Excess Properties of Binary Mixtures of Esters of Carbonic Acid + Three Aryl Alcohols at 308.15 K

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Excess molar enthalpies, H_m^E, and excess molar volumes, V_m^E, of binary mixtures containing dimethyl carbonate, or diethyl carbonate, or propylene carbonate + benzyl alcohol, + 2-phenylethyl alcohol, and + 3-phenyl-1-propyl alcohol have been studied over the entire range of composition at 308.15 K and at atmospheric pressure using a flow microcalorimeter and a digital density meter. The H_m^E values are positive, increasing as the chain length of aryl alcohol increases and showing a maximum varying from 1040 J·mol^{-1} (diethyl carbonate + benzyl alcohol) up to 1730 J·mol^{-1} (dimethyl carbonate + 3-phenyl-1-propyl alcohol). V_m^E values are negative or positive with no definite trend. The mixture containing propylene carbonate + 2-phenylethyl alcohol shows an inversion of sign. All V_m^E curves for mixtures containing dimethyl carbonate and diethyl carbonate are quasi-symmetrical, whereas those containing propylene carbonate are asymmetrical. Results were fitted to the Redlich−Kister polynomial. The results are qualitatively discussed in terms of molecular interactions.

Introduction

The thermodynamic study of esters of carbonic acids is arousing increasing interest owing to their uses in extractions of industrial importance, for many synthetic and natural resins and polymers,[1,2] in the synthesis of pharmaceuticals,[3] and in agricultural chemistry.

Particularly, there is strong interest in the dimethyl carbonate and propylene carbonate applications to storage batteries,[4−6] based on the intercalation of solutions of carbonates with lithium and bromine in graphite. Such batteries can be recharged thermally at relatively low temperatures.

Binary mixtures containing propylene carbonate and other organic solvents, especially aprotic ones, also have great technological and theoretical interest.[7]

Estimations of molecular interactions of binary mixtures and information needed to test existing theories of solutions may be related to the magnitude of excess thermodynamic properties such as excess molar enthalpies, H_m^E, and excess molar volumes, V_m^E. This paper aims to determine both properties with the purpose of obtaining some insight into interactions between molecules and studying the influence of the increasing chain length in aryl alcohols on H_m^E and V_m^E.

This paper reports experimental H_m^E and V_m^E for binary mixtures containing dimethyl carbonate (DMC), or diethyl carbonate (DEC), or propylene carbonate (PC) + three aryl alcohols, viz., benzyl alcohol (BA), 2-phenylethyl alcohol (PEA), and 3-phenyl-1-propyl alcohol (PPA). Measurements were made at 308.15 K and at atmospheric pressure.

Excess values were correlated using the Redlich−Kister polynomial, and the standard deviation, σ, between experimental and calculated values is reported.

No data on excess enthalpy or excess volume have been found in the literature for the mixtures studied in this paper.

Table 1. Mole Percent Purities, Sources, and Densities, ρ, of Pure Components at 308.15 K and Comparison with Literature Values and Vaporization Enthalpy of Components (1)

			ρ/g·cm^{-3}		
component	source	T/K	this paper	lit.	$\Delta_{vap}H$/ kJ·mol^{-1}
dimethyl carbonate (99+%)	Aldrich	298.15	1.06335	1.0632[8]	31.9[a]
		308.15	1.05004		
diethyl carbonate (99.8%)	Aldrich	298.15	0.96923	0.96926[10]	43.6[b,g]
		308.15	0.95773	0.9579[11]	40.3[c,g] 36.2[d,h]
propylene carbonate (99.7%)	Aldrich	298.15	1.19957	1.1995[12]	55.2[e,h]
		308.15	1.18889		49.8[f,h]
benzyl alcohol (99.8%)	Riedel de Haen	298.15	1.04146	1.04127[10]	
		308.15	1.03369	1.0330[13]	
2-phenyl alcohol (≥99%)	Fluka	308.15	1.00872		
3-phenyl-1-propyl alcohol	Lancaster	308.15	0.99081		

[a] At $t = 90.5$ °C (from Trouton law). [b] At $t = 25$ °C (see footnote g). [c] 25 °C $< t <$ 125 °C (see footnote g). [d] At $t = 126$ °C (see footnote h). [e] $t = 150$ °C (see footnote h). [f] $t = 242$ °C (see footnote h). [g] D. R. Lide. [h] Riddick et al.

