Densities, Excess Molar Volumes, and Isobaric Thermal Expansibilities for 1,2-Ethanediol + 1-Butanol, or 1-Hexanol, or 1-Octanol in the Temperature Range from (293.15 to 313.15) K

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Densities for 1,2-ethanediol + 1-butanol, or 1-hexanol, or 1-octanol were measured over the entire composition range in the temperature range from (293.15 to 313.15) K at 5 K intervals. From the experimental values, the excess molar volumes and isobaric thermal expansibilities were calculated. The Redlich–Kister polynomials were fitted to the results, and smooth representations of the results are presented. In all cases, the positive values of the excess molar volumes were obtained for 1-hexanol and 1-octanol mixtures, whereas for 1-butanol mixtures, negative values were observed. The experimental data were used also for the calculation by analytical differentiation of the isobaric coefficient of thermal expansion.

Introduction

Binary systems are an important class of solvents and solutions. Unfortunately, the behavior of some of their properties is still not clear. Especially interesting are highly associated binary systems. At the same time, interpretation of the mixing effects is extremely difficult because of various possible interactions and size effects.

The aim of this work is to study the composition and temperature dependence of volume effects for 1,2-ethanediol + 1-butanol, or 1-hexanol, or 1-octanol systems, i.e., for systems with two strong self-associated hydroxyl compounds. The type and extent of the association is essentially determined by the two components of the alcohol molecule, i.e., the hydrocarbon chain (length and/or degree of branching) and the hydroxyl groups (number and positions). In the mixtures of alcohols, interplay of molecules of the same kind (self-association) and of different kinds (complexation) occurs.

To the best of our knowledge, the literature contains values for the investigated systems that are in practice available only for 1,2-ethanediol + 1-butanol.[1,2]

Experimental

Materials. 1,2-Ethanediol (puriss, p.a. mass fraction > 0.995, Fluka), 1-butanol (puriss, p.a. mass fraction > 0.995, ACS, Sigma-Aldrich), 1-hexanol (mass fraction > 0.98, Riedel de Haen), and 1-octanol (mass fraction > 0.99, Lancaster) were used without further purification. The purities of the alcohols were checked by comparing measured densities and refractive indices with those reported in the literature (see Table 1). The mass fraction of water determined by the Karl Fischer method was $2.7 \cdot 10^{-4}$ (1,2-ethanediol), $3.7 \cdot 10^{-4}$ (1-butanol), $2.4 \cdot 10^{-4}$ (1-hexanol), and $6.1 \cdot 10^{-4}$ (1-octanol), respectively.

Safety note. 1-Butanol is potentially more toxic than any of the lower alcohols, but the actual hazard is reduced by its low vapor pressure. Exposure to concentrations of 25 ppm resulted in mild irritation of the nose, throat, and eyes.

Methods. Solutions were prepared by mass (precision $\pm$ $6 \cdot 10^{-7}$ kg) using an OHAUS Analytical Standard balance (model AS 200) and stored in sealed flasks. The uncertainty of the determination for the mole fraction was estimated to be of $\pm$ $5 \cdot 10^{-5}$.

The densities were measured with a vibrating-tube densimeter Unilab MG-2. Before the measurements, the solutions and components were partially degassed (20 min) using an ultrasound (ultrasonic cleaner Unitra-Unima, model UH-4, $f = 25$ kHz). The densimeter was calibrated using the air and deionized and degassed (as above) water with an electrolytic conductivity of $1.2 \cdot 10^{-4}\ \Omega^{-1}\mathrm{m}^{-1}$ as the standard. The density of water was calculated by means of the equation proposed by Kell.[3] The temperature of the water bath in the densimetric experiment was measured by means of a calibrated (NIST certified) platinum resistance thermometer (ERTCO-HART, model 850, Pt100) with uncertainty better than $\pm$ 0.05 K and precision of $\pm$ 0.01 K. The expected uncertainty and precision of the measured densities is $\pm$ $5 \cdot 10^{-2}\ \mathrm{kg \cdot m^{-3}}$. More details concerning the density measurements can be found in a paper given by Marczak.[4] It is worth noting that the apparatus used in the present study was checked using standard liquids (aqueous solutions of NaCl, K_2CrO_4, and $K_2Cr_2O_7$) whose densities were previously determined using bicapillary pycnometers by Ernst et al.[5] The differences in the measured densities did not exceed $7 \cdot 10^{-2}\ \mathrm{kg \cdot m^{-3}}$.

