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Excess Enthalpy, Density, Speed of Sound, and Viscosity for 2-Methyltetrahydrofuran + 1-Butanol at (283.15, 298.15, and, 313.15) K

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Excess molar enthalpies, densities, speeds of sound, and viscosities at the temperatures (283.15, 298.15, and 313.15) K have been measured for the binary mixture 2-methyltetrahydrofuran + 1-butanol. From the experimental data, excess molar volumes, excess isentropic compressibilities, excess speeds of sound, and viscosity deviations have been calculated. These excess properties and deviations have been fitted to Redlich−Kister equations.

Introduction

Excess thermodynamic properties and deviations of properties for mixtures of liquids have both practical and theoretical interest: They have to be known to design industrial processes properly. They can be used to develop models that allow us to predict other properties. Finally, they can also reveal the existence of specific molecular interactions.

1-Butanol[1,2] is an aliphatic alcohol that is extensively used in different fields such as the pharmaceutical industry[3] as a solvent or extractant agent, in clothing and plastics manufacture, and in wastewater treatment.[4] It is also frequent in theoretical studies about hydrogen bond and structural effects in the excess thermodynamic properties.[5,6]

In this last respect, the knowledge of the behavior of mixtures of 1-butanol with other compounds carrying oxygen atoms is of interest. In a previous paper,[7] we reported a study of some thermodynamic and transport properties for the mixture tetrahydropyran + 1-butanol. This one presents a similar study of the same or similar properties for the mixture 2-methyltetrahydrofuran + 1-butanol. Tetrahydropyran and 2-methyltetrahydrofuran are isomeric cyclic ethers, but the last has a ring of five carbon atoms instead of a ring of six carbon atoms and shows a less symmetrical structure due to the presence of a branching methyl group. Our aim is to compare the effect of this loss of symmetry in the excess properties of these mixtures.

Experimental Section

Materials. The compounds used were 2-methyltetrahydrofuran and 1-butanol, both from Aldrich (purities > 99 %). The claimed purities of these liquids were verified by gas−liquid chromatography (HP 6890) using a flame ionization detector. Experimental values of density, viscosity, and speed of sound for the pure components at the three temperatures are reported in Table 1 along with the available bibliographic values.[8−16]

Apparatus and Procedure. The excess molar enthalpies (H_m^E) were determined with a Thermometric 2277 thermal activity monitor maintained at ± 0.0002 K that operates under constant-flow conditions. Two Shimadzu LC-10ADVP HPLC pumps were used to drive the liquids into the calorimeter. The pumps were calibrated for each liquid in order to relate the real flow to the programmed one. The uncertainty in the mole fractions of the mixtures calculated from the uncertainty in the flow delivered by the pumps was estimated to be ± 0.001. The calibration of the calorimeter was achieved with reference to the recommended values[17] of the mixture hexane + cyclohexane. The deviation lies within ± 1 %, so the uncertainty in the determination of excess enthalpies is estimated as ± 1 % as well.

Densities (ρ) and speeds of sound (u) of the pure compounds and their mixtures were determined with a vibrating-tube densimeter and sound analyzer Anton Paar DSA48 automatically thermostated within ± 0.01 K. The corresponding uncertainties are ± 0.00001 $\text{g}\cdot\text{cm}^{-3}$ for densities and ± 0.1 $\text{m}\cdot\text{s}^{-1}$ for speeds of sound, respectively.

Kinematic viscosities (ν) were measured using a capillary Ubbelohde viscometer connected to a Schott-Geräte AVS-440 unit, whose uncertainty in the flow time measurement is ± 0.01 s. The temperature was kept constant with ± 0.01 K by means of a Schott-Geräte thermostat. The corresponding uncertainty in the kinematic viscosity was ± 0.002 $\text{mm}^2\cdot\text{s}^{-1}$. At least four flow time measurements differing each other not more than 0.05 % were performed for each composition and temperature, and the results were averaged. Next, together with densities previously determined, dynamic viscosities were calculated ($\eta = \nu\cdot\rho$). The uncertainties in dynamic viscosities are about ± 0.002 mPa·s.

