

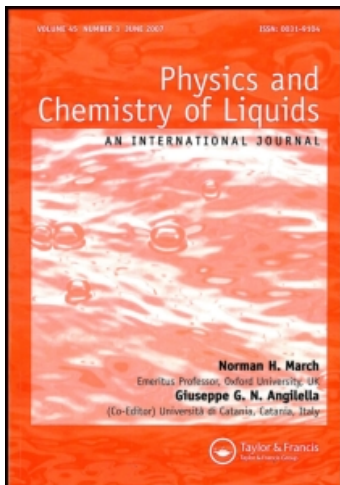
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EXCESS MOLAR VOLUME OF 2,2'- OXYBIS[PROPANE] WITH BENZENE, TOLUENE, CYCLOHEXANE, AND METHYL ETHANOATE AT 298.15 K

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Densities of the binary systems 2,2'-oxybis[propane] with benzene, toluene, cyclohexane, and methyl ethanoate have been measured as a function of the composition, at 298.15 K and atmospheric pressure, using an Anton Paar DMA 5000 oscillating U-tube densitometer. The calculated excess volumes were correlated with the Redlich-Kister equation and with Legendre's polynomials. The excess volumes are negative for the systems of 2,2'-oxybis[propane] with benzene and toluene and positive with cyclohexane and methyl ethanoate.

Keywords: Excess thermodynamic properties; Liquid mixtures

INTRODUCTION

Amendment of the US Clean Air Act in 1990 have mandated that new gasoline formulations be sold in highly polluted areas of the country, with oxygenated gasolines being supplied particularly during the winter. Methyl 1,1-dimethylethyl ether (MTBE), the primary oxygenated compound currently used to reformulate gasolines is being phased out because of evidence that it accumulates in surface and

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underground water. Other branched ethers that have been considered in the past are ethyl 1,1-dimethylethyl ether (ETBE) and methyl 1,1-dimethylpropyl ether (TAME). Additional candidates may be methanol, ethanol, 2-methyl-2-butanol, methyl acetate and 2,2'-oxybis[propane] (diisopropyl ether, DIPE).

Densities of binaries mixtures of oxygenates with gasoline components and others are needed to guide density correlations for gasoline + oxygenate mixtures, in addition to calculating transport properties.

The information available in the literature indicates that the excess volumes of mixing of MTBE and TAME with straight and branched hydrocarbons, benzene and cyclohexane, are positive (*e.g.*, Blanco *et al.*, 1994; Linek, 1987; Pinnnick *et al.*, 1995; Sharma *et al.*, 1992 and Witek *et al.*, 1997). Negative excess volumes of mixing have been reported for the system MTBE + benzene and + toluene (Sharma *et al.*, 1992) and for MTBE, ETBE and TAME + toluene by Domanska (1996). According to Linek (1995) at 298.15 K the volumes of mixing for the system ethyl propanoate + butyl methyl ether are almost zero ($< 1.4 \cdot 10^{-2} \text{ mL} \cdot \text{mol}^{-1}$).

Excess thermodynamic properties of mixtures are useful in the study of molecular interactions. The aim of this work is to provide data for the characterization of the molecular interactions, in particular the influence of the chemical structure of the solute in the systems under consideration.

EXPERIMENTAL SECTION

Materials

DIPE (99+ mass %), benzene (99.9 mass %), toluene (99.8 mass %), cyclohexane (99+ mass %) and methyl ethanoate (99.5 mass %), were purchased from Aldrich and used without further purification, except for degassing by boiling. The purity of the solvents was further ascertained by comparing their densities at 298.15 K with the values reported in the literature (Tab. I).

Density Measurements

The density of the samples was measured with an Anton Paar model DMA 5000 oscillating U-tube densitometer, provided with automatic

TABLE I Purity and densities of pure components at 298.15 K

Component	Purity (mass %)	Density/g · cm ⁻³	
		Measured	Lit. ^a
DIPE	99. +	0.718565	0.72198
Benzene	99.9	0.873455	0.87302
Toluene	99.8	0.862111	0.86403
Cyclohexane	99. +	0.773788	0.77394
Methyl ethanoate	99.5	0.926831	0.92805

^aDaubert and Donner (1989).

viscosity correction, two integrated Pt 100 platinum thermometers (DKD traceable), and a stated accuracy of $5 \cdot 10^{-6} \text{ g} \cdot \text{cm}^{-3}$. The temperature in the cell was regulated to $\pm 0.001 \text{ K}$ with a solid-state thermostat. The apparatus was calibrated once a day with dry air and bi-distilled freshly degassed water.

