Densities, Speeds of Sound, Refractive Indices, and the Corresponding Changes of Mixing at 25 °C and Atmospheric Pressure for Systems Composed by Ethyl Acetate, Hexane, and Acetone

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Densities, speeds of sound, and refractive indices of the ternary system composed of ethyl acetate, hexane, and acetone and the binary combinations of these constituents have been measured. The excess molar volumes and isentropic compressibility and molar refraction changes of mixing were calculated and correlated using the Redlich−Kister expression.

1. Introduction

Pure solvents and their mixtures have broad applications in the pharmaceutical industry, usually in the purification processes. Because of this, it is of interest to determine their physical properties. For example, the mixture of ethyl acetate + hexane + acetone is obtained as the result of the column chromatographic purification of the Prostaglandin F2α (PGF2α),[1] an intermediate in the production of Prostacyclin (of important medical applications). Despite the industrial importance of this mixture, in the open literature we have found only some physical properties for binary system ethyl acetate + hexane[2] at 25 °C and hexane + acetone[3] at 35 °C. We here report the densities, speeds of sound, and refractive indices of the ternary system composed of ethyl acetate, hexane, and acetone and its component binary subsystems. The results are used to calculate excess molar volumes and isentropic compressibility and molar refraction changes of mixing over the entire mole fraction range at 25 °C and atmospheric pressure. These data are fitted using the Redlich−Kister polynomial.

2. Experimental Section

Materials. Ethyl acetate (99.9% mass, HPLC grade) and acetone (99.9% mass, HPLC grade) were purchased from Sigma and hexane (>99.5% mass, HPLC grade) was supplied from Fluka. Gas chromatographic analysis of these solvents showed no impurities. Thus, they were used without further purification. Properties of pure solvents are shown in Table 1, together with published values for these parameters.[4−7]

Apparatus and Procedure. The samples were prepared by mass, using a Mettler AE 240 balance with a precision of ±0.1 mg. Densities of the mixtures were measured with a precision of ±0.000 01 g·cm^{-3} in an Anton Paar DMA-60/602 vibrating tube densimeter. The speeds of sound were measured using an Anton Paar DSA-48 densimeter and sound analyzer with a precision of ±1 m·s^{-1}. This equipment was calibrated with air and water.

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Table 1. Densities ρ, Speeds of Sound u, and Refractive Indices n_D of Pure Components at 25 °C and Atmospheric Pressure

	ρ (g·cm^{-3})		u (m·s^{-1})		n_D	
component	exptl	lit.	exptl	lit.	exptl	lit.
ethyl acetate	0.89431	0.89455[4]	1140	1144[5]	1.36986	1.36978[4]
hexane	0.65507	0.65484[4]	1077	1078[7]	1.37236	1.37226[4]
acetone	0.78483	0.78440[4]	1163	1161[6]	1.35599	1.35596[4]

Refractive indices were measured using an ATAGO RX-5000 refractometer, the precision being ±0.000 04. Temperature was kept at (298.15 ± 0.02) K with a Hetotherm Thermostat.

3. Results and Data Treatment

The experimental values obtained for the densities (ρ), sound speeds (u), and refractive indices (n_D) for binary and ternary systems are listed in Tables 2 and 3, respectively. These tables include the calculated values of isentropic compressibilities (κ_s), excess molar volumes (V^E), and molar refraction and isentropic compressibility changes of mixing (ΔR, $\Delta\kappa_S$).

From the densities, the excess molar volumes have been calculated as

$$V^E = V - \sum_i x_i V_i \tag{1}$$

where V_i represents the molar volume of pure component i and V refers to the molar volume of the mixture calculated as

$$V = \frac{\sum x_i M_i}{\rho} \tag{2}$$

Here, x_i and M_i are the mole fraction and molar mass of component i in the mixture.

Molar refraction R was calculated using the Lorentz−Lorenz equation.

$$R = V\frac{(n_D^2 - 1)}{(n_D^2 + 2)} \tag{3}$$

where n_D is the refractive index.

