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Vapor−Liquid Equilibria, Excess Molar Enthalpies, and Excess Molar Volumes of Dialkyl Carbonates + Methyl *tert*-Butyl Ether at 298.15 K

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Vapor−liquid equilibria, VLE, excess molar enthalpies, H_m^E, and excess molar volumes, V_m^E, for dimethyl and diethyl carbonate + methyl *tert*-butyl ether have been determined at 298.15 K and at atmospheric pressure. VLE data were tested for thermodynamic consistency and were correlated by the Wilson, NRTL, and Redlich−Kister equations. The Redlich−Kister polynomial was used to correlate H_m^E and V_m^E values. Parameters and least-squares analysis of the results have been reported.

Introduction

The knowledge of the physical properties of mixtures is important in understanding the nature of molecular interactions.

Continuing our research program involving substances of practical interest, we report in this paper vapor−liquid equilibrium (VLE) measurements, excess molar enthalpies, H_m^E, and excess molar volumes, V_m^E, of dimethyl carbonate (DMC) and diethyl carbonate (DEC) + methyl *tert*-butyl ether (MTBE) at 298.15 K and at atmospheric pressure.

Dialkyl carbonates find a wide range of applications as solvents for many synthetic and natural resins, in the separation of sour gases, in the synthesis of pharmaceuticals, and in agricultural chemistry.

In the recent years, the importance of methyl *tert*-butyl ether (2-methoxy-2-methylpropane or MTBE) as an oxygenating agent in gasoline technology has increased, with the publication of several papers reporting measurements on binary or ternary mixtures with hydrocarbons and alcohols (Wang and Benson, 1993; Arce et al., 1995, 1996; McHall et al., 1996).

To our knowledge no excess properties have been previously reported for mixtures containing esters of carbonic acid + MTBE.

Experimental Section

Materials. All chemicals were supplied from Aldrich Chemical Co. Dialkyl carbonates, stated purity 99 mol % from purchaser, were fractionally distilled with sodium following the procedure used in a previous paper (Francesconi and Comelli, 1996). After purification, GLC analysis indicated that chemicals had a purity grade of 99.9 mol %. MTBE, stated purity 99.8 mol %, was used as received.

The purities were checked also by comparing some of the measured properties of pure components with the values of literature and are reported in Table 1.

Before measurements, the liquids were kept in dark bottles, dried over molecular sieves (Union Carbide, type 4A, 1/16 in. pellets), and degassed by ultrasound (ultrasonic bath, Hellma, type 460, Milan, Italy).

Table 1. Densities, ρ, Vapor Pressures, P°, and Normal Boiling Points, T_b, of Pure Compounds in Comparison with Literature Data

component	ρ(298.15 K)/g·cm^{-3} exptl	ρ(298.15 K)/g·cm^{-3} lit.	P°(298.15 K)/kPa exptl	P°(298.15 K)/kPa lit.	T_b/K exptl	T_b/K lit.
DMC	1.063 00	1.063 059[a]	7.14	7.190[b]	363.60	363.65[c]
DEC	0.969 24	0.969 26[d]	1.52	1.530[d]	399.90	399.95[e]
MTBE	0.735 31	0.735 6[f]	33.36	33.447[g]	328.20	328.11[f]

[a] Negadi et al., 1993. [b] Cocero et al., 1989a. [c] CRC Handbook, 1979. [d] Riddick et al., 1986. [e] Cocero et al., 1989b. [f] Arce et al., 1996. [g] Belaribi et al., 1992.

Procedure. Binary VLE measurements were determined using a glass dynamic vapor-recirculating still (Fritz Gmbh, Normag, Hofheim, Germany) as described elsewere (Gmehling et al., 1980), the same as used in our previous papers (Francesconi and Comelli, 1996; Comelli et al., 1996).

Each sample was kept at the boiling point for 20 min or more to ensure the stationary state, and the liquid and vapor phase samples were withdrawn using proper syringes. Samples were analyzed by density technique using a digital density meter (Anton Paar, model DMA 60, Graz, Austria) equipped with a measuring cell (Anton Paar, type 602).

The uncertainties for pressure and temperature in the still were ±0.05 kPa and ±0.05 K respectively, measured by digital instruments (Fritz Gmbh, Normag) supplied with the glass apparatus. Composition uncertainties were estimated better than 0.002, in mole fraction of dialkyl carbonate (component 1).