All temperatures are reported in terms of ITS-90 and the molar values in terms of the 1996 IUPAC relative atomic masses.[6]

Results and Discussion

Densities, ρ, measured over the entire composition range and at the temperatures T ranging from (293.15 to 313.15) K at 5 K intervals are given in Tables 1 and 2.

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Table 1. Densities (ρ), Refractive Indices (n_D), and Isobaric Coefficients of Thermal Expansion (α_p) of Pure Components and Comparison with Selected Literature Data

	ρ/kg·m^{-3}		n_D		$\alpha_p \cdot 10^4$/K^{-1}	
T/K	exptl	lit.	exptl[a]	lit.	exptl	lit.
		1,2-Ethanediol				
293.15	1113.37	1113.44[b], 1113.47[c], 1113.5[d]	1.4323	1.4318[d]	6.20	
298.15	1109.88	1109.91[b], 1110.0[d], 1110.09[c], 1109.6[i],	1.4309	1.4306[d]	6.26	6.25[d]
303.15	1106.40	1106.51[b], 1106.35[d], 1106.65[c]			6.33	
308.15	1102.89	1103.09[b], 1102.96[c]			6.39	
313.15	1099.35	1099.75[b]			6.46	
		1-Butanol				
293.15	809.60	809.56[d], 810.34[c]	1.3997	1.39929[d]	9.23	
298.15	805.84	805.67[c], 805.9[i], 805.75[d], 805.737[e]	1.3978	1.39741[d]	9.38	9.48[d]
303.15	802.04	801.95[c], 801.97[f], 801.94[h]			9.53	
308.15	798.20	798.12[c]			9.69	
313.15	794.31	794.23[f], 794.05[h]			9.84	
		1-Hexanol				
293.15	818.74	818.75[d]	1.4183	1.4172[d]	8.48	
298.15	815.28	815.34[d], 815.32[g]	1.4161	1.4161[d],	8.62	8.78[d]
303.15	811.71	811.95[h]			8.77	
308.15	808.13				8.91	
313.15	804.53	804.14[h]			9.06	
		1-Octanol				
293.15	825.12	824.99[d]	1.4298	1.4296[d]	8.25	
298.15	821.75	821.57[d], 821.62[g]	1.4280	1.4276[d],	8.32	8.27[d]
303.15	818.31	818.31[h]			8.40	
308.15	814.80				8.47	
313.15	811.41	811.88[h]			8.55	

[a] PZO, RL2 Abbe refractometer. [b] Ref 7. [c] Ref 2. [d] Ref 8. [e] Ref 9. [f] Ref 10. [g] Ref 11. [h] Ref 12. [i] Ref 1.

Table 2. Densities (ρ) for the Investigated System at Temperatures (T)

	ρ/(kg·m^{-3}) at the following temperatures T/K				
x	293.15	298.15	303.15	308.15	313.15
	x 1,2-Ethanediol + (1 − x) 1-Butanol				
0.08923	826.85	823.02	819.29	815.49	811.51
0.24902	861.02	857.21	853.42	849.65	845.69
0.32059	878.11	874.25	870.64	866.86	862.94
0.40186	898.74	894.96	891.24	887.45	883.57
0.53472	936.02	932.22	928.63	924.91	921.05
0.65712	975.06	971.34	967.77	964.18	960.49
0.69744	989.14	985.27	981.88	978.14	974.54
0.77530	1017.69	1013.86	1010.51	1006.95	1003.28
0.81003	1030.76	1027.06	1023.76	1020.29	1016.76
0.91207	1073.19	1069.67	1066.15	1062.56	1059.01
	x 1,2-Ethanediol + (1 − x) 1-Hexanol				
0.08275	829.72	826.21	822.63	819.02	815.44
0.15846	840.78	837.17	833.58	829.96	826.33
0.23738	853.56	849.99	846.39	842.67	839.04
0.32651	869.87	866.29	862.69	859.01	855.36
0.49049	905.80	902.20	898.58	894.88	891.27
0.54448	919.79	916.19	912.56	908.90	905.23
0.59646	934.39	930.83	927.24	923.53	919.95
0.69315	965.41	961.84	958.23	954.54	950.95
0.77570	996.60	993.11	989.49	985.79	982.18
0.90534	1057.08	1053.67	1050.10	1046.52	1042.98
	x 1,2-Ethanediol + (1 − x) 1-Octanol				
0.07997	833.01	829.64	826.15	822.68	819.15
0.15901	842.00	838.63	835.13	831.61	828.07
0.21593	849.21	845.74	842.24	838.76	835.18
0.36005	870.86	867.38	863.92	860.33	856.83
0.48253	894.13	890.64	887.11	883.47	879.91
0.59121	920.03	916.52	912.94	909.33	905.71
0.62990	930.80	927.19	923.70	920.08	916.50
0.74368	968.64	965.11	961.50	957.92	954.32
0.80631	994.57	991.03	987.45	983.80	980.25
0.88918	1036.57	1033.02	1029.52	1025.99	1022.37

