Density, Viscosity, Refractive Index, and Speed of Sound in the Binary Mixtures of Ethyl Chloroacetate + Cyclohexanone, + Chlorobenzene, + Bromobenzene, or + Benzyl Alcohol at (298.15, 303.15, and 308.15) K

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Densities, viscosities, and refractive indices at (298.15, 303.15, and 308.15) K, and the speed of sound at 298.15 K, are presented as a function of mixture composition for the binary mixtures of ethyl chloroacetate + cyclohexanone, + chlorobenzene, + bromobenzene, or + benzyl alcohol. Using these data, excess molar volume and deviations in viscosity, molar refraction, and speed of sound have been calculated. These results have been correlated with the Redlich and Kister polynomial equation to derive the coefficients and standard errors. Variations in the calculated excess quantities have been studied for mixtures of ethyl chloroacetate with component liquids.

Introduction

In our earlier papers, we have studied the thermodynamic and hydrodynamic properties of binary mixtures of ethyl chloroacetate with alkanes[1] as well as substituted monocyclic aromatic liquids.[2] In continuation of this research, we now present additional data on density (ρ), viscosity (η), refractive index (n_D), for the sodium D-line at (298.15, 303.15, and 308.15) K, and speed of sound (u) at 298.15 K for the binary mixtures of ethyl chloroacetate with cyclohexanone, chlorobenzene, bromobenzene, or benzyl alcohol. Such a database will have great relevance in process engineering as well as other industrial sectors. Moreover, we are not aware of any physical property data in the earlier literature on these mixtures, and hence, an attempt has been made to measure the properties and then compute excess molar volume (V^E) and deviations in viscosity ($\Delta\eta$), molar refraction (ΔR), and speed of sound (Δu). The computed results have been fitted to the Redlich and Kister equation[3] to derive the binary coefficients and estimate the standard errors. These results have been presented for the mixtures.

Experimental Section

Materials. High purity laboratory reagent grade samples of chlorobenzene, cyclohexanone, bromobenzene, and benzyl alcohol were procured from s.d. fine Chemicals, Mumbai, India. Ethyl chloroacetate was purchased from BASCO, Mumbai, India. The mole percent purities of these liquids as determined by GC (HP 6890) using a FID detector were >99 and are reported in Table 1 along with the density and refractive index data measured at 298.15 K for the pure liquids, and these are compared with the literature values.

Binary mixtures were prepared by mass in specially designed airtight glass bottles.[4] The mass measurements accurate to ±0.01 mg were performed on a digital electronic balance (Mettler, AE 240, Switzerland). A set of nine compositions was prepared for each mixture, and their physical properties were measured at the respective compositions in the mole fraction scale from 0.1 to 0.9 in steps of 0.1. In all cases, the possible error in mole fraction was less than 0.0002.

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

liquid	mol % purity	ρ/kg·m^{-3} expt	ρ/kg·m^{-3} lit.	n_D expt	n_D lit.
ethyl chloroacetate	>99.0	1157.0[a]	1158.5[a]	1.4211[a]	1.4215[a]
cyclohexanone	>99.0	942.4	945.2[15]	1.4480	1.4500[15]
chlorobenzene	>99.5	110.1	110.0[15]	1.5222	1.5218[15]
bromobenzene	>97.0	148.9	148.8[15]	1.5577	1.5570[15]
benzyl alcohol	>99.7	1041.6	1041.3[15]	1.5378	1.5383[15]

[a] Measured and compared at 293.15 K.

Methods. Densities of liquids and liquid mixtures were measured to an accuracy of ±0.0001 g·cm^{-3} using a capillary-type pycnometer of capacity 10 cm^3 volume. Experimental details of density measurements are the same as reported earlier.[4−6]

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 accurate to ±0.001 mPa·s. Calibrations of the pycnometer remain the same as described previously.[4,5]

Refractive indices for the sodium D-line were measured using a thermostatically controlled Abbe refractometer (Atago 3T, Japan). A minimum of three independent readings was taken for each composition, and their average value was considered in all the calculations. Refractive index data are accurate to ±0.0001 units.

Speed of sound was measured by 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

† CEPS Communication no. 10.

