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Composition, Density, Viscosity, Electrical Conductivity, and Refractive Index of Saturated Solutions of Lithium Formate + Water + Ethanol

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The composition, density, viscosity, electrical conductivity, and refractive index of saturated solutions of lithium formate in aqueous solutions of ethanol have been determined over the temperature range 283.15−313.15 K and in the mass fraction of ethanol range 0−0.8. Equations are given for these properties as a function of temperature and the mass fraction of ethanol.

Introduction

The addition of organic solvents such as alcohols to an aqueous salt solution usually decreases the salt solubility, so selective precipitation may occur by a drowning-out process. Miscibility with the solvent of the original solution, limited solubility of the solute in the second solvent, and economical separability from water are some required properties of the cosolvent (Mullin, 1993). The process may offer certain advantages over other modes of crystallization (Söhnel and Garside, 1992) and attracts increasing attention of technologists in chemical (Mydlarz and Jones, 1989; Fleischmann and Mersmann, 1984) and pharmaceutical industries (Fernandez Lozano, 1976; Mahajan *et al.*, 1991).

Among the crystalline inorganic formates, lithium formate monohydrate exhibits interesting piezoelectric properties (Chumakov and Koptsik, 1959; Aleksandrovski *et al.*, 1974) and is a potentially useful nonlinear material for optical device applications (Singh *et al.*, 1970; Baldini *et al.*, 1975). Some properties of this material, such us the crystalline structure (Krishnan and Ramanujam, 1971; Mohana Rao and Viswamitra, 1971), the infrared and Raman spectra (Agarwal *et al.*, 1983; Vierne and Cadene, 1973), the elastic constants (Zaitseva *et al.*, 1983), and the thermal phase transformations (Masuda *et al.*, 1990; Müller *et al.*, 1992), can be found in the literature. This work considers the possibility of purification by drowning-out with ethanol.

We report in this paper the solubility, density, viscosity, electrical conductivity, and refractive index for saturated solutions of lithium formate in water and aqueous ethanol mixtures at different temperatures. All of these properties are useful in studying the crystallization process.

Experimental Section

Lithium formate monohydrate was prepared by neutralization of Panreac PRS grade 85% formic acid with Panreac (99.5+%) lithium carbonate to a weakly acidic reaction according to the equation

$$2HCOOH + Li_2CO_3 + 2H_2O \rightarrow 2\ HCOOLi{\cdot}H_2O + CO_2$$

The starting solutions were filtered through a 0.22 μm Millipore filter and then were evaporated slowly until long needle-like crystals were deposited. The crystals were filtered off and dried in an oven at about 423 K in order to obtain anhydrous lithium formate. The water of hydration is lost at 367.15 K, and the melting point, 544.15 K, compares well with the value of 545.15 K found in the literature (Müller *et al.*, 1992). The anhydrous salt, without further purification, was used to prepare the saturated solutions.

The saturation of aqueous and ternary solutions was achieved by stirring for more than 48 h, in closed 100 mL glass vessels, known masses of ethanol, water, and anhydrous lithium formate, the latter being in excess to ensure the saturation of the solutions. Distilled water passed through a Millipore ultrapure cartridge kit and PRS grade absolute ethanol (Panreac, 99.5+%) stored over 3A molecular sieves were used. All flasks, fitted with a magnetic stirrer, were immersed in a thermostated water bath controlled to ±0.05 K. The equilibrium temperature was measured by a digital thermometer (Yokogawa 7563) with a precision of ±0.01 K. At the end of each run, the solutions were allowed to stand for some time, at constant temperature, until clear. The clear liquor was collected from the flasks by means of a Masterflex peristaltic pump and then filtered through a 0.22 μm filter fitted to the pump and discharged directly into the different apparatus to measure the aforementioned properties. Possible solvent evaporation was eliminated by this procedure. All sampling equipment was adequately thermostated in order to avoid salt precipitation when transporting the solutions.

