

Dynamic Viscosity and Speed of Sound of the Ternary Mixture Methyl Acetate + Methanol + Ethanol at 298.15 K

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Dynamic viscosity and speed of sound of the ternary mixture (methyl acetate + methanol + ethanol) at 298.15 K and atmospheric pressure have been measured over the whole range of mixture compositions. Viscosity deviations and deviation in isentropic compressibility for the ternary system were calculated and fitted to Cibulka's equation to estimate the fitting parameters and the root-mean-square deviations between calculated and experimental data. To correlate viscosities of the ternary system from binary data, different equations have been used and their parameters are shown. To predict the viscosity of the ternary mixture, several methods have been proposed; some of them are based on contribution groups, and the other ones use the binary correlation parameters. Some methods that predict viscosity deviations by means of binary contributions have been applied, and their root-mean-square deviations are presented.

1. Introduction

A survey of the literature shows that very few measurements have been made on the viscosities and speeds of sounds of ternary mixtures containing alkanols and ester (Rodríguez et al., 1998). In this work, in the scope of a program to determine thermodynamic properties of a family of alkanols from methanol to *tert*-amyl alcohol with methyl acetate (Rodríguez et al., 1996, 1997; Canosa et al., 1997a,b), we present dynamic viscosities and speeds of sound of the ternary system (methyl acetate + methanol + ethanol) at 298.15 K and atmospheric pressure. Viscosity deviations and deviation in isentropic compressibility for binary mixtures were correlated using Redlich–Kister's equation (1948). Once these parameters are obtained, they are applied to the Cibulka's equation (1982), where ternary excess values are correlated.

After these viscosities were measured, the correlation equations of Katti–Chaudhri (1964), Nissan–Grunberg (1949), Hind et al. (1960), Frenkel (1946), and McAllister (1960) were applied, for ternary system, using their binary parameters of correlation, and a comparison between theoretical and experimental values is made. The fitting parameters for binary and ternary systems and the root-mean-square deviations are presented.

Several predictive equations have been applied to compare, in terms of root-mean-square deviations, the difference between predictive and experimental viscosity, some of them using binary parameters [UNIMOD (Cao et al., 1993a) and Noda et al. (1982)] and other ones using the pure components and group interaction parameters [UNIFAC-VISCO (Gaston-Bonhomme et al., 1994) and GC-UNIMOD (Cao et al., 1993b)] to estimate the viscosity. Furthermore, we have used equations (Iglesias et al., 1996) that estimate viscosity deviations values and deviation in isentropic compressibility from the Redlich–Kister fitting parameters of binary systems and their root-mean-square deviations shown below.

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Table 1. Comparison of Data with Literature Data for Pure Liquids at 298.15 K

component	η /(mPa·s)		u /(m·s ⁻¹)	
	exptl	lit.	exptl	lit.
methyl acetate	0.380	0.3640 ^b	1150.6	
methanol	0.553	0.5513 ^a	1102.1	1102.0 ^e
		0.5450 ^b		
ethanol	1.105	1.0826 ^a	1142.6	1142.4 ^d
		1.1060 ^c		

^a TRC Thermodynamic Tables (1994). ^b Riddick et al. (1986). ^c Kumar et al. (1981). ^d Papaloannou et al. (1991). ^e Arce et al. (1996).

Table 2. Speed of Sound u , Isentropic Compressibility k_s , and Deviation in Isentropic Compressibility Δk_s for Binary Mixtures at 298.15 K