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10.1021/je0201828 CCC: $25.00
Published on Web 04/08/2003

Table 2. Experimental Density (ρ/kg·m^{-3}), Viscosity (η/mPa·s), Refractive Index (n_D), and Speed of Sound (u/m·s^{-1}) of Binary Mixtures at Different Temperatures

x_1	ρ	η	n_D	u	x_1	ρ	η	n_D	u	x_1	ρ	η	n_D	u	x_1	ρ	η	n_D	u
Ethyl Chloroacetate (1) + Cyclohexanone(2)					Ethyl Chloroacetate (1) + Chlorobenzene(2)					Ethyl Chloroacetate (1) + Bromobenzene(2)					Ethyl Chloroacetate (1) + Benzyl Alcohol(2)				
298.15 K					298.15 K					298.15 K					298.15 K				
0.0000	942.5	1.963	1.4486	1417	0.0000	1101.0	0.773	1.5223	1271	0.0000	1489.0	1.080	1.5577	1162	0.0000	1041.0	5.313	1.5378	1541
0.1084	964.3	1.745	1.4453	1396	0.1064	1106.0	0.799	1.5108	1266	0.1089	1451.0	1.079	1.5419	1170	0.1072	1053.0	4.078	1.5258	1503
0.2069	984.4	1.604	1.4424	1379	0.2055	1111.0	0.828	1.5002	1264	0.2034	1418.0	1.079	1.5284	1176	0.2035	1064.0	3.295	1.5142	1472
0.3060	1004.0	1.493	1.4395	1361	0.3000	1115.0	0.858	1.4902	1262	0.3024	1383.0	1.080	1.5145	1183	0.2998	1075.0	2.729	1.5030	1442
0.3993	1023.0	1.403	1.4367	1345	0.3972	1120.0	0.889	1.4802	1261	0.3976	1351.0	1.082	1.5011	1190	0.3950	1085.0	2.285	1.4918	1412
0.5032	1044.0	1.352	1.4337	1328	0.5005	1124.0	0.925	1.4696	1261	0.5011	1315.0	1.085	1.4868	1199	0.5000	1096.0	1.923	1.4794	1380
0.6002	1064.0	1.283	1.4309	1312	0.5976	1129.0	0.959	1.4596	1261	0.6018	1281.0	1.087	1.4730	1209	0.5984	1106.0	1.669	1.4675	1352
0.6945	1083.0	1.230	1.4283	1297	0.6985	1133.0	0.996	1.4493	1260	0.6992	1247.0	1.092	1.4598	1218	0.6981	1116.0	1.459	1.4556	1325
0.7947	1104.0	1.191	1.4256	1283	0.7953	1137.0	1.031	1.4390	1258	0.7963	1214.0	1.096	1.4469	1222	0.7972	1126.0	1.326	1.4437	1299
0.8975	1124.0	1.144	1.4228	1267	0.8963	1141.0	1.064	1.4294	1254	0.8948	1181.0	1.098	1.4338	1239	0.8941	1135.0	1.188	1.4320	1275
1.0000	1145.0	1.095	1.4200	1250	1.0000	1145.0	1.095	1.4200	1250	1.0000	1145.0	1.095	1.4200	1250	1.0000	1145.0	1.095	1.4200	1250
303.15 K					303.15 K					303.15 K					303.15 K				
0.0000	937.3	1.768	1.4461		0.0000	1095.0	0.725	1.5195		0.0000	1481.0	1.013	1.5542		0.0000	1037.0	4.515	1.5354	
0.1084	959.1	1.595	1.4430		0.1064	1100.0	0.752	1.5076		0.1089	1443.0	1.012	1.5385		0.1072	1050.0	3.567	1.5233	
