

# Experimental Viscosities and Viscosity Predictions of the Ternary Mixture (Cyclohexane + 1,3-Dioxolane + 2-Butanol) at 298.15 and 313.15 K

Ignacio Gascón, Ana M. Mainar, Félix M. Royo, and José S. Urieta\*

Departamento de Química Orgánica-Química Física, Facultad de Ciencias, Universidad de Zaragoza, Ciudad Universitaria, Zaragoza 50009, Spain

L. Alvarez-Cerdeirina

Departamento de Física Aplicada, Facultad de Ciencias, Universidad de Vigo, Campus de Orense, Orense 32004, Spain

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Viscosities of the ternary mixture cyclohexane + 1,3-dioxolane + 2-butanol and the constituent binary mixtures cyclohexane + 2-butanol and 1,3-dioxolane + 2-butanol have been measured at atmospheric pressure at 298.15 and 313.15 K. Viscosity deviations for the binary and ternary mixtures were calculated from experimental data and fitted by Redlich–Kister and Cibulka equations. The group contribution method proposed by Wu has been used to predict the viscosity of all the mixtures.

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## Introduction

Continuing our work about transport properties of ternary mixtures (Domínguez et al., 1996, 1998, 2000, Gascón et al., 1999, 2000), we present viscosity measurements of the ternary mixture cyclohexane + 1,3-dioxolane + 2-butanol and for the binary mixtures cyclohexane + 2-butanol and 1,3-dioxolane + 2-butanol at atmospheric pressure at 298.15 K and 313.15 K. In a previous paper (Gascón et al., 2000) we have reported viscosities of the ternary system cyclohexane + 1,3-dioxolane + 1-butanol and for the binary mixtures cyclohexane + 1-butanol and 1,3-dioxolane + 1-butanol. Here we will compare the behavior of all the mixtures.

The group contribution method proposed by Wu (1986) has been used to predict the viscosity of binary and ternary systems.

## Experimental Section

**Materials.** The compounds used were cyclohexane (>99.9%), 1,3-dioxolane (>99%) and 2-butanol (>99%) obtained from Aldrich. The purities of these compounds were checked by comparing the measured densities with those reported in the literature and also by a chromatographic method, using a semicapillary methyl silicone column (o.d. 530  $\mu\text{m}$ ) and a flame ion detector. These confirmed the absence of other significant compounds, so no further purification was attempted. The 2-butanol was dried with active molecular sieve type 0.3 nm from Merck. The pure compounds properties at 298.15 K and 313.15 K, along with literature values, are given in Table 1.

**Apparatus and Procedure.** Kinematic viscosities,  $\nu$ , of the pure compounds and their mixtures were determined using an Ubbelohde viscosimeter (i.d. 0.63 mm) with a Schott-Gerate automatic measuring unit, model AVS-440, for which the uncertainty of the flow time measurement

is  $\pm 0.01$  s and the corresponding uncertainty in the kinematic viscosity was  $\pm 1 \times 10^{-4}$   $\text{mm}^2 \cdot \text{s}^{-1}$ . At least four time flow measurements were performed for each composition and temperature, and the results were averaged.

The temperature was kept constant within  $\pm 0.01$  K by means of a Schott-Gerate thermostat. Densities,  $\rho$ , required to calculate absolute viscosities,  $\eta = \rho\nu$ , were measured with an Anton Paar DMA-58 vibrating tube densimeter. The accuracy of the density measurements was  $\pm 1 \times 10^{-2}$   $\text{kg} \cdot \text{m}^{-3}$ .

Calibration of viscosimeter was carried out with deionized doubly distilled water ( $\eta = 0.8902$  mPa·s at 298.15 K), as it is recommended by Marsh (1987). More details of calibration and procedures for the viscosimeter and densimeter can be found in an earlier paper, Blasco et al. (1993).

The compositions of the mixtures are given in mole fraction and were determined by mass using a Mettler H20T balance with a precision of  $\pm 0.01$  mg. The precision of the mole fraction is estimated to be better than  $\pm 1 \times 10^{-4}$ .