All liquids were boiled to remove dissolved air. Solutions of different compositions were prepared by mass in a 10 cm^3 rubber-stoppered vial to prevent evaporation, using a Mettler AG 204 balance accurate to $\pm 10^{-4} \text{ g}$. To minimize the errors in composition, the heavier component was charged first and the sample kept in ice water. Accuracy in the mole fraction is $5 \cdot 10^{-5}$, of the density (duplicate) measurement $\pm 2 \cdot 10^{-6} \text{ g} \cdot \text{cm}^{-3}$, and of the temperature $\pm 0.002 \text{ K}$.

RESULTS AND DISCUSSION

Twenty-one density measurements were performed (with repetition) for each binary system, in the full concentration range ($0 \leq x \leq 1$).

The excess volumes V^E of the solutions of molar composition x were calculated from the densities of the pure liquids and their mixtures according the following equation:

$$V^E = [xM_1 + (1 - x)M_2]/\rho - [xM_1/\rho_1 + (1 - x)M_2/\rho_2] \quad (1)$$

where ρ , ρ_1 and ρ_2 are the densities of the solution and pure components 1 and 2, respectively, and M_1 and M_2 the molar masses of the pure components. The corresponding values of ρ and V^E are reported in Tables II to V and Figure 1. The values of V^E were

TABLE II Experimental densities, volumes, calculated excess volumes, and deviations δV^E for the system DIPE(1) + benzene(2) at 298.15 K

x_1	$\rho/g \cdot cm^3$	$V/$	$V^E/$	$\delta V^E \cdot 10^3/$
		$mol \cdot cm^{-3}$		
0	0.873455	8.944E+01	0.000E+00	0
0.0668	0.85851	9.287E+01	-9.558E-02	4
0.1067	0.84998	9.493E+01	-1.384E-01	6
0.1551	0.840324	9.741E+01	-2.168E-01	-3
0.1985	0.83186	9.965E+01	-2.606E-01	1
0.2456	0.822996	1.021E+02	-2.939E-01	13
0.3188	0.810184	1.059E+02	-3.687E-01	-6
0.332	0.808076	1.066E+02	-3.958E-01	-25
0.4006	0.796617	1.102E+02	-4.113E-01	-9
0.4481	0.78906	1.127E+02	-4.131E-01	2
0.5083	0.779918	1.158E+02	-4.110E-01	9
0.5662	0.771548	1.189E+02	-4.036E-01	8
0.6184	0.764288	1.217E+02	-3.858E-01	8
0.6526	0.75973	1.235E+02	-3.782E-01	-1
0.6996	0.753543	1.260E+02	-3.416E-01	6
0.7511	0.747094	1.288E+02	-3.129E-01	-6
0.8028	0.740766	1.315E+02	-2.627E-01	-5
0.8447	0.735767	1.338E+02	-2.089E-01	3
0.9117	0.728144	1.374E+02	-1.300E-01	-2
0.9504	0.723948	1.395E+02	-8.814E-02	-14
1	0.718565	1.422E+02	0.000E+00	0

$$\delta V^E = V^E_{\text{expt}} - V^E_{\text{calc}}$$

TABLE III Experimental densities, volumes, calculated excess volumes, and deviations δV^E for the system DIPE(1) + toluene(2) at 298.15 K

x_1	$\rho/g \cdot cm^3$	$V/$	$V^E/$	$\delta V^E \cdot 10^3/$
0.0000	0.862111	1.069E+02	0.0000	0
0.0492	0.853893	1.085E+02	-0.1300	0
0.0993	0.845513	1.102E+02	-0.2313	19
0.1509	0.837403	1.119E+02	-0.3683	-8
0.2001	0.829529	1.135E+02	-0.4489	3
0.2506	0.821771	1.152E+02	-0.5439	-13
0.3002	0.814077	1.169E+02	-0.5942	2
0.3499	0.80653	1.186E+02	-0.6374	10
0.4003	0.799069	1.203E+02	-0.6766	8
0.4497	0.792031	1.220E+02	-0.7267	-20
0.5001	0.784704	1.238E+02	-0.7230	-8
0.5493	0.777648	1.256E+02	-0.7032	5
0.6000	0.770607	1.274E+02	-0.6858	1
0.6491	0.763841	1.292E+02	-0.6459	6
0.7000	0.756942	1.310E+02	-0.5924	8
0.7507	0.750228	1.329E+02	-0.5304	4
0.8004	0.743726	1.347E+02	-0.4547	1
0.8501	0.737316	1.365E+02	-0.3633	0
0.9006	0.730996	1.384E+02	-0.2731	-19
0.9505	0.724657	1.403E+02	-0.1334	0
1.0000	0.718565	1.422E+02	0.0000	0