Solution concentrations were determined by evaporating a known mass of saturated solution (between 15 and 30 g) to the anhydrous salt, at 423 K. All masses were measured on an analytical balance with a precision of $\pm 10^{-4}$ g. A constant mass of anhydrous salt was considered only when successive measurements of a sample differed by less than 10^{-3} g. On the basis of repeated determinations, the reproducibility of the gravimetric method was $\pm 10^{-4}$ g of lithium formate/g of solution.

The temperature of the following measurements was regulated through a cascade bath apparatus with a stability within ±0.02 K.

Densities were measured with an Anton Paar DMA 602 vibrating tube densimeter. Ultrapure degassed water and air were used as references for calibration. Three runs were made for each sample, the reproducibility being better than $\pm 10^{-2}$ kg·m^{-3}. The accuracy was $\pm 2 \times 10^{-2}$ kg·m^{-3}, estimated from comparison of NaCl solutions using the density data of Lo Surdo *et al.* (1982).

Viscosities were measured with a Haake B/BH falling-ball viscometer, using two different sphere diameters: one (A) for solutions of viscosity <3.3 mPa·s and the other (B) for those of higher viscosities. Typical time scales were 170−330 s for measurements with ball A and 45−130 s

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Table 1. Solubility s (x_1), Density ρ, Viscosity η, Electrical Conductivity κ, and Refractive Index n(D,T) of Lithium Formate Saturated Solutions for Various Mass Fractions w in (1 − w) Water + w Ethanol at 283.15, 293.15, 298.15, 303.15, and 313.15 K

w	s/(kg/100 kg of soln)	x_1	ρ/(kg·m^{-3})	$10^3\eta$/(Pa·s)	κ/(S·m^{-1})	n(D,T)
			T = 283.15 K			
0.0000	26.73	0.1123	1135.88	6.86	3.55	1.371 36
0.1011	23.09	0.0999	1105.77	8.10	2.55	1.373 20
0.2012	20.12	0.0903	1076.58	9.04	1.897	1.374 91
0.3043	16.37	0.0769	1040.40	9.10	1.415	1.375 66
0.4047	13.62	0.0676	1006.20	8.48	1.113	1.376 44
0.5172	10.63	0.0568	967.28	7.25	0.826	1.376 04
0.6119	8.00	0.0459	934.24	5.97	0.619	1.374 96
0.7163	5.15	0.0323	898.95	4.55	0.415	1.374 01
0.8135	3.20	0.0222	865.11	3.36	0.280	1.373 19
			T = 293.15 K			
0.0000	28.34	0.1206	1142.44	5.83	4.36	1.372 98
0.1029	25.15	0.1106	1110.64	6.80	3.24	1.374 12
0.2176	21.09	0.0965	1074.84	7.22	2.45	1.375 02
0.3052	18.36	0.0874	1043.26	7.09	1.968	1.375 37
0.4053	15.21	0.0763	1006.94	6.49	1.555	1.374 97
0.5076	11.95	0.0638	969.61	5.60	1.199	1.373 89
0.6070	8.94	0.0512	931.87	4.56	0.904	1.372 52
0.7160	5.97	0.0376	892.20	3.58	0.614	1.370 72
0.8129	3.54	0.0246	858.68	2.63	0.395	1.368 60
			T = 298.15 K			
0.0000	29.17	0.1250	1146.70	5.53	4.83	1.373 74
0.1000	25.87	0.1141	1115.03	6.31	3.75	1.374 70
0.2024	22.74	0.1043	1082.38	6.55	2.89	1.375 20
0.3028	19.40	0.0928	1046.60	6.45	2.28	1.375 48
0.4060	16.22	0.0819	1009.15	5.92	1.856	1.374 61
0.5122	12.51	0.0672	967.42	4.98	1.401	1.373 42
0.6090	9.70	0.0559	931.37	4.12	1.059	1.371 71
0.7170	6.27	0.0395	890.22	3.18	0.707	1.369 59
0.8164	3.81	0.0266	856.89	2.39	0.460	1.367 29
			T = 303.15 K			
0.0000	30.36	0.1313	1150.10	5.26	5.25	1.374 63
0.1040	26.99	0.1204	1117.95	5.90	4.03	1.375 29
0.2130	23.36	0.1083	1081.61	6.08	3.14	1.375 71
0.3220	20.04	0.0976	1043.28	5.84	2.46	1.375 48
0.4410	15.87	0.0821	996.41	5.02	1.901	1.373 82
0.5520	12.32	0.0684	954.51	4.21	1.421	1.372 37
0.6380	9.41	0.0556	920.23	3.43	1.102	1.370 11
0.7470	5.81	0.0378	876.57	2.60	0.708	1.367 27
0.8560	3.12	0.0228	839.02	1.90	0.416	1.364 72
			T = 313.15 K			
0.0000	32.72	0.1443	1156.67	4.61	6.20	1.376 26
0.1000	29.70	0.1349	1127.35	5.18	4.89	1.377 01
0.2100	26.07	0.1230	1090.05	5.29	3.79	1.376 60
0.3100	22.66	0.1113	1052.75	5.00	3.16	1.375 61
0.4270	18.51	0.0962	1005.90	4.30	2.48	1.373 42
0.5180	14.79	0.0808	965.88	3.63	2.01	1.371 03
0.6170	11.16	0.0652	925.06	2.92	1.491	1.368 47
0.7160	7.65	0.0485	884.75	2.27	1.050	1.365 66
0.8190	4.54	0.0318	844.88	1.69	0.636	1.362 49

