

Density, Viscosity, Refractive Index, and Speed of Sound in the Binary Mixtures of 1,4-Dioxane + Ethanediol, + Hexane, + Tributylamine, or + Triethylamine at (298.15, 303.15, and 308.15) K

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Densities, viscosities, and refractive indices of the binary mixtures of 1,4-dioxane with ethanediol, hexane, tributylamine, and triethylamine have been measured at (298.15, 303.15, and 308.15) K, while the speed of sound data are measured at 298.15 K. From these results, excess molar volume and deviations in viscosity, molar refraction, speed of sound, and isentropic compressibility have been calculated. These results are fitted to the polynomial equation to derive the binary coefficients. Standard errors have been calculated between the fitted results and the calculated data. These data have been used to discuss the mixing behavior of the components.

Introduction

Dioxane is a cyclic molecule used in a variety of applications in industrial sectors. Its interactions with different types of liquids such as ethanediol, hexane, tributylamine, and triethylamine are important from a fundamental viewpoint. In continuation of our earlier paper¹ on the properties of binary mixtures of tributylamine with a variety of other liquids as well as dioxane containing binary mixtures,^{2–8} we present here the experimental data on density, ρ , viscosity, η , and refractive index for the sodium D-line, n_D , of the binary mixtures of 1,4-dioxane + ethanediol, + hexane, + tributylamine, or + triethylamine at (298.15, 303.15, and 308.15) K and the data for speed of sound, u , at 298.15 K. From these data, excess molar volume, V^E , deviation in viscosity, $\Delta\eta$, deviation in molar refraction, ΔR , deviation in sound velocity, Δu , and deviation in isentropic compressibility, Δk_s , have been computed and the results are discussed in terms of the nature of molecular interactions between the mixing components.

Experimental Section

Materials. High-purity analytical reagent grade samples of ethanediol, hexane, and triethylamine and a laboratory reagent grade sample of tributylamine were procured from S.D. Fine Chemicals, Mumbai, India. 1,4-Dioxane is a high-purity sample procured from S.D. Fine Chemicals. The mol % purities of these liquids as determined by gas chromatography (GC) (HP 6890) using a flame ionization detector (FID) were >99% and are reported in Table 1 along with density and refractive index data measured at 298.15 K for pure liquids and a comparison with the literature values.

Binary mixtures were prepared by mass⁹ in specially designed conical flasks. The mass measurements accurate to ± 0.01 mg were performed on a digital electronic balance (Mettler, AE 240, Switzerland). A set of nine compositions were prepared for each mixture, and their physical properties were measured at the respective composition in the

Table 1. Experimental Densities (ρ) and Refractive Indices (n_D) of Pure Liquids Compared with Literature Values at 298.15 K

liquid	mol% purity	$\rho/(\text{kg}\cdot\text{m}^{-3})$		n_D	
		expt	lit (ref)	expt	lit (ref)
1,4-dioxane	>99.0	1027.6	1028.0 (2)	1.4201	1.4200 (5)
ethanediol	>99.0	1111.3	1110.0 (20)	1.4307	1.4306 (19)
hexane	>99.0	654.8	654.8 (11)	1.3728	1.3723 (11)
tributylamine	>99.0	774.3	774.0 (21)	1.4274	1.4280 (19)
triethylamine	>99.5	723.1	722.8 (19)	1.3983	1.3980 (19)

mole fraction scale from 0.1 to 0.9 in steps of 0.1. The possible uncertainty in mole fraction was less than 0.0002.

Methods. Densities of liquids and liquid mixtures were measured within a precision of ± 0.0001 $\text{g}\cdot\text{cm}^{-3}$ using the capillary-type pycnometer of capacity 10 cm^3 volume. Experimental details and calibrations of the pycnometer and measurements are the same as reported earlier.^{9–11}

Viscosities were measured using a Cannon Fenske viscometer (size 75, ASTM D 445, Industrial Research Glassware Ltd., Roselle, NJ). An electronic digital stopwatch with a readability of ± 0.01 s was used for the flow time measurements. The measured viscosity values are precise up to ± 0.001 $\text{mPa}\cdot\text{s}$. Calibration procedures of the pycnometer remain the same as described previously.^{9,10}

Refractive indices for the sodium D-line were measured using a thermostatically controlled Abbe refractometer (Atago 3T, Japan). A minimum of three independent readings were taken for each composition, and the average value was considered in all the calculations. Refractive index data are accurate to ± 0.001 units.