Experimental Section

Chemicals. The mass fraction purities of all chemicals, declared by the manufacturers, were ≥99% for the pure components, which were used without further purification.

Before use, the components were degassed ultrasonically (ultrasonic bath, Hellma, type 460, Milan, Italy) and dried over molecular sieves (Aldrich, type 3A) to remove any traces of moisture. The purities of all products were checked using a Hewlett-Packard GC model 5890 with an HP (cross-linked 5% ME syloxane) capillary column, and the obtained values complied with purchaser specifications. The densities of the pure components were determined experimentally as a test of component quality. Table 1 reports purities, sources, and densities in comparison with literature data.[8−13]

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Calorimetric Measurements. A flow microcalorimeter (LKB, model 2107, Producer AB, Bromma, Sweden), thermostated at (308.15 ± 0.01) K, was used to measure the excess molar enthalpies, H_m^E. The apparatus consists of a flow-mixing cell, a reference cell, a thermostatic water bath, a data acquisition unit, and two automatic burets (ABU, Radiometer, Copenhagen, Denmark) that are necessary to pump the pure liquids into the mixing cell of the calorimeter. The temperature of the bath was controlled within ±0.01 K. The calibration of the apparatus and its operating procedure were described elsewhere.[14,15] The performance and reliability of the microcalorimeter were checked by the test mixtures hexane + cyclohexane, benzene + cyclohexane, and methanol + water and agreed with literature data.[16] The miscibility of the components was tested prior to measurements, and the components were found to be completely miscible over the whole concentration range. Mole fractions of mixtures were computed from densities. Volumetric flow rates of components, selected to cover the entire weight fraction range, were stated by the automatic burets. The total flow rates are usually kept at about 0.4 $cm^3 \cdot min^{-1}$, but in the dilute region the total flow rates may increase up to 0.8 $cm^3 \cdot min^{-1}$. The experimental uncertainties in H^E were estimated to be less than 0.5% over most of the composition range.

Density measurements. Excess molar volumes, V_m^E, reproducible to ±0.003 $cm^3 \cdot mol^{-1}$, have been determined from density measurements with the help of a digital density meter (Anton Paar, model DMA 60, Graz, Austria) equipped with a measuring cell (Anton Paar, type 602) whose operating procedure has been described elsewhere.[17]

All measurements have been determined at a constant temperature using an external ultrathermostat bath circulator (Heto, type 01 DTB 623, Birkeròd, Denmark), precision ±0.005 K, and temperatures have been detected by a digital thermometer (Anton Paar, type CTK 100). Mole fractions, x_1, of esters of carbonic acid (component 1) have been obtained by mass using a digital balance (Mettler, model AE 160, Switzerland) with an accuracy of ±0.0001 g. All masses have been corrected for buoyancy and the evaporation of components. All molar quantities have been based on the relative atomic mass table by IUPAC.[18]

Before each series of measurements, the apparatus was calibrated at atmospheric pressure using double-distilled water and dry air, whose densities were taken from the literature.[19,20] The uncertainty in density was $\pm 1.5 \times 10^{-5}$ $g \cdot cm^{-3}$ at 308.15 K. Correspondingly, the uncertainty in V_m^E is estimated to be <1%. Before measurements, the apparatus was checked by determining V_m^E using the test mixture benzene + cyclohexane at 298.15 K. Our results agree with those of the literature,[21] showing a discrepancy of ±0.5% in the central range of the mole fraction of benzene.