The composition of the binary mixtures used to measure densities, speeds of sound, and viscosities was determined by mass using a Sartorius analytical balance with a precision of ± 10^{-5} g. Hence the uncertainty in the mole fraction was estimated to be less than ± 0.0001.

Results and Discussion

Excess molar volumes (V_m^E) were calculated from the density of the mixture (ρ), densities (ρ_i), and molar masses (M_i) of the pure compounds, and the corresponding molar fractions (x_i) by means of the equation:

$$V_m^E/\text{m}^3\cdot\text{mol}^{-1} = x_1M_1\left(\frac{1}{\rho} - \frac{1}{\rho_1}\right) + x_2M_2\left(\frac{1}{\rho} - \frac{1}{\rho_2}\right) \qquad (1)$$

Isentropic and excess isentropic compressibilities were estimated from densities (ρ) and speeds of sound (u) by using the following

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Table 1. Densities (ρ), Viscosities (η), and Speeds of Sound (u) of Pure Compounds at (283.15, 298.15, and 313.15) K: Comparison with Literature Data

		ρ/kg·m^{-3}		η/mPa·s		u/m·s^{-1}	
T/K		2-methyl-tetrahydrofuran	1-butanol	2-methyl-tetrahydrofuran	1-butanol	2-methyl-tetrahydrofuran	1-butanol
283.15	exptl	864.17	816.92	0.5618	3.8690	1267.6	1292.1
	lit	864.99^{b}	817.0^{a}	0.5625^{b}	3.87^{a}		
298.15	exptl	849.04	805.73	0.4776	2.5609	1202.2	1242.9
	lit	849.27^{c}	806.0^{a}	0.4750^{c}	2.58^{a}		1239.39^{f}
		848.52^{d}	805.75^{e}	0.4753^{d}	2.5710^{e}		1239.3^{g}
			805.56^{c}		2.5720^{c}		1241^{h}
			805.6^{g}		2.5647^{i}		
313.15	exptl	833.76	794.38	0.4088	1.7567	1129.9	1186.3
	lit	834.01^{c}	794.6^{a}	0.4100^{c}	1.78^{a}		1189.5^{g}
		833.65^{j}	794.1^{g}	0.4060^{j}	1.7574^{i}		1190^{h}

[a] Ref 8. [b] Extrapolated from ref 9. [c] Ref 10. [d] Ref 11. [e] Ref 12. [f] Ref 13. [g] Ref 14. [h] Ref 15. [i] Ref 16. [j] Interpolated from ref 9.

Table 2. Isentropic Compressibilities (κ_S), Molar Volumes (V), Thermal Expansion Coefficients (α), and Molar Heat Capacities (C_p) of Pure Compounds at (283.15, 298.15, and 313.15) K

property	T/K	2-methyl-tetrahydrofuran	1-butanol
$\kappa_{S,i} \times 10^{12}$/Pa^{-1} ^{a}	283.15	720.17	733.21
	298.15	814.93	803.41
	313.15	939.46	894.50
$V_i \times 10^6$/m^3·mol^{-1} ^{a}	283.15	99.67	90.73
	298.15	101.4	91.99
	313.15	103.3	93.31
$\alpha_i \times 10^3$/K^{-1} ^{a}	283.15	1.139	0.875
	298.15	1.170	0.909
	313.15	1.203	0.958
$C_{p,i}$/J·mol^{-1}·K^{-1}	283.15	152.21^{b}	167.68^{c}
	298.15	156.89^{b}	177.32^{c}
	313.15	161.60^{b}	189.08^{c}

[a] Calculated from experimental data. [b] Ref 19. [c] Ref 8.

