J. Chem. Eng. Data **1996,** *41,* 1397−1400 **1397**

Excess Enthalpies and Excess Volumes of the Liquid Binary Mixtures of Propylene Carbonate + Six Alkanols at 298.15 K

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The excess molar enthalpies and the excess molar volumes for binary mixtures of propylene carbonate with methanol, ethanol, 1- and 2-propanol, and 1- and 2-butanol have been determined as a function of composition at 298.15 K and atmospheric pressure, using an LKB flow microcalorimeter and an Anton Paar density meter. The excess enthalpies are positive for all mixtures, while excess volumes are negative for methanol and ethanol, positive for 1- and 2-butanol and show a change of sign, as a function of composition, for 1- and 2-propanol. Both excess properties increase in magnitude as the alkanol chain length increases. Results were correlated by means of the Redlich−Kister equation and a qualitative molecular interpretation is presented.

Introduction

Alkylene carbonates are widely used solvents in extraction in the chemical industry. Particularly, propylene carbonate (4-methyl-1,3-dioxolan-2-one) is well-known for a variety of applications in high-energy batteries. Few binary mixtures containing nonaqueous solvents of practical importance have been investigated. Our recent studies have been concerned with the measurement, analysis, and interpretation of the excess thermodynamic properties of binary mixtures containing esters of carbonic acid and a second component with different functional groups (Comelli and Francesconi, 1995; Francesconi and Comelli, 1995a,b).

We report in this paper the excess molar enthalpies, H_m^E, and the excess molar volumes, V_m^E, of binary mixtures containing propylene carbonate + six alkanols at 298.15 K and atmospheric pressure. The alkanols considered are methanol, ethanol, 1- and 2-propanol, and 1- and 2-butanol. Mixtures containing alkanols with longer chain lengths could not be studied owing to the partial miscibility of propylene carbonate in these solvents. Other authors (Hong et al., 1982) have determined densities at 298.15 K of propylene carbonate + ethanol using standard pycnometers, with the purpose to study the possibility of recovering ethanol from aqueous solutions.

We have repeated these measurements with a density meter, and our values show differences, with respect to the previous literature, of ±0.2% in the central range of mole fraction.

To our knownledge, no other literature values are available for these mixtures.

Experimental Section

Chemicals. Table 1 reports origins and stated purities, while Table 2 shows measured densities, ρ, and refractive indices, n_D, at 298.15 K in comparison with literature values.

Table 1. Origins and Stated Purities of the Pure Components

component	origin	stated purity/ mol %
propylene carbonate	Aldrich Chemical Co.	99.7 (HPLC)
methanol	Aldrich Chemical Co.	99.8 (HPLC)
ethanol	Fluka Chemie AG, CH	99.8 (GC)
1-propanol	Aldrich Chemical Co.	99.5 (ACS)
2-propanol	E. Merck AB, D	99.7 (PA)
1-butanol	Aldrich Chemical Co.	99.8 (HPLC)
2-butanol	E. Merck AB, D	99.5 (PA)

Table 2. Comparison of Densities, ρ, and Refractive Indices, n_D, of Pure Components with Literature Values at 298.15 K

	ρ/g·cm^{-3}		n_D	
component	exptl	lit.	exptl	lit.
propylene carbonate	1.199 57	1.199 5[a] 1.199 2[b]	1.4195	1.419 4[a] 1.419 9[c]
methanol	0.786 55	0.786 37[c] 0.786 6[d]	1.3264	1.326 52[c]
ethanol	0.785 24	0.784 93[c] 0.785 3[e] 0.785 4[f]	1.3593	1.359 41[c]
1-propanol	0.799 61	0.799 60[c] 0.799 65[f]	1.3838	1.383 70[c]
2-propanol	0.780 96	0.781 26[c] 0.781 02[g]	1.3751	1.375 2[c]
1-butanol	0.805 77	0.805 75[c] 0.805 85[h]	1.3975	1.397 41[c]
2-butanol	0.802 49	0.802 41[c] 0.802 40[h]	1.3954	1.395 30[c]

[a] Moumouzias et al., 1991. [b] Wilhelm et al., 1991. [c] Riddick et al., 1986. [d] Martin and Mato, 1995. [e] El-Banna and Ramadan, 1995. [f] Hiaki et al., 1995. [g] Singh et al., 1994. [h] Artigas et al., 1995.