x_1	u /(m·s ⁻¹)	k_s /(TPa ⁻¹)	Δk_s /(TPa ⁻¹)
Methyl Acetate (1) + Methanol (2)			
0.0967	1109.4	1001.1	-23.1
0.1943	1115.2	965.7	-35.9
0.2869	1120.2	938.0	-42.2
0.3956	1125.9	910.3	-44.7
0.5002	1131.3	887.3	-43.4
0.5947	1136.0	869.1	-39.8
0.7702	1144.7	839.6	-28.7
0.8318	1147.2	831.1	-22.9
0.8810	1148.9	825.2	-17.5
Methyl Acetate (1) + Ethanol (2)			
0.1117	1138.9	958.0	0.3
0.1941	1136.8	945.6	1.0
0.3043	1135.3	928.4	1.5
0.3392	1135.0	922.9	1.7
0.4928	1135.7	897.9	1.4
0.5676	1137.0	885.5	0.9
0.7054	1140.9	861.7	-0.7
0.8194	1144.9	842.4	-1.7
0.8985	1147.8	829.6	-1.8

2. Experimental Section

Chemicals are supplied by Merck, all with LiChrosolv quality, except methyl acetate, which is for synthesis. They were recently acquired and kept in an argon atmosphere (N-55, with a maximum of water contents 2 ppmv), degassed in an ultrasound bath, and stored over freshly activated molecular sieves Type 4 Å for methyl acetate and ethanol and Type 3 Å for methanol for several days before using. Chromatographic (GLC) tests of the solvents pre-

Table 3. Viscosity, Viscosity Deviation, Speed of Sound, Isentropic Compressibility, and Deviation in Isentropic Compressibility Ternary Mixture at 298 K