when using the second sphere. Ultrapure degassed water and aqueous solutions of recrystallized sucrose were used for calibration depending on the operation range of each sphere. The viscosities and densities of water, in the temperature range 278.15−298.15 K, were taken from Riddick *et al.* (1986) and Marsh (1987), respectively. The viscosities and densities at 293.15 K of several aqueous solutions of sucrose of concentrations within the range 20−50% were taken from Swindells *et al.* (1958) and Spieweck and Emmerich (1994), respectively. The exact compositions of the sucrose + water samples were checked by comparing the experimental density measurements with those found by Spieweck and Emmerich (1994). All solutions were maintained at the desired temperature by circulating water, from the constant-temperature water bath, through the outer jacket of the viscosimeter. The flow times were determined with an estimated precision of ±0.1 s using a digital stopwatch. The results given in Table 1 are the average of 10 determinations for each solution, measurements of flow time being reproducible within ±0.1%. The reproducibility of the measured viscosities was better than ±0.5%. On the basis of comparison with reliable data from the literature (Cartón *et al.*, 1995), the accuracy of the viscosity was better than ±1%.

Electrical conductivities were measured with a Crison 522 conductimeter provided with a conductivity cell of platinum electrodes. Conductivity measurements were made after completing the viscosity measurements, by introducing the conductivity cell into the central tube of the viscometer. The electrical conductivities at 298.15 K of aqueous KCl solutions in the composition range 0.01−1.0 M (Diaz Peña and Roig, 1976) were used for calibration of the cell. In order to verify the accuracy of the conductivity measurements, measurements were made on several aqueous KBr solutions in the temperature range 293.15−318.15 K (Isono, 1984). The resulting agreement was better than ±0.5%. At least five measurements of each sample were made. The resulting mean value of the reproducibility was ±0.4%.

Table 2. Coefficients of Eqs 1−5

a_{i0}	a_{i1}	a_{i2}	a_{i3}	a_{i4}	b_{i0}	b_{i1}	b_{i2}
44.97×10^{-2}	31.42×10^{-1}	-19.89×10^{-1}	-28.23×10^{-1}		-74.81×10	-12.83×10^{2}	10.01×10^{2}
92.12×10	-65.03	17.19×10	13.47×10		75.41×10^{-2}	-70.18×10^{-2}	-12.81×10^{-1}
-88.93×10^{-1}	-77.51×10^{-1}	27.25×10^{-1}	12.59×10^{-1}		11.03×10^{2}	28.43×10^{2}	-21.52×10^{2}
-21.28	15.73	21.62	-26.42	11.64	87.62×10^{-3}	-98.04×10^{-3}	72.10×10^{-4}
132.15×10^{-2}	189.98×10^{-3}	-976.25×10^{-5}			175.92×10^{-6}	-596.81×10^{-6}	-505.13×10^{-7}