Chemicals were used as received, kept in dark bottles, in an inert argon atmosphere, and dried over molecular sieves (Union Carbide type 4A, 1/16 in. pellets) with the exception of ethanol, originally supplied with sieves. No further purification was attempted owing to their high-purity grade which was checked by gas chromatography. Before measurements, all chemicals were partially degassed using an ultrasonic bath (Hellma, type 460, Milan, Italy).

Calorimetric Measurements. Excess molar enthalpies, H_m^E, were determined using an LKB flow microcalorimeter (LKB Produkter, model 2107, Bromma, Sweden): details of the equipment and its operating procedure have been described previously (Monk and Wadso, 1968;

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S0021-9568(96)00186-0 CCC: $12.00

Table 3. Excess Molar Enthalpies, H_m^E, of Propylene Carbonate (1) + Alkanols (2) at 298.15 K

x_1	H_m^E/J·mol^{-1}	x_1	H_m^E/J·mol^{-1}
Propylene Carbonate (1) + Methanol (2)			
0.0196	155	0.4891	1365
0.0384	288	0.5895	1237
0.0739	486	0.6569	1067
0.1376	784	0.7415	751
0.1931	972	0.7929	527
0.2419	1107	0.8517	272
0.3237	1264	0.9199	29
0.4179	1360	0.9387	3
Propylene Carbonate (1) + Ethanol (2)			
0.0279	286	0.5084	1882
0.0543	521	0.5796	1852
0.0793	719	0.6740	1706
0.1030	881	0.7338	1555
0.1470	1140	0.8053	1273
0.1868	1310	0.8465	1062
0.2563	1531	0.8922	795
0.3148	1678	0.9430	446
0.4080	1825		
Propylene Carbonate (1) + 1-Propanol (2)			
0.0355	434	0.5698	2140
0.0685	763	0.6384	2064
0.0994	1011	0.7259	1868
0.1282	1208	0.7793	1657
0.1808	1479	0.8412	1317
0.2273	1665	0.8759	1076
0.3062	1887	0.9137	796
0.3704	2012	0.9549	439
0.4688	2120		
Propylene Carbonate (1) + 2-Propanol (2)			
0.0363	510	0.5465	2383
0.0700	888	0.5756	2364
0.1015	1172	0.6438	2279
0.1309	1396	0.7306	2019
0.1843	1674	0.7833	1769
0.2306	1881	0.8443	1384
0.3112	2129	0.8785	1119
0.3760	2235	0.9156	812
0.4747	2363	0.9559	437
Propylene Carbonate (1) + 1-Butanol (2)			
0.0431	533	0.5903	2233
0.0826	930	0.6837	2111
0.1190	1190	0.7643	1852
0.1526	1390	0.8121	1641
0.2127	1656	0.8664	1292
0.2648	1823	0.8963	1053
0.3508	2005	0.9284	784
0.4188	2112	0.9629	421
0.5194	2219		
Propylene Carbonate (1) + 2-Butanol (2)			
0.0425	668	0.6195	2373
0.0829	1127	0.6846	2257
0.1194	1440	0.7650	1963
0.1531	1642	0.8128	1700
0.2134	1932	0.8669	1313
0.2656	2088	0.8967	1055
0.3517	2255	0.9287	755
0.4197	2340	0.9630	404
0.5204	2413		

Francesconi and Comelli, 1986). Two identical automatic burets (ABU, Radiometer, Copenhagen, Denmark) were used to pump liquids into the mixing cell of the calorimeter. The apparatus was electrically calibrated before use and the calibration subsequently checked by using the standard cyclohexane + hexane test mixture (Gmehling, 1993): discrepancies between our and literature values are better than 0.5% over the central range of composition and correspond to uncertainties of H_m^E less than 1 J·mol^{-1}.