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Table 2. Densities ρ, Speeds of Sound u, Isentropic Compressibilities κ_S, Refractive Indices n_D, Excess Molar Volumes V^E, and $\Delta\kappa_S$ and ΔR Changes of Mixing for Binary Systems at 25 °C

x_1	ρ	u	κ_S	n_D	V^E	$\Delta\kappa_S$	ΔR
	g·cm^{-3}	m·s^{-1}	TPa^{-1}		cm^3·mol^{-1}	TPa^{-1}	cm^3·mol^{-1}
Ethyl Acetate (1) + Hexane (2)							
0.0615	0.66464	1073	1308	1.37125	0.315	13	0.001
0.1294	0.67600	1069	1295	1.37045	0.570	24	0.014
0.1618	0.68172	1068	1287	1.37009	0.662	29	0.015
0.2026	0.68915	1066	1277	1.36981	0.754	33	0.024
0.2534	0.69879	1065	1261	1.36930	0.841	38	0.018
0.3182	0.71165	1065	1239	1.36882	0.915	41	0.015
0.3690	0.72220	1065	1220	1.36855	0.949	43	0.015
0.4035	0.72959	1066	1206	1.36839	0.959	43	0.014
0.4569	0.74142	1068	1182	1.36815	0.961	42	0.010
0.5061	0.75276	1071	1159	1.36797	0.945	41	0.005
0.5458	0.76222	1073	1139	1.36791	0.918	39	0.004
0.6121	0.77870	1079	1104	1.36786	0.853	35	0.000
0.6524	0.78913	1082	1082	1.36786	0.798	32	−0.003
0.6938	0.80022	1087	1058	1.36798	0.730	29	−0.002
0.7460	0.81476	1093	1027	1.36812	0.628	24	−0.006
0.8044	0.83175	1102	991	1.36840	0.496	19	−0.007
0.8531	0.84652	1109	960	1.36867	0.374	14	−0.009
0.8883	0.85755	1116	937	1.36891	0.281	11	−0.010
0.9385	0.87379	1126	903	1.36929	0.147	6	−0.009
Ethyl Acetate (1) + Acetone (2)							
0.0615	0.79368	1160	936	1.35711	−0.006	−1	−0.001
0.1098	0.80040	1159	930	1.35794	−0.012	−1	−0.003
0.1560	0.80661	1157	926	1.35871	−0.015	−1	−0.004
0.2099	0.81362	1156	920	1.35958	−0.018	−1	−0.005
0.2600	0.81992	1154	916	1.36038	−0.019	−1	−0.005
0.2977	0.82451	1153	912	1.36096	−0.019	−1	−0.005
0.3498	0.83070	1152	908	1.36175	−0.018	−1	−0.005
0.4011	0.83659	1150	903	1.36250	−0.015	−1	−0.004
0.4495	0.84198	1149	900	1.36320	−0.013	0	−0.003
0.5003	0.84748	1148	896	1.36390	−0.010	0	−0.002
0.5449	0.85218	1147	892	1.36450	−0.007	0	−0.002
0.5967	0.85748	1146	888	1.36519	−0.004	0	0.000
0.6339	0.86121	1145	886	1.36566	−0.002	0	0.000
0.6893	0.86662	1144	882	1.36635	−0.001	0	0.000
0.7398	0.87142	1143	878	1.36697	0.001	0	0.001
0.7785	0.87502	1143	875	1.36743	0.002	0	0.001
0.8301	0.87971	1142	872	1.36803	0.002	0	0.002
0.9092	0.88669	1141	866	1.36892	−0.002	0	0.001
0.9393	0.88928	1141	864	1.36925	−0.004	0	0.001
Hexane (1) + Acetone (2)							
0.0602	0.76931	1143	995	1.35631	0.228	13	−0.008
0.1022	0.75952	1131	1029	1.35680	0.365	23	−0.005
0.1597	0.74731	1117	1073	1.35750	0.530	36	−0.003
0.2009	0.73928	1108	1101	1.35799	0.633	43	−0.004
0.2469	0.73096	1100	1130	1.35864	0.735	50	−0.001
0.3074	0.72092	1092	1164	1.35952	0.850	57	0.002
0.3449	0.71517	1087	1183	1.36013	0.909	60	0.008
0.3956	0.70791	1082	1207	1.36093	0.976	64	0.012
0.4553	0.70003	1076	1233	1.36189	1.036	67	0.017
0.4879	0.69603	1074	1246	1.36243	1.058	68	0.020
0.5532	0.68857	1070	1268	1.36354	1.081	69	0.027
0.5831	0.68540	1069	1277	1.36402	1.082	68	0.027
0.6447	0.67932	1067	1294	1.36505	1.056	66	0.028
0.6820	0.67592	1066	1302	1.36570	1.025	63	0.028
0.7331	0.67159	1066	1311	1.36663	0.958	59	0.029
0.7868	0.66746	1066	1318	1.36767	0.853	51	0.028
0.8243	0.66481	1067	1321	1.36838	0.758	45	0.024
0.8866	0.66087	1069	1323	1.36973	0.551	32	0.020
0.9207	0.65894	1071	1322	1.37044	0.410	24	0.013