The speed of sound was measured using a variable path single-crystal interferometer (Mittal Enterprises, model M-84, New Delhi). A crystal-controlled high-frequency generator was used to excite the transducer at a frequency of 1 MHz. The frequency was measured within an accuracy of 1 in 10^4 using a digital frequency meter. The interferometer cell was filled with the test liquid, and water was circulated around the measuring cell from a constant-temperature bath maintained at (298.15 \pm 0.01) K. Details of the speed of sound measurements have been given earlier,¹² and these data are precise up to ± 2 in 1000 $\text{m}\cdot\text{s}^{-1}$.

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Table 2. Experimental Density (ρ), Viscosity (η), Refractive Index (n_D), and Speed of Sound (u) of the Binary Mixtures at Different Temperatures

temp/K	x_1	$\rho/(\text{kg}\cdot\text{m}^{-3})$	$\eta/(\text{mPa}\cdot\text{s})$	n_D	$u/(\text{m}\cdot\text{s}^{-1})$	temp/K	x_1	$\rho/(\text{kg}\cdot\text{m}^{-3})$	$\eta/(\text{mPa}\cdot\text{s})$	n_D	$u/(\text{m}\cdot\text{s}^{-1})$
1,4-Dioxane (1) + Ethanediol (2)						1,4-Dioxane (1) + Tributylamine (2)					
298.15	0.0	1111.3	12.62	1.4307	1659	298.15	0.0	774.4	1.360	1.4274	1261
	0.1031	1100.6	9.007	1.4294	1620		0.1201	785.1	1.245	1.4266	1253
	0.2072	1090.6	6.285	1.4284	1577		0.2122	794.7	1.190	1.4259	1254
	0.3021	1082.0	4.654	1.4275	1537		0.3072	806.0	1.156	1.4250	1257
	0.4246	1071.1	3.177	1.4260	1485		0.4085	820.5	1.137	1.4244	1264
	0.5042	1064.5	2.566	1.4248	1456		0.5014	836.6	1.123	1.4234	1272
	0.5986	1056.8	2.012	1.4238	1426		0.5972	856.9	1.118	1.4226	1281
	0.6914	1049.6	1.663	1.4230	1400		0.6998	884.0	1.112	1.4217	1292
	0.7906	1042.4	1.388	1.4220	1381		0.7999	917.9	1.114	1.4210	1305
	0.8909	1035.2	1.212	1.4210	1369		0.8947	960.6	1.133	1.4205	1320
	1.0	1027.6	1.166	1.4201	1347		1.0	1027.6	1.166	1.4201	1343
303.15	0.0	1107.3	9.618	1.4269		303.15	0.0	770.1	1.221	1.4251	
	0.1031	1096.6	7.262	1.4279			0.1201	780.9	1.142	1.4240	
	0.2072	1086.4	5.149	1.4268			0.2122	790.2	1.089	1.4236	
	0.3021	1077.6	3.849	1.4256			0.3072	801.6	1.059	1.4226	
	0.4246	1066.5	2.680	1.4240			0.4085	815.9	1.044	1.4218	
	0.5042	1059.7	2.184	1.4226			0.5014	832.1	1.032	1.4209	
	0.5986	1051.7	1.728	1.4216			0.5972	852.1	1.026	1.4201	
	0.6914	1044.5	1.447	1.4207			0.6998	879.2	1.022	1.4192	
	0.7906	1037.1	1.226	1.4196			0.7999	912.6	1.025	1.4185	
	0.8909	1029.9	1.081	1.4187			0.8947	955.2	1.042	1.4180	
	1.0	1021.9	1.069	1.4170			1.0	1021.9	1.069	1.4170	
308.15	0.0	1103.5	7.539	1.4246		308.15	0.0	766.2	1.125	1.4226	