## Results and Discussion

The experimental viscosities of the binary mixtures cyclohexane + 2-butanol and 1,3-dioxolane + 2-butanol at 298.15 K and 313.15 K are given in Table 2 and the viscosities of the ternary mixture cyclohexane + 1,3-dioxolane + 2-butanol at the same temperatures are shown in Table 3. Experimental viscosities of the binary mixture cyclohexane + 1,3-dioxolane have been reported in the previous paper of Guzmán et al. (1999).

The viscosity deviations,  $\Delta\eta$ , for the binary and ternary mixtures were determined using the equation

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

\* To whom all the correspondence should be addressed. Telephone: +34-976-76-1298. Fax: +34-976-76-1202. E-mail: urieta@posta.unizar.es.

**Table 1. Densities,  $\rho$ , and Viscosities,  $\eta$ , of Pure Components at 298.15 and 313.15 K and Comparison with Literature Data at 298.15 K**

component	T = 298.15K				T = 313.15K	
	$\rho/(\text{kg}\cdot\text{m}^{-3})$		$\eta/(\text{mPa}\cdot\text{s})$		$\rho/(\text{kg}\cdot\text{m}^{-3})$	$\eta/(\text{mPa}\cdot\text{s})$
	exp	lit.	exp	lit.	exp	exp
cyclohexane	773.72	773.89 <sup>a</sup>	0.8859	0.898 <sup>a</sup>	759.47	0.6983
1,3-dioxolane	1058.62	1058.66 <sup>b</sup>	0.5886		1039.97	0.4985
2-butanol	802.20	802.41 <sup>a</sup>	3.0427	2.998 <sup>a</sup>	789.59	1.7913

<sup>a</sup> Riddick et al. (1986). <sup>b</sup> Brocos et al. (1998).

**Table 2. Experimental Absolute Viscosities,  $\eta$ , and Viscosity Deviations,  $\Delta\eta$ , of the Binary Mixtures Cyclohexane + 2-Butanol and 1,3-Dioxolane + 2-Butanol at 298.15 K and 313.15 K**

$x_1$	T = 298.15 K		T = 313.15 K	
	$\eta/(\text{mPa}\cdot\text{s})$	$\Delta\eta/(\text{mPa}\cdot\text{s})$	$\eta/(\text{mPa}\cdot\text{s})$	$\Delta\eta/(\text{mPa}\cdot\text{s})$
Cyclohexane (1) + 2-Butanol (2)				
0.0998	2.3340	-0.4935	1.4420	-0.2402
0.2045	1.8410	-0.7607	1.1896	-0.3781
0.3002	1.5240	-0.8713	1.0231	-0.4401
0.4007	1.2845	-0.8940	0.8968	-0.4565
0.4995	1.1158	-0.8496	0.8098	-0.4355
0.6000	1.0011	-0.7476	0.7497	-0.3858
0.7028	0.9323	-0.5946	0.7106	-0.3125
0.8005	0.8895	-0.4267	0.6896	-0.2267
0.9036	0.8720	-0.2218	0.6801	-0.1235
1,3-Dioxolane (1) + 2-Butanol (2)				
0.0987	1.9968	-0.8037	1.2905	-0.3732
0.1966	1.4543	-1.1059	1.0156	-0.5215
0.2957	1.1490	-1.1680	0.8449	-0.5641
0.3994	0.9497	-1.1128	0.7262	-0.5487
0.5008	0.8242	-0.9895	0.6486	-0.4952
0.6023	0.7412	-0.8234	0.5930	-0.4196
0.699	0.6698	-0.6575	0.5500	-0.3376
0.8014	0.6265	-0.4495	0.5218	-0.2334
0.9023	0.5997	-0.2287	0.5052	-0.1195