The density meter, whose operating procedure has been described elsewere (Fermeglia and Lapasin, 1988), has been used also to measure the densities ρ necessary to determine the excess molar volumes, V_m^E, according to

$$V_m^E/\mathrm{cm^3{\cdot}mol^{-1}} = (x_1M_1 + x_2M_2)/\rho - x_1M_1/\rho_1 - x_2M_2/\rho_2 \qquad (1)$$

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

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Table 2. Clausius−Clapeyron Coefficients *A* and *B*, Eq 1, Correlation Coefficients | *R* |, and Standard Deviation $\sigma(P°)$ of Pure Compounds

component	temp range/K	A	B	$\lvert R \rvert$	$\sigma(P°)$
DMC	283.15−343.15	7.398 38	−1951.23	0.999 94	0.34
DEC	293.15−363.55	7.610 67	−2215.23	0.999 77	0.19
MTBE	283.75−328.20	6.805 98	−1575.04	0.999 97	0.26

Table 3. Pressures, *P*/kPa, Mole Fractions, x_1 and y_1, in Liquid and Vapor Phases, and Activity Coefficients, γ_i, Eq 3, for Dialkyl Carbonates + MTBE at 298.15 K

P/kPa	x_1	y_1	γ_1	γ_2
		DMC (1) + MTBE (2)		
32.60	0.059	0.026	1.97	1.01
31.45	0.133	0.056	1.82	1.03
30.35	0.202	0.086	1.78	1.04
28.85	0.296	0.114	1.54	1.09
26.90	0.396	0.158	1.49	1.13
24.65	0.513	0.195	1.30	1.23
23.05	0.583	0.235	1.29	1.28
21.50	0.650	0.267	1.23	1.36
19.80	0.713	0.297	1.15	1.47
18.10	0.767	0.345	1.13	1.54
16.30	0.819	0.391	1.08	1.66
15.20	0.849	0.432	1.08	1.74
13.70	0.891	0.501	1.07	1.90
10.35	0.960	0.708	1.07	2.15
9.70	0.969	0.753	1.05	2.20
9.20	0.976	0.800	1.05	2.23
8.90	0.980	0.817	1.04	2.26
8.60	0.983	0.855	1.04	2.27
8.45	0.985	0.864	1.04	2.28
8.25	0.987	0.885	1.03	2.30
8.05	0.990	0.906	1.03	2.31
7.80	0.993	0.935	1.02	2.33
		DEC (1) + MTBE (2)		
32.75	0.031	0.002	1.33	1.01
31.90	0.063	0.004	1.30	1.02
30.70	0.107	0.007	1.29	1.02
29.30	0.161	0.011	1.28	1.04
27.30	0.229	0.017	1.28	1.05
25.65	0.286	0.022	1.27	1.06
24.40	0.333	0.028	1.26	1.07
23.15	0.370	0.030	1.21	1.08
21.85	0.412	0.034	1.15	1.09
20.45	0.457	0.039	1.13	1.09
19.05	0.503	0.043	1.07	1.11
17.60	0.555	0.052	1.07	1.14
15.25	0.625	0.068	1.07	1.15
12.70	0.706	0.091	1.07	1.19
11.50	0.735	0.105	1.07	1.19
9.20	0.801	0.143	1.07	1.21
7.60	0.845	0.180	1.06	1.23
6.45	0.875	0.220	1.06	1.23
5.50	0.901	0.260	1.04	1.23
3.50	0.945	0.385	1.00	1.25
2.50	0.965	0.505	1.00	1.25

Solutions were prepared by mass using a Mettler balance (model AE 160) with a precision of ±0.0001 g, charging the heavier component first to minimize vaporization.

Taking these precautions, the error in mole fraction is less than 0.0002.

The period of oscillation τ of the vibrating U-shaped tube filled with the samples has a precision of $\pm 1 \times 10^{-6}$ s, and densities were determined with an accuracy less than 2×10^{-5} g·cm^{-3}.

For each set of measurements the apparatus was calibrated with doubly distilled and degassed water and dry air and was connected with an external constant-temperature bath circulator (Heto, type 01 DBT 623, Birkerød, Denmark), with an accuracy of ±0.005 K. The mixture benzene + cyclohexane was used to check the density meter. Our V_m^E values are in agreement with those of the literature (Wilhelm, 1985) (our value of V_m^E at 0.5 mole fraction is 0.652 cm^3·mol^{-1}, lit. value = 0.6514).

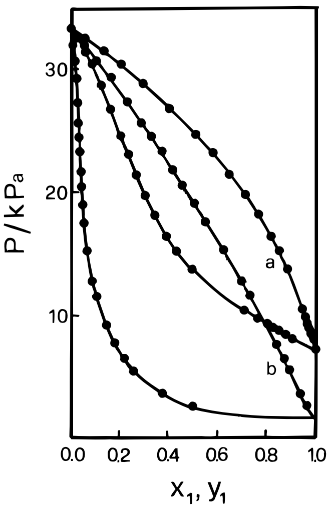

Figure 1. $P-x_1-y_1$ plots for the dialkyl carbonates (1) + MTBE (2) mixtures at 298.15 K. Closed circles, experimental points; a, b curves refer to DMC or DEC + MTBE mixtures, respectively.