Values of mole fractions of the mixtures were determined from the calibrated flow rates and the fluid density and are estimated to be precise to 1 × 10^{-4}: two replicate measurements were made for each determination.

Table 4. Densities, ρ, and Excess Molar Volumes, V_m^E, of Propylene Carbonate + Alkanols at 298.15 K

x_1	ρ/g·cm^{-3}	V_m^E/ cm^3·mol^{-1}	x_1	ρ/g·cm^{-3}	V_m^E/ cm^3·mol^{-1}
Propylene Carbonate (1) + Methanol (1)					
0.0148	0.799 92	−0.043	0.3593	1.016 06	−0.371
0.0510	0.830 80	−0.134	0.3985	1.032 86	−0.369
0.0754	0.850 31	−0.187	0.4572	1.056 01	−0.352
0.1130	0.878 03	−0.244	0.5135	1.076 35	−0.334
0.1717	0.917 20	−0.308	0.6317	1.113 76	−0.266
0.2136	0.942 46	−0.341	0.7523	1.146 16	−0.182
0.2529	0.964 38	−0.364	0.8694	1.173 22	−0.091
0.2874	0.982 16	−0.367	0.9587	1.191 58	−0.025
0.3276	1.001 60	−0.372			
Propylene Carbonate (1) + Ethanol (2)					
0.0206	0.798 08	−0.040	0.4541	1.014 87	−0.212
0.0627	0.823 29	−0.102	0.4845	1.027 23	−0.205
0.1252	0.858 76	−0.164	0.5445	1.050 74	−0.188
0.1604	0.877 76	−0.186	0.6270	1.081 38	−0.159
0.2310	0.914 04	−0.214	0.6946	1.105 04	−0.128
0.2901	0.942 67	−0.228	0.8298	1.149 09	−0.057
0.3186	0.955 98	−0.229	0.8776	1.163 77	−0.036
0.3724	0.980 15	−0.226	0.9638	1.189 19	−0.005
0.4092	0.996 07	−0.222			
Propylene Carbonate (1) + 1-Propanol (2)					
0.0209	0.809 15	−0.007	0.5062	1.014 92	−0.034
0.0616	0.827 48	−0.019	0.5495	1.031 98	−0.030
0.1356	0.860 34	−0.034	0.6148	1.057 37	−0.022
0.2155	0.894 97	−0.042	0.6721	1.079 31	−0.015
0.2762	0.920 82	−0.045	0.7549	1.110 49	−0.004
0.3439	0.949 12	−0.044	0.8505	1.145 72	0.006
0.3805	0.964 19	−0.044	0.9304	1.174 65	0.007
0.4219	0.981 09	−0.042	0.9778	1.191 65	0.004
0.4706	1.000 71	−0.038			
Propylene Carbonate (1) + 2-Propanol (2)					
0.0424	0.800 64	−0.012	0.5152	1.007 91	−0.058
0.0666	0.811 79	−0.018	0.5679	1.029 64	−0.050
0.1555	0.852 25	−0.041	0.6080	1.045 99	−0.041
0.2087	0.876 08	−0.051	0.6792	1.074 69	−0.027
0.2656	0.901 29	−0.601	0.7571	1.105 62	−0.012
0.3474	0.936 93	−0.067	0.8605	1.146 00	0.007
0.3956	0.957 62	−0.067	0.9114	1.165 67	0.009
0.4301	0.972 25	−0.066	0.9837	1.193 36	0.003
0.4758	0.991 47	−0.062			
Propylene Carbonate (1) + 1-Buanol (2)					
0.0177	0.812 17	0.007	0.5740	1.023 49	0.068
0.1009	0.842 50	0.033	0.5941	1.031 52	0.068
0.1721	0.868 84	0.047	0.6527	1.054 98	0.066
0.2426	0.895 22	0.056	0.7175	1.081 22	0.063
0.3273	0.927 38	0.062	0.7846	1.108 76	0.057
0.3725	0.944 72	0.064	0.8798	1.148 36	0.040
0.4352	0.968 96	0.065	0.9319	1.170 39	0.026
0.4788	0.985 97	0.067	0.9907	1.195 55	0.005
0.5180	1.001 35	0.068			
Propylene Carbonate (1) + 2-Butanol (2)					
0.0586	0.823 41	0.070	0.5580	1.013 95	0.182
0.0919	0.835 42	0.103	0.6123	1.035 78	0.176
0.1614	0.860 93	0.146	0.6520	1.051 92	0.169
0.2455	0.892 39	0.174	0.7153	1.077 89	0.155
0.3222	0.921 54	0.187	0.7925	1.110 10	0.126
0.3793	0.943 53	0.191	0.8916	1.152 21	0.081
0.4405	0.967 36	0.191	0.9247	1.166 56	0.059
0.4860	0.985 38	0.189	0.9807	1.191 02	0.018
0.5158	0.997 11	0.187			