Table 3. Values of the Parameters a_i for Redlich–Kister Type Equation 2 and Standard Deviations δ

	$V^E \cdot 10^6$/(m^3·mol^{-1})			
T/K	a_1	a_2	a_3	δ
	x 1,2-Ethanediol + (1 − x) 1-Butanol			
293.15	−0.41959	0.48734	−0.21575	0.0084
298.15	−0.40978	0.49342	−0.16964	0.0059
303.15	−0.46967	0.55012	−0.24467	0.0085
308.15	−0.50293	0.56647	−0.30520	0.0085
313.15	−0.52485	0.66162	−0.28085	0.0092
	x 1,2-Ethanediol + (1 − x) 1-Hexanol			
293.15	0.60402	0.32910		0.0043
298.15	0.63679	0.41847		0.0060
303.15	0.64052	0.41918		0.0057
308.15	0.67312	0.44692		0.0064
313.15	0.66403	0.47374		0.0075
	x 1,2-Ethanediol + (1 − x) 1-Octanol			
293.15	1.15148	0.30677	0.23579	0.0047
298.15	1.19281	0.30785	0.19799	0.0057
303.15	1.20500	0.32199	0.26318	0.0069
308.15	1.23931	0.30077	0.15156	0.0046
313.15	1.28467	0.39732	0.30580	0.0099

The values of the excess molar volumes V^E were determined from the equation

$$V^E = V - \sum_{i=1}^{2} x_i V_i \tag{1}$$

where V is the molar volume; x_i is the mole fraction of component i of the system; and V_i is the molar volume of component i of the system. The corresponding values of V^E are reported in Supporting Information Table 1.

Each set of values of V^E was fitted to the Redlich–Kister type equation

$$V^E/(\mathrm{m}^3 \cdot \mathrm{mol}^{-1}) = x(1 - x) \cdot \sum_{i=1}^{n} a_i \cdot (1 - 2x)^{i-1} \tag{2}$$

where x is the mole fraction of 1,2-ethanediol. Values of the parameters a_i, determined by the unweighted least-squares method, are listed in Table 3 where standard deviations δ are also given; the optimum number of the coefficients was ascertained from an examination of δ.

The results shown in Supporting Information Table 1 and Figures 1 and 2 reveal that $V^E > 0$ for (1,2-ethanediol + 1-hexanol) and (1,2-ethanediol + 1-octanol), whereas for (1,2-ethanediol + 1-butanol), $V^E < 0$ over the whole composition

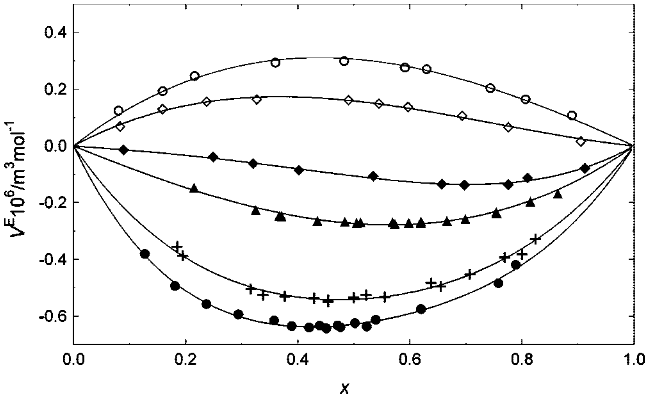

Figure 1. Excess molar volumes, V^E, plotted against mole fraction, x, for ($x \cdot$1,2-ethanediol + (1 − x)$\cdot$1-alkanol) systems at the temperature 298.15 K: •, methanol of Jimenez and Paz Andrade;[1] +, ethanol of Jimenez and Paz Andrade;[1] ▲, 1-propanol of Jimenez and Paz Andrade;[1] ♦, 1-butanol of this work; ◇, 1-hexanol of this work; and ○, 1-octanol of this work. Curves calculated by using the Redlich–Kister eq 2.