Samples were prepared in ≈15 cm^3 capacity flasks following the procedure described by Fermeglia and Lapasin (1988); corrections for buoyancy and evaporation of the components were also applied to achieve greater accuracy. By such procedure, the accuracies of V_m^E are less than 0.003 cm^3·mol^{-1}.

A flow microcalorimeter (LKB produkter, model 2107, Bromma, Sweden) was used to determine the excess molar enthalpies, H_m^E.

Details of the electrical calibration and operating procedure have been described elsewere (Monk and Wadso, 1968; Francesconi and Comelli, 1968). Two automatic burets (ABU, Radiometer, Copenhagen, Denmark) were used to pump the pure liquids into the mixing cell of the calorimeter.

Mole fractions were determined from fluxes and density of compounds.

The temperature of the calorimeter was kept constant at (298.15 ± 0.01) K, and before measurements the apparatus was calibrated by determining H_m^E of the hexane + cyclohexane test mixture (Gmehling, 1993). Measurements were reproduced within ±0.5% over the central range of mole fraction of hexane.

The mole fractions and H_m^E values of DMC and DEC + MTBE mixtures were determined with an accuracy of ±0.0005 and 0.5%, respectively.

Results and Discussion

Vapor pressures $P_i°$ of the pure compounds were obtained with the still used for VLE data of mixtures and were fitted to the Clausius−Clapeyron equation

$$\log(P_i°/\mathrm{kPa}) = A + B/T(\mathrm{K}) \qquad (2)$$

Constants A and B for the three components are summarized in Table 2.

The experimental VLE values of the two binary mixtures are given in Table 3 and graphically represented in Figures 1 and 2.

The activity coefficients γ_i, which are shown in Table 3, were evaluated from the formula

$$\gamma_i = Py_i \,/\, x_iP_i \qquad (3)$$

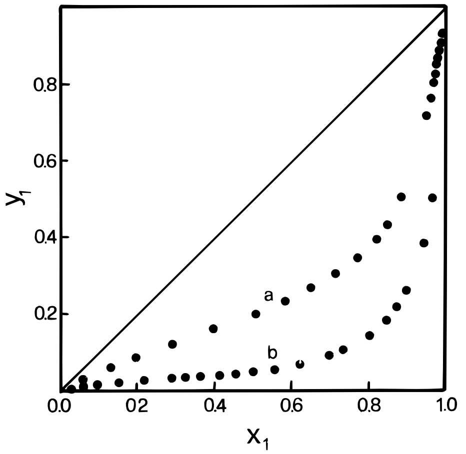

Figure 2. x_1−y_1 plots for dialkyl carbonate (1) + MTBE (2) mixtures at 298.15 K. Closed circles, experimental points; a, b curves refer to DMC or DEC + MTBE mixtures, respectively.

Table 4. Correlation Parameters, a_k, for Activity Coefficients, Eq 7, Standard Deviations, σ, Integral | I |, and Uncertainty δI, Eq 4, for Diaklyl Carbonates + MTBE Mixtures at 298.15 K

model	$a_0{}^c$	$a_1{}^c$	a_2	α_{12}	σ	$\lvert I \rvert$	δ
			DMC (1) + MTBE (2)				
Wilson	1190.9^a	1002.7^a			0.056	0.053	0.016
NRTL	911.94^a	539.06^a		−1.54^b	0.079		
Redlich−Kister	0.7235^b	0.0922^b	0.0481$_b$		0.058		
			DEC (1) + MTBE (2)				
Wilson	538.62^a	171.23^a			0.042		
NRTL	346.46^a	329.46^a		0.067^b	0.043		
Redlich−Kister	0.2809^b	−0.0147^b	−0.0378^b		0.041	0.022	0.079

^{a} J·mol^{-1}. ^{b} Dimensionless. ^{c} For Wilson and NRTL a_0, $a_1 = a_{12}$, a_{21}.

where P_i is the vapor pressure of pure compound multiplied by a fugacity term (Prausnitz, 1969) obtained from the virial coefficients calculated following the procedure described in a previous paper (Francesconi and Cojutti, 1971).

The thermodynamic consistency of VLE data was checked by a modified area test used in our previous papers (Francesconi and Comelli, 1996; Comelli et al., 1996).