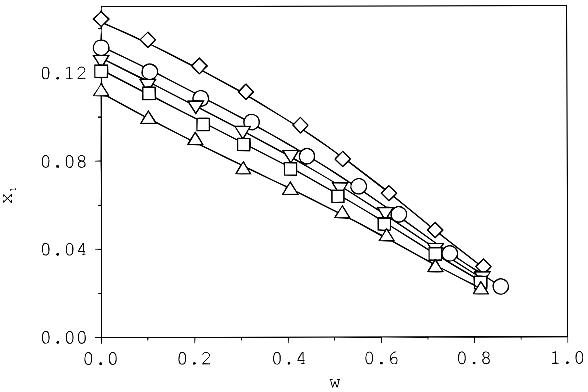

Figure 1. Solubility of lithium formate in w ethanol + $(1 - w)$ water at different temperatures: △, $T = 283.15$ K; □, $T = 293.15$ K; ▽, $T = 298.15$; ○, $T = 303.15$ K; ◇, $T = 313.15$ K.

Refractive indices for the sodium-D line were measured on an Abbe 60/ED refractometer with a resolution of ±0.000 01 unit. The glass test piece supplied by the manufacturer was used for calibration. The refractive index measurements were repeated at least three times without appreciable variation. The average of these readings was used to calculate the refractive index. The accuracy established by determining the refractive index of pure water at the temperatures used in this work (Riddick *et al.*, 1986) was ±0.003%.

Results

The solubility, density, viscosity, electrical conductivity, and refractive index of the saturated solutions for lithium formate + water + ethanol determined in the range 283.15−313.15 K are given in Table 1, and plotted, respectively, in Figures 1, 2, 4, 5 and 6.

The solubility results, expressed as the mole fraction of lithium formate x_1, may be correlated, with both ethanol composition and temperature, according to the equation

$$\ln x_1 = A_0(w) + B_0(w)/(T/\mathrm{K}) \qquad (1)$$

with

$$A_0(w) = a_{00} + a_{01}w + a_{02}w^2 + a_{03}w^3$$

$$B_0(w) = b_{00} + b_{01}w + b_{02}w^2$$

The a_{0i} and b_{0i} coefficient values are presented in Table 2. The coefficients of the empirical equation of two independent variables have been exclusively determined in order to minimize the mean quadratic deviation of the fitting to the experimental values. The mean relative standard deviation between all experimental and calculated solubility values is 1.2%. The maximum relative deviation is 2.7%. Solid lines in Figure 1 show the fitting resulting from eq 1. At each temperature, there is a reduction of the solubility of lithium formate by the addition of ethanol. Table 3 shows a comparison of the solubility results with those taken from the literature (Lin *et al.*, 1989; Shkovskaya *et al.*, 1977; Portnova and Itkina, 1979). In the narrow range of possible comparison, the agreement is within ±2.5%.

Table 3. Comparison of Solubility *s*/(kg/100 kg of soln) Results with Literature Values

298.15 K			303.15 K			313.15 K		
w	exptl	lit.	*w*	exptl	lit.[a]	*w*	exptl	lit.[a]
0.0000	29.17	28.13[a] 30.54[b] 27.99[c]	0.0000	30.36	29.67	0.0000	32.72	32.80
0.1000	25.87	25.10[a]	0.1040	26.99	26.44	0.1000	29.70	29.59
0.2024	22.74	21.82[a]	0.2130	23.36	22.86	0.2100	26.07	25.83
0.3028	19.40	18.45[a]	0.3220	20.04	19.08	0.3100	22.66	22.21

[a] Lin *et al.* (1989). [b] Shkovskaya *et al.* (1977). [c] Portnova and Itkina (1979).