(a) Viscosity η and Viscosity Deviation $\Delta\eta$									
x_1	x_2	$\eta/(\text{mPa}\cdot\text{s})$	$\Delta\eta/(\text{mPa}\cdot\text{s})$	x_1	x_2	$\eta/(\text{mPa}\cdot\text{s})$	$\Delta\eta/(\text{mPa}\cdot\text{s})$		
Methyl Acetate (1) + Methanol (2) + Ethanol (3)									
0.0751	0.4064	0.713	-0.113	0.4408	0.3094	0.442	-0.173		
0.0736	0.5205	0.670	-0.094	0.4374	0.4299	0.428	-0.123		
0.0707	0.6300	0.629	-0.077	0.5520	0.1853	0.424	-0.179		
0.0600	0.8415	0.561	-0.036	0.5469	0.3153	0.412	-0.122		
0.1476	0.1710	0.700	-0.204	0.6641	0.1821	0.400	-0.123		
0.1441	0.2860	0.662	-0.181	0.0415	0.0779	0.944	-0.088		
0.1392	0.3735	0.640	-0.158	0.1140	0.0802	0.795	-0.183		
0.1358	0.4948	0.605	-0.128	0.2740	0.0818	0.588	-0.273		
0.1358	0.6034	0.570	-0.103	0.3597	0.0889	0.521	-0.274		
0.1267	0.6833	0.553	-0.083	0.5457	0.0932	0.439	-0.219		
0.1466	0.7485	0.514	-0.072	0.6481	0.0953	0.415	-0.172		
0.1925	0.1652	0.648	-0.226	0.7579	0.0976	0.396	-0.106		
0.1911	0.3068	0.606	-0.191	0.1857	0.0772	0.688	-0.240		
0.2135	0.4008	0.560	-0.169	0.0721	0.2444	0.792	-0.126		
0.2062	0.4960	0.538	-0.144	0.4491	0.0989	0.473	-0.252		
0.1835	0.6116	0.525	-0.109	0.4736	0.1533	0.457	-0.220		
0.1759	0.7124	0.504	-0.080	0.7485	0.1784	0.390	-0.074		
0.2820	0.1888	0.552	-0.244	0.5944	0.3371	0.400	-0.088		
0.2589	0.3845	0.526	-0.179	0.5055	0.4358	0.408	-0.090		
0.2720	0.4073	0.511	-0.172	0.3820	0.5558	0.430	-0.091		
0.2697	0.5061	0.494	-0.136	0.2959	0.6449	0.447	-0.087		
0.2539	0.6172	0.476	-0.104	0.1436	0.8054	0.498	-0.058		
0.3905	0.1540	0.493	-0.244	0.5750	0.2465	0.413	-0.139		
0.3544	0.2930	0.488	-0.198	0.6705	0.2582	0.391	-0.085		
0.3280	0.4496	0.474	-0.145	0.0533	0.7568	0.602	-0.047		
0.3575	0.5085	0.449	-0.116	0.0793	0.1560	0.820	-0.141		
(b) Speed of Sound u , Isentropic Compressibility κ_S , and Deviation in Isentropic Compressibility $\Delta\kappa_S$									
x_1	x_2	$u/(\text{m}\cdot\text{s}^{-1})$	$\kappa_S/(\text{TPa}^{-1})$	$\Delta\kappa_S/(\text{TPa}^{-1})$	x_1	x_2	$u/(\text{m}\cdot\text{s}^{-1})$	$\kappa_S/(\text{TPa}^{-1})$	$\Delta\kappa_S/(\text{TPa}^{-1})$
Methyl Acetate (1) + Methanol (2) + Ethanol (3)									
0.8803	0.0733	1148.3	828.0	-11.5	0.2931	0.6055	1134.7	935.2	-36.4
0.7849	0.1210	1144.5	842.4	-15.8	0.1915	0.1127	1132.4	947.8	-5.1
0.6889	0.1165	1140.4	860.4	-12.9	0.1984	0.2071	1130.1	948.6	-9.9
0.6818	0.2235	1140.2	857.6	-24.4	0.2013	0.3082	1127.6	950.1	-15.2
0.5915	0.1161	1137.2	878.1	-10.8	0.1991	0.4006	1124.8	952.8	-19.3
0.6065	0.2024	1137.2	873.9	-18.7	0.2027	0.5050	1121.9	954.6	-24.4
0.5862	0.2994	1136.2	873.9	-28.8	0.2011	0.6043	1119.1	957.6	-28.6
0.4908	0.1172	1135.0	896.1	-9.1	0.2016	0.6889	1136.7	960.2	-31.9
0.4963	0.2014	1134.4	893.6	-16.7	0.1001	0.1094	1136.7	963.1	-4.2
0.4518	0.3401	1131.9	900.0	-27.2	0.0980	0.2106	1134.0	967.0	-7.8
0.4945	0.4101	1132.2	890.2	-35.2	0.0945	0.3017	1131.5	971.2	-10.7
0.3666	0.1755	1132.9	917.5	-11.8	0.0993	0.4011	1128.3	974.5	-13.7
0.3942	0.2023	1132.8	912.3	-14.4	0.0958	0.5072	1124.7	980.3	-16.0
0.3944	0.3158	1131.1	911.7	-23.1	0.0945	0.6022	1121.4	985.1	-18.1
0.3908	0.4072	1129.5	912.0	-29.8	0.0999	0.7013	1117.7	988.7	-20.6
0.3926	0.5067	1127.8	911.0	-37.6	0.0699	0.8248	1112.5	1004.8	-18.2
0.2947	0.1130	1133.8	930.0	-6.3	0.0555	0.8895	1109.5	1013.9	-15.9
0.2941	0.2076	1132.1	930.7	-12.5	0.0515	0.0539	1139.3	969.7	-1.5
0.2985	0.3176	1129.8	931.2	-19.1	0.1566	0.7444	1116.7	974.6	-28.7
0.3081	0.3881	1125.9	929.6	-24.1	0.4363	0.0684	1134.7	906.2	-4.2
0.3002	0.4972	1123.2	932.2	-30.5					

sented purities that fulfilled purchaser specifications. Their mass fraction purities were more than 99.8 mass % for methanol and ethanol and more than 99 mass % for methyl acetate with a maximum water contents of 1.5×10^{-2} , 1.6×10^{-2} , and 6.8×10^{-3} %, respectively, determined using a Metrohm 737 KF coulometer. The solvent purities was assessed by contrasting with recommended and published recent values (Table 1).

