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## EXCESS MOLAR VOLUMES OF TETRAHYDROFURAN WITH ETHYL ACRYLATE, BUTYL ACRYLATE, METHYL METHACRYLATE, AND STYRENE AT 298.15 K

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Densities of the binary systems of tetrahydrofuran (THF) with ethyl acrylate, butyl acrylate, methyl methacrylate, and styrene 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 densimeter. The calculated excess volumes were correlated with the Redlich–Kister equation and with a series of Legendre polynomials. The excess volumes are negative for all the systems studied. The system THF + methyl methacrylate behaves almost ideally.

*Keywords:* Densities; Excess volumes; Monomers; Tetrahydrofuran

### INTRODUCTION

The mixing of different compounds gives rise to different excess properties, which reflect the extent of the deviations from nonideality. Excess thermodynamic properties of mixtures are defined as the difference between the actual value of the property and the value it would have if the solution behaved ideally. Excess properties are useful in the study of molecular interactions and can be used to calculate other properties such as the activity coefficient and the conditions for phase separation.

This work is part of our program to provide data for the characterization of the molecular interactions between solvents and commercially important monomers, in particular the influence of the chemical structure of the solute in the systems under consideration.

Sastry and Dave measured the excess volumes, isentropic compressibilities, and dielectric behavior of fifteen binary mixtures of alkyl (methyl, ethyl, and butyl) methacrylates with hexane, heptane, carbon tetrachloride, chlorobenzene, and

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*o*-dichlorobenzene at 308.15 K and found that with aliphatic hydrocarbons the results were controlled by dispersing interactions while with chlorinated solvents the controlling factors were specific interactions (O–Cl and  $n$ – $\pi$  types) [1,2]. Sastry and Valand also measured the excess volumes of mixtures of alkyl (methyl, ethyl, and butyl) acrylates in several alkanols at 298.15 and 308.15 K, and found that they were always positive. These results were explained on the basis of nonspecific interactions between the components [3].

No literature data are available on the excess volumes of the systems analyzed in this work.

## EXPERIMENTAL SECTION

### Materials

Tetrahydrofuran, THF (HPLC grade, 99.9+ mass%), ethyl acrylate, EA (99+ mass%), butyl acrylate, BA (99+ mass%), methyl methacrylate, MMA (99+ mass%), and styrene (99 mass%), were purchased from Aldrich and vacuum distilled previous to use to eliminate the stabilizer (about 0.002% mass of hydroquinone monomethyl ether). Styrene, containing 10–15 ppm of 4-*tert*-butylcatechol as stabilizer, was not distilled to avoid polymerization but was degassed by freezing and heating. The purity of the solvents was further ascertained by comparing their densities at 298.15 K with the values reported in the literature (Table I).

### Density Measurements

The density of the samples was measured with an Anton Paar model DMA 5000 oscillating U-tube densimeter, provided with automatic viscosity correction, two integrated Pt 100 platinum thermometers (DKD traceable), and a stated accuracy of  $5 \times 10^{-6} \text{ g} \cdot \text{cm}^{-3}$ . 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 or heated to remove dissolved air. Solutions of different compositions were prepared by mass in a  $10 \text{ cm}^3$  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  $5 \times 10^{-5}$ , of the density (duplicate) measurement  $\pm 2 \times 10^{-6} \text{ g} \cdot \text{cm}^{-3}$ , and of the temperature  $\pm 0.002 \text{ K}$ .

Proper safety measures were taken when handling all the materials.

TABLE I Purity and densities of pure components at 298.15 K

Component	Purity (mass%)	Density ( $\text{g} \cdot \text{cm}^{-3}$ )	
		Measured	Literature
THF (1)	99.9+	0.882592	0.88193 [9]
Butyl acrylate (2)	99+	0.893666	0.8941 [3]
Ethyl acrylate (3)	99	0.915930	0.9163 [3]
Methyl methacrylate (4)	99	0.937669	0.93766 [10]
Styrene (5)	99	0.901972	0.9016 [11]

## RESULTS AND DISCUSSION

At least 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 mole fraction  $x$  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  $\rho$ ,  $\rho_1$ , and  $\rho_2$  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  $\rho$  and  $V^E$  are reported in Tables II–V and Fig. 1. The values of  $V^E$  were correlated with composition using two procedures;

(a) The Redlich–Kister expression [4]

$$V^E = x_1x_2 \sum_{k=0}^n A_k(x_1 - x_2)^k \quad (2)$$

where the  $A_k$ 's are the adjustable parameters of the model.