$$^a \delta V^E = V^E_{\text{expt}} - V^E_{\text{calc}}$$

TABLE IV Experimental densities, volumes, calculated excess volumes, and deviations δV^E for the system DIPE(1) + cyclohexane (2) at 298.15K

x_1	$\rho/g \cdot cm^3$	$V/$	$V^E/$	$\delta V^E \cdot 10^3/$
		$cm^3 \cdot mol^{-1}$		
0	0.773788	108.764	0.0000	0
0.0474	0.770193	110.380	0.0318	2
0.11081	0.765623	112.532	0.0629	0
0.14226	0.763398	113.602	0.0817	5
0.20468	0.759244	115.705	0.0976	-2
0.2511	0.756228	117.273	0.1130	0
0.30377	0.753026	119.032	0.1110	-13
0.3432	0.750538	120.373	0.1339	5
0.4134	0.746579	122.706	0.1194	-14
0.44676	0.744549	123.848	0.1459	12
0.50291	0.741496	125.722	0.1430	12
0.53809	0.739729	126.879	0.1240	-4
0.59704	0.736646	128.852	0.1260	6
0.64601	0.734222	130.480	0.1159	5
0.68592	0.732295	131.805	0.1069	5
0.75871	0.729014	134.198	0.0654	-17
0.80467	0.72692	135.723	0.0546	-14
0.84563	0.725042	137.093	0.0546	-1
0.90053	0.722574	138.931	0.0563	20
0.92541	0.721689	139.722	0.0160	-12
0.94631	0.720694	140.438	0.0327	13
1	0.718565	142.200	0.0000	0

$$^a \delta V^E = V_{\text{expt}}^E - V_{\text{calc}}^E$$

correlated using the Redlich-Kister expression (1948)

$$V^E = x_1 x_2 \sum_{j=0}^n A_j (x_1 - x_2)^j \quad (2)$$

where the A_j 's are the adjustable parameters of the model.

Use of a polynomial like that of Redlich-Kister to represent the data presents the disadvantage that addition of new terms changes the constants (A_j 's) and hence the possible physical interpretation of the same. For this reason the data have also been correlated using the following series of Legendre's polynomials, $L_j(x_1)$, ($j=0, 1, 2$),

$$V^E = x_1 x_2 [A_0 + A_1(2x_1 - 1) + A_2(6x_1^2 - 6x_1 + 1)] \quad (3)$$

Legendre polynomials present the advantage of orthogonality: increasing the number of members in Eq. (3) change the coefficients very little if at all.

TABLE V Experimental densities, volumes, calculated excess volumes, and deviations δV^E for the system DIPE(1)+methylacetoate(2) at 298.15 K

x_1	$\rho/g \cdot cm^3$	$V/$	$V^E/$	$\delta V^E \cdot 10^3/$
		$mol \cdot cm^{-3}$		
0	0.926831	79.928	0.0000	0
0.0695	0.90219	84.277	0.0190	3
0.1013	0.891998	86.242	0.0034	-21
0.1443	0.878578	88.934	0.0188	-16
0.2006	0.862017	92.477	0.0577	8
0.2501	0.848731	95.562	0.0624	-1
0.2966	0.836805	98.488	0.0883	12
0.333	0.828009	100.769	0.1031	16
0.3966	0.81358	104.752	0.1273	23
0.4494	0.802823	108.003	0.0919	-26
0.4785	0.796778	109.850	0.1242	-1
0.5482	0.783488	114.211	0.1482	9
0.5958	0.77524	117.153	0.1235	-23
0.6383	0.767904	119.828	0.1511	1
0.679	0.7615	122.338	0.1262	-25
0.7377	0.752193	126.045	0.1772	31
0.7986	0.743626	129.799	0.1377	4
0.8498	0.736845	132.945	0.0974	-16
0.8877	0.731791	135.317	0.1111	17
1	0.718565	142.200	0.0000	0