The temperature of the calorimeter was kept constant to ±0.01 K by digital instruments. A further water bath (Julabo GMBH, type 12 B, Seelbach, Germany) has been used to pump water into the external jacket of the calorimetric bath at a temperatue of 10 °C up to the experimental temperature to supply the heat necessary to reach the constancy of the temperature.

Density Measurements. Density values were determined using a vibrating tube density meter (Anton Paar, model DMA 60, Graz, Austria) equipped with a density measuring cell (type 602) with a precision on the measured period τ on the order of 1 × 10^{-6} s.

Table 5. Least-Squares Parameters, a_k, and Standard Deviations, σ, Eq 3, of Propylene Carbonate + Alkanols at 298.15 K

mixture	function	a_0	a_1	a_2	a_3	σ
propylene carbonate						
+methanol	H^E_m/J·mol^{-1}	5445.2	−1212.7	−2245.5	−3867.7	5.5
	V^E_m/cm^3·mol^{-1}	−1.3448	0.8134	−0.4277	0.4798	0.0025
+ethanol	H^E_m/J·mol^{-1}	7506.7	285.47	2201.0	−1728.3	5.4
	V^E_m/cm^3·mol^{-1}	−0.8085	0.5405	−0.1880	0.4177	0.0015
+1-propanol	H^E_m/J·mol^{-1}	8553.6	817.26	3294.3	−2496.0	4.6
	V^E_m/cm^3·mol^{-1}	−0.1485	0.1864	0.0417	0.0968	0.0002
+2-propanol	H^E_m/J·mol^{-1}	9501.2	835.44	3414.6	−3614.9	6.8
	V^E_m/cm^3·mol^{-1}	−0.2395	0.2475	0.2121		0.0006
+1-butanol	H^E_m/J·mol^{-1}	8802.1	1412.9	4314.8	−2563.0	8.6
	V^E_m/cm^3·mol^{-1}	0.2692	0.0226	0.1707		0.0005
+2-butanol	H^E_m/J·mol^{-1}	9618.7	1002.7	5015.3	−4319.0	5.6
	V^E_m/cm^3·mol^{-1}	0.7508	−0.1313	0.4116	−0.1535	0.0011

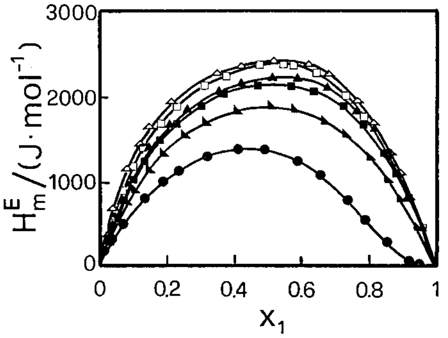

Figure 1. Excess molar enthalpies, H^E_m, at 298.15 K of propylene carbonate + methanol (●), +ethanol (◪), +1-propanol (■), +1-butanol (▲), +2-propanol (□), +2-butanol (△). Solid lines, calculated with eq 2.