0.2069	979.1	1.470	1.4399		0.2055	1105.0	0.778	1.4972		0.2034	1410.0	1.011	1.5256		0.2035	1060.0	2.887	1.5114	
0.3060	999.6	1.376	1.4370		0.3000	1109.0	0.804	1.4878		0.3024	1376.0	1.010	1.5118		0.2998	1070.0	2.428	1.5000	
0.3993	1018.0	1.298	1.4342		0.3972	1113.0	0.834	1.4773		0.3976	1344.0	1.011	1.4982		0.3950	1080.0	2.036	1.4890	
0.5032	1038.0	1.244	1.4312		0.5005	1118.0	0.865	1.4665		0.5011	1308.0	1.012	1.4839		0.5000	1091.0	1.747	1.4765	
0.6002	1058.0	1.184	1.4286		0.5976	1122.0	0.895	1.4566		0.6018	1274.0	1.014	1.4705		0.5984	1100.0	1.516	1.4706	
0.6945	1077.0	1.139	1.4255		0.6985	1126.0	0.927	1.4467		0.6992	1241.0	1.017	1.4578		0.6981	1113.0	1.334	1.4528	
0.7947	1097.0	1.101	1.4230		0.7953	1130.0	0.959	1.4366		0.7963	1207.0	1.018	1.4444		0.7972	1119.0	1.209	1.4410	
0.8975	1118.0	1.058	1.4203		0.8963	1134.0	0.986	1.4270		0.8948	1174.0	1.010	1.4313		0.8941	1128.0	1.092	1.4295	
1.0000	1139.0	1.013	1.4173		1.0000	1139.0	1.013	1.4173		1.0000	1139.0	1.013	1.4173		1.0000	1139.0	1.013	1.4173	
308.15 K					308.15 K					308.15 K					308.15 K				
0.0000	932.3	1.602	1.4440		0.0000	1090.0	0.683	1.5166		0.0000	1473.0	0.953	1.5514		0.0000	1033.0	3.877	1.5318	
0.1084	954.3	1.444	1.4406		0.1064	1094.0	0.706	1.5050		0.1089	1436.0	0.947	1.5358		0.1072	1045.0	3.058	1.5208	
0.2069	974.1	1.336	1.4375		0.2055	1099.0	0.728	1.4946		0.2034	1403.0	0.943	1.5224		0.2035	1055.0	2.480	1.5097	
0.3060	994.0	1.259	1.4346		0.3000	1103.0	0.751	1.4846		0.3024	1369.0	0.940	1.5086		0.2998	1065.0	2.127	1.4982	
0.3993	1012.0	1.190	1.4319		0.3972	1107.0	0.776	1.4745		0.3976	1337.0	0.938	1.4954		0.3950	1075.0	1.815	1.4866	
0.5032	1033.0	1.135	1.4289		0.5005	1112.0	0.804	1.4639		0.5011	1301.0	0.938	1.4812		0.5000	1085.0	1.572	1.4738	
0.6002	1052.0	1.087	1.4262		0.5976	1116.0	0.831	1.4539		0.6018	1267.0	0.942	1.4675		0.5984	1095.0	1.365	1.4620	
0.6945	1071.0	1.047	1.4236		0.6985	1120.0	0.858	1.4438		0.6992	1234.0	0.941	1.4543		0.6981	1104.0	1.209	1.4501	
0.7947	1091.0	1.011	1.4207		0.7953	1124.0	0.885	1.4338		0.7963	1201.0	0.941	1.4413		0.7972	1114.0	1.093	1.4384	
0.8975	1112.0	0.972	1.4178		0.8963	1128.0	0.910	1.4243		0.8948	1167.0	0.939	1.4284		0.8941	1122.0	0.997	1.4270	
1.0000	1132.0	0.930	1.4148		1.0000	1132.0	0.930	1.4148		1.0000	1132.0	0.930	1.4148		1.0000	1132.0	0.930	1.4148	