Table 2. Excess Molar Enthalpies, H_m^E, for Binary Mixtures Containing Esters of Carbonic Acid + Three Aryl Alcohols

x_1	$H_m^E/J \cdot mol^{-1}$	x_1	$H_m^E/J \cdot mol^{-1}$	x_1	$H_m^E/J \cdot mol^{-1}$
Dimethyl Carbonate (1) + Benzyl Alcohol (2)					
0.0483	202	0.3787	1254	0.8298	1002
0.0922	393	0.4483	1311	0.8798	841
0.1322	565	0.5494	1366	0.9070	733
0.1689	692	0.6466	1308	0.9360	560
0.2336	938	0.7092	1252	0.9670	311
0.2890	1089	0.7854	1132		
Dimethyl Carbonate (1) + 2-Phenylethyl Alcohol (2)					
0.0555	220	0.4138	1423	0.8495	1096
0.1052	453	0.4847	1489	0.8944	912
0.1500	657	0.5852	1494	0.9186	755
0.1904	831	0.6792	1427	0.9443	565
0.2608	1110	0.7384	1350	0.9713	320
0.3200	1254	0.8090	1218		
Dimethyl Carbonate (1) + 3-Phenyl-1-propyl Alcohol (2)					
0.0626	318	0.4448	1674	0.8650	1187
0.1178	599	0.5164	1712	0.9017	975
0.1668	851	0.6157	1701	0.9276	784
0.2107	1033	0.7062	1598	0.9506	579
0.2860	1328	0.7621	1520	0.9747	324
0.3481	1487	0.8278	1334		
Diethyl Carbonate (1) + Benzyl Alcohol (2)					
0.0341	94	0.2978	786	0.7724	927
0.0660	204	0.3612	874	0.8358	820
0.0959	267	0.4590	987	0.8716	731
0.1236	358	0.5600	1023	0.9106	578
0.1750	504	0.6292	1031	0.9532	349
0.2204	623	0.7179	987		
Diethyl Carbonate (1) + 2-Phenylethyl Alcohol (2)					
0.0393	141	0.3293	922	0.7971	1040
0.0756	246	0.3957	1023	0.8549	877
0.1093	373	0.4955	1166	0.8871	756
0.1404	480	0.5957	1199	0.9218	576
0.1971	643	0.6626	1206	0.9593	335
0.2466	759	0.7466	1118		
Diethyl Carbonate (1) + 3-Phenyl-1-propyl Alcohol (2)					
0.0444	138	0.3579	1062	0.8168	1107
0.0850	276	0.4263	1164	0.8645	960
0.1223	410	0.5272	1246	0.8992	801
0.1564	534	0.6258	1293	0.9304	618
0.2179	726	0.6903	1292	0.9640	359
0.2709	890	0.7698	1202		
Propylene Carbonate (1) + Benzyl Alcohol (2)					
0.0483	219	0.3788	1003	0.8299	697
0.0922	398	0.4484	1057	0.8798	545
0.1323	532	0.5491	1079	0.9070	427
0.1689	610	0.6467	1025	0.9361	313
0.2337	792	0.7092	955	0.9670	170
0.2890	912	0.7854	807		
Propylene Carbonate (1) + 2-Phenylethyl Alcohol (2)					
0.0556	263	0.0556	263		
0.1053	462	0.4138	1070	0.8496	686
0.1500	587	0.4849	1107	0.8944	534
0.1905	718	0.5854	1120	0.9187	433
0.2609	876	0.6793	1049	0.9443	297
0.3200	986	0.7385	971	0.9713	168
0.4138	1070	0.8090	811		
Propylene Carbonate (1) + 3-Phenyl-1-propyl Alcohol (2)					
0.0626	373	0.4448	1238	0.8651	808
0.1178	621	0.5165	1245	0.9058	630
0.1669	773	0.6159	1261	0.9276	508
0.2108	908	0.7063	1176	0.9506	350
0.2861	1060	0.7622	1122	0.9747	197
0.3482	1157	0.8278	959		

Results and Discussion

The experimental results of the excess molar enthalpies, H_m^E, and the excess molar volumes, V_m^E, at atmospheric pressure and at 308.15 K are listed in Tables 2 and 3 as a function of the mole fraction x_1 (ester of carbonic acid) and are graphically represented in Figures 1 to 4.