The Redlich–Kister regressor is very powerful and frequently used to correlate vapor–liquid equilibrium data and excess properties. Notwithstanding, it suffers from the important drawback that the values of its adjustable parameters change as the number of terms in the series is increased.

TABLE II Experimental densities, volumes, calculated excess volumes, and deviations  $\delta V^E$  for the system THF (1) + butyl acrylate (2) at 298.15 K

$x_1$	$\rho$ (g · cm <sup>3</sup> )	$V \times 10^{-2}$	$V^E \times 10^2$	$\delta V^E \times 10^3$
			(mol cm <sup>-3</sup> )	
0	0.893666	1.4342	0.0000	0
0.0522	0.893492	1.4017	-2.5606	0
0.0997	0.893276	1.3723	-4.1014	0
0.1505	0.893018	1.3408	-5.5110	-1
0.2015	0.892713	1.3092	-6.4340	1
0.2496	0.89243	1.2794	-7.5714	-1
0.3001	0.892084	1.2481	-8.2918	1
0.3502	0.89173	1.2172	-9.0819	1
0.3999	0.891359	1.1864	-9.8609	-1
0.4500	0.890942	1.1554	-10.3435	0
0.5001	0.890493	1.1245	-10.7011	0
0.5509	0.889994	1.0931	-10.8574	0
0.6007	0.889452	1.0624	-10.7263	1
0.6509	0.888868	1.0315	-10.5184	0
0.7008	0.88823	1.0007	-10.0876	0
0.7500	0.887558	0.9704	-9.6070	-1
0.8001	0.886782	0.9396	-8.6251	0
0.8502	0.885938	0.9087	-7.4943	0
0.9002	0.884994	0.8780	-5.9344	-1
0.9499	0.883884	0.8476	-3.3908	1
1	0.882592	0.8170	0.0000	0

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

TABLE III Experimental densities, volumes, calculated excess volumes, and deviations  $\delta V^E$  for the system THF (1) + ethyl acrylate (3) at 298.15 K

$x_1$	$\rho$ (g · cm <sup>3</sup> )	$(\text{mol} \cdot \text{cm}^{-3})$		
		$V \times 10^{-1}$	$V^E \times 10^2$	$\delta V^E \times 10^3$
0.0000	0.915930	10.93	0.000	0
0.0681	0.914296	10.74	-1.090	-1
0.1019	0.913439	10.65	-1.334	1
0.1521	0.912156	10.51	-1.933	1
0.2010	0.910878	10.37	-2.563	-1
0.2562	0.909364	10.22	-2.929	0
0.3080	0.907908	10.08	-3.337	0
0.3504	0.906698	9.960	-3.802	-2
0.4003	0.905186	9.822	-3.836	1
0.4499	0.903674	9.685	-4.190	0
0.4998	0.902099	9.547	-4.459	-1
0.5502	0.900420	9.408	-4.307	2
0.6000	0.898756	9.270	-4.613	-1
0.6500	0.896990	9.132	-4.461	0
0.7002	0.895159	8.994	-4.287	0
0.7501	0.893273	8.856	-3.987	1
0.8020	0.891226	8.713	-3.502	1
0.8500	0.889275	8.581	-3.038	0
0.9001	0.887157	8.444	-2.414	-1
0.9504	0.884923	8.306	-1.467	-2
1.0000	0.882592	8.170	0.000	0

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

TABLE IV Experimental densities, volumes, calculated excess volumes, and deviations  $\delta V^E$  for the system THF (1) + methyl methacrylate (4) at 298.15 K

$x_1$	$\rho$ (g · cm <sup>-3</sup> )	$(\text{cm}^3 \cdot \text{mol}^{-1})$		
		$V \times 10^{-2}$	$V^E \times 10^2$	$\delta V^E \times 10^3$
0	0.937669	1.068	0.000	0
0.0495	0.935679	1.055	-1.369	0
0.1000	0.933475	1.043	-1.343	0
0.1537	0.931019	1.029	-0.7805	0
0.2008	0.928807	1.017	-0.2168	0
0.2503	0.926446	1.005	0.1950	0
0.3003	0.92415	0.9924	-1.020	0
0.3510	0.921674	0.9796	-1.318	0
0.4004	0.919106	0.9673	-0.6376	0
0.4498	0.916604	0.9548	-1.347	0
0.5008	0.913881	0.9420	-1.357	0
0.5004	0.913843	0.9422	-7.538	0
0.5501	0.911166	0.9297	-1.206	0
0.5501	0.911166	0.9297	-1.206	0
0.6002	0.908409	0.9171	-1.850	0
0.6499	0.905469	0.9047	-1.214	0
0.7005	0.90247	0.8920	-1.313	0
0.7507	0.899397	0.8794	-1.312	0
0.8001	0.896322	0.8670	-1.699	0
0.8500	0.89308	0.8545	-1.617	0
1	0.882592	0.8170	0.000	0