The mixtures were prepared by weighing amounts of the pure liquids by syringing into stoppered bottles to prevent evaporation and reducing possible errors in mole fraction calculations. A Mettler AT-261 Delta Range balance was used with a precision of 10^{-5} g, covering the whole composition range of the mixture.

The densities of mixture and pure liquids were measured with an Anton Paar DMA-60/602 densimeter with a precision of $\pm 10^{-5}$ g \cdot cm $^{-3}$, calibrated with Millipore quality water and degassed and dried Fluka quality heptane, both as reference liquids. Viscosities were measured with an automated AMV 200 Anton Paar microviscometer and a precision better of 1% mPa \cdot s. The viscometer is based on

the rolling ball principle: a gold-covered steel ball rolls down inside an inclined, sample-filled glass capillary. The apparatus is equipped with an automatic timer (± 0.01 s), and the time taken for the ball to roll a fixed distance between two magnetic sensors allows one to evaluate the viscosity of the fluid. The time measuring range is from 12 to 250 s; the accuracy and the precision in the time measuring range are ± 0.01 s and ± 0.1 s, respectively. The capillary was placed in a block, thermostatically controlled by a PolyScience controller bath model 9010 with a temperature stability of $\pm 10^{-2}$ K. The apparatus was frequently calibrated, testing three fluids of known viscosity and known density. Then, the calibration constants of the appropriate measuring system were determined and stored. The calibration constant has to be determined for each inclination angle used for measurement. Each capillary must be calibrated separately. To avoid incorrect measuring results, we made measurements periodically using a liquid of known viscosity. If the results differ more than $\pm 1\%$ from the actual value, a new calibration has to be performed. The viscometer was calibrated with Millipore

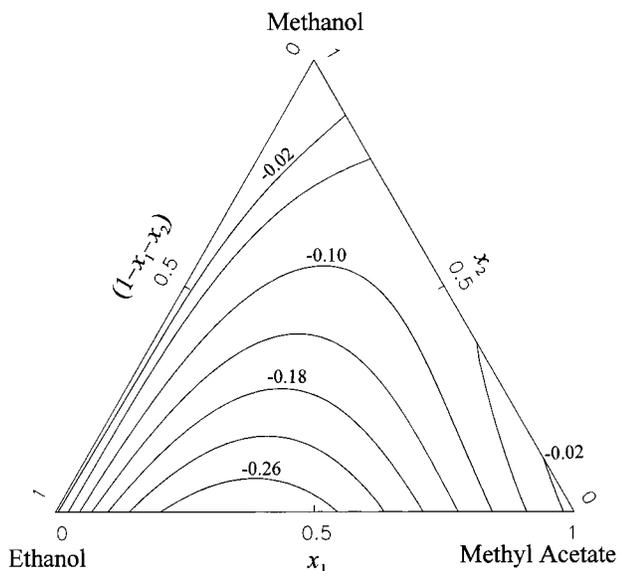


Figure 1. Curves of constant viscosity deviations ($\Delta\eta/\text{mPa}\cdot\text{s}$) from Cibulka's equation, eq 4, for the ternary system methyl acetate (1) + methanol (2) + ethanol (3) at 298.15 K.