The molar refraction changes of mixing, ΔR, were calculated from

$$\Delta R = R - \sum_i x_i R_i \tag{4}$$

R_i being the molar refraction of pure component i and R representing the molar refraction of the mixture.

The isentropic compressibilities, κ_S, were calculated from the densities and the corresponding speeds of sound using the equation

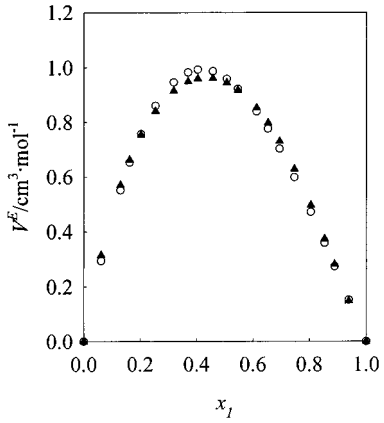

Figure 1. Excess molar volume for the ethyl acetate + hexane system at 25 °C and atmospheric pressure: (▲) this work; (○) Dusart.[2]

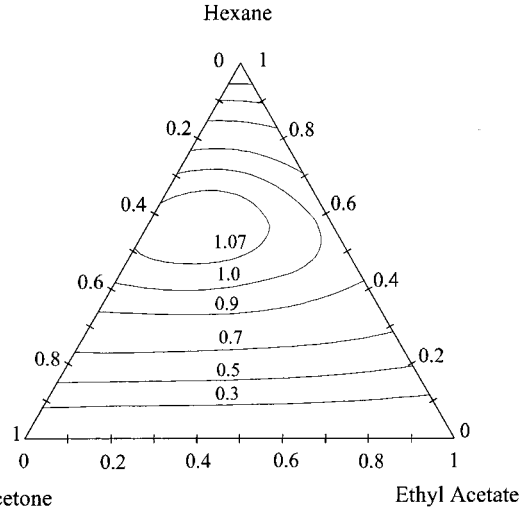

Figure 2. Excess molar volume isolines for the ethyl acetate + hexane + acetone system at 25 °C and atmospheric pressure.

$$\kappa_S = u^{-2}\rho^{-1} \tag{5}$$

and the isentropic compressibility changes of mixing ($\Delta\kappa_S$) were obtained using the expression

$$\Delta\kappa_S = \kappa_S - \sum_i \phi_i \kappa_{Si} \tag{6}$$

where κ_S and κ_{Si} are the isentropic compressibilities of the mixture and component i, respectively, and ϕ_i is the volume fraction of component i in the mixture and was calculated from

$$\phi_i = \frac{x_i V}{\sum_j x_j V_j} \tag{7}$$

where j refers to all components of the mixture.