$$\delta V^E = V_{\text{expt}}^E - V_{\text{calc}}^E$$

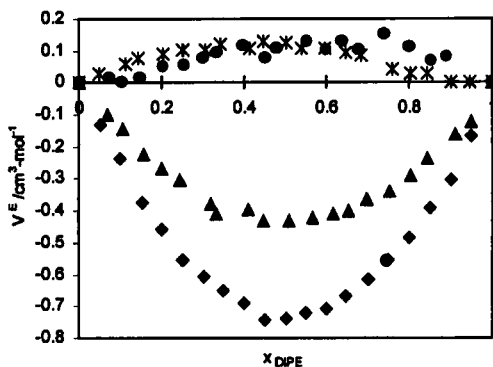


FIGURE 1 Excess volumes at 298.15 K: * DIPE + cyclohexane; Δ DIPE + benzene; \blacklozenge DIPE + toluene; \bullet DIPE + methylacetate.

Equations (3) and (4) were fit using a least-squares optimization procedure, with all points weighted equally and minimizing the following objective function (OF)

$$OF = \sum_1^N (V_{i, \text{expt}}^E - V_{i, \text{calc}}^E)^2 \tag{4}$$

where N is the number of observations. The values of parameters A_j are reported in Table VI for different values of j , together with the pertinent statistics. The standard deviation s was calculated as

$$s = \left[\sum (V_{i, \text{expt}}^E - V_{i, \text{calc}}^E)^2 / (N - k) \right]^{1/2} \tag{5}$$

where k is the number of adjustable parameters. The statistical significance of adding one or more terms after the third, was examined using a χ^2 -based test, coupled to the requirement that the residues be randomly distributed, as suggested by Wisniak and Polishuk (1999).

The values of the function $V^E/x_1 x_2$ at infinite dilution represent the values of the partial excess volume at infinite dilution, $\bar{V}_i^{E, \infty}$ (Van Ness and Abbott, 1982) and can be calculated from the adjustable parameters A_j , as follows:

(a) Redlich-Kister

$$\bar{V}_1^{E, \infty} = A_0 - A_1 + A_2 - \dots \tag{6}$$

$$\bar{V}_2^{E, \infty} = A_0 + A_1 + A_2 + \dots \tag{7}$$

(b) Legendre: $\bar{V}_1^{E, \infty}$ is given by Eq. (6) also, and $\bar{V}_2^{E, \infty}$ by

$$\bar{V}_2^{E, \infty} = A_0 - 2A_1 + A_2 \tag{8}$$

The pertinent values of $V_i^{E, \infty}$ are also shown in Tables VI and VII.

TABLE VI Coefficients A_j (Eq. (2)), standard deviation (Eq. (4)), Durbin-Watson statistic d , $(V^E)_{x=0.5}$, and $\bar{V}_i^{E, \infty}$ at 298.15 K

System	$A_0 \cdot 10^1$	$A_1 \cdot 10^2$	$A_2 \cdot 10^2$	$A_3 \cdot 10^1$	$s \cdot 10^3$	d^a	$(V^E)_{x=0.5} \quad \bar{V}_1^{E, \infty} / \quad \bar{V}_2^{E, \infty} /$		
							$cm^3 \cdot mol^{-1}$		
1+2	-16.79	13.73	11.42	-3.751	8.2	1.94	-0.42	-1.33	-1.80
1+3	-28.59	5.968	6.26	-2.635	9.3	2.35	-0.71	-2.59	-3.00
1+4	5.301	-14.48	-2.423	-0.0531	10.4	2.45	0.13	0.652	0.359
1+5	4.569	32.60	1.398		17.4	2.51	0.11	0.145	0.797

^a $d = \sum_{u=2}^N (e_u - e_{u-1})^2 / \sum_{u=1}^N e_u^2$
 $e_u = V_{u, \text{calc}}^E - V_{u, \text{expt}}^E$

TABLE VII Coefficients A_{ij} (Eq. (7)), standard deviation (Eq. (4)), $(V^E)_{x=0.5}$, and $\bar{V}_1^{E,\infty}$ at 298.15 K