	0.1031	1092.8	5.518	1.4262			0.1201	776.8	1.039	1.4215	
	0.2072	1082.4	4.013	1.4250			0.2122	785.9	0.989	1.4210	
	0.3021	1073.1	3.044	1.4236			0.3072	797.1	0.963	1.4201	
	0.4246	1061.7	2.181	1.4219			0.4085	811.4	0.950	1.4196	
	0.5042	1054.7	1.802	1.4204			0.5014	827.5	0.940	1.4184	
	0.5986	1046.8	1.444	1.4193			0.5972	847.4	0.934	1.4174	
	0.6914	1039.5	1.229	1.4186			0.6998	874.1	0.933	1.4168	
	0.7906	1031.8	1.063	1.4172			0.7999	907.4	0.936	1.4160	
	0.8909	1024.5	0.949	1.4163			0.8947	949.9	0.952	1.4154	
	1.0	1016.2	0.984	1.4143			1.0	1016.2	0.984	1.4143	
1,4-Dioxane (1) + Hexane (2)						1,4-Dioxane (1) + Triethylamine (2)					
298.15	0.0	654.8	0.315	1.3728	1083	298.15	0.0	723.1	0.363	1.3983	1127
	0.1135	682.0	0.333	1.3758	1087		0.1104	744.6	0.392	1.3996	1139
	0.2057	706.4	0.357	1.3787	1099		0.2044	764.5	0.418	1.4014	1155
	0.3099	736.2	0.391	1.3824	1116		0.3028	787.3	0.451	1.4032	1172
	0.4032	765.7	0.429	1.3864	1132		0.3980	811.3	0.494	1.4048	1189
	0.5010	799.6	0.482	1.3906	1154		0.4946	838.0	0.542	1.4068	1210
	0.6028	838.1	0.549	1.3954	1185		0.5986	869.7	0.613	1.4095	1234
	0.7006	878.7	0.634	1.4014	1220		0.6952	902.3	0.697	1.4117	1260
	0.7991	924.0	0.753	1.4078	1257		0.7996	941.2	0.815	1.4147	1290
	0.8962	972.1	0.912	1.4140	1303		0.8934	979.8	0.958	1.4176	1318
	1.0	1027.6	1.166	1.4201	1347		1.0	1027.6	1.166	1.4201	1343
303.15	0.0	649.7	0.299	1.3698		303.15	0.0	718.5	0.347	1.3955	
	0.1135	677.3	0.319	1.3728			0.1104	739.7	0.372	1.3969	
	0.2057	701.4	0.340	1.3756			0.2044	759.5	0.397	1.3985	
	0.3099	731.2	0.372	1.3795			0.3028	782.2	0.428	1.4002	
	0.4032	760.5	0.408	1.3838			0.3980	806.1	0.466	1.4020	
	0.5010	794.3	0.455	1.3878			0.4946	832.8	0.512	1.4040	
	0.6028	832.7	0.517	1.3928			0.5986	864.3	0.575	1.4067	
	0.7006	873.3	0.596	1.3986			0.6952	896.8	0.652	1.4090	
	0.7991	918.3	0.705	1.4048			0.7996	935.6	0.760	1.4120	
	0.8962	966.4	0.849	1.4119			0.8934	974.2	0.889	1.4148	
	1.0	1021.9	1.069	1.4170			1.0	1021.9	1.069	1.4170	
308.15	0.0	644.8	0.286	1.3670		308.15	0.0	713.6	0.330	1.3926	
	0.1135	672.5	0.304	1.3700			0.1104	734.8	0.353	1.3942	
	0.2057	696.5	0.323	1.3726			0.2044	754.6	0.377	1.3956	
	0.3099	726.1	0.353	1.3767			0.3028	777.1	0.404	1.3972	
	0.4032	755.3	0.386	1.3814			0.3980	800.9	0.438	1.3994	
	0.5010	788.9	0.429	1.3850			0.4946	827.5	0.481	1.4013	
	0.6028	827.2	0.485	1.3903			0.5986	859.0	0.538	1.4041	
	0.7006	868.0	0.557	1.3959			0.6952	891.5	0.608	1.4062	
	0.7991	912.4	0.656	1.4017			0.7996	930.0	0.705	1.4093	
	0.8962	960.8	0.787	1.4098			0.8934	968.5	0.821	1.4121	
	1.0	1016.2	0.984	1.4143			1.0	1016.2	0.984	1.4143	