Figure 1 shows excess molar volumes for the ethyl acetate + hexane binary system obtained in this work, together with correlated values published by Dusart.[2] For the ternary system, Figure 2 shows the excess molar volume isolines (system compositions in mole fraction, x_i), and Figure 3 shows the isentropic compressibility changes of mixing isolines (system composition in volume fraction, ϕ_i).

Table 3. Densities ρ, Speeds of Sound u, Isentropic Compressibilities κ_S, Refractive Indices n_D, Excess Molar Volumes V^E, and $\Delta\kappa_S$ and ΔR Changes of Mixing for Ethyl Acetate (1) + Hexane (2) + Acetone (3) at 25 °C

x_1	x_2	ρ	u	κ_S	n_D	V^E	$\Delta\kappa_S$	ΔR
		g·cm^{-3}	m·s^{-1}	TPa^{-1}		cm^3·mol^{-1}	TPa^{-1}	cm^3·mol^{-1}
0.8967	0.0000	0.88570	1141	868	1.36881	−0.012	1	0.002
0.7936	0.1150	0.84839	1116	947	1.36803	0.278	12	−0.007
0.7023	0.2168	0.81805	1099	1012	1.36754	0.525	22	−0.007
0.6193	0.3094	0.79268	1087	1068	1.36731	0.705	30	−0.006
0.5388	0.3991	0.76995	1078	1118	1.36732	0.830	37	−0.003
0.4430	0.5060	0.74505	1070	1172	1.36761	0.906	42	0.001
0.3716	0.5855	0.72788	1067	1207	1.36799	0.915	44	0.006
0.2777	0.6903	0.70687	1065	1246	1.36872	0.862	41	0.013
0.1920	0.7859	0.68922	1066	1276	1.36958	0.733	34	0.018
0.1011	0.8872	0.67209	1070	1299	1.37073	0.483	21	0.017
0.7973	0.0000	0.87670	1142	874	1.36765	0.006	1	0.002
0.7066	0.1138	0.84030	1116	955	1.36705	0.297	13	−0.003
0.6284	0.2119	0.81158	1100	1019	1.36667	0.546	24	−0.003
0.5563	0.3023	0.78732	1088	1073	1.36655	0.735	32	0.000
0.4795	0.3986	0.76365	1078	1127	1.36668	0.878	40	0.005
0.4072	0.4893	0.74321	1071	1173	1.36702	0.952	45	0.010
0.3130	0.6074	0.71896	1066	1224	1.36772	0.960	47	0.015
0.2355	0.7046	0.70076	1065	1259	1.36847	0.897	44	0.018
0.1603	0.7990	0.68454	1066	1285	1.36937	0.757	36	0.017
0.0935	0.8827	0.67137	1070	1302	1.37038	0.537	24	0.012
0.6921	0.0000	0.86690	1144	881	1.36641	−0.001	0	0.002
0.6138	0.1132	0.83079	1118	964	1.36573	0.356	15	−0.003
0.5510	0.2038	0.80500	1102	1024	1.36564	0.575	25	0.001
0.4892	0.2931	0.78185	1089	1078	1.36575	0.742	34	0.005
0.4189	0.3947	0.75785	1078	1135	1.36606	0.881	43	0.009
0.3529	0.4900	0.73730	1071	1182	1.36649	0.964	48	0.012
0.2710	0.6085	0.71417	1066	1232	1.36726	0.993	50	0.017
0.2093	0.6975	0.69843	1065	1262	1.36805	0.939	47	0.020
0.1421	0.7946	0.68282	1066	1288	1.36915	0.787	38	0.023
0.0644	0.9069	0.66676	1071	1308	1.37076	0.450	21	0.018