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 thermostat maintained at (298.15 ± 0.01) K. Details of the speed of sound measurements have been given earlier,[1,2] and these values are accurate to ±2 in 1000 m·s^{-1}.

In all the property measurements, temperature was controlled to 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 circulating bath liquid. The immersion probe was connected to the instrument with a flexible and insulated tube. To prevent the immersion probe from icing, it was completely immersed in the bath liquid.

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

Results and Discussion

From the density results, excess molar volumes (V^E) have 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 pure components, and x_i represents the mole fraction of the ith component of the mixture. In a similar manner, the results of $\Delta\eta$, ΔR, and Δu have been calculated using the values of η, n_D, and u from a general relationship of the type used earlier[1,2,7−10]:

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

In the above equation, ΔY represents $\Delta\eta$, ΔR, and Δu, respectively, while Y_m represents the respective mixture properties, viz., molar refractivity (R) (calculated from the Lorenz−Lorentz relation), viscosity (η), and speed of sound (u) of the binary mixture; the symbol Y_i refers to the same properties of the pure components in the mixtures. While calculating ΔR, volume fraction (ϕ_i) was used,[5,9,11−13,] but for calculating $\Delta\eta$ and Δu, the mole fraction (x_i) was used.

All the quantities (V^E, $\Delta\eta$, ΔR, and Δu) have been fitted to the Redlich and Kister[3] equation by the method of least-squares using the Marquardt algorithm,[14] to derive the binary coefficient (A_i) and standard deviation (σ):

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

In each case, the optimum number of coefficients A_j was determined from an examination of the variation of standard deviation (σ) as calculated by

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

where 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, and Δu are given in Table 3. In all the cases, the best fit was found by using only three adjustable fitting coefficients in eq 3.

Table 3. Estimated Parameters of Eq 3 for Various Functions of the Binary Mixtures at Different Temperatures

function	temp/K	A_1	A_2	A_3	σ
Ethyl Chloroacetate (1) + Cyclohexanone (2)					
$V^E/10^{-6}$(m^3·mol^{-1})	298.15	0.636	0.138	−0.125	0.006
	303.15	0.555	−0.019	0.085	0.043
	308.15	0.275	0.193	−0.391	0.003
$\Delta\eta$/mPa·s	298.15	−0.747	−0.505	−0.193	0.008
	303.15	−0.609	−0.374	−0.089	0.004
	308.15	−0.523	−0.342	−0.107	0.002
$10^6\Delta R$/m^3·mol^{-1}	298.15	0.142	−0.046	−0.033	0.001
	303.15	0.130	−0.008	0.134	0.018
	308.15	0.073	0.043	−0.132	0.002
Δu/m·s^{-1}	298.15	−21.42	−6.91	12.37	0.885
Ethyl Chloroacetate (1) +Chlorobenzene (2)					
$V^E/10^{-6}$(m^3·mol^{-1})	298.15	−0.338	0.019	0.062	0.005
	303.15	−0.175	−0.357	0.972	0.029
	308.15	−0.210	0.138	0.316	0.004
$\Delta\eta$/mPa·s	298.15	−0.036	−0.063	0.015	0.000
	303.15	−0.019	−0.049	0.027	0.000
	308.15	−0.013	−0.058	0.030	0.001
$10^6\Delta R$/m^3·mol^{-1}	298.15	0.229	−0.166	−0.371	0.014
	303.15	0.252	0.007	0.002	0.013
	308.15	0.233	−0.190	−0.227	0.010
Δu/m·s^{-1}	298.15	2.02	−34.76	−8.99	0.495
Ethyl Chloroacetate (1) + Bromobenzene (2)					
$V^E/10^{-6}$(m^3·mol^{-1})	298.15	0.079	0.130	−0.114	0.002
	303.15	0.095	−0.218	0.736	0.028
	308.15	−0.235	−0.005	0.145	0.001
$\Delta\eta$/mPa·s	298.15	−0.014	−0.039	0.035	0.001
	303.15	−0.001	−0.025	0.005	0.005
	308.15	−0.011	−0.063	0.037	0.001
$10^6\Delta R$/m^3·mol^{-1}	298.15	0.189	−0.037	−0.028	0.001
	303.15	0.299	0.121	0.321	0.017
	308.15	0.152	−0.027	−0.088	0.002
Δu/m·s^{-1}	298.15	−27.35	0.82	13.63	0.317
Ethyl Chloroacetate (1) + Benzyl Alcohol (2)					
$V^E/10^{-6}$(m^3·mol^{-1})	298.15	−0.727	−0.185	0.208	0.011
	303.15	−0.766	−0.065	0.025	0.213
	308.15	−0.819	0.109	−0.091	0.004
$\Delta\eta$/mPa·s	298.15	−5.106	−2.677	−1.257	0.019
	303.15	−4.106	−1.944	−0.779	0.019
	308.15	−3.362	−1.706	−1.063	0.027
$10^6\Delta R$/m^3·mol^{-1}	298.15	0.505	−0.116	−0.094	0.009
	303.15	0.799	0.270	−0.952	0.128
	308.15	0.557	−0.490	0.333	0.006
Δu/m·s^{-1}	298.15	−60.66	3.71	−4.41	0.714