This method is based on the determination of integral

$$I = \int_0^1 [\ln (\gamma_1/\gamma_2) + V_m^E/RT \cdot dP/dx_1] dx_1 \qquad (4)$$

and its uncertainty δI.

The thermodynamic consistency is established when | I | and its uncertainty δI are of the same order or $\delta I \geq$ | I |.

The contribution of the $V_m^E/RT \cdot dP/dx_1$ term to the integral I was evaluated from data of Tables 3 and 4 and was found to be less than the estimated error δI.

The criterion (eq 4) is well verified for DEC + MTBE, whereas values of I for DMC + MTBE are somewhat large, probably because the y_1 values of many experimental points are very close to unity. This entails a corresponding large value of uncertainty in the term $y_1/(1 - y_1)$ entering the ratio $\ln(\gamma_1/\gamma_2)$ in the integral I, or rather larger than the average uncertainty in the mole fractions.

The excess molar Gibbs energies, G_m^E, were then calculated using the equation

$$G_m^E = RT(x_1 \ln \gamma_1 + x_2 \ln \gamma_2) \qquad (5)$$

and are graphically represented in Figure 3 (dashed lines).

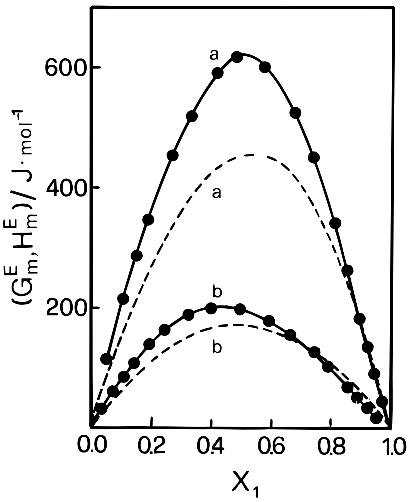

Figure 3. Excess molar enthalpies, H_m^E, eq 8, and excess molar Gibbs energies, G_m^E (dashed curves), eq 5, for dialkyl carbonates (1) + MTBE (2) mixtures at 298.15 K. Closed circles, experimental H_m^E points; solid lines, Redlich−Kister equation. a, b curves refer to DMC or DEC + MTBE, respectively.

Table 5. Mole Fractions, x_1, and Experimental Excess Molar Enthalpies, H_m^E, of Dialkyl Carbonates + MTBE at 298.15 K

x_1	H_m^E/J·mol^{-1}	x_1	H_m^E/J·mol^{-1}
	DMC (1) + MTBE(2)		
0.0556	114	0.6797	524
0.1054	215	0.7389	449
0.1503	286	0.8093	334
0.1908	346	0.8498	263
0.2613	455	0.8946	180
0.3204	518	0.9188	134
0.4143	591	0.9444	90
0.4853	616	0.9714	43
0.5859	599		
	DEC (1) + MTBE (2)		
0.0394	32	0.5960	172
0.0757	61	0.6630	155
0.1095	82	0.7469	119
0.1408	104	0.7997	94
0.1974	138	0.8551	66
0.2469	163	0.8872	51
0.3297	185	0.9219	34
0.396	192	0.9594	17
0.4959	195		

The activity coefficients γ_i were correlated with the Wilson, NRTL, and three-parameter Redlich−Kister equations. The best fits were obtained from Wilson and Redlich−Kister models for DMC + MTBE and DEC + MTBE, respectively. Results of least-squares analysis are reported in Table 4. Critical values were obtained from literature (Cocero et al., 1989a,b; Bennett et al., 1993).

The objective function to minimize was

$$\Phi = \sum_{k=1}^{N} \eta_k^2 \qquad (6)$$

where N is the number of experimental points and η the residual

$$\eta = \ln(\gamma_1/\gamma_2)_{\text{exptl}} - \ln(\gamma_1/\gamma_2)_{\text{calcd}} \qquad (7)$$

with $\gamma_{i,\text{exptl}}$ evaluated by means of eq 3 and $\gamma_{i,\text{calcd}}$ obtained from Wilson or Redlich−Kister expressions.