0.5963	0.0000	0.85740	1146	888	1.36520	0.000	0	0.002
0.5281	0.1144	0.82162	1118	974	1.36466	0.368	17	−0.003
0.4751	0.2033	0.79709	1102	1033	1.36466	0.578	27	−0.004
0.4221	0.2922	0.77487	1090	1087	1.36488	0.739	36	−0.002
0.3557	0.4036	0.74977	1078	1148	1.36538	0.883	45	0.001
0.2902	0.5133	0.72757	1070	1200	1.36608	0.968	51	0.006
0.2342	0.6072	0.71038	1066	1238	1.36685	0.985	52	0.012
0.1725	0.7108	0.69323	1065	1272	1.36792	0.925	48	0.020
0.1245	0.7912	0.68118	1066	1292	1.36893	0.797	40	0.023
0.0622	0.8957	0.66717	1070	1309	1.37050	0.499	24	0.020
0.5020	0.0000	0.84770	1148	895	1.36392	−0.014	0	−0.003
0.4448	0.1139	0.81282	1119	982	1.36368	0.361	18	−0.001
0.3987	0.2057	0.78824	1103	1043	1.36384	0.588	29	0.002
0.3502	0.3024	0.76508	1089	1102	1.36419	0.772	39	0.003
0.3000	0.4024	0.74361	1078	1156	1.36479	0.908	48	0.008
0.2499	0.5023	0.72433	1071	1204	1.36560	0.992	53	0.017
0.2052	0.5911	0.70880	1067	1239	1.36647	1.013	55	0.026
0.1593	0.6826	0.69431	1065	1269	1.36748	0.970	52	0.032
0.1030	0.7949	0.67850	1066	1296	1.36892	0.802	41	0.033
0.0573	0.8859	0.66723	1070	1310	1.37032	0.538	26	0.026
0.3946	0.0000	0.83580	1150	904	1.36239	−0.011	−1	−0.004
0.3509	0.1107	0.80288	1121	991	1.36241	0.360	19	0.000
0.3125	0.2081	0.77788	1103	1056	1.36279	0.603	32	0.004
0.2761	0.3002	0.75693	1090	1112	1.36334	0.779	42	0.008
0.2363	0.4011	0.73648	1079	1166	1.36410	0.918	51	0.012
0.1949	0.5061	0.71755	1071	1215	1.36505	1.008	57	0.019
0.1534	0.6112	0.70074	1066	1255	1.36619	1.025	57	0.026
0.1176	0.7021	0.68778	1065	1281	1.36734	0.970	53	0.032
0.0797	0.7979	0.67563	1066	1301	1.36874	0.810	42	0.033
0.0433	0.8903	0.66538	1070	1313	1.37028	0.535	26	0.027
0.3037	0.0000	0.82520	1153	912	1.36105	−0.015	−1	−0.004
0.2710	0.1078	0.79407	1123	998	1.36127	0.353	20	0.000
0.2400	0.2098	0.76886	1104	1068	1.36174	0.619	34	0.000
0.2134	0.2974	0.74986	1091	1121	1.36241	0.793	44	0.005
0.1820	0.4007	0.73003	1079	1176	1.36342	0.941	54	0.015
0.1563	0.4855	0.71554	1072	1215	1.36437	1.017	59	0.024
0.1236	0.5929	0.69923	1067	1256	1.36568	1.040	60	0.032
0.0899	0.7040	0.68451	1065	1287	1.36724	0.967	55	0.039
0.0624	0.7947	0.67402	1066	1304	1.36871	0.810	44	0.044
0.0368	0.8787	0.66547	1070	1314	1.37023	0.560	29	0.042
0.2027	0.0000	0.81260	1156	921	1.35944	−0.008	−1	−0.004
0.1808	0.1082	0.78253	1126	1009	1.35989	0.381	21	0.002
0.1593	0.2140	0.75793	1104	1083	1.36069	0.650	38	0.005
0.1425	0.2967	0.74121	1091	1133	1.36150	0.806	48	0.008