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

TABLE V Experimental densities, volumes, calculated excess volumes, and deviations  $\delta V^E$  for the system THF (1) + styrene (5) at 298.15 K

$x_1$	$\rho$ ( $\text{g} \cdot \text{cm}^{-3}$ )	$V$	$V^E$	
			(mol $\cdot$ cm $^{-3}$ )	
0	0.901972	1.1547	0.0000	0
0.0500	0.902000	1.1369	-0.9130	-6
0.1032	0.901861	1.1182	-1.6744	5
0.1506	0.901797	1.1014	-2.4293	4
0.2005	0.901740	1.0838	-3.2406	-5
0.2499	0.901511	1.0665	-3.8423	1
0.3004	0.901291	1.0488	-4.4819	-5
0.3504	0.900894	1.0314	-4.9189	0
0.4000	0.900433	1.0143	-5.2908	2
0.4499	0.899863	0.9972	-5.5670	2
0.5007	0.899149	0.9799	-5.7236	3
0.5501	0.898333	0.9632	-5.7719	2
0.6006	0.897378	0.9461	-5.7252	-3
0.6505	0.896228	0.9296	-5.4990	-2
0.6503	0.896181	0.9297	-5.4471	3
0.7000	0.894975	0.9131	-5.2039	-9
0.7502	0.893400	0.8967	-4.6465	-3
0.8001	0.891596	0.8806	-3.9146	6
0.8500	0.889671	0.8646	-3.1302	6
0.9000	0.887558	0.8485	-2.2392	3
0.9250	0.886451	0.8406	-1.7737	-2
0.9500	0.885246	0.8327	-1.2358	-3
0.9749	0.883985	0.8248	-0.6665	-5
1	0.882592	0.8170	0.0000	0

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

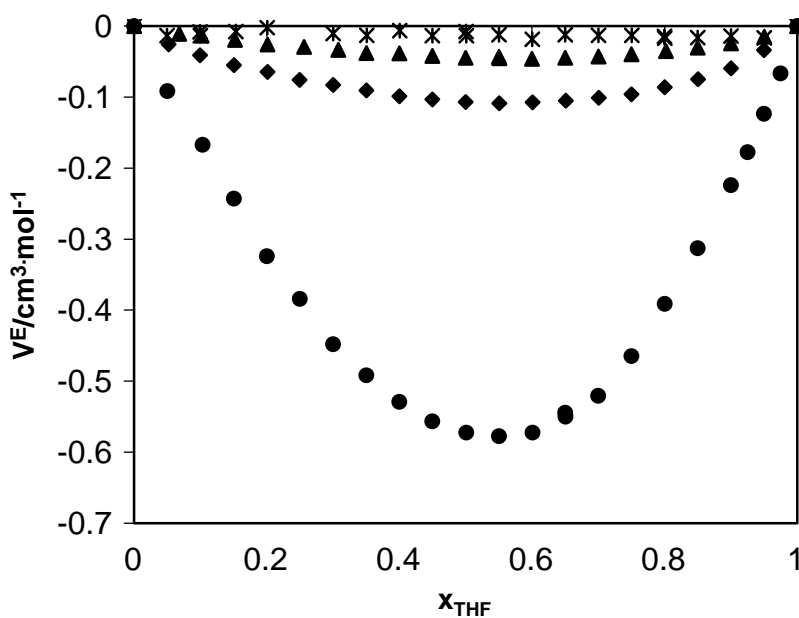


FIGURE 1 Excess volumes at 298.15 K: \* THF + MMA;  $\blacktriangle$  THF + EA;  $\blacklozenge$  THF + BA;  $\bullet$  THF + styrene.