Table 3. Excess Molar Enthalpies for the Mixture 2-Methyltetrahydrofuran (1) + 1-Butanol (2) at (283.15, 298.15, and 313.15) K

	H_m^E/J·mol $^{-1}$		
x_1	T/K = 283.15	T/K = 298.15	T/K = 313.15
0.054	99	110	116
0.104	206	220	234
0.206	385	425	455
0.308	548	587	632
0.411	646	694	748
0.510	695	748	806
0.609	693	746	803
0.708	631	678	728
0.806	505	541	578
0.907	291	310	329
0.953	159	170	179

equations:

$$\kappa_S/\text{Pa}^{-1} = \frac{1}{\rho \cdot u^2} \tag{2}$$

$$\kappa_S^E/\text{Pa}^{-1} = \kappa_S - \kappa_S^{\text{id}} \tag{3}$$

where κ_S^{id} was obtained according to Benson and Kiyohara:[18]

$$\kappa_S^{\text{id}} = \sum_i \phi_i \left\{ \kappa_{S,i} + \frac{TV_i(\alpha_i)^2}{C_{p,i}} \right\} - T\left(\sum_i x_i V_i\right) \frac{\left(\sum_i \phi_i \alpha_i\right)^2}{\left(\sum_i x_i C_{p,i}\right)} \tag{4}$$

where ϕ_i is the volume fraction of component i in the mixture referred to the unmixed stated; x_i is the corresponding mole fraction; T is the absolute temperature; and $\kappa_{S,i}$, V_i, α_i, and $C_{p,i}$ are the isentropic compressibility, the molar volume, the thermal expansion coefficient, and the molar heat capacity of the pure component (i), respectively. All these properties of the pure substances are listed in Table 2.

Excess speed of sound (u^E) was obtained according to Junquera et al.:[20]

$$u^E/\text{m}\cdot\text{s}^{-1} = u - u^{\text{id}} \tag{5}$$

$$u^{\text{id}} = (\rho^{\text{id}} \kappa_S^{\text{id}})^{-1/2} \tag{6}$$

$$\rho^{\text{id}} = \frac{(x_1 M_1 + x_2 M_2)}{(x_1 V_1 + x_2 V_2)} \tag{7}$$

Viscosity deviations ($\Delta\eta$) were calculated from the viscosity of the mixture (η) and viscosities of the pure compounds (η_i) by using

$$\Delta\eta = \eta - x_1\eta_1 - x_2\eta_2 \tag{8}$$

The excess molar enthalpies are reported in Table 3. The densities and excess molar volumes are gathered in Table 4. The speeds of sound, excess isentropic compressibilities, and excess speeds of sound are reported in Table 5. The dynamic viscosities and its deviations are gathered in Table 6. All the excess properties and deviations of the property at (283.15, 298.15, and 313.15) K are graphically represented in Figures 1 to 5. They have been fitted to the Redlich−Kister equation:[21]

$$Y^E \text{ (or } \Delta Y) = x_1 x_2 \sum_{i=1}^{p} A_i (x_1 - x_2)^{i-1} \tag{9}$$

where Y^E denotes the excess property, ΔY denotes the deviation of the property in each case, and A_i are the fitting coefficients determined by the least-squares method. The parameters obtained together with the standard deviations (σ) calculated with eq 4 are summarized in Table 7:

$$\sigma = \left[\frac{\sum (Y_{\text{exptl}}^E - Y_{\text{cal}}^E)^2}{N - p} \right]^{1/2} \tag{10}$$

where N and p are the number of experimental points and parameters, respectively.

Some experimental data on viscosities and densities have already been reported for this system at (298.15 and 313.15) K.[10] For the dynamic viscosities, the mean relative deviation between our data and the literature ones were better than 1.3 %. The densities reported in this work are more suitable for calculating excess molar volumes because of their better fit to eq 9 that provides standard deviations four times lower.