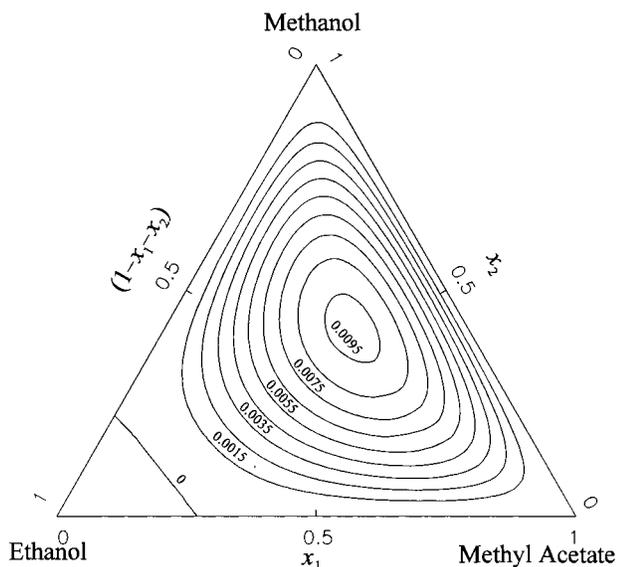


Figure 2. Curves of ternary contribution for viscosity deviations ($\Delta\eta - \Delta\eta_{\text{bin}}$) from Cibulka's equation, eq 4, for the ternary mixture methyl acetate (1) + methanol (2) + ethanol (3) at 298.15 K.

quality water and degassed and dried Fluka quality octane and 1-pentanol. The speeds of sound of the pure liquids and mixtures were measured with an Anton Paar DSA-48 density and sound analyzer with a precision of $\pm 1 \text{ m}\cdot\text{s}^{-1}$. Before each series of measurements, this instrument was calibrated with Millipore quality water and ambient air, in accordance with the instructions.

The accuracy in the calculation of viscosity deviations, mole fraction and deviation in isentropic compressibility were estimated as better than $2 \times 10^{-2} \text{ mPa}\cdot\text{s}$, 5×10^{-5} , and $2 \text{ m}\cdot\text{s}^{-1}$, respectively.

3. Results and Discussion

Speeds of sound of binary mixtures methyl acetate + methanol and methyl acetate + ethanol and viscosities and speeds of sound of the ternary mixture methyl acetate + methanol + ethanol were measured at 298.15 K and atmospheric pressure and are given in Tables 2 and 3a,b. In previous papers, the density of the ternary liquid

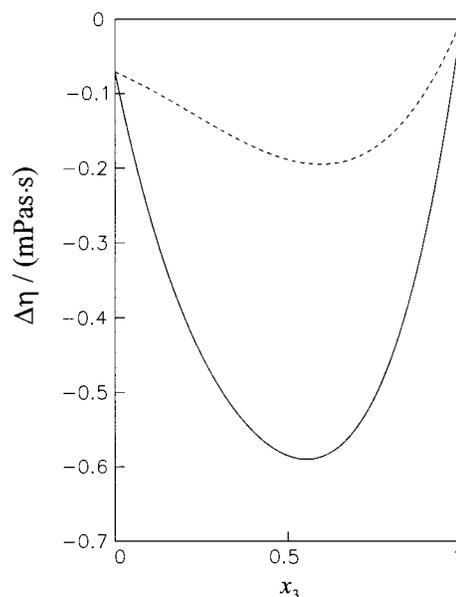


Figure 3. Curves of constant viscosity deviations when $x_1 = x_2$ for the ternary mixture: (a) (---) methyl acetate (1) + methanol (2) + ethanol (3) and (b) (—) methyl acetate (1) + methanol (2) + 1-propanol (3) at 298.15 K.

mixture (Rodríguez et al., 1996) and the speed of sound of the binary methanol + ethanol (Iglesias et al., 1997) were measured. The viscosity deviations are calculated from dynamic viscosities by eq 1

$$\Delta\eta = \eta - \sum_{i=1}^N (\eta_i^\circ x_i) \quad (1)$$

where η and η_i° are the dynamic viscosity of the mixture and the pure component, respectively, and x_i represents the mole fraction of the pure component. Isentropic compressibility (determined by means of Laplace equation, $k_S = \rho^{-1} \cdot u^{-2}$) and deviation in isentropic compressibility Δk_S of the ternary mixture are given in the last columns of Table 3b. The deviation in isentropic compressibility values were calculated as

$$\Delta k_S = k_S - \sum_{i=1}^N x_i k_{S,i} \quad (2)$$

where k_S is the isentropic compressibility of the mixture and $k_{S,i}$ is the isentropic compressibility of the pure component.