In all the property measurements, temperature was controlled within an accuracy of ± 0.01 K using a constant-temperature bath. A Julabo immersion cooler (FT 200,

Julabo Labortechnik, GmbH, Germany) was used to cool the water bath. This unit was installed at the intake of a heating circulator to draw the heat away from the circulator.

Table 3. Derived Parameters of Equation 3 for Various Functions at Different Temperatures

function	temp/K	A_1	A_2	A_3	σ
1,4-Dioxane (1) + Ethanediol (2)					
$V^E/10^{-6}(\text{m}^3\cdot\text{mol}^{-1})$	298.15	-1.099	-0.024	-0.037	0.0104
	303.15	-1.174	-0.046	-0.218	0.0106
	308.15	-1.226	-0.015	-0.432	0.0114
$\Delta\eta/(\text{mPa}\cdot\text{s})$	298.15	-17.25	-9.156	-3.606	0.0453
	303.15	-12.65	-5.325	-0.761	0.0634
	308.15	-9.800	-4.515	-2.079	0.0173
$\Delta R \times 10^6/(\text{m}^3\cdot\text{mol}^{-1})$	298.15	-3.216	-0.778	-0.245	0.0066
	303.15	-3.062	-0.923	0.163	0.0063
	308.15	-3.041	-0.987	0.254	0.0066
$\Delta u/(\text{m}\cdot\text{s}^{-1})$	298.15	-187.4	66.04	115.9	2.040
$\Delta k_s/(\text{TPa}^{-1})$	298.15	-59.87	61.21	-0.87	4.139
1,4-Dioxane (1) + Hexane (2)					
$V^E/10^{-6}(\text{m}^3\cdot\text{mol}^{-1})$	298.15	1.522	2.073	-0.869	0.0167
	303.15	1.418	1.800	-1.244	0.0177
	308.15	1.391	1.664	-1.470	0.0241
$\Delta\eta/(\text{mPa}\cdot\text{s})$	298.15	-1.039	0.588	-0.366	0.0054
	303.15	-0.924	0.495	-0.268	0.0025
	308.15	-0.827	0.439	-0.228	0.0021
$\Delta R \times 10^6/(\text{m}^3\cdot\text{mol}^{-1})$	298.15	-3.340	0.742	0.124	0.0186
	303.15	-3.301	0.886	0.161	0.0215
	308.15	-3.310	0.953	0.092	0.0358
$\Delta u/(\text{m}\cdot\text{s}^{-1})$	298.15	-242.2	-14.54	27.26	3.036
$\Delta k_s/(\text{TPa}^{-1})$	298.15	-286.1	-136.6	94.50	4.725
1,4-Dioxane (1) + Tributylamine (2)					
$V^E/10^{-6}(\text{m}^3\cdot\text{mol}^{-1})$	298.15	3.722	-0.988	0.177	0.0337
	303.15	3.774	-1.053	0.244	0.0505
	308.15	4.030	-0.858	0.429	0.0757
$\Delta\eta/(\text{mPa}\cdot\text{s})$	298.15	-0.557	-0.184	-0.309	0.0025
	303.15	-0.457	-0.106	-0.157	0.0036
	308.15	-0.462	-0.132	-0.243	0.0030
$\Delta R \times 10^6/(\text{m}^3\cdot\text{mol}^{-1})$	298.15	-37.14	18.75	-11.07	0.0923
	303.15	-37.10	18.92	-11.17	0.1211
	308.15	-37.04	18.83	-11.02	0.1233
$\Delta u/(\text{m}\cdot\text{s}^{-1})$	298.15	-120.47	-13.50	-67.48	0.607
$\Delta k_s/(\text{TPa}^{-1})$	298.15	-57.5	-53.7	193.5	3.199
1,4-Dioxane (1) + Triethylamine (2)					
$V^E/10^{-6}(\text{m}^3\cdot\text{mol}^{-1})$	298.15	-0.406	1.054	-0.631	0.0133
	303.15	-0.333	1.170	-0.472	0.0160
	308.15	-0.362	1.133	-0.621	0.0092
$\Delta\eta/(\text{mPa}\cdot\text{s})$	298.15	-0.877	0.426	-0.145	0.0012
	303.15	-0.777	0.350	-0.084	0.0016
	308.15	-0.699	0.313	-0.079	0.0011
$\Delta R \times 10^6/(\text{m}^3\cdot\text{mol}^{-1})$	298.15	-5.821	1.408	-0.322	0.0216
	303.15	-5.776	1.486	-0.278	0.0109
	308.15	-5.772	1.468	-0.266	0.0146
$\Delta u/(\text{m}\cdot\text{s}^{-1})$	298.15	-96.39	-34.11	47.25	2.058
$\Delta k_s/(\text{TPa}^{-1})$	298.15	-289.8	-11.14	-4.38	1.183