where  $\eta$  is the absolute viscosity of the mixture,  $n$  is the number of components,  $x_i$  is the mole fraction in component  $i$ , and  $\eta_i$  is the absolute viscosity of pure component  $i$ . We have fitted the viscosity deviations of those systems to a Redlich–Kister equation:

$$\Delta\eta_{ij} = x_i x_j \sum_{p=0}^n A_{p,ij} (x_i - x_j)^p \quad (2)$$

where  $x_i$  denotes the mole fraction of the component  $i$  in the mixture and  $A_{p,ij}$  are adjustable parameters obtained by the least-squares method. The choice of the number of parameters was based on the standard deviations,  $\sigma$ , and the F-test as criterion of goodness of fit, with an error lower than 1%. These parameters, along with the standard deviations,  $\sigma$ , are given in Table 4. The viscosity deviations for the binary mixtures are shown in Table 2 and graphically represented in Figures 1 and 2.

The viscosity deviations for the ternary mixture are shown in Table 3. We have fitted these to the Cibulka equation (Cibulka, 1982):

$$\Delta\eta = \Delta\eta_{\text{bin}} + x_1 x_2 (1 - x_1 - x_2) [B_1 - B_2 x_1 - B_3 x_2] \quad (3)$$

where

$$\Delta\eta_{\text{bin}} = x_1 x_2 \sum_{p=0}^n A_{p,12} (x_1 - x_2)^p + x_1 x_3 \sum_{q=0}^m A_{q,13} (x_1 - x_2)^q + x_1 x_2 \sum_{r=0}^l A_{r,23} (x_2 - x_3)^r \quad (4)$$

**Table 3. Experimental Absolute Viscosities,  $\eta$ , and Viscosity Deviations,  $\Delta\eta$ , of the Ternary Mixture Cyclohexane (1) + 1,3-Dioxolane (2) + 2-Butanol (3) at 298.15 and 313.15 K**

$x_1$	$x_2$	T = 298.15 K		T = 313.15 K	
		$\eta/(\text{mPa}\cdot\text{s})$	$\Delta\eta/(\text{mPa}\cdot\text{s})$	$\eta/(\text{mPa}\cdot\text{s})$	$\Delta\eta/(\text{mPa}\cdot\text{s})$
0.0515	0.0508	2.1031	-0.7039	1.3454	-0.3239
0.0498	0.0984	1.7681	-0.9257	1.2102	-0.3994
0.0488	0.8494	0.5987	-0.2543	0.5027	-0.1372
0.0499	0.9032	0.5908	-0.1277	0.4984	-0.0706
0.1003	0.0517	1.8789	-0.8206	1.2271	-0.3877
0.1009	0.0983	1.5987	-0.9852	1.0879	-0.4660
0.1013	0.2026	1.2391	-1.0879	0.8828	-0.5359
0.0984	0.3037	0.9960	-1.0892	0.7587	-0.5324
0.1003	0.3992	0.8449	-1.0019	0.6596	-0.5060
0.1021	0.4992	0.7454	-0.8521	0.5969	-0.4374
0.0990	0.6012	0.6764	-0.6773	0.5522	-0.3536
0.1012	0.7000	0.6292	-0.4774	0.5213	-0.2544
0.1009	0.8007	0.5989	-0.2612	0.5022	-0.1436
0.1015	0.8523	0.5970	-0.1351	0.5036	-0.0748
0.2020	0.0965	1.3435	-1.0267	0.9596	-0.4861
0.2002	0.2017	1.0459	-1.0700	0.7784	-0.5333
0.2005	0.2983	0.8818	-0.9964	0.6805	-0.5060
0.1997	0.4010	0.7687	-0.8592	0.6102	-0.4444
0.1985	0.4988	0.6981	-0.6924	0.5613	-0.3681
0.1816	0.6313	0.6363	-0.4654	0.5256	-0.2510
0.2021	0.6994	0.6079	-0.2825	0.5073	-0.1588
0.3045	0.0943	1.1461	-1.0084	0.8342	-0.5023
0.3022	0.1975	0.9314	-0.9748	0.7095	-0.4961
0.3012	0.3001	0.8005	-0.8561	0.6287	-0.4454
0.3023	0.3962	0.7172	-0.7012	0.5783	-0.3704
0.3019	0.4997	0.6557	-0.5096	0.5359	-0.2794
0.3002	0.6023	0.6192	-0.2979	0.5125	-0.1720
0.4019	0.0985	1.0045	-0.9297	0.7500	-0.4747
0.4028	0.1956	0.8507	-0.8433	0.6593	-0.4388
0.4014	0.3007	0.7457	-0.6933	0.5930	-0.3707
0.4022	0.3987	0.6790	-0.5178	0.5558	-0.2804
0.4040	0.4996	0.6358	-0.3095	0.5229	-0.1809
0.5050	0.0996	0.9075	-0.8016	0.6955	-0.4150
0.5020	0.1976	0.7918	-0.6833	0.6231	-0.3641
0.5035	0.3006	0.7108	-0.5082	0.5707	-0.2816
0.5021	0.3962	0.6620	-0.3255	0.5401	-0.1901
0.6048	0.0938	0.8510	-0.6571	0.6616	-0.3474
0.6013	0.2020	0.7501	-0.5000	0.5979	-0.2750
0.6056	0.2952	0.6963	-0.3158	0.5609	-0.1868
0.7073	0.0943	0.8127	-0.4731	0.6407	-0.2555
0.7049	0.1967	0.7336	-0.3061	0.5888	-0.1777
0.8051	0.0955	0.7925	-0.2794	0.6286	-0.1592
0.8510	0.0495	0.8277	-0.2581	0.6515	-0.1456
0.8549	0.0966	0.7892	-0.1726	0.6288	-0.1032
0.9035	0.0510	0.8315	-0.1374	0.6520	-0.0858