Table 6. Mole Fractions, x_1, Experimental Densities, ρ, and Excess Molar Volumes, V_m^E, of Dialkyl Carbonates + MTBE 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}
		DMC (1) + MTBE (2)			
0.0169	0.739 14	−0.005	0.6245	0.913 58	−0.136
0.1065	0.760 86	−0.034	0.6664	0.928 32	−0.132
0.1652	0.775 76	−0.052	0.7067	0.942 95	−0.127
0.3011	0.812 36	−0.090	0.7733	0.968 04	−0.112
0.4009	0.841 35	−0.116	0.8363	0.992 86	−0.090
0.4383	0.852 71	−0.124	0.9037	1.020 75	−0.060
0.5026	0.872 86	−0.128	0.9407	1.036 63	−0.039
0.5519	0.888 93	−0.135	0.9848	1.056 13	−0.011
		DEC (1) + MTBE (2)			
0.0526	0.748 309	−0.076	0.5382	0.864 66	−0.348
0.0845	0.756 14	−0.120	0.5940	0.877 63	−0.334
0.1626	0.775 20	−0.203	0.6307	0.886 12	−0.323
0.2167	0.788 31	−0.250	0.6979	0.901 51	−0.286
0.2949	0.807 14	−0.303	0.7822	0.920 66	−0.226
0.3680	0.824 61	−0.335	0.8578	0.937 68	−0.160
0.3980	0.831 71	−0.342	0.9226	0.952 12	−0.092
0.4681	0.848 27	−0.354	0.9752	0.963 74	−0.027

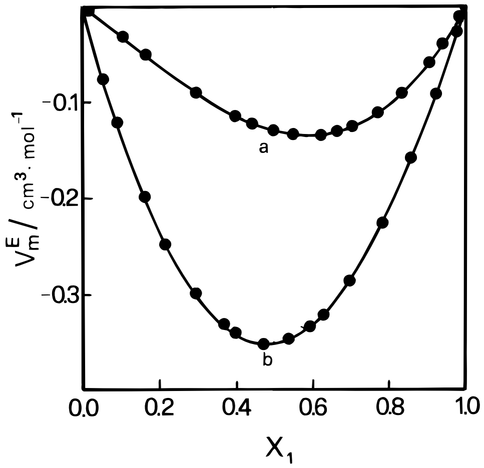

Figure 4. Excess molar volumes, V_m^E, eq 8, for dialkyl carbonates (1) + MTBE (2) mixtures at 298.15 K. Closed circles, experimental points; solid lines, Redlich−Kister equation. a, b curves refer to DMC or DEC + MTBE mixtures, respectively.

Experimental H_m^E and V_m^E are shown in Tables 5 and 6 and are graphically represented in Figures 3 and 4. Values were fitted to the Redlich−Kister polynomial

$$Q_m^E = x_1 x_2 \sum_{k \geq 0} a_k (x_1 - x_2)^k \tag{8}$$

where $Q_m^E = H_m^E$/J·mol^{-1}) or V_m^E/cm^3·mol^{-1} with all points weighted equally, x_1, x_2 are the mole fractions of components, and a_k are the adjustable parameters.

Values of a_k and standard deviation $\sigma(Q_m^E)$ are given in Table 7.

As can be seen from Figures 1−4, mixtures of dialkyl carbonates + MTBE show positive deviations from ideality (positive G_m^E with activity coefficients up to 2.3), positive H_m^E, and negative V_m^E.

It is interesting to remark that in going from dimethyl to diethyl carbonate, H_m^E and G_m^E become about three times smaller, whereas absolute values of V_m^E become about three times larger.

These results agree with the assumption of an increasing of the interaction energy E_{12} between dissimilar molecules, which leads to smaller heat of mixing and larger volume reduction after mixing.

Table 7. Adjustable Parameters, a_k, Eq 8, and Standard Deviations, $\sigma(Q_m^E)$, of Dialkyl Carbonates + MTBE at 298.15 K

function	a_0	a_1	a_2	a_3	$\sigma(Q_m^E)$
		DMC (1) + MTBE (2)			
H_m^E/J·mol^{-1}	2474.6	102.08	−652.57	−493.59	2.7
$V_m{}^E$/cm^3·mol^{-1}	−0.5220	−0.2132			0.0012
		DEC (1) + MTBE (2)			
H_m^E/J·mol^{-1}	774.33	−232.23	−140.84		1.7
V_m^E/cm^3·mol^{-1}	−1.4079	0.1297			0.0017

Since interactions between dialkyl carbonates and the ether MTBE cannot imply hydrogen bonding, the relatively larger value of the molecular dipole of diethyl carbonate ($\epsilon_r = 90$, compared with $\epsilon_r = 82$ of dimethyl carbonate) may be responsible of the increase of E_{12} when ethyl groups substitute for the methyl ones in the carbonate molecule.

From Figure 3, the values of excess entropy S_m^E may be obtained, remembering that $S_m^E = (H_m^E - G_m^E)/T$.

The S_m^E's are always positive and much larger for the system DMC + MTBE and this again indicates the absence of strong bonding between components which would lead to a decrease of entropy after mixing.

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