(b) A series of Legendre polynomials  $L_k(x_1)$

$$V^E = x_1 x_2 \sum_{k=1}^N a_k L_k(x_1) \quad (3)$$

which for the three first terms ( $k=0, 1, 2$ ) becomes

$$V^E = x_1 x_2 [a_0 + a_1(2x_1 - 1) + a_2(6x_1^2 - 6x_1 + 1)] \quad (4)$$

Legendre polynomials belong to the category of orthogonal functions such as Fourier, Bessel, and Chebyshev, which have the important characteristic that for a continuous series of observations (infinite) the values of the coefficients do not change as the number of terms in the series is increased. This is an important property because if a physical explanation can be assigned to one of its coefficients, its value remains constant. For the case of discrete measurements, such as determination of volumes of mixing, the values of the coefficients will vary, but slightly.

The series of Legendre polynomials have the additional important characteristic that the structure of its first three terms is the same as that of the first three terms of the Redlich–Kister expansion.

Equations (2) and (3) were fitted using a least-squares optimization procedure, with all points weighted equally and minimizing the following objective function (OF)

$$\text{OF} = \sum_1^N (V_{i, \text{expt}}^E - V_{i, \text{calc}}^E)^2 \quad (5)$$

where  $N$  is the number of observations. The values of the different adjustable parameters,  $A_k$  and  $a_k$ , are reported in Tables VI and VII for different values of  $k$ , together

TABLE VI Coefficients  $A_k$  (Eq. (2)), standard deviation (Eq. (6)), Durbin–Watson statistic  $d$ ,  $(V^E)_{x=0.5}$ , and  $\bar{V}_i^{E,\infty}$  at 298.15 K

System	$A_0$	$A_1$	$A_2$	$A_3$	$A_4$	$s \times 10^3$	$d^a$	$(\bar{V}^E)_{x=0.5}$	$\bar{V}_1^{E,\infty}$	$\bar{V}_2^{E,\infty}$
								$(\text{cm}^3 \cdot \text{mol}^{-1})$		
1+2	-0.4270	-0.1020	-0.04118	-0.02705	-0.2503	0.7	3.04	-0.107	-0.59	-0.848
1+3	-0.1740	-0.06032	-0.04861			1.05	2.12	-0.043	-0.1623	-0.283
1+4										
1+5	-2.301	-0.4095	0.1747			4.3	1.82	-0.575	-1.717	-2.536

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

TABLE VII Coefficients  $a_k$  (Eq. (3)), standard deviation (Eq. (6)),  $(V^E)_{x=0.5}$ , and  $\bar{V}_i^{E,\infty}$  at 298.15 K

System	$a_0$	$a_1$	$a_2$	$a_3$	$a_4$	$s \times 10^3$	$d^a$	$(\bar{V}^E)_{x=0.5}$	$\bar{V}_1^{E,\infty}$	$\bar{V}_2^{E,\infty}$
								$(\text{cm}^3 \cdot \text{mol}^{-1})$		
1+2	-0.4908	-0.1182	-0.1705	-0.01082	-0.0572	0.74	3.04	-0.107	-0.59	-0.848
1+3	-0.1902	-0.06032	-0.03241			1.08	2.12	-0.0435	-0.162	-0.283
1+4										
1+5	-2.252	-0.4044	0.0988				4.6	-0.575	-1.749	-2.557

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

with the pertinent statistics. The standard deviation  $s$  was calculated as

$$s = \left[ \sum (V_{i, \text{expt}}^E - V_{i, \text{calc}}^E)^2 / (N - k) \right]^{1/2} \quad (6)$$

where  $k$  is the number of adjustable parameters. The statistical significance of adding one or more terms after the third, was examined using a  $\chi^2$ -based test, coupled to the requirement that the residues be randomly distributed, as suggested by Wisniak and Polishuk [5]. It was not deemed necessary to perform a step-wise regression.