Table 4. Densities and Excess Molar Volumes for the Mixture 2-Methyltetrahydrofuran (1) + 1-Butanol (2) at (283.15, 298.15, and 313.15) K

T/K = 283.15			T/K = 298.15			T/K = 313.15		
x_1	ρ / $\text{kg}\cdot\text{m}^{-3}$	$10^6 \times V_m^E$ / $\text{m}^3\cdot\text{mol}^{-1}$	x_1	ρ / $\text{kg}\cdot\text{m}^{-3}$	$10^6 \times V_m^E$ / $\text{m}^3\cdot\text{mol}^{-1}$	x_1	ρ / $\text{kg}\cdot\text{m}^{-3}$	$10^6 \times V_m^E$ / $\text{m}^3\cdot\text{mol}^{-1}$
0.0539	820.03	−0.037	0.1020	811.16	−0.070	0.1038	799.46	−0.071
0.1047	822.92	−0.069	0.2068	816.38	−0.113	0.2098	804.34	−0.120
0.2068	828.51	−0.120	0.3108	821.37	−0.145	0.3139	808.86	−0.148
0.3108	833.90	−0.149	0.4152	826.08	−0.155	0.4135	813.00	−0.163
0.4147	839.04	−0.162	0.4848	829.16	−0.160	0.4906	816.02	−0.159
0.4893	842.56	−0.160	0.6191	834.74	−0.142	0.6175	820.80	−0.142
0.6195	848.41	−0.135	0.7245	838.88	−0.111	0.7256	824.59	−0.105
0.7240	852.97	−0.111	0.8157	842.28	−0.072	0.8172	827.71	−0.070
0.8923	859.92	−0.049	0.8911	845.05	−0.039	0.8909	830.17	−0.041

Table 5. Speeds of Sound, Excess Isentropic Compressibilities, and Excess Speeds of Sound for the Mixture 2-Methyl-Tetrahydrofuran (1) + 1-Butanol (2) at (283.15, 298.15, and 313.15) K

T/K = 283.15				T/K = 298.15				T/K = 313.15			
x_1	u / $\text{m}\cdot\text{s}^{-1}$	$\kappa_S^E \times 10^{12}$ / Pa^{-1}	u^E / $\text{m}\cdot\text{s}^{-1}$	x_1	u / $\text{m}\cdot\text{s}^{-1}$	$\kappa_S{}^E \times 10^{12}$ / Pa^{-1}	u^E / $\text{m}\cdot\text{s}^{-1}$	x_1	u / $\text{m}\cdot\text{s}^{-1}$	$\kappa_S^E \times 10^{12}$ / Pa^{-1}	u^E / $\text{m}\cdot\text{s}^{-1}$
0.0447	1292.4	−3.5	2.9	0.0341	1242.6	−3.3	2.3	0.0519	1184.6	−5.3	3.2
0.1154	1292.6	−8.4	6.9	0.1054	1241.6	−9.1	6.5	0.1237	1181.8	−11.3	6.8
0.2143	1291.5	−12.9	10.5	0.2085	1238.8	−14.8	10.5	0.2168	1177.7	−17.2	10.4
0.2451	1291.0	−13.9	11.3	0.2968	1235.7	−17.8	12.6	0.3097	1172.9	−20.9	12.5
0.4221	1286.8	−16.5	13.4	0.3535	1233.4	−18.9	13.3	0.4133	1167.2	−23.1	13.7
0.5204	1284.0	−16.2	13.2	0.4283	1230.3	−19.6	13.8	0.5110	1161.3	−23.0	13.5
0.6172	1281.2	−15.0	12.3	0.5058	1226.8	−19.4	13.7	0.6561	1152.6	−20.5	12.0
0.7128	1278.1	−12.7	10.5	0.6075	1222.2	−18.2	12.7	0.7265	1148.3	−18.1	10.6
0.8078	1275.2	−9.9	8.2	0.7077	1217.6	−15.7	11.0	0.8151	1142.7	−13.9	8.1
0.8952	1272.2	−6.3	5.3	0.8111	1212.6	−11.7	8.3	0.8995	1137.1	−8.5	5.0
0.9364	1270.3	−3.8	3.2	0.8947	1208.3	−7.3	5.2	0.9476	1133.5	−4.3	2.5
				0.9337	1206.1	−4.8	3.5				