ing bath liquid. An immersion probe was connected to the instrument with a flexible and insulated tube, which maintained the constant temperature of the bath.

Results and Discussion

At least three independent readings of all the physical property measurements on ρ , η , n_D , and u were taken for each composition, and the averages of these experimental values are presented in Table 2.

From the density results, excess molar volume, V^E , has been calculated as

$$V^E = V_m - V_1x_1 - V_2x_2 \quad (1)$$

Here, V_m is the molar volume of the mixture, V_1 and V_2 are the molar volumes of the respective pure components, and x_i represents the mole fraction of the i th component of the mixture. In a similar manner, the results of $\Delta\eta$, ΔR , Δu , and Δk_s have been calculated using the values of η , n_D , and u from a general relationship of the type used earlier,¹²⁻¹⁴

$$\Delta Y = Y_m - Y_1x_1 - Y_2x_2 \quad (2)$$

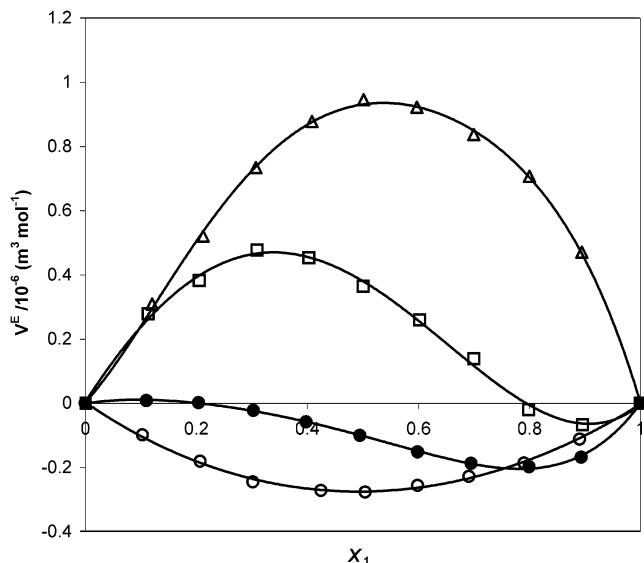


Figure 1. Excess molar volume vs mole fraction of 1,4-dioxane with (○) ethanediol, (□) hexane, (△) tributylamine, and (●) triethylamine at 298.15 K.

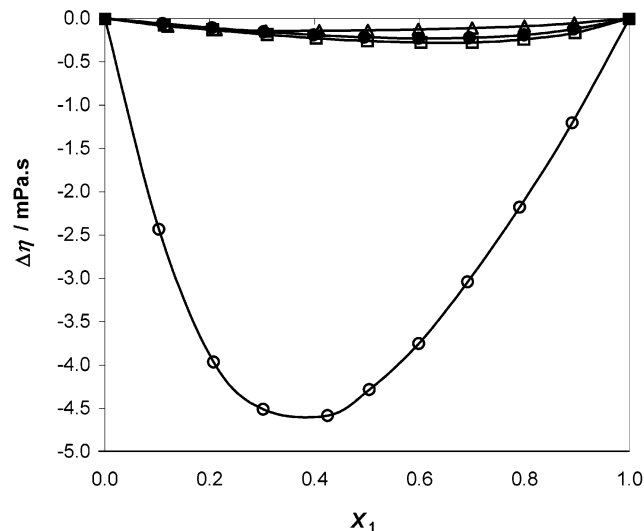


Figure 2. Deviations in viscosity vs mole fraction at 298.15 K for the same mixtures presented in Figure 1.