Density values of the mixtures, ρ, have been used to calculate the excess molar volumes, V_m^E, with the following equation

$$V_m^E = \frac{x_1 M_1 + x_2 M_2}{\rho} - \frac{x_1 M_1}{\rho_1} - \frac{x_2 M_2}{\rho_2} \tag{1}$$

where x_i, M_i, and ρ_i ($i = 1, 2$) are the mole fractions, molecular weights, and densities of pure components 1 and 2, respectively.

Each set of experimental values of H_m^E and V_m^E is fitted to a Redlich−Kister polynomial of the type

$$Q_m^E = x_1 x_2 \sum_{k \geq 0} a_k (x_1 - x_2)^k \tag{2}$$

Table 3. Densities, ρ, and Excess Molar Volumes, V_m^E, for Binary Mixtures Containing Esters of Carbonic Acid + Three Aryl Alcohols

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}
Dimethyl Carbonate (1) + Benzyl Alcohol (2)					
0.0478	1.03462	0.029	0.6255	1.04481	0.148
0.0879	1.03540	0.051	0.7081	1.04606	0.130
0.2007	1.03752	0.102	0.7924	1.04728	0.102
0.2997	1.03930	0.130	0.8424	1.04796	0.081
0.3788	1.04070	0.146	0.9314	1.04917	0.040
0.5552	1.04369	0.156	0.9574	1.04951	0.025
Dimethyl Carbonate (1) + 2-Phenylethyl Alcohol (2)					
0.0774	1.01118	−0.017	0.5880	1.02995	−0.045
0.1146	1.01240	−0.025	0.7335	1.03637	−0.030
0.2190	1.01592	−0.040	0.8154	1.04026	−0.020
0.3247	1.01968	−0.049	0.8704	1.04302	−0.013
0.4211	1.02328	−0.053	0.9473	1.04710	−0.005
0.4991	1.02631	−0.050	0.9705	1.04838	−0.002
Dimethyl Carbonate (1) + 3-Phenyl-1-propyl Alcohol (2)					
0.0675	0.99325	0.015	0.6176	1.01948	0.107
0.1509	0.99638	0.044	0.7605	1.02910	0.100
0.2665	1.00118	0.070	0.8260	1.03417	0.082
0.3760	1.00621	0.091	0.8950	1.03992	0.062
0.4574	1.01033	0.099	0.9445	1.04446	0.038
0.5285	1.01422	0.104	0.9680	1.04674	0.024
Diethyl Carbonate (1) + Benzyl Alcohol (2)					
0.0461	1.03049	0.092	0.5738	0.99128	0.489
0.0626	1.02934	0.123	0.6238	0.98738	0.467
0.1509	1.02307	0.267	0.7110	0.98055	0.405
0.2205	1.01800	0.354	0.8166	0.97224	0.293
0.2959	1.01243	0.426	0.9056	0.96519	0.165
0.4666	0.99954	0.502	0.9440	0.96215	0.102
Diethyl Carbonate (1) + 3-Phenylethyl Alcohol (2)					
0.0617	1.00629	0.093	0.5827	0.98179	0.371
0.1025	1.00463	0.147	0.6519	0.97802	0.343
0.1776	1.00144	0.232	0.7544	0.97227	0.275
0.2457	0.99843	0.293	0.8291	0.96795	0.206
0.3209	0.99496	0.343	0.9071	0.96332	0.117
0.4937	0.98643	0.384	0.9590	0.96021	0.053
Diethyl Carbonate (1) + 3-Phenyl-1-propyl Alcohol (2)					
0.0297	0.99019	−0.033	0.5247	0.97656	−0.293
0.0786	0.98910	−0.086	0.6119	0.97354	−0.279
0.1968	0.98623	−0.187	0.6917	0.97058	−0.246
0.2732	0.98421	−0.235	0.7678	0.96762	−0.203
0.3516	0.98200	−0.271	0.8510	0.96420	−0.139
0.4434	0.97921	−0.292	0.9538	0.95978	−0.045
Propylene Carbonate (1) + Benzyl Alcohol (2)					
0.0670	1.04252	0.010	0.5509	1.11138	0.135
0.1226	1.04995	0.023	0.6243	1.12292	0.141
0.2053	1.06118	0.046	0.7096	1.13680	0.137
0.2974	1.07398	0.075	0.7868	1.14986	0.122
0.3710	1.08451	0.951	0.8608	1.16290	0.093
0.4658	1.09841	0.122	0.9111	1.17203	0.687
Propylene Carbonate (1) + 2-Phenylethyl Alcohol (2)					
0.0824	1.01974	−0.020	0.5820	1.09772	0.048
0.1091	1.02337	−0.024	0.6619	1.11280	0.059
0.2327	1.04087	−0.023	0.7441	1.12928	0.066
0.3160	1.05333	−0.011	0.8014	1.14147	0.063
0.4136	1.06872	0.010	0.8747	1.15796	0.051
0.4860	1.08078	0.027	0.9551	1.17739	0.023
Propylene Carbonate (1) + 3-Phenyl-1-propyl Alcohol (2)					
0.0731	1.00011	0.001	0.6100	1.08690	0.180
0.1339	1.00812	0.028	0.6937	1.10494	0.180
0.2574	1.02555	0.071	0.7618	1.12088	0.172
0.3540	1.04041	0.112	0.8268	1.13743	0.146
0.4362	1.05414	0.139	0.8890	1.15458	0.105
0.5404	1.07309	0.169	0.9548	1.17429	0.050