Table 6. Dynamic Viscosities and Viscosity Deviations for the Mixture 2-Methyltetrahydrofuran (1) + 1-Butanol (2) at (283.15, 298.15, and 313.15) K

	η/mPa·s			$\Delta\eta$/mPa·s		
x_1	T/K = 283.15	T/K = 298.15	T/K =313.15	T/K = 283.15	T/K = 298.15	T/K = 313.15
0.0518	3.231	2.212	1.555	−0.467	−0.241	−0.132
0.1048	2.670	1.914	1.361	−0.852	−0.428	−0.254
0.2123	1.958	1.446	1.085	−1.210	−0.673	−0.386
0.3159	1.507	1.145	0.896	−1.318	−0.758	−0.435
0.4197	1.218	0.951	0.759	−1.263	−0.736	−0.432
0.5195	1.013	0.806	0.651	−1.138	−0.672	−0.406
0.6211	0.865	0.701	0.574	−0.950	−0.566	−0.345
0.7116	0.760	0.626	0.518	−0.756	−0.453	−0.280
0.8103	0.675	0.563	0.473	−0.514	−0.310	−0.192
0.9246	0.607	0.507	0.432	−0.205	−0.128	−0.078
0.9585	0.584	0.502	0.425	−0.115	−0.062	−0.040

The graphics of the excess properties and deviations show that the excess molar enthalpies are positive over the whole composition range, that they are greater when the temperature increases while the excess molar volumes are negative, and that they hardly change with temperature. The excess isentropic compressibilities are negative for all the temperatures and show a minimum that increases with temperature. The excess speeds of sound are always positive, and they hardly change with temperature. Finally, the viscosity deviations are negative throughout and the higher the temperature, the less negative the values.

The positive values of the excess molar enthalpies indicate that the breaking of the interactions existing in the pure compounds, especially the breaking of the hydrogen bonds existing between butanol molecules, is the main source of the energetic behavior. The effect of this breaking is presumably mitigated by the formation of new hydrogen bonds between the hydroxyl group in 1-butanol and the oxygen atom in 2-methyltetrahydrofuran. The viscosity deviations are negative as usual with associated compounds.[22−25] In fact some authors[26,27] have pointed out that when one of the compounds of a binary mixture is strongly associated, the deviations of viscosity are usually negative. This can be attributed to the fact that the breaking of association leads to smaller groups of molecules, which in most cases flow

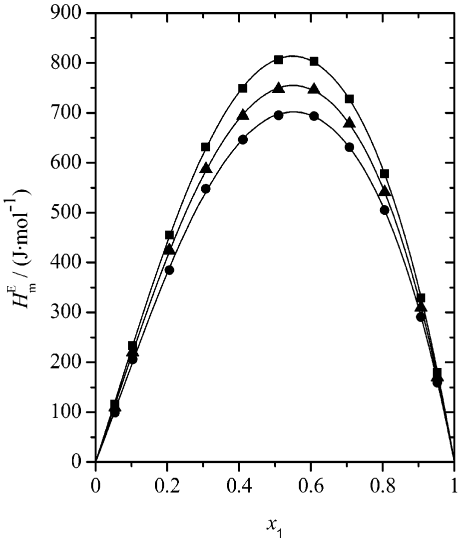

Figure 1. Excess molar enthalpies for the mixture 2-methyltetrahydrofuran (1) + 1-butanol (2): ●, 283.15 K; ▲, 298.15 K; ■, 313.15 K.

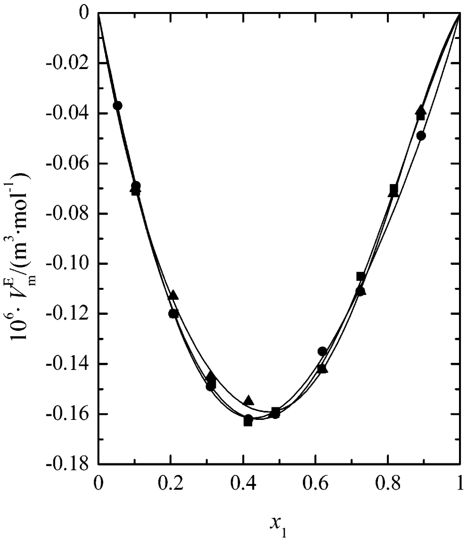

Figure 2. Excess molar volumes for the mixture 2-methyltetrahydrofuran (1) + 1-butanol (2): ●, 283.15 K; ▲, 298.15 K; ■, 313.15 K.