In the above equation, ΔY represents $\Delta\eta$, ΔR , Δu , and Δk_s , respectively, while Y_m represents the respective mixture properties, viz., viscosity, η , molar refractivity, R (calculated from the Lorentz–Lorentz relation), speed of sound, u , and isentropic compressibility, k_s ($=1/u^2\rho$) of the binary mixture; the symbol Y_i refers to the same properties for the pure components of the mixture. For calculation of ΔR and Δk_s , volume fraction, ϕ_i ($=\sum x_i v_i / \sum_{j=1}^2 x_j v_j$), was used,^{10,15,16} but for calculation of $\Delta\eta$ and Δu , the mole fraction, x_i , was used.

All the quantities (V^E , $\Delta\eta$, ΔR , Δu , and Δk_s) have been fitted to the Redlich and Kister¹⁷ equation by the method of least-squares using the Marquardt algorithm¹⁸ to derive the binary coefficients, A_j , and the standard deviation, σ , as follows.

$$V^E(\Delta Y) = x_1x_2 \sum_{j=1}^k A_j(x_2 - x_1)^{j-1} \quad (3)$$

In each case, the optimum number of coefficients, A_j , was determined from an examination of the variation of stan-

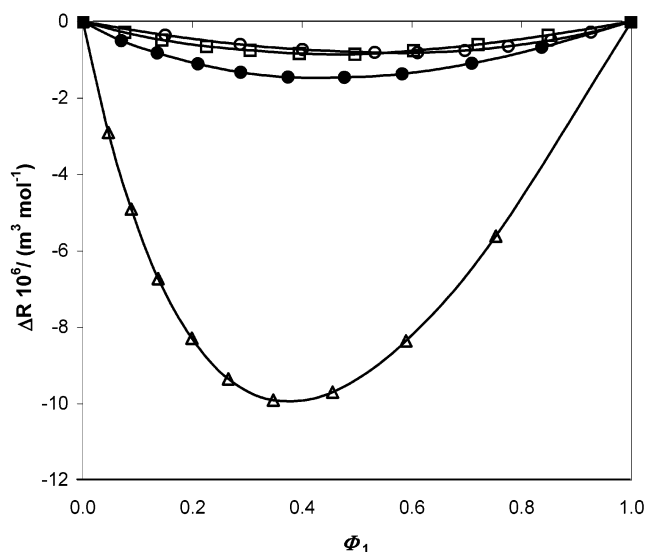


Figure 3. Deviations in molar refraction (ΔR) vs volume fraction at 298.15 K for the same mixtures presented in Figure 1.

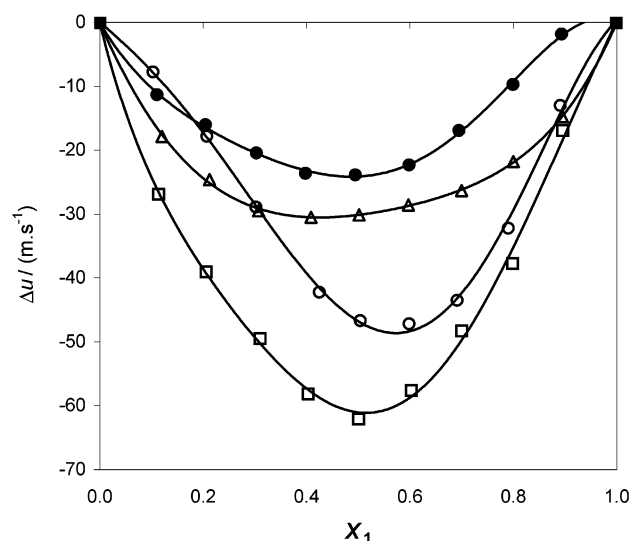


Figure 4. Deviations in speed of sound (Δu) vs mole fraction at 298.15 K for the same mixtures presented in Figure 1.