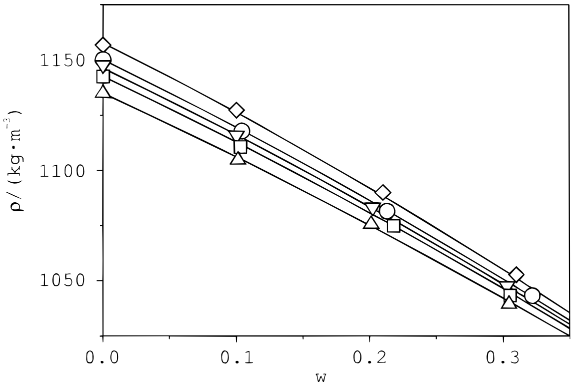

Figure 2. Density of lithium formate in w ethanol + $(1 - w)$ water at the same temperatures used in Figure 1.

The density results may be correlated, with both ethanol composition and temperature, according to the equation

$$\varrho/(\mathrm{kg{\cdot}m^{-3}}) = A_1(w) + B_1(w)(T/\mathrm{K}) \qquad (2)$$

with

$$A_1(w) = a_{10} + a_{11}w + a_{12}w^2 + a_{13}w^3$$

$$B_1(w) = b_{10} + b_{11}w + b_{12}w^2$$

The a_{1i} and b_{1i} coefficient values are presented in Table 2. The mean relative standard deviation between all experimental and calculated density values is 0.09%. The maximum relative deviation is about 0.2%. For the purpose of achieving a better visualization, the density results from $w = 0$ to $w = 0.3$ are represented in Figure 2. Solid lines in this figure show the fitting resulting from eq 2. For the aqueous systems at different temperatures, the densities are compared with those from the literature (Karniewicz *et al.*, 1982) in Figure 3. Agreement is within ±0.2%.

The viscosity results may be correlated, with both ethanol composition and temperature, according to the equation

$$\ln \eta/(\mathrm{Pa{\cdot}s}) = A_2(w) + B_2(w)/(T/\mathrm{K}) \qquad (3)$$

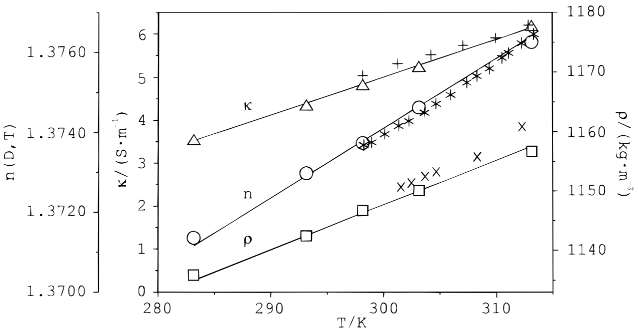

Figure 3. Density *ρ*, electrical conductivity *κ*, and refractive index *n*(D,T) of lithium formate in aqueous solutions as a function of temperature: □, △, and ○, this study; ×, +, and ∗, Karniewicz (1982).

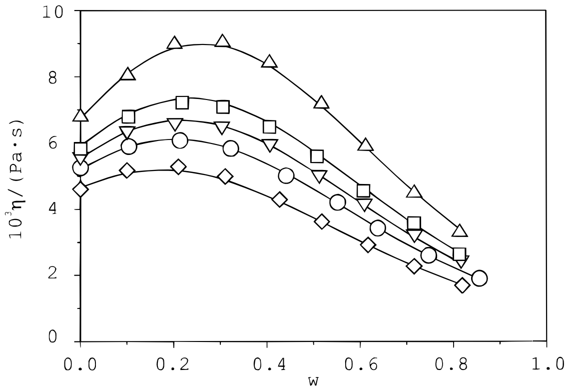

Figure 4. Viscosity of lithium formate in *w* ethanol + (1 − *w*) water at the same temperatures used in Figure 1.

with

$$A_2(w) = a_{20} + a_{21}w + a_{22}w^2 + a_{23}w^3$$

$$B_2(w) = b_{20} + b_{21}w + b_{22}w^2$$

The a_{2i} and b_{2i} coefficient values are presented in Table 2. The mean relative standard deviation between all experimental and calculated viscosity values is 1.1%. The maximum relative deviation is about 2.3%. Solid lines in Figure 4 show the fitting resulting from eq 3.

