quad (12)$$

for a particular value of C_{HX} obtained in the above manner.

In the present correlation of conductance values, a trial and error procedure was adopted for L-ascorbic acid, with different sets of values for K_{HX} , $K_{\text{HX}_2^-}$, and K_{d} and the plots of $\log \Lambda_{\text{C}_{\text{HX}}}$ versus $\log C_{\text{HX}}$ were generated using the values of λ_{H^+} , λ_{X^-} , and $\lambda_{\text{HX}_2^-}$. The simulated plots were then compared with the experimental ones, using the criterion of best fit. The specific set of values of K_{HX} , $K_{\text{HX}_2^-}$, and K_{d} for best fit are considered to be representative of any particular acid system.

Table 3. Molar Conductance Λ at Various Concentrations c in DMSO + Water Mixtures at 25 °C

$10^4 c/\text{mol}\cdot\text{dm}^{-3}$	$\Lambda/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$	$10^4 c/\text{mol}\cdot\text{dm}^{-3}$	$\Lambda/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$
Sodium Ascorbate			
0 mass % DMSO		20 mass % DMSO	
62.66	64.98	134.87	43.43
44.66	68.33	85.74	45.00
25.29	72.80	30.28	47.57
10.82	77.12	19.99	48.25
8.69	77.86	14.55	48.84
6.54	79.12	11.75	49.24
2.20	82.77	8.90	49.41
		3.02	50.20
40 mass % DMSO		60 mass % DMSO	
122.24	29.07	44.89	19.62
82.28	30.18	33.21	20.20
64.77	30.77	22.38	20.80
48.64	31.31	13.20	21.36
35.05	31.76	9.91	21.70
25.15	32.26	5.09	22.26
14.50	32.98	2.94	22.51
80 mass % DMSO		100 mass % DMSO	
54.86	16.70	31.49	17.59
35.66	17.51	24.65	18.37
11.00	19.14	15.61	19.85
8.83	19.22	9.91	21.10
6.97	19.36	7.33	21.59
4.70	19.71	2.50	23.09
3.16	19.85	1.27	23.84
2.38	20.07	0.95	24.03
1.59	20.20	0.32	25.28
80 mass % DMSO			
tetrabutylammonium iodide		sodium tetraphenylboride	
16.94	17.72	16.01	14.81
12.61	18.05	11.37	15.03
9.33	18.34	8.48	15.13
7.86	18.37	5.80	15.28
6.12	18.65	4.32	15.41
4.42	18.85	2.72	15.48
2.59	19.09	1.88	15.60
1.62	19.32	1.44	15.65
		1.09	15.70
sodium iodide		sodium chloride	
13.45	23.20	117.99	17.91
11.83	23.38	91.01	18.30
10.10	23.62	70.66	18.59
8.25	23.83	51.88	18.98
6.27	24.05	30.36	19.49
4.14	24.42	18.38	19.90
3.01	24.60	12.04	20.17
1.84	24.92	9.44	20.22
1.60	24.97	8.12	20.30
Hydrochloric Acid			
80 mass % DMSO		100 mass % DMSO	
125.02	39.18	110.07	24.65
99.67	39.82	99.45	25.20
84.00	40.22	67.07	27.47
73.35	40.47	56.11	28.50
57.07	40.95	49.07	29.18
46.01	41.35	45.07	29.65
40.42	41.46	33.93	31.20
34.78	41.75	22.71	33.17
29.09	42.02	11.40	35.83

For the determination of the limiting ionic conductivity of ascorbate ion (λ_{X^-}) in all the above solvent systems, of sodium and chloride ions in 80 mass % DMSO, and of hydrogen ions (λ_{H^+}) in 80 and 100 mass % DMSO, further studies were initiated as follows.

The conductivity results for sodium ascorbate, tetrabutylammonium iodide, sodium tetraphenylboride, sodium iodide, sodium chloride, and hydrochloric acid were analyzed by the Fuoss conductance–concentration equation (Fuoss, 1978). Table 3 gives the variation of the molar conductance Λ as a function of the concentration c for the above systems. The computer program SCAN ON RHO was

Table 4. Derived Conductance Parameters for Sodium Ascorbate, at 25 °C in DMSO + Water Mixtures

DMSO mass %	$\Lambda_0 \pm \Delta\Lambda_0/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$	K_A	$R/\text{\AA}$	$\sigma/\%$	$\Lambda_0\eta$
Sodium Ascorbate					
00	84.26 ± 0.38	185.27	29.4	0.57	66.74
20	50.74 ± 0.05	072.48	23.6	0.16	67.99
40	34.01 ± 0.07	033.26	14.4	0.21	75.50
60	22.94 ± 0.03	148.68	30.8	0.20	78.45
80	20.44 ± 0.03	140.11	26.2	0.32	70.51
100	24.41 ± 0.07	247.81	24.0	0.42	48.57
Tetrabutylammonium Iodide					
80	19.69 ± 0.03	377.05	43.7	0.20	67.93
Sodium Tetraphenylboride					
80	15.84 ± 0.01	376.56	46.1	0.14	54.64
Sodium Iodide					
80	25.43 ± 0.02	337.74	41.6	0.12	87.73
Sodium Chloride					
80	20.83 ± 0.01	61.30	20.90	0.10	71.86
Hydrochloric Acid					
80	43.70 ± 0.04	20.95	12.0	0.09	150.76
100	40.69 ± 0.05	157.02	14.4	0.07	80.97