Table 3 (Continued)

x_1	x_2	ρ / g·cm^{-3}	u / m·s^{-1}	κ_S / TPa^{-1}	n_D	V^E / cm^3·mol^{-1}	$\Delta\kappa_S$ / TPa^{-1}	ΔR / cm^3·mol^{-1}
0.1230	0.3932	0.72397	1081	1183	1.36260	0.938	56	0.014
0.1031	0.4915	0.70849	1073	1226	1.36387	1.022	61	0.024
0.0798	0.6064	0.69268	1067	1267	1.36551	1.046	62	0.038
0.0586	0.7108	0.68027	1065	1295	1.36713	0.975	56	0.048
0.0411	0.7971	0.67133	1066	1310	1.36858	0.828	46	0.050
0.0177	0.9127	0.66125	1071	1319	1.37066	0.455	24	0.034
0.0976	0.0000	0.79870	1159	932	1.35771	−0.009	−1	−0.003
0.0870	0.1081	0.77048	1128	1020	1.35850	0.357	21	−0.001
0.0779	0.2015	0.74981	1107	1087	1.35938	0.620	39	0.003
0.0680	0.3028	0.73063	1091	1149	1.36049	0.835	52	0.007
0.0589	0.3967	0.71533	1081	1197	1.36178	0.970	60	0.016
0.0495	0.4932	0.70163	1073	1238	1.36332	1.047	65	0.031
0.0400	0.5902	0.68961	1068	1271	1.36494	1.059	65	0.045
0.0285	0.7082	0.67699	1066	1301	1.36691	0.988	59	0.057
0.0193	0.8025	0.66840	1067	1315	1.36857	0.834	47	0.059
0.0082	0.9160	0.65990	1071	1321	1.37079	0.467	24	0.049

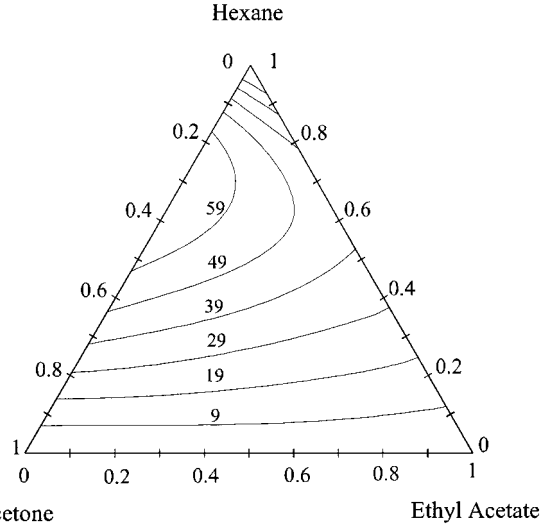

Figure 3. Isentropic compressibility changes of mixing isolines for the ethyl acetate + hexane + acetone system at 25 °C and atmospheric pressure.

Correlation. For binary systems, the V^E, $\Delta\kappa_S$, and ΔR data were correlated with composition using the Redlich−Kister[8] polynomial

$$Q_{ij} = x_i x_j \sum_k A_k (x_i - x_j)^k \qquad (8)$$

where Q_{ij} is V^E or ΔR and x_i is the mole fraction of component i or Q_{ij} is $\Delta\kappa_S$, x_i being the volume fraction of component i. A_k is the polynomial coefficient, and k is the number of the polynomial coefficient.

For ternary systems the corresponding equation, as a function of the composition, x_i (in mole or volume fraction), is

$$Q_{123} = Q_{12} + Q_{23} + Q_{13} + x_1x_2x_3(A + B(x_1 - x_2) + C(x_2 - x_3) + D(x_1 - x_3) + E(x_1 - x_2)^2 + F(x_2 - x_3)^2 + G(x_1 - x_3)^2 + \ldots \qquad (9)$$

where Q_{123} represents V^E, ΔR, or $\Delta\kappa_S$ for the ternary mixture ethyl acetate (1) + hexane (2) + acetone (3) and Q_{ij} is the Redlich−Kister polynomial for the same property fitted to the binary systems data.