by an unweighted least-squares method, where Q_m^E refers to H_m^E/J·mol^{-1} or V_m^E/cm^3·mol^{-1}, x_1 and x_2 are the molar fractions of component 1 (esters of carbonic acid) and component 2 (aryl alcohols), and a_k are the adjustable parameters. The a_k values are given in Table 4 together with the standard deviation $\sigma(Q_m^E)$ defined as

$$\sigma(Q_m^E) = \left|\frac{\phi_{\min}}{N - n}\right|^{0.5} \tag{3}$$

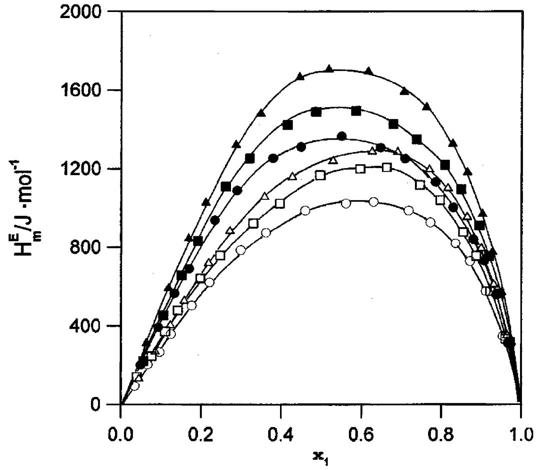

Figure 1. Experimental excess molar enthalpies, H_m^E, for the following binary mixtures: ●, DMC (1) + BA (2); ■, DMC (1) + PEA (2); ▲, DMC (1) + PPA (2); ○, DEC (1) + BA (2); □, DEC + PEA (2); and △, DEC (1) + PPA (2) at 308.15 K. Solid curves, Redlich−Kister equation.

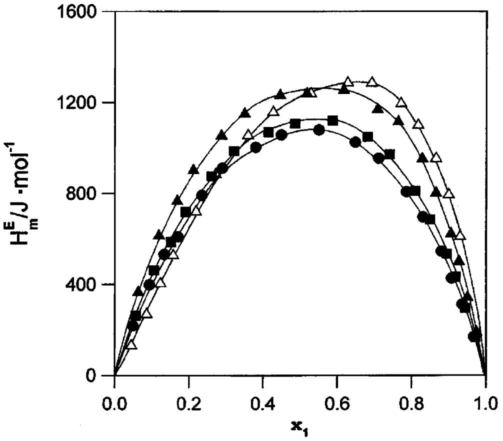

Figure 2. Experimental excess molar enthalpies, H_m^E, for the following binary mixtures: ●, PC (1) + BA (2); ■, PC (1) + PEA (2); ▲, PC (1) + PPA (2); and △, DEC (1) + PPA (2) at 308.15 K. Solid curves, Redlich−Kister equation.

with N and n the number of experimental points and parameters, whereas $\phi_{\min}$ is the minimum value of the objective function ϕ defined as

$$\phi = \sum_{k=1}^{N} {\eta_k}^2 \tag{4}$$

where $\eta_k = Q_{m,calcd}^E - Q_m^E$; Q_m^E is the experimental value, and $Q_{m,calcd}^E$ is evaluated through eq 2.