The electrical conductivity results may be correlated, with both ethanol composition and temperature, according to the equation

$$\kappa/(\mathrm{S{\cdot}m^{-1}}) = A_3(w) + B_3(w)(T/\mathrm{K}) \qquad (4)$$

with

$$A_3(w) = a_{30} + a_{31}w + a_{32}w^2 + a_{33}w^3 + a_{34}w^4$$

$$B_3(w) = b_{30} + b_{31}w + b_{32}w^2$$

The a_{3i} and b_{3i} coefficient values are presented in Table 2. The mean relative standard deviation between all experimental and calculated electrical conductivity values is 1.2%. The maximum relative deviation is 2.9%. Solid lines in Figure 5 show the fitting resulting from eq 4. For the aqueous systems, a comparison between the conductivity results and those found in the literature (Karniewicz *et al.*, 1982) is also shown in Figure 3. Agreement is within ±2.4%.

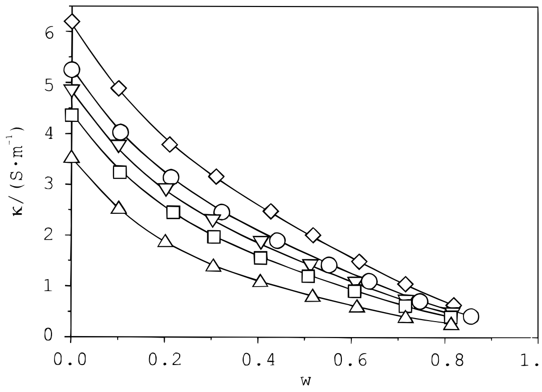

Figure 5. Electrical conductivity of lithium formate in *w* ethanol + (1 − *w*) water at the same temperatures used in Figure 1.

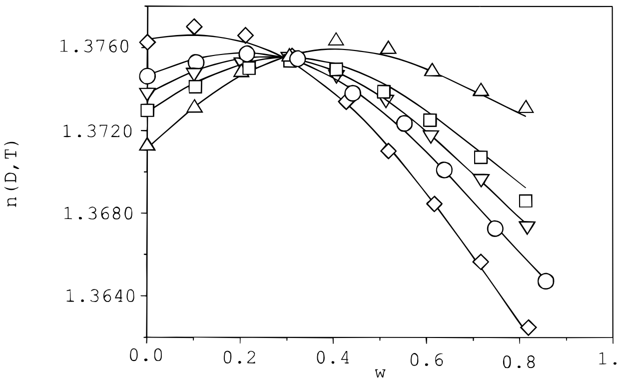

Figure 6. Refractive index of lithium formate in *w* ethanol + (1 − *w*) water at the same temperatures used in Figure 1.

The refractive index results may be correlated, with both ethanol composition and temperature, according to the equation

$$n(D,T) = A_4(w) + B_4(w)(T/\mathrm{K}) \qquad (5)$$

with

$$A_4(w) = a_{40} + a_{41}w + a_{42}w^2$$

$$B_4(w) = b_{40} + b_{41}w + b_{42}w^2$$

The a_{4i} and b_{4i} coefficient values are presented in Table 2. The mean relative standard deviation between all experimental and calculated refractive index values is 0.02%. The maximum relative deviation is 0.05%. Solid lines in Figure 6 show the fitting resulting from eq 5. For the aqueous systems at different temperatures, Figure 3 shows a comparison of the refractive indices with those found in the literature (Karniewicz *et al.*, 1982). Agreement is within ±0.010%.

Registry Numbers Supplied by the Author. $LiCH_2O_2{\cdot}H_2O$, 6108-23-2; $LiCH_2O_2$, 556-63-8; ethanol, 64-17-5.

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