System	$A_0 \cdot 10^1$	$A_1 \cdot 10^2$	$A_2 \cdot 10^2$	$s \cdot 10^3$	$(V^E)_{x=0.5}$		
					$\bar{V}_1^{E,\infty} /$	$\bar{V}_2^{E,\infty} /$	$cm^3 \cdot mol^{-1}$
1+2	-16.39	-13.13	7.809	10.0	-0.42	-1.57	-1.35
1+3	-28.84	2.801	4.152	10.1	-0.71	-2.77	-2.85
1+4	5.219	14.662	-1.633	10.5	0.12	0.65	0.21
1+5	4.615	-32.60	0.931	16.9	0.11	0.14	1.12

$$^a d = \frac{\sum_{i=1}^N (e_i - e_{i-1})^2}{\sum_{i=1}^N e_i^2}$$

$$e_i = \bar{V}_{i,calc}^E - \bar{V}_{i,expt}^E$$

Inspection of the results of Tables II to V and Figure 1 indicates that the excess volumes for the systems of DIPE with benzene and toluene are negative for the whole composition range, while those of DIPE + cyclohexane are positive. The results for cyclohexane and toluene are consistent with those reported in the literature for MTBE, ETBE and TAME, that is, mixtures of branched ethers with cyclohexane expand on mixing, while those with toluene contract. Mixtures of benzene with a branched ether behave in a more complex manner, with MTBE and DIPE they present negative values of V^E while for TAME they are positive. Table V shows that the excess volumes for the system DIPE + methyl ethanoate are very small ($< 1.5 \cdot 10^{-2} \text{ cm}^3 \cdot \text{mol}^{-1}$), a result similar to that obtained by Linek (1995) for the system ethyl propanoate + butyl methyl ether ($V^E < 1.4 \cdot 10^{-4} \text{ mL} \cdot \text{mol}^{-1}$). This is an interesting result because Wisniak *et al.* (2000) measured the vapor–liquid equilibria of the system and found that it deviates strongly from ideal behavior, presenting a minimum boiling azeotrope at 327.9 K and containing 78% mole methyl ethanoate.

As regards to the symmetry of the excess function, it appears that a symmetric $V^E(x)$ function is observed for all the systems studied here. The curves in Figure 1 show the presence of a clear maximum. The maximum becomes less positive and always centered at $x \approx 0.5$ confirming that the maximum specific interaction occurs at about equimolar composition.

The total volume change in mixing is made up of at least two contributions: (a) a positive one due to the break-up of the structure of one or both components (originating from non-chemical or chemical interactions such as hydrogen bonding or complex-forming interactions) and (b), a negative one due to physical interactions or geometric

fitting of one component into the second, leading to a more efficient packing. The second contribution should be more predominant the more spherical the molecule of the solute is and the higher the molar volume of the solvent.

Structurally DIPE may be considered the homomorph of 2,4 dimethylpentane, aprotic and almost non-polar. Interactions between a polar component and such an ether are considered to occur *via* complex formation between the two species or $n-\pi$ interaction (Sharma, 1992).

The magnitude and sign of V^E is a reflection of the type of interactions taking place in the mixture and is very well exhibited by the mixtures studied here, with $(V^E)_{x=0.5}$ ranging from +0.1 to $-0.71 \text{ cm}^3/\text{mol}$ (the pertinent value of V^E is given by $A_0/4$).

For the systems studied here the V^E curves are either positive or negative, depending on the nature of the solvent, and most of them are symmetrical at equimolar compositions. The DIPE + benzene and benzene + toluene systems show negative deviations from ideality, indicating a net packing effect. The overall magnitude of V^E is a result of the effect of breaking the ether's dipole-dipole association. Interactions between an aromatic hydrocarbon and an ether have been described as much stronger than those between a cyclic hydrocarbon and an ether.

In contrast with benzene and toluene the system DIPE + cyclohexane presents excess volumes that are positive for the whole composition range. This fact can be explained with a decrease in packing in the ether (due to dipole-dipole interaction) caused by the cyclohexane coming in between the molecules. In our case the solute has a strong steric interaction with the ether, and therefore the V^E value is always positive.