The results of excess molar volume displayed at 298.15 K in Figure 1 show wide variations depending upon the nature of the second component in the mixture. In the case of the ethyl chloroacetate + cyclohexanone mixture, positive values of excess molar volume are observed, signifying mild dispersion type interactions between the ketonic group of cyclohexanone and the ester moiety of ethyl chloroacetate. The excess molar volume data of mixtures of ethyl chloroacetate + bromobenzene are quite small and exhibit an incipient inversion. At higher amounts of ethyl chloroacetate (i.e., $x_1 = 0.8$ and 0.9), the V^E curve exhibits a slight negative trend, but it is still very close to the ideal behavior. This indicates the varying interactions between the components with the mixture composition. In the case of mixtures of ethyl chloroacetate + chlorobenzene, the values of V^E are negative but are larger in magnitude when compared to those for the ethyl chloroacetate + benzyl alcohol mixtures, for which the V^E values are more negative, which may be due to the formation of weak molecular complexes, even when compared to all the remaining mixtures. The V^E results at other higher temperatures follow the same trends but show different values, and these data are not displayed to minimize the number of plots.

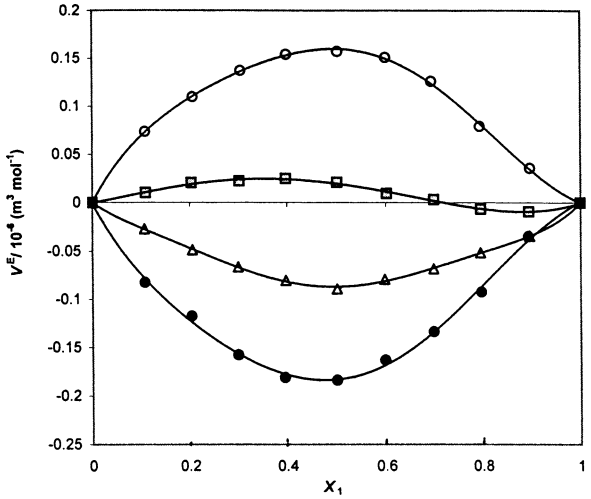

Figure 1. Excess molar volume vs mole fraction of ethyl chloroacetate with (○) cyclohexanone, (△) chlorobenzene, (□) bromobenzene, and (●) benzyl alcohol at 298.15 K.

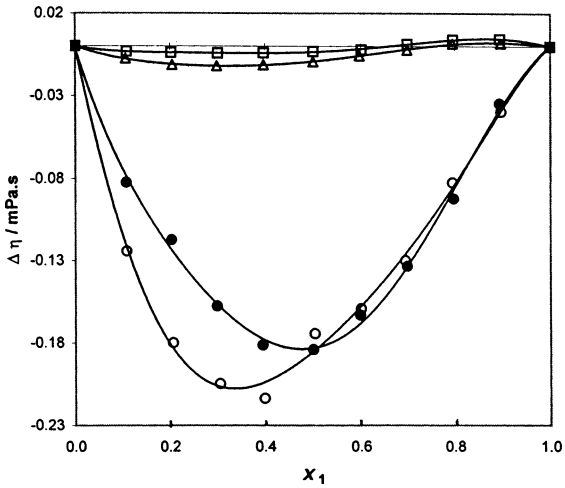

Figure 2. Deviations in viscosity vs mole fraction at 298.15 K for the same mixtures presented in Figure 1.