standard deviation, σ , as calculated by

$$\sigma = \left(\frac{\sum (Y_{\text{cal}}^E - Y_{\text{obs}}^E)^2}{n - m} \right)^{1/2} \quad (4)$$

Here n represents the number of measurements and m is the number of coefficients used in fitting the data. The estimated values of A_j and σ for V^E , $\Delta\eta$, ΔR , Δu , and Δk_s are presented in Table 3. In all the cases, the best fit was found by using only three adjustable fitting coefficients in eq 3.

The results of V^E at 298.15 K presented in Figure 1 display widely varying trends. For instance, a large positive V^E is observed for mixtures of 1,4-dioxane + tributylamine, while a negative V^E is observed for the 1,4-dioxane + ethanediol mixture. On the other hand, for mixtures of 1,4-dioxane + hexane or 1,4-dioxane + triethylamine, V^E plots exhibit slightly sigmoidal trends. Particularly, with 1,4-dioxane + triethylamine, the V^E values are negative over the entire range of mixture composition, whereas with 1,4-dioxane + n -hexane, the V^E values are positive up to $x_1 =$

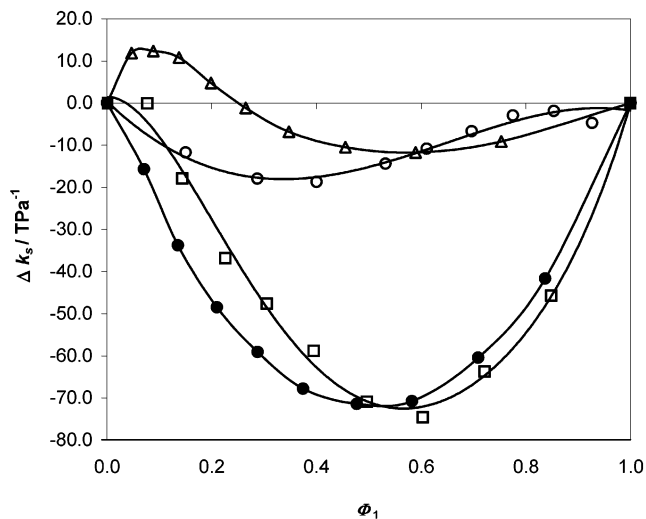


Figure 5. Deviations in isentropic compressibility (Δk_s) vs volume fraction at 298.15 K for the same mixtures presented in Figure 1.

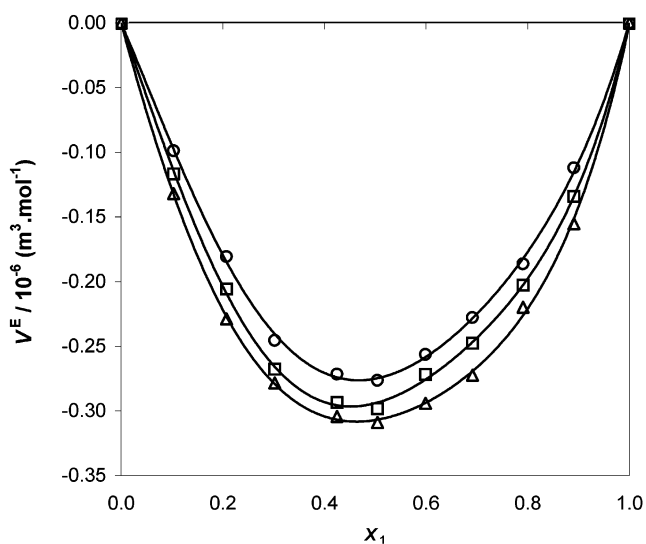


Figure 6. Effect of temperature on V^E for the 1,4-dioxane + ethanediol mixture at (○) 298.15 K, (□) 303.15 K, and (△) 308.15 K.