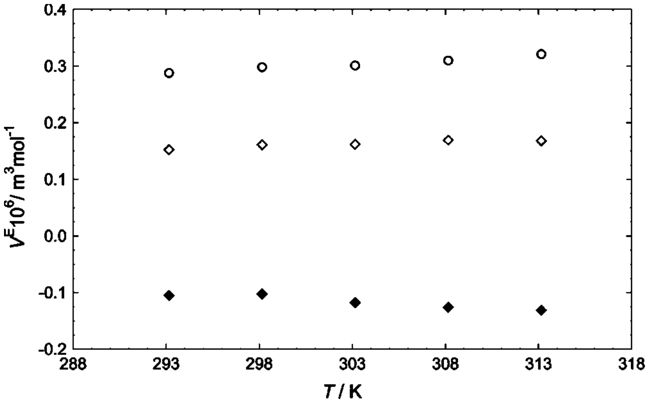

Figure 2. Temperature dependence of the excess molar volumes V^E at equimolar composition for the investigated systems of ($x \cdot$1,2-ethanediol + (1 − x)$\cdot$1-alkanol): ♦, 1-butanol; ◇, 1-hexanol; and ○, 1-octanol.

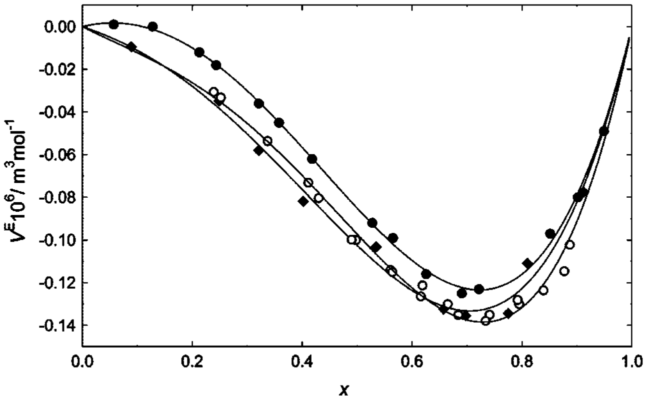

Figure 3. Excess molar volumes, V^E, plotted against mole fraction, x, for the ($x \cdot$1,2-ethanediol + (1−x)$\cdot$1-butanol) system at the temperature 298.15 K: ♦, this work; •, Jimenez and Paz Andrade;[1] and ○, Jimenez et al.[2] Curves calculated by using the Redlich–Kister eq 2.

Table 4. Values of the Parameters a_{ij} for Equation 3 in the Temperature Range from (293.15 to 313.15) K

a_{11}	a_{12}	a_{21}	a_{22}	a_{32}	δ
		$V^E \cdot 10^6/m^3 \cdot mol^{-1}$			
		x 1,2-Ethanediol + (1−x) 1-Butanol			
1.5438	$-6.628 \cdot 10^{-3}$	−2.2518	$9.265 \cdot 10^{-3}$	$-0.778 \cdot 10^{-3}$	0.0085
		x 1,2-Ethanediol + (1−x) 1-Hexanol			
−0.30431	$3.127 \cdot 10^{-3}$	−1.5089	$6.355 \cdot 10^{-3}$		0.0062
		x 1,2-Ethanediol + (1−x) 1-Octanol			
−0.70405	$6.329 \cdot 10^{-3}$		$1.0798 \cdot 10^{-3}$	$0.762 \cdot 10^{-3}$	0.0069