Table 5. Limiting Ionic Conductivity of Ions in DMSO + Water Mixtures at 25 °C

DMSO mass %	$\lambda_{\pm}^{\circ}/\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1}$			
	$\text{H}^+{}^a$	$\text{Na}^+{}^a$	ascorbate ⁻	$\text{Cl}^-{}^a$
00	349.90	50.11	34.15	76.4
20	238.80	35.30	15.44	49.2
40	145.50	24.00	10.01	29.5
60	73.50	14.60	8.34	18.0
80	34.01 ^b	11.14 ^b	9.66	9.69 ^b
100	16.29 ^b	13.80 ^c	10.61	24.4 ^d

^a Feakins and Shaughnessy, 1978. ^b Present work. ^c Sears et al., 1956. ^d Conway and Bockris, 1972.

used, as described elsewhere (Srivastava and Shankar, 1998), to determine the best fit conductivity parameters such as limiting molar conductivity (Λ_0), association constant (K_A), and cosphere diameter (R) for these systems, as given in Table 4.

Using the values of limiting molar conductivity thus obtained and considering that the limiting molar conductivity is an algebraic sum of the ionic conductivities of all the ions present in the molecule, we calculated the value of the limiting molar conductivity for tetrabutylammonium tetraphenylboride by the following relation:

$$\Lambda_0(\text{Bu}_4\text{NBPh}_4) = \Lambda_0(\text{Bu}_4\text{NI}) + \Lambda_0(\text{NaBPh}_4) - \Lambda_0(\text{NaI}) \quad (13)$$

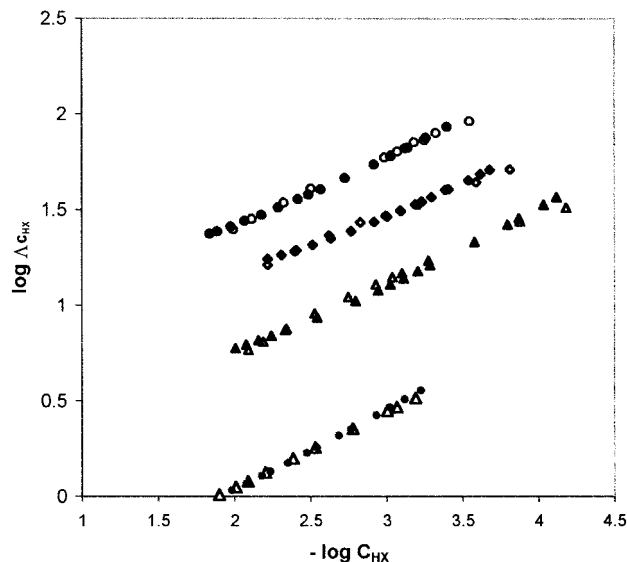
Tetrabutylammonium tetraphenylboride was used as a reference electrolyte (Coetzee and McGuire, 1963; Krumgalz, 1983) for the determination of the limiting ionic conductivities. According to the assumption (James and Fuoss, 1975), the limiting transference number of the Bu_4N^+ ion in Bu_4NBPh_4 is 0.519 and independent of the kind of solvent used, so the limiting ionic conductivity was calculated on the basis of the following relation:

$$\lambda^{\circ}(\text{Bu}_4\text{N}^+) = 0.519\Lambda_0(\text{Bu}_4\text{NBPh}_4) \quad (14)$$

The value of the ionic conductivity of Bu_4N^+ thus obtained has been used to calculate single-ion limiting molar conductivities for various ions mentioned in Table 5. By subtracting $\lambda^{\circ}(\text{Bu}_4\text{N}^+)$ from $\Lambda_0(\text{Bu}_4\text{NI})$, the $\lambda^{\circ}(\text{I}^-)$ value was determined, which in turn was used to determine the ionic conductivities $\lambda^{\circ}(\text{Na}^+)$, $\lambda^{\circ}(\text{Cl}^-)$, and $\lambda^{\circ}(\text{H}^+)$ on

Table 6. K_{HX} , $K_{HX_2^-}$, and K_d Values for L-Ascorbic Acid in DMSO + Water Mixtures at 25 °C

DMSO mass %	$10^{-5}K_{HX}$	$K_{HX_2^-}$	K_d
0	0.90	5	0
20	0.93	10	0
40	1.38	6	0
60	1.60	2	0
80	1.32	1	0
100	6.07	1	14

**Figure 1.** Plot of $\log \Lambda_{C_{HX}}$ versus $-\log C_{HX}$ for L-ascorbic acid in 0 wt % DMSO: [(○) experimental; (●) calculated], 20 wt % DMSO [(◇) experimental; (◆) calculated], 40 wt % DMSO (△) experimental; (▲) calculated, and 60 wt % DMSO [(△) experimental; (●) calculated].