Data reported in Table 2 and Figures 1 and 2 show that H_m^E's are positive for all mixtures and increase with the increasing chain length of the alcohol. DEC and PC lead to values of H_m^E smaller than the corresponding values for DMC mixtures. DEC and PC curves intersect and have the same order of H_m^E for all mixtures. These results may be qualitatively explained by considering the approximate expression $H_m^E \approx E_{11} + E_{22} - 2E_{12}$ and the relative values of the energy E_{ij} between molecules i and j, with 1 and 2 being the carbonates and alcohols, respectively. In fact, interaction energy E_{22} of alcohols $HO(CH_2)_n$-C_6H_5 are dominant because of hydrogen bonding, and thus positive values of H_m^E are found. Moreover, the hydrogen bonding strength decreases with the increase of n from 1 to 3

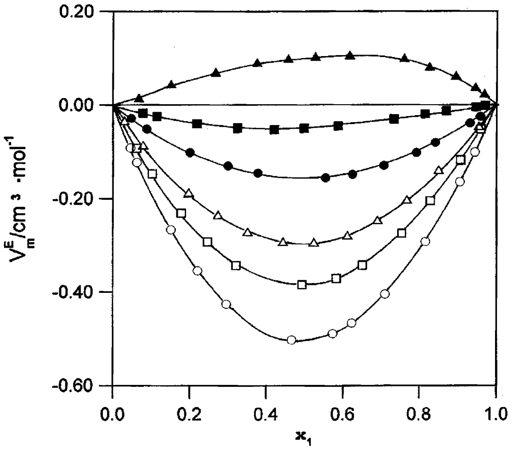

Figure 3. Experimental excess molar volumes, V^{E}_{m}, for the following binary mixtures: ●, DMC (1) + BA (2); ■, DMC (1) + PEA (2); ▲, DMC (1) + PPA (2); ○, DEC (1) + BA (2); □, DEC + PEA (2); and △, DEC (1) + PPA (2) at 308.15 K. Solid curves, Redlich−Kister equation.

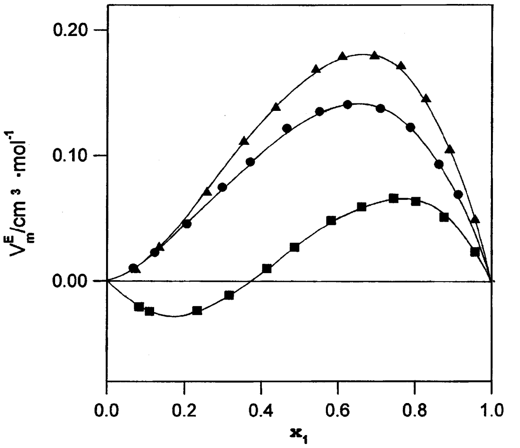

Figure 4. Experimental excess molar volumes, V^{E}_{m}, for the following binary mixtures: ●, PC (1) + BA (2); ■, PC (1) + PEA (2); and ▲, PC (1) + PPA (2) at 308.15 K. Solid curves, Redlich−Kister equation.

because a longer alkyl chain exhibits a larger electron donor effect.[22] When, for each carbonate, the length of the alkyl chain is increased, the difference $E_{22} - 2E_{12}$ increases because both terms E_{22} and E_{12} decrease, E_{12} more markedly than E_{22}, probably because of steric effects.