range and at all temperatures. For the last mentioned system, a comparison (at $T = 298.15$ K) with the literature data[1,2] is shown in Figure 3. As can be seen, the shape and symmetry of the all presented $V^E(x)$ curves show a good agreement. However, some quantitative differences are observed. On one hand, the values of V^E reported in this work are in good agreement with the values reported by Jimenez and Paz Andrade[1] (at $x = 0.5$ the V^E values are equal to $-0.1024 \cdot 10^{-6}$ m$^3 \cdot$mol^{-1} and $-0.0972 \cdot 10^{-6}$ m$^3 \cdot$mol^{-1}, respectively). On the other hand, the agreement between the V^E values of this work and those given by Jimenez et al.[2] is worse (at $x = 0.5$ the V^E values are equal to $-0.1024 \cdot 10^{-6}$ m$^3 \cdot$mol^{-1} and $-0.085 \cdot 10^{-6}$ m$^3 \cdot$mol^{-1}, respectively). It should be mentioned that the deviations found between the V^E values of this work and those determined by Jimenez et al.[2] at T = (303.15 and 308.15) K are of the same order as those at T = 298.15 K. However, the deviations at T = 293.15 K are considerable. In this case, Jimenez et al.'s[2] V^E values are positive for $x \leq 0.4$ and negative for $x > 0.4$ (i.e., $V^E(x)$ curve is s-shaped) contrary to the negative V^E values of this work (Supporting Information Table 1). For both of the other systems, the lack of literature data precludes any kind of comparison.

Taking into account the literature data,[1,2,13,15] the V^E values for (1,2-ethanediol + 1-alkanol) for a given x decrease at T = 298.15 K in sequence:

1-octanol > 1-hexanol > 0 ≈ 1-pentanol > 1-butanol > 1-propanol > ethanol > methanol.

The magnitude and sign of V^E is sensitive to the carbon chain lengths of the 1-alkanol molecules. So, as the length of 1-alkanol is increased, the V^E increases; however, for the lower 1-alkanols (C_1 to C_4), V^E is negative over the whole concentration range, while for 1-hexanol and 1-octanol, it is positive. Instead, in the case of 1-pentanol, the V^E values are very small, close to zero, but unfortunately, the results are ambiguous (the fluctuations of the order of 10^{-8} m$^3 \cdot$mol^{-1} near zero are observed[13]). The above changes of V^E with increasing chain length of 1-alkanol can be related to the increase difference in size and shape of the component molecules, as well as to the increase difference in the intermolecular interaction energy between like molecules. As known, the negative V^E indicates more compact packing of the molecules in the binary mixture than those in pure

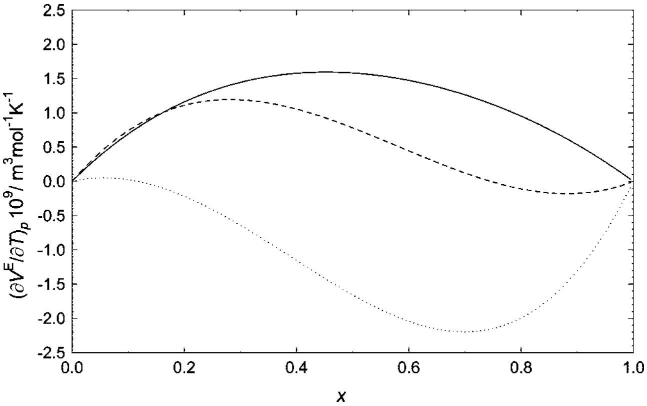

Figure 4. $(\partial V^E/\partial T)_p$ plotted against the mole fraction, x, for the investigated systems of ($x \cdot$1,2-ethanediol + (1−x)$\cdot$1-alkanol) for the temperatures in the range (298.15 to 313.15) K: dotted line, 1-butanol; dashed line, 1-hexanol; and continuous line, 1-octanol.

components, whereas the positive V^E indicates a loose packing. It seems that the positive V^E in the case of 1-hexanol and 1-octanol results mainly from the disruption of the hydrogen-bonded structures upon mixing.

It is worth adding that for two systems, i.e. (1,2-ethanediol + methanol) and (1,2-ethanediol + ethanol), the literature data are inconsistent. For the first system, the results presented by Albuquerqe et al.[14] differ from the results obtained by Jimenez and Paz Andrade[1] by $0.165 \cdot 10^{-6}$ $m^3 \cdot mol^{-1}$ at equimolar composition, and for the second one even by $0.313 \cdot 10^{-6}$ $m^3 \cdot mol^{-1}$. In our opinion, the data reported by the last-mentioned authors are more reliable because a fair agreement was found independently for (1,2-ethanediol + 1-butanol) (this work, see above) as well as for (1,2-ethanediol + 1-propanol). Here, the results of Pal and Sharma[15] are in excellent agreement with values obtained by Jimenez et al.[2] as well as by Jimenez and Paz Andrade.[1] At equimolar composition ($T = 298.15$ K), the V^E values are equal to ($-0.2657 \cdot 10^{-6}$, $-0.2628 \cdot 10^{-6}$, and $-0.2700 \cdot 10^{-6}$) $m^3 \cdot mol^{-1}$, respectively.