0.9, beyond which the V^E curve falls in the negative scale. This may be due to dispersion forces between the mixing components. Such widely differing trends are attributed to different types of interactions depending upon the dipole moment, dielectric constant, and molar volumes of the second mixing component of the mixture. For instance, ethanediol has the highest values of dipole moment ($\mu = 2.31$) and dielectric constant ($D = 37.70$) among the liquids considered here. The large differences of these values between 1,4-dioxane and ethanediol indicate the specific interactions between them leading to negative V^E over the whole range of mixture composition. The large positive V^E observed for 1,4-dioxane + tributylamine is attributed to their large differences in molar volumes ($V_1 = 85.74 \text{ cm}^3/\text{mol}$ and $V_2 = 239.3 \text{ cm}^3/\text{mol}$) as well as their almost identical values of dielectric constants ($D_1 = 2.21$ and $D_2 = 2.29$) leading to expansion of volume upon mixing.

Variation of $\Delta\eta$ versus x_1 at 298.15 K, displayed in Figure 2, shows a large negative $\Delta\eta$ for mixtures of 1,4-dioxane + triethylamine, whereas for all the remaining mixtures, negative values of $\Delta\eta$ are almost identical over the entire mixture composition. A similar trend is observed for the dependence of ΔR versus ϕ_1 as shown in Figure 3, except

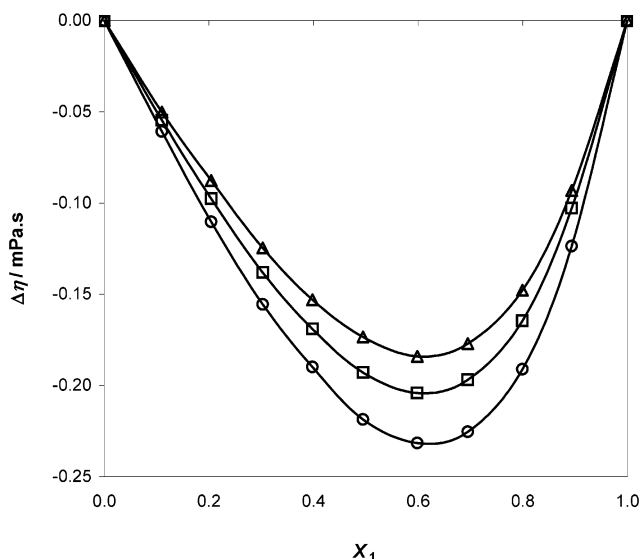


Figure 7. Effect of temperature on $\Delta\eta$ for the 1,4-dioxane + ethanediol mixture at (○) 298.15 K, (□) 303.15 K, and (△) 308.15 K.

that the largest negative ΔR is observed for mixtures of 1,4-dioxane with tributylamine.

The results of Δu versus x_1 at 298.15 K are displayed in Figure 4. It is observed that for all the mixtures, Δu values are negative, and these negative values vary in the following order: hexane > ethanediol > tributylamine > triethylamine. The results of Δk_s versus ϕ_1 at 298.15 K displayed in Figure 5 are slightly different. For instance, with mixtures of 1,4-dioxane + triethylamine or 1,4-dioxane + hexane, Δk_s values are negative over the entire mixture composition. On the other hand, for mixtures of 1,4-dioxane with ethanediol or tributylamine, the Δk_s versus ϕ_1 curves exhibit somewhat sigmoidal trends. For 1,4-dioxane + ethanediol mixtures, these variations are in the negative scale, whereas with 1,4-dioxane + tributylamine, the values of Δk_s vary in both the positive and negative scales.

The effect of temperature on V^E and $\Delta\eta$ is quite systematic as displayed typically in Figures 6 and 7, in the case of the dioxane + ethanediol mixture. It is found that V^E results become more negative with increasing temperature, whereas $\Delta\eta$ values show the reverse trend.

In all the plots, points represent the quantities calculated from eqs 1 and 2, while the smooth curves are drawn from the best-fitted values calculated from eq 3.

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