On the other side, when we compare the H^{E}_{m}'s for mixtures with the same alcohol and varying carbonates, the difference $E_{11} - 2E_{12}$ must be accounted for. Because E_{ii} is proportional to the vaporization enthalpy, $\Delta_{vap}H$, we report in Table 1 the values of ΔH_v for DMC, DEC, and PC. However, no value of $\Delta_{vap}H$ for DMC is quoted in the literature, and thus an estimate was obtained from Trouton's law. Allowing for the decrease of ΔH_v with the increase of temperature, from data of Table 1 we may conclude that $E_{11} \approx \Delta_{vap}H$ values follow the order DMC < DEC < PC. Only a variation of E_{12} in the same order can agree with the decrease of H^{E}_{m} in going from DMC to DEC and PC, and really, an alkyl chain is a stronger electron donor when the length is increased or cyclization occurs, with a consequent increase of the negative charge on the oxygen atom of the C=O group and an increase of the hydrogen bonding C=O···OH between carbonate and alcohol.

Table 4. Adjustable Parameters, a_k, (Equation 4) and Standard Deviations $\sigma(Q^E)$ (Equation 3) of Esters of Carbonic Acids + Aryl Alcohols at 308.15 K

function	a_0	a_1	a_2	a_3	$\sigma(Q^E)$
		DMC + BA			
H^E/J·mol^{-1}	5073.2	401.1	1790.9	2962.4	12.9
V^E/cm^3·mol^{-1}	−0.2019	0.0834	0.0414		0.0003
H^E/J·mol^{-1}	4006.5	926.5	1621.9	2126.7	8.0
		DMC + PEA			
V^E/cm^3·mol^{-1}	−2.0115	0.09285			0.0006
H^E/J·mol^{-1}	5943.5	492.9	1916.4	4030.3	9.0
V^E/cm^3·mol^{-1}	−0.6261	0.0013			0.0012
		DMC + PPA			
H^E/J·mol^{-1}	6782.7	870.7	2270.9	3831.7	11.6
V^E/cm^3·mol^{-1}	0.4135	0.1877	0.1249		0.0030
		DEC + PEA			
H^E/J·mol^{-1}	4607.7	1607.4	1760.6	1249.0	10.1
V^E/cm^3·mol^{-1}	−1.5328	0.1019			0.0024
		DEC + PPA			
H^E/J·mol^{-1}	4954.7	1513.5	2086.8	2680.5	6.6
V^E/cm^3·mol^{-1}	−1.1817	0.0482	0.0838		0.0012
		PC + BA			
H^E/J·mol^{-1}	4318.1	315.6	909.6		9.4
V^E/cm^3·mol^{-1}	0.5086	0.3836			0.0011
		PC + PEA			
H^E/J·mol^{-1}	4485.6	488.7	1200.1		8.1
V^E/cm^3·mol^{-1}	0.1188	0.4636			0.0006
		PC + PPA			
H^E/J·mol^{-1}	5022.0	743.1	2582.4		11.7
V^E/cm^3·mol^{-1}	0.6417	0.5562			0.0018

The intersection of the curves for DEC and PC with the three alcohols points out values of H^{E}_{m} for PC larger than those of DEC in the carbonate-poor region and smaller in the carbonate-rich region, with a maximum difference in H^{E}_{m} of 150 J·mol^{-1}, about 15% of the thermal effect.

This relatively small difference makes any discussion of the structural and energy contributions affecting H^{E}_{m} difficult.

As to volumetric data, Figures 3 and 4 show values of V^{E}_{m} for DMC larger than those for DEC, as was observed for the thermal data. The V^{E}_{m} for the mixtures containing PC do not preserve this latter trend and show a sign inversion for the 2-phenylethyl-alcohol and a maximum value twice that of DMC. Only the mixtures DMC + PPA, PC + BA, and PC + PPA have positive values of V^{E}_{m}. Thus, this variety in the curves, with positive and negative values for DMC, negative values for DEC, and positive values for PC, with the exception of the mixtures with 2-phenylethyl alcohol, indicates the complexity of the systems, where the interaction energy, steric effects, and packaging of molecules contribute to the final volume of the mixture.

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