It is worthy of notice that because the degrees of the Redlich–Kister polynomials given by Jimenez and Paz Andrade[1] are too high (the results are evidently strongly overparametrized) we have executed a recalculation according to eq 2. The results are listed in Supporting Information Table 2 and used in this work for presentation in Figures 1 and 3.

As shown in Figure 2, the effect of temperature on the V^E values is generally rather small for all systems studied in this work. As known, eq 2 can be extended to represent V^E as a function of concentration as well as of temperature T and can be used in the form:

$$V^E/(m^3 \cdot mol^{-1}) = x(1 - x) \cdot \sum_{i=1}^{k} \sum_{j=1}^{n} a_{ij} \cdot (1 - 2x)^{i-1} (T/K)^{j-1} \quad (3)$$

Taking into account a concentration dependence and precision of our V^E studies, six a_{ij} parameters ($11 \le ij \le 32$) should be sufficient. It appears that for (1,2-ethanediol + 1-octanol) and (1,2-ethanediol + 1-hexanol) only four significant a_{ij} values (estimated by the unweighted least-squared method) were found, and for (1,2-ethanediol + 1-butanol) five values were found. Analytical derivation of eq 3 with the estimated (as above) parameters gives the values of $(\partial V^E/\partial T)_p$ as a function of composition in the form

$$(\partial V^E/\partial T)_p/(m^3 \cdot mol^{-1} \cdot K^{-1}) = x(1 - x) \cdot \sum_{i=1}^{3} a_{i2} \cdot (1 - 2x)^{i-1} \quad (4)$$

where a_{i2} are coefficients collected in Table 4. The obtained $(\partial V^E/\partial T)_p(x)$ curves are shown in Figure 4.

As can be seen, the $(\partial V^E/\partial T)_p$ values (in the range from (293.15 to 313.15) K) for the test systems of 1,2-ethanediol with 1-octanol, 1-hexanol, and 1-butanol decrease in sequence:

1-octanol > 1-hexanol > 1-butanol,

and at $x = 0.5$ are equal ($1.6 \cdot 10^{-9}$, $0.78 \cdot 10^{-9}$, and $-1.7 \cdot 10^{-9}$) $m^3 \cdot mol^{-1} \cdot K^{-1}$, respectively. Simultaneously, the least changes in V^E with temperature are observed for (1,2-ethanediol + 1-hexanol). This rather weak temperature effect on the V^E values is most likely due to the fact that the increase in kinetic energy is not enough to change in an appreciable manner the interaction of the H-bond type.

Because the reliable experimental values of the isobaric coefficient of thermal expansion α_p (α_p is one of the three so-called thermophysical material constants) are desirable among others for the calculations of the isochoric heat capacity from the isobaric heat capacity and of the isothermal compressibility from the isentropic compressibility, we have calculated additionally the α_p values. The calculated values for pure compounds are collected in Table 1 and for mixtures in Supporting Information Table 3. It can be seen that α_p increases with the temperature. The greatest temperature dependence of α_p is with 1-butanol, and the least with 1-octanol.

The isobaric coefficients of thermal expansion were calculated from

$$\alpha_p = -(\partial \ln \rho/\partial T)_p = \rho(\partial \rho^{-1}/\partial T)_p \quad (5)$$

by analytical differentiation of the temperature dependencies of the densities. Needed dependencies of the density on the temperature were approximated by the equation

$$\rho/(kg \cdot m^{-3}) = \sum_{i=1}^{3} a_i \cdot (T/100 \cdot K)^{i-1} \quad (6)$$

where a_i are coefficients calculated by the least-squares method. A scaling factor of 100 improved the numerical stability. In calculations, the density values smoothed in this way were used.

Supporting Information Available:

The values of excess molar volumes V^E and isobaric coefficients of thermal expansion α_p for the investigated systems as well as recalculated Redlich–Kister parameters a_i for Jimenez and Paz Andrade data.[1] This material is available free of charge via the Internet at http://pubs.acs.org.

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