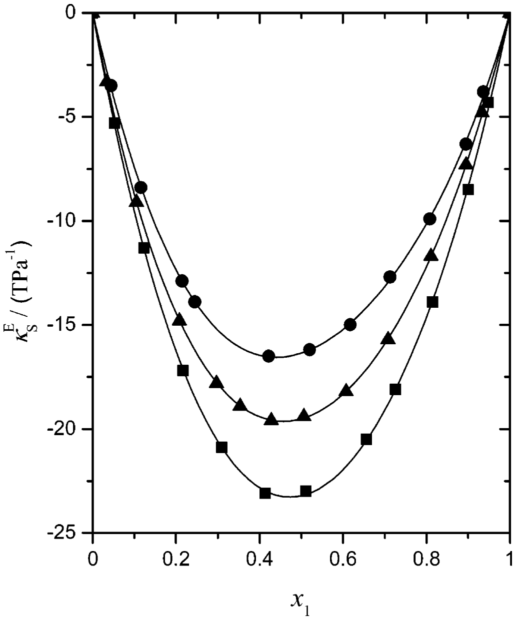

Figure 3. Excess isentropic compressibilities for the mixture 2-methyltetrahydrofuran (1) + 1-butanol (2): ●, 283.15 K; ▲, 298.15 K; ■, 313.15 K.

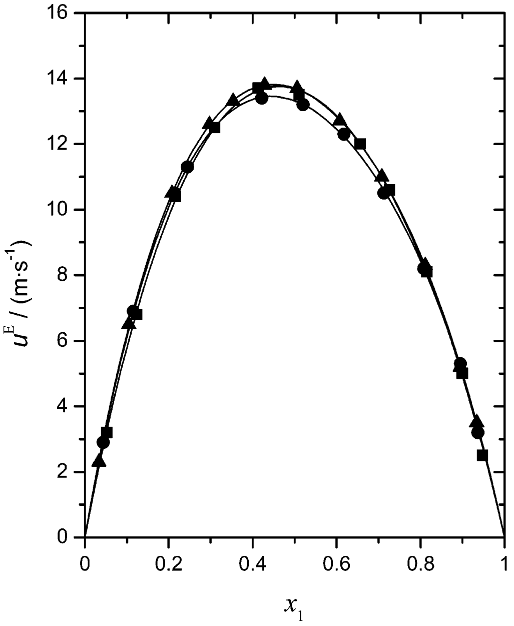

Figure 4. Excess speed of sound for the mixture 2-methyltetrahydrofuran (1) + 1-butanol (2): ●, 283.15 K; ▲, 298.15 K; ■, 313.15 K.

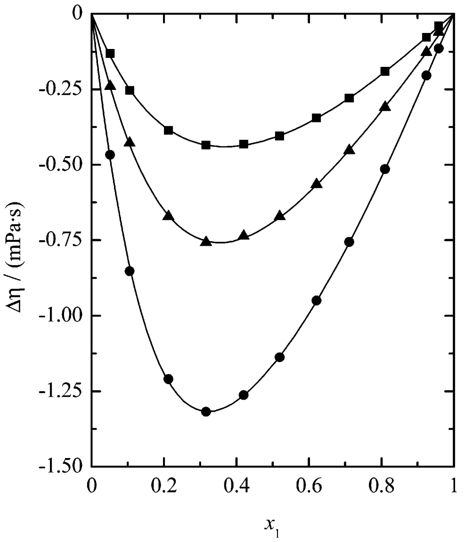

Figure 5. Viscosity deviations for the mixture 2-methyltetrahydrofuran (1) + 1-butanol (2): ●, 283.15 K; ▲, 298.15 K; ■, 313.15 K.

better yielding therefore a viscosity lower than the linear prediction.