similar lines from the molecular conductivities of their salts. The $\lambda(HX_2^-)$ value is taken as half of the $\lambda^{\circ}(X^-)$ value in the respective DMSO + water mixtures. [The mobility of the HX_2^- species is reduced by half (Harned and Owen, 1958) on association of X^- and HX species.] The results of the conductance measurements of L-ascorbic acid were calculated using the Debye–Hückel constant A equal to 0.5119, 0.5158, 0.5311, 0.5651, 0.6815, and 1.1221 for 0, 20, 40, 60, 80, and 100 mass % DMSO, respectively. Table 6 gives the calculated values of K_{HX} , $K_{HX_2^-}$, and K_d obtained from the present investigation. Figures 1 and 2 show the plots of $\log \Lambda_{C_{HX}}$ versus $\log C_{HX}$ along with the calculated plots yielding the best fit on the basis of preceding treatments.

The molar conductance values are found to decrease with increasing mass % of DMSO. The addition of DMSO to water results in a dramatic decrease in the molar conductance, due to the breakdown of the three-dimensional structure characteristic of pure water and consequent impairment of the proton-jump mechanism for the transport of hydrogen ion.

It is interesting to note that the value of the homoconjugation constant, which reflects the individual tendencies for intermolecular hydrogen bonding, is found to initially increase with the addition of DMSO to water up to 20 mass % and further steadily decrease as DMSO is added progressively. The dimerization constant is uniquely observed to be zero in the entire series, except in pure DMSO, where some dimerization is observed.

The K_a values of L-ascorbic acid are found to initially decrease from 0 to 60 mass % DMSO, with an increase at 80 mass % which further decreases at 100 mass % DMSO.

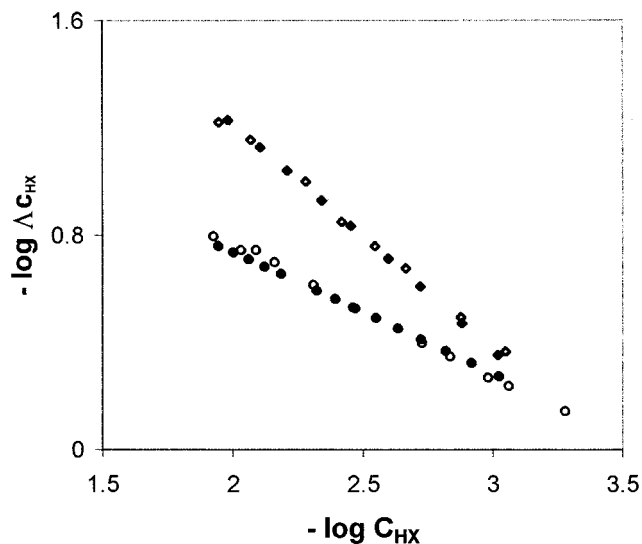
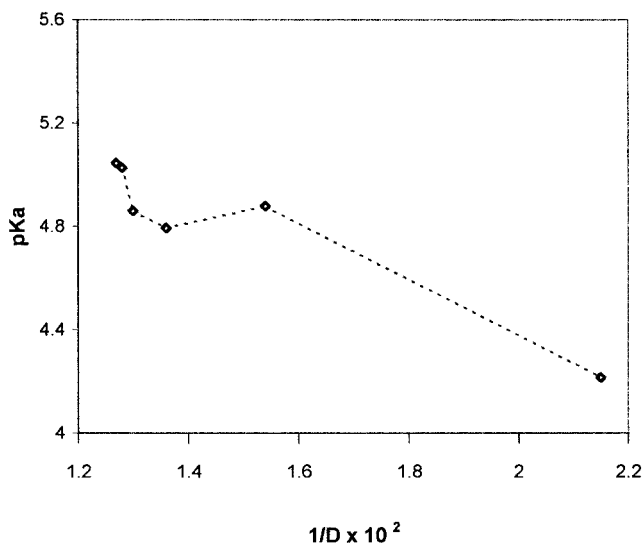
**Figure 2.** Plot of $-\log \Lambda_{C_{HX}}$ versus $-\log C_{HX}$ for L-ascorbic acid in 80 wt % DMSO [(◇) experimental; (◆) calculated] and pure DMSO [(○) experimental; (●) calculated].**Figure 3.** Plot of pK_a versus the reciprocal of the dielectric constant for L-ascorbic acid.

Figure 3 records the plot of the pK_a values of L-ascorbic acid as a function of the reciprocal of the dielectric constant of 0 to 100 mass % DMSO + water mixtures. No linear variation was observed. The figure reveals that, initially from 0 to 20 mass % DMSO, the pK_a value remains almost constant with slight decrease at 40 mass % to remain almost constant up to 60 mass %. Further, an increase in the pK_a value at 80 mass % is observed, which is expected, since, with the viscosity being maximum at 80 mass % DMSO, dissociation in the molecule will be less. An abrupt decrease in the pK_a value from 80 mass % to pure DMSO could be well attributed to the abrupt decrease in the dielectric constant of the medium from 80 to 100 mass % DMSO.

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