The values of the function  $V^E/x_1x_2$  at infinite dilution represent the values of the partial excess volume at infinite dilution,  $\bar{V}_i^{E, \infty}$  [6] and can be calculated from the adjustable parameters  $A_k$  and  $a_k$  as follows:

(a) Redlich–Kister

$$\bar{V}_1^{E, \infty} = A_0 - A_1 + A_2 - \dots = \bar{V}_1 - V_1^0 \quad (7)$$

$$\bar{V}_2^{E, \infty} = A_0 + A_1 + A_2 + \dots = \bar{V}_2 - V_2^0 \quad (8)$$

(b) Legendre

$$\bar{V}_1^{E, \infty} = a_0 - a_1 + a_2 - \dots = \bar{V}_1 - V_1^0 \quad (9)$$

$$\bar{V}_2^{E, \infty} = a_0 + a_1 + a_2 + \dots = \bar{V}_2 - V_2^0 \quad (10)$$

where  $V_i^0$  is the molar volume of pure component  $i$ . The pertinent values of  $\bar{V}_i^{E, \infty}$  are also shown in Table VI. The same values of  $\bar{V}_i^{E, \infty}$  are obtained when using either Eqs. (7) and (8) or (8) and (9).

Figure 2 shows a typical distribution of the residuals, which is random as declared by the Durbin–Watson statistic.

Inspection of the results of Tables II–V and Fig. 1 indicates that the excess volumes for the four systems THF + monomer are negative for the whole composition range and, in addition, the system THF + methyl methacrylate behaves almost ideally. The results for THF and styrene are consistent with those reported in the literature for the systems of cyclohexane and toluene with MTBE, ETBE, and TAME, that is, mixtures of branched ethers with cyclohexane expand on mixing, while those with toluene contract.

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 Fig. 1 show the presence of a clear minimum. The minimum becomes more negative 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 nonchemical or chemical interactions such as hydrogen bonding or



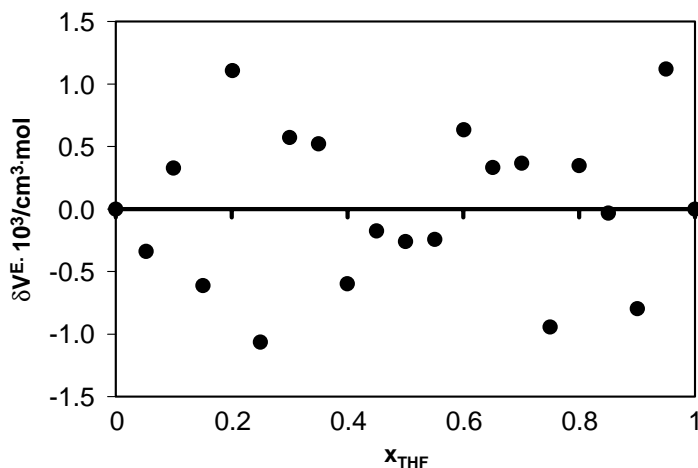


FIGURE 2 Residual distribution plot for the system THF + BA according to the fit given in Table VI.

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 THF may be considered the homomorph of cyclopentane, aprotic, and almost nonpolar. Interactions between a polar component and such ether are considered to occur via complex formation between the two species, or  $n-\pi$  interaction [7].

The magnitude and sign of  $V^E$  is a reflection of the type of interactions taking place in the mixture. This is very well exhibited by the mixtures studied here, with  $(V^E)_{x=0.5}$  ranging from about  $-0.1$  to  $-0.58 \text{ cm}^3/\text{mol}$  (the pertinent value of  $V^E$  is given by  $A_0/4$ ).

For the systems studied here the  $V^E$  curves are all negative, the relative intensity depending on the nature of the solute (monomer) and of the solvent. While the overall magnitude of  $V^E$  is a result of the effect of breaking the ether's dipole-dipole association, the negative sign indicates a net packing effect contributed by structural effects arising from interstitial accommodation. These results are similar to those obtained by Pal and Kumar [8] for the excess volumes of mixtures of polyethers and ester. In addition, the curves are almost symmetrical, reflecting lack of homoassociation of each of the components.

Interactions between an aromatic hydrocarbon (such as styrene) and an ether have been described as much stronger than those between a cyclic hydrocarbon (such as cyclohexane) and an ether. For example, the inductive action of the methyl group in toluene enhances the  $n-\pi$  interaction and leads to mixing volumes larger than for benzene. The large negative values for the system THF + styrene indicate that the disruption of the dipolar association of the cyclic ether is considerably less than the additional specific interaction between its pairs of electrons and the  $\pi$  electrons of the aromatic ring. The substitution of a vinyl group on the benzene ring may prevent specific interactions because of the increased bulkiness of the branch.

The observed molar excess volumes exhibit an interesting dependence with respect to the structure of the unsaturated ester: their values increase as the length of the ester chain increases from methyl to butyl, signaling an increase of the contribution of the interstitial accommodation.

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