On the other hand, the negative values of the excess molar volumes and exces isentropic compressibilities and the positive values of the excess speeds of sound indicate that this mixture is better packed than the pure compounds, but this effect is small because the absolute values are low. In any case, this is not a very usual behavior especially taking into account that the mixture is quite endothermic (from 700 to 800 J·mol^{-1} depending on the temperature). Commonly, the breaking of association produces an expansion of the mixture as the intermolecular distance grows with less or weaker interactions. The values obtained for excess molar volumes and excess speed of sound would indicate that the molecules of 1-butanol and 2-methyltetrahydrofuran intertwine occupying more effectively than expected a given volume.

If these results are compared with those of tetrahydropyran + 1-butanol[7] reported in our previous paper, it can be noticed that the values of excess molar enthalpies and viscosity deviations are quite similar in both cases; therefore, the structure of the ether has little effect in these properties, and the extent of breaking of hydrogen bonds of 1-butanol and formation of new hydrogen bonds between alcohol and ether is similar for both mixtures. Specifically, if the excess molar enthalpies are considered, the overall breaking of interactions is greater for tetrahydropyran than for 2-methyltetrahydrofuran.

However, attending to the excess molar volumes, important differences are observed. The excess molar volumes for 1-butanol + tetrahydropyran are sigmoidal-shaped being negative in the zone rich in ether and positive in the zone rich in butanol. In any case the values are small, which indicates, taking into account the quite high value of the excess molar enthalpy (near 1 kJ·mol^{-1}), that also in this mixture there would be an intertwining of ether and alcohol molecules. But this intertwining is not so effective as is in the case of 2-methyltetrahydrofuran due to the fact that probably pure tetrahydropyran is better packed than 2-methyltetrahydrofuran because the branching methyl group would pose a steric hindrance to the interactions

Table 7. Fitting Coefficients of the Redlich−Kister Equation for the Properties at the Temperatures (283.15, 298.15, and 313.15) K and Standard Deviations

property	T/K	A_1	A_2	A_3	A_4	σ
H^E_m/J·mol^{-1}	283.15	2779	572	24	365	3.2×10^{-3}
	298.15	2989	608	41	338	2.8×10^{-3}
	313.15	3223	646	−32	355	2.8×10^{-3}
$V^E_m \times 10^6$/m^3·mol^{-1}	283.15	−0.631	0.205	0.009	−0.105	1.4×10^{-3}
	298.15	−0.633	0.091	0.095	0.221	1.5×10^{-3}
	313.15	−0.639	0.169	0.091	0.088	1.5×10^{-3}
$\kappa^E_S \times 10^{12}$/Pa^{-1}	283.15	−65.4	13.2	−13.4	−4.5	0.11
	298.15	−78.1	11.4	−14.3	1.0	0.04
	313.15	−92.9	9.1	−10.0	−1.4	0.15
u^E/m·s^{-1}	283.15	53.3	−9.6	12.6	2.7	0.09
	298.15	54.8	−9.0	11.5	1.2	0.04
	313.15	54.7	−7.3	7.0	1.8	0.11
$\Delta\eta$/mPa·s	283.15	−4.655	2.974	−2.110	1.146	8.9×10^{-3}
	298.15	−2.764	1.650	−0.764	0.139	4.5×10^{-3}
	313.15	−1.647	0.811	−0.409	0.233	4.5×10^{-3}

in the pure compound. Agreeing with this possibility, the density of tetrahydropyran is clearly greater than that of 2-methyltetrahydrofuran. The existence of that steric hindrance is supported by the excess enthalpy data as the lower values for the mixture of 1-butanol + 2-methyltetrahydrofuran would correspond to either weaker ether−ether interactions (that are also broken) in the pure compound or weaker hydrogen bonds between ether and alcohol or likely to a combination of both effects.

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