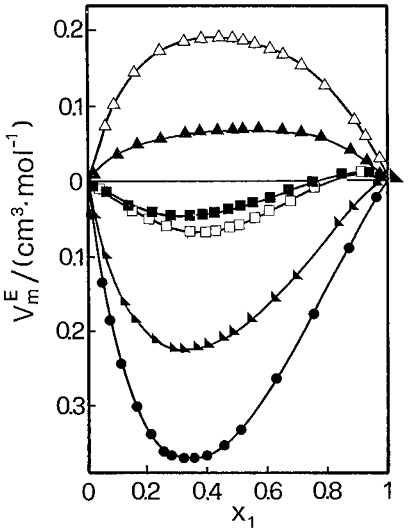

Figure 2. Excess molar volumes, V^E_m, at 298.15 K of propylene carbonate + methanol (●), +ethanol (◪), +2-propanol (□), +1-propanol (■), +1-butanol (▲), +2-butanol (△). Solid lines, calculated with eq 2.

A detailed description of the apparatus and experimental procedure is described elsewhere (Fermeglia and Lapasin, 1988).

The samples were prepared by mass using a Mettler balance (model AE 160, precision $\pm 1 \times 10^{-4}$ g), charging the heavier component first to prevent evaporation. We have also followed the same directions indicated by Fermeglia and Lapasin for determining mole fractions. The volume of the density bottles used for analysis was approximately ≈15 cm^3. In the density measurements, a digital thermometer (Anton Paar, DT-25), precision ±0.01 K, inserted into the jacket of the measuring cell was used. The accuracies in density and in excess molar volume are 5×10^{-5} g·cm^{-3} and 3×10^{-3} cm^3·mol^{-1}, respectively.

Before measurements, the apparatus was calibrated with double distilled water and dry air (Wagenbreth and Blanke, 1971; Kohlrausch, 1968) and checked with the test mixture benzene + cyclohexane (Wilhelm, 1985). Our results showed a discrepancy of less than 0.5% over the central range of the mole fraction (at $x = 0.5$ our value of V^E_m is 0.652 cm^3·mol^{-1}, lit. value 0.6514 cm^3·mol^{-1}).

The densities, ρ, of mixtures are used to calculate the excess molar volumes, V^E_m, according to

$$V^E_m/\mathrm{cm^3\cdot mol^{-1}} = (x_1M_1 + x_2M_2)/\rho - x_1M_1/\rho_1 - x_2M_2/\rho_2 \quad (1)$$

where x_i, M_i, and ρ_i are the molar fraction, molar mass, and density of component i, respectively.

Corrections for buoyancy and evaporation of the components were carried out, but only a negligible contribution to the mole fraction was detected, on the order of 1×10^{-4} at the wrong.

Results and Discussion

The experimental values of H^E_m and V^E_m are listed in Tables 3 and 4 and graphically represented in Figures 1 and 2. All experimental data were fitted by a least-squares method to the Redlich–Kister equation

$$Q^E_m = x_1x_2\sum_{k\geq 0} a_k(x_1 - x_2)^k \quad (2)$$

where $Q^E_m = H^E_m/\mathrm{J\cdot mol^{-1}}$ or $V^E_m/\mathrm{cm^3\cdot mol^{-1}}$ with all points weighted equally. a_k are the adjustable parameters, and in each case the optimum number of coefficients is ascertained from an examination of the variation in the standard deviation $\sigma(Q^E_m)$ as given by