The observed molar excess volumes exhibit an interesting dependence with respect to the structure of the branched ether. Figures 2 to 4 compare the values of V^E for solutions of benzene, toluene and cyclohexane with the branched ether MTBE, ETBE, TAME and DIPE. The first three ethers are asymmetric whilst the latter is symmetric around the oxygen atom. Figure 1 shows that the interactions between an aromatic hydrocarbon (benzene, toluene) are much stronger than with a cyclic hydrocarbon, and result in a change of sign for V^E , from negative for benzene and toluene, to positive

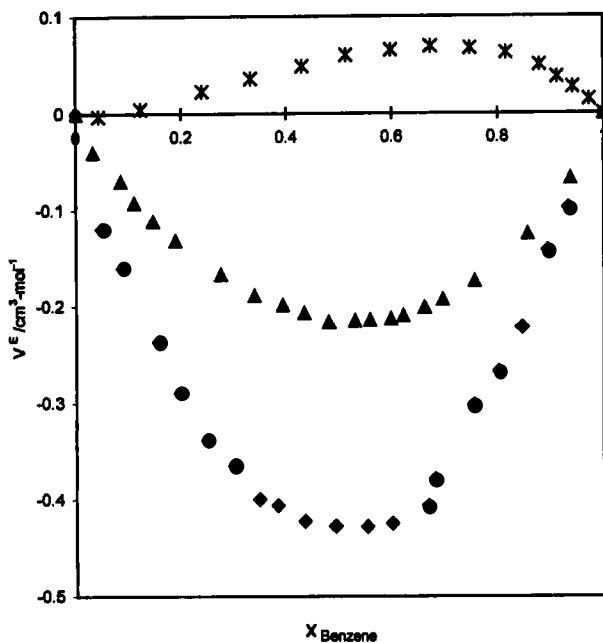


FIGURE 2 Effect of structure on V^E : Δ MTBE + benzene (Sharma, 1972); \blacklozenge DIPE + benzene (this work); $*$ TAME + benzene (Linek, 1987).

for cyclohexane. The inductive action of the methyl group in toluene enhances the $n-\pi$ interaction resulting in mixing volumes larger than for benzene.

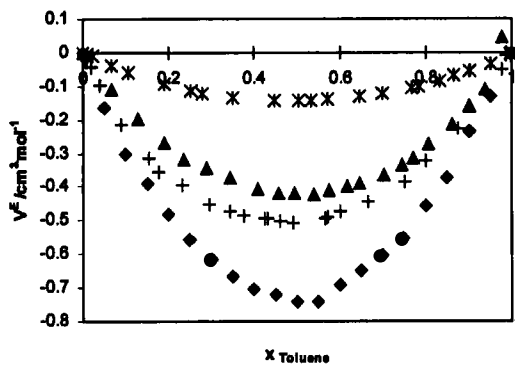


FIGURE 3 Effect of structure on V^E : Δ MTBE + toluene (Sharma, 1992); \blacklozenge DIPE + toluene (this work); $+$ ETBE + toluene (Domanska, 1996); $*$ TAME + toluene (Domanska, 1996).

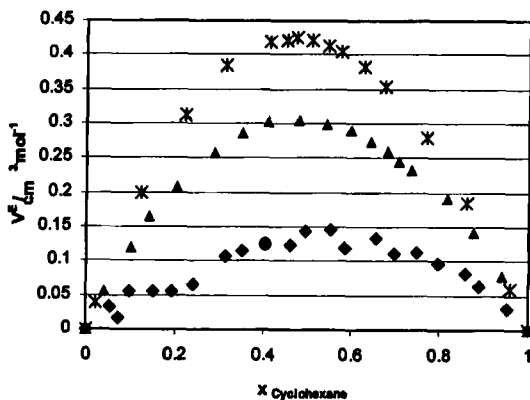


FIGURE 4 Effect of structure on V^E : Δ MTBE + cyclohexane (Sharma, 1992); \blacklozenge DIPE + cyclohexane (this work); * TAME + cyclohexane (Witek, 1997).

Figures 2 and 3 show more clearly the effect of the lateral branch of the ether. With benzene (Fig. 2) the symmetric ether (DIPE) allows for the largest $n-\pi$ interaction; with TAME the steric effect of the *t*-amyl group is large enough to block completely this interaction and switch the mode from chemical to physical. With toluene (Fig. 3) as a solvent the overall behavior is similar to that with benzene, but the activation of the ring caused by the inductive effect of the methyl group is large enough to retain part of the $n-\pi$ interaction, in spite of the size of the lateral branch of the ether.

The overall positive magnitude of V^E in mixtures with cyclohexane is a result of breaking to different degrees the packing of pure cyclohexane. As shown in Figure 4, the longer and the more branched the lateral ether the radical (from MTBE to TAME) the larger the disruption and the larger the value of V^E .

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