The A_k values for binary and ternary systems are summarized in Tables 4 and 5, respectively, together with the corresponding standard deviations of fit. All these coefficients were obtained by fitting, to the appropriate parameters, eqs 8 and 9 by a least-squares regression, and the Fisher's F-tests were used to decide which coefficients to use.

Table 4. Polynomial Coefficients (A_k) and Standard Deviations (σ) Obtained for the Fits of Equation 8 to the V^E, $\Delta\kappa_S$, and ΔR Composition Data for the Binary Systems (for $\Delta\kappa_S$, System Compositions Were in Volume Fractions, ϕ_i)

property	A_0	A_1	A_2	A_3	σ
Ethyl Acetate (1) + Hexane (2)					
V^E/cm^3·mol^{-1}	3.7916	−0.9130	0.3494	−0.9758	0.001
$\Delta\kappa_S$/TPa^{-1}	150.17	−116.50	42.60		0.3
ΔR/cm^3·mol^{-1}	0.0291	−0.1341			0.002
Ethyl Acetate (1) + Acetone (2)					
V^E/cm^3·mol^{-1}	−0.0385	0.0978	−0.0392		0.002
$\Delta\kappa_S$/TPa^{-1}	−2.18				0.5
ΔR/cm^3·mol^{-1}	−0.0097	0.0318			0.001
Hexane (1) + Acetone (2)					
V^E/cm^3·mol^{-1}	4.2590	0.9912	0.7814		0.001
$\Delta\kappa_S$/TPa^{-1}	246.56	140.50	100.42	109.01	0.3
ΔR/cm^3·mol^{-1}	0.0844	0.1567	−0.0385		0.001

Table 5. Polynomial Coefficients and Standard Deviations (σ) Obtained for Fits of Equation 9 to the V^E, $\Delta\kappa_S$, and ΔR Composition Data for the Ternary System Ethyl Acetate (1) + Hexane (2) + Acetone (3) (for $\Delta\kappa_S$, System Compositions Were in Volume Fractions, ϕ_i)

property	A	B	C	D	σ
V^E/cm^3·mol^{-1}	−1.9039	1.2700	−2.5183	−1.2483	0.01
$\Delta\kappa_S$/TPa^{-1}	−237.10	165.31	−158.49	6.82	0.57
ΔR/cm^3·mol^{-1}	−0.0928	−0.3001	−0.1024	−0.4026	0.006

4. Discussion

The values of V^E are positive for the binary mixtures that consisted of ethyl acetate + hexane and hexane + acetone, with a maximum value of around 0.97 cm^3·mol^{-1} and 1.08 cm^3·mol^{-1}, respectively. In the case of the ethyl acetate + acetone system, the V^E vs x_1 curve has an S-shape form with small values going from (−0.018 to +0.010) cm^3·mol^{-1}.

Similar results are observed in the isentropic compressibility changes of mixing, with maximum values of 43 TPa^{-1} and 69 TPa^{-1} for the ethyl acetate + hexane and hexane + acetone systems, respectively. A behavior close to ideal for ethyl acetate + acetone system was also observed.

For all binary systems the molar refraction changes of mixing takes positive and negative values, the maximun and minimum being (+0.020 and −0.007, +0.004 and −0.004, and +0.030 and −0.06) cm^3·mol^{-1}, for ethyl acetate

+ hexane, ethyl acetate + acetone and hexane + acetone systems, respectively.

The ternary system exhibits large and positive excess molar volume values for the entire range of composition except for the binary system cited above, the maximum value being around 1.07 $cm^3 \cdot mol^{-1}$. Likewise, the isentropic compressibility changes of mixing are always positive except for the ethyl acetate + acetone system and larger values corresponding to the *n*-hexane acetone binary system. The values of ΔR are small, the maximum being 0.06 $cm^3 \cdot mol^{-1}$.

In all cases, the Redlich−Kister polynomial allowed satisfactory correlation of binary and ternary data.

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