$$\sigma(Q^E_m) = |\Phi/(N - n)|^{0.5} \quad (3)$$

with N the number of experimental points and n the number of adjustable parameters. Φ is the objective function defined as

$$\Phi = \sum_{k\geq 0}^{N} {\eta_k}^2 \quad (4)$$

where $\eta = Q^E_{m,calcd} - Q^E_m$, $Q^E_{m,calcd}$ being determined from the right hand side of eq 2. The adjustable parameters a_k and the standard deviation σ as computed from eqs 2 and 3 are presented in Table 5. Figures 3 and 4 show $H^E_m(x_1 = 0.5)$ or $V^E_m(x_1 = 0.5)$, the equimolar excess enthalpy or the equimolar excess volume plotted against the number n_c of carbon atoms of n-alkanols.

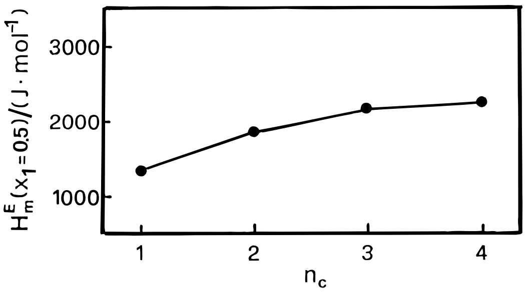

Figure 3. Values of equimolar $H^E_m(x_1 = 0.5)$ at 298.15 K as a function of the numbers of carbon atoms of alkanols, n_c, of propylene carbonate + 1-alkanols.

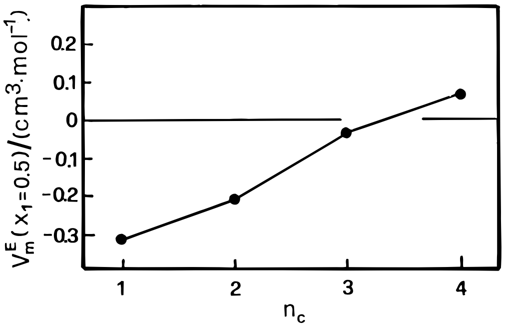

Figure 4. Values of equimolar $V^E_m(x_1 = 0.5)$ at 298.15 K as a function of the numbers of carbon atoms of alkanols of propylene carbonate + 1-alkanols.

Figure 1 shows high positive values of H^E_m for all alkanols, as a consequence of breaking of their hydrogen bonds during mixing. The increase of H^E_m with the increased chain length is qualitatively the same observed for mixtures of these alkanols with epoxybutane (Comelli and Francesconi, 1996) and cyclohexane derivatives (Letcher and Nevines, 1996).

A sign inversion of H^E_m in the propylene carbonate rich region is predicted by the fitting polynomial, eq 2, for the mixture containing methanol.

2-Propanol and 2-butanol have H^E_m larger than the ones of the corresponding 1-alkanols in accordance with the smaller observed equilibrium constant for the association of isoalkanols (Prausnitz, 1969) and the consequent smaller value of the interaction energy between dissimilar molecules, E_{12}.

Also V^E_m vs x_1 curves show values increasing with the increase of chain length. However, V^E_m for methanol and ethanol are negative and for 1-butanol are positive, whereas for 1-propanol a change of sign as a function of mole fraction is observed.

The propylene carbonate + 2-butanol has V^E_m larger than those of the system containing 1-butanol, whereas the propylene carbonate + 2-propanol has V^E_m smaller than those of the system containing 1-propanol.

The mixtures containing epoxybutane with alkanols, previously studied, showed a pattern similar as to the increase of V^E_m in passing from methanol to 1-butanol, though only the V^E_m's for methanol are negative and those for 1-propanol are always positive, with no sign inversion.

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Received for review May 29, 1996. Accepted August 13, 1996.⊗

JE960186A

⊗ Abstract published in *Advance ACS Abstracts,* September 15, 1996.