From the curves of $\Delta\eta$ versus x_1 at 298.15 K displayed in Figure 2, we find that the $\Delta\eta$ values for mixtures of ethyl chloroacetate + cyclohexanone or + benzyl alcohol are quite negative when compared to those for the other mixtures. However, for mixtures of ethyl chloroacetate + bromobenzene or + chlorobenzene, the variations of $\Delta\eta$ values with mixture composition are quite identical and exhibit slightly negative $\Delta\eta$ values up to $x_1 = 0.7$, but after this composition, the curves show slightly positive values. The temperature variation plots of $\Delta\eta$ versus x_1 are not displayed due to slight variations with temperature. However, there is a general decrease in viscosity with increasing temperature.

The results of ΔR versus ϕ_1 (volume fraction of ethyl chloroacetate) at 298.15 K are presented in Figure 3. The positive ΔR curves for mixtures of ethyl chloroacetate + cyclohexanone, or + bromobenzene, vary almost identically over the entire composition range. The large positive ΔR values are observed for mixtures of ethyl chloroacetate + benzyl alcohol, while, in the case of mixtures of ethyl chloroacetate + chlorobenzene, the ΔR versus ϕ_1 dependence shows a sigmoidal trend. The effect of temperature on ΔR is not considerable for all the binary mixtures, and hence, this dependence is not displayed.

The plot of Δu versus x_1 shown in Figure 4 is almost linear for the ethyl chloroacetate + benzyl alcohol mixture. On the other hand, for ethyl chloroacetate + chlorobenzene,

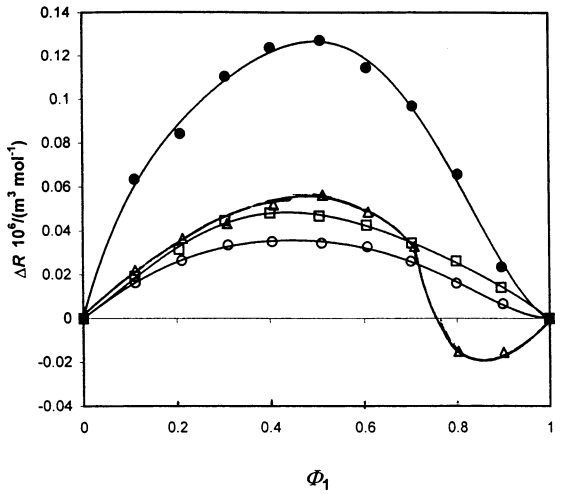

Figure 3. Deviations in molar refraction (ΔR) vs volume fraction at 298.15 K for the same mixtures presented in Figure 1.

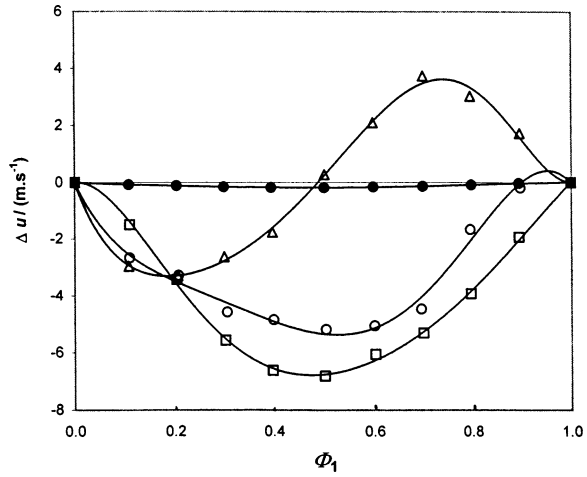

Figure 4. Deviations in speed of sound vs mole fraction at 298.15 K for the same mixtures presented in Figure 1.

the Δu versus x_1 curve displays a sigmoidal trend, but in quite the opposite direction to that exhibited for the ΔR dependence of this mixture (see Figure 3). However, for mixtures of ethyl chloroacetate + cyclohexanone or + bromobenzene, Δu curves are in the negative globe and also display a quite opposite behavior to that observed for ΔR versus ϕ_1 as shown in Figure 3.

It may be noted that, in all the plots, the 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.

Acknowledgment

This research was funded by the Department of Science and Technology, New Delhi, India (SP/S1/H-09/2000).

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Received for review September 18, 2002. Accepted January 10, 2003.

JE0201828