The binary mixture viscosity deviations and deviation in isentropic compressibility were fitted to a Redlich-Kister type equation

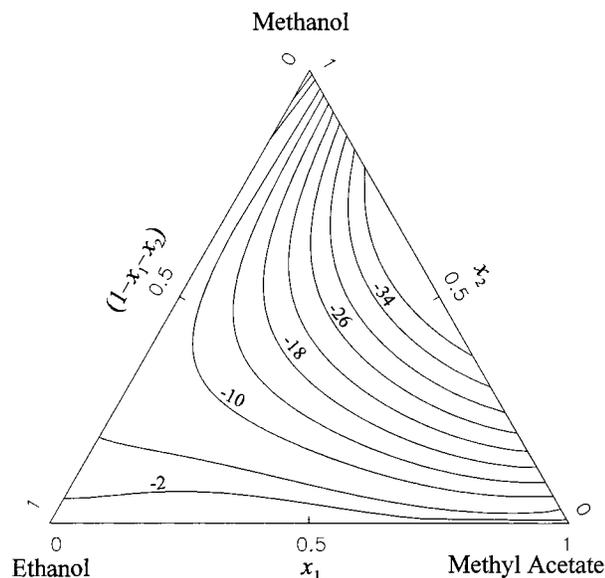
$$\Delta Q_{ij} = x_i x_j \sum_{p=0}^M B_p (x_i - x_j)^p \quad (3)$$

where ΔQ_{ij} is the excess property, x is the mole fraction, B_p is the fitting parameter, and M is the degree of the polynomial expansion, which was optimized using the F -test (Bevington, 1969). Viscosity deviations and deviation in isentropic compressibility of the ternary system were correlated with Cibulka's equation

$$\Delta Q_{123} = \Delta Q_{12} + \Delta Q_{13} + \Delta Q_{23} + x_1 x_2 (1 - x_1 - x_2) \times (C_1 + C_2 \cdot x_1 + C_3 \cdot x_2) \quad (4)$$

Table 4. Parameters and Root-Mean-Square Deviations σ

	Methyl Acetate (1) + Methanol (2)						
$\Delta\kappa_S/(\text{TPa}^{-1})$	$B_0 = -173.0493$	$B_1 = 54.9324$	$B_2 = -62.8985$				$\sigma = 0.25$
	Methyl Acetate (1) + Ethanol (3)						
$\Delta\kappa_S/(\text{TPa}^{-1})$	$B_0 = 5.6477$	$B_1 = -13.5000$	$B_2 = -21.1458$				$\sigma = 0.08$
	Methyl Acetate (1) + Methanol (2) + Ethanol (3)						
$\eta/(\text{mPa}\cdot\text{s})$	$A_1 = 0.3828$	$A_2 = 0.5530$	$A_3 = 1.1046$	$B_{12} = -0.9307$	$B_{13} = -1.0400$	$B_{23} = -0.5421$	
	$C_{122} = 0.9782$	$C_{132} = 1.6568$	$C_{212} = 0.4311$	$C_{232} = 0.8954$	$C_{312} = -0.9984$	$C_{322} = -0.0168$	
	$C_{123} = -0.2757$	$C_{133} = -1.3866$	$C_{213} = 0.0521$	$C_{233} = -0.4249$	$C_{313} = -0.1121$	$C_{323} = 0.4025$	
							$\sigma = 0.0019$
$\Delta\eta/(\text{mPa}\cdot\text{s})$	$C_1 = -0.2007$	$C_2 = 0.5696$	$C_3 = 0.6833$				$\sigma = 0.0025$
$\Delta\kappa_S/(\text{TPa}^{-1})$	$C_1 = 125.820$	$C_2 = -118.710$	$C_3 = -16.2932$				$\sigma = 0.38$



introduce a new parameter to correlate ternary systems in McAllister's equation. We, in this paper, have taken this new contribution into account and have expanded the other equations, the result being:

McAllister

$$\ln(\eta \cdot V) = \sum_i^n x_i \ln(\eta_i \cdot V_i) + 3 \cdot \sum_i^n \sum_{j \neq i}^n x_i^2 \cdot x_j \cdot \ln(A_{ij}) + 6 \cdot \sum_i^n \sum_{j > i}^n \sum_{k > j}^n x_i \cdot x_j \cdot x_k \cdot \ln(A_{ijk}) \quad (6)$$

Nissan–Grunberg

$$\ln(\eta) = \sum_i^n x_i \ln(\eta_i) + \sum_i^n \sum_{j > i}^n x_i \cdot x_j \cdot A_{ij} + \sum_i^n \sum_{j > i}^n \sum_{k > j}^n x_i \cdot x_j \cdot x_k \cdot A_{ijk} \quad (7)$$

Katti–Chaudhri

$$\ln(\eta \cdot V) = \sum_i^n x_i \ln(\eta_i \cdot V_i) + \sum_i^n \sum_{j > i}^n x_i \cdot x_j \cdot A_{ij} + \sum_i^n \sum_{j > i}^n \sum_{k > j}^n x_i \cdot x_j \cdot x_k \cdot A_{ijk} \quad (8)$$

Hind et al.

$$\eta = \sum_i^n x_i^2 \cdot \eta_i + 2 \cdot \left(\sum_i^n \sum_{j > i}^n x_i \cdot x_j \cdot A_{ij} + \sum_i^n \sum_{j > i}^n \sum_{k > i}^n x_i \cdot x_j \cdot x_k \cdot A_{ijk} \right) \quad (9)$$

Frenkel

$$\ln(\eta) = \sum_i^n x_i \ln(\eta_i) + 2 \cdot \left(\sum_i^n \sum_{j > i}^n x_i \cdot x_j \cdot \ln(A_{ij}) + \sum_i^n \sum_{j > i}^n \sum_{k > j}^n x_i \cdot x_j \cdot x_k \cdot \ln(A_{ijk}) \right) \quad (10)$$

where, for each equation, x is the mole fraction, η and η_i are the dynamic viscosity of the mixture and the pure components, respectively, V and V_i are the molar volume of the mixture and the pure component respectively, A_{ij} is the binary correlation parameter, and A_{ijk} is the ternary correlation parameter.

The best correlation method for this ternary system methyl acetate + methanol + ethanol corresponds to McAllister's equation and the worst is Hind's model [data previously published (Canosa et al., 1998)].

Comparison between viscosity values of the ternary mixture and predictive values is shown in Table 6. Here it is observed that predictive methods of dynamic viscosity that use binary correlation parameters obtain better results for the ternary system. When predictive methods based on contribution groups are applied, the UNIFAC-VISCO model obtains better results for the ternary system studied. We must take into account, when observing these results, the fact that the UNIFAC-VISCO and GC-UNIMOD model are predictive ones and that they do not require determining of adjustable parameters that would reduce this deviation.

Figure 5 shows a comparison between experimental and predictive values for all the models studied. The worst results are observed when the GC-UNIMOD model is applied, since it is impossible to reproduce the curve that

exists when the mole fraction is approximately equimolar. This is due to the group interaction energy parameters used that have been obtained from VLE data, whereas the UNIFAC-VISCO model applies its own parameters.

The predictive methods for excess properties of Kohler, Jacob and Fitzner, Colinet, Tsao and Smith, Toop, Scatchard et al. and Rastogi (Iglesias et al., 1996) were used to determine ternary viscosity deviations values and deviation in isentropic compressibility by means of additive binary contributions. The experimental and estimated viscosity deviations and deviation in isentropic compressibility are compared, showing the best estimated viscosity deviations results are obtained when Type a asymmetric equations are used, and for deviation in isentropic compressibilities the same behavior is observed.

Acknowledgment

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Registry No. Supplied by the Author: Methyl acetate, 79-20-9; methanol, 67-56-1; ethanol, 64-17-5.

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