and  $x_i$  is the mole fraction of the component  $i$  in the ternary data point.

The coefficients  $B_p$  and the standard deviations,  $\sigma$ , obtained by the least-squares method are shown in Table 4.

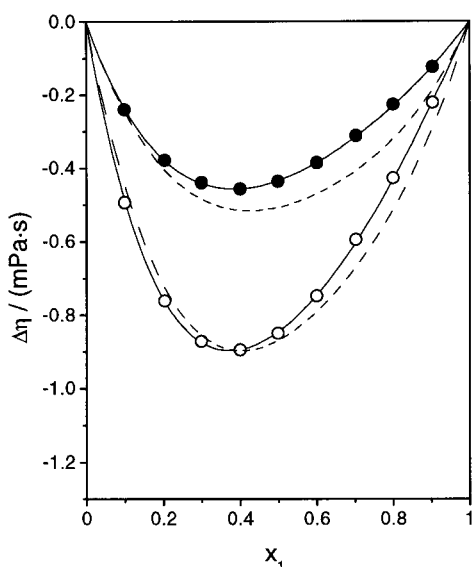
Isolines at constant values of calculated  $\Delta\eta$  from Cibulka's eq 3 for the ternary system at both temperatures have been drawn in Figure 3.

The viscosity deviations for the two binary mixtures are negatives over the whole composition range. A rise of temperature makes  $\Delta\eta$  increase (less negative values). Both

**Table 4. Coefficients of Redlich–Kister's Eq 2,  $A_p$ , and Cibulka's Eq 3,  $B_p$ , and the Corresponding Standard Deviations,  $\sigma$ , for Viscosity Deviations of the Binary and Ternary Mixtures**

$T$ (K)	$A_0$	$A_1$	$A_2$	$A_3$	$\sigma(\Delta\eta)$ /(mPa·s)
Cyclohexane (1) + 1,3-Dioxolane (2) <sup>a</sup>					
298.15	-0.4138	-0.1613	-0.1951	-0.0452	0.0005
313.15	-0.2971	-0.0905	-0.1178	-0.0266	0.0006
Cyclohexane (1) + 2-Butanol (2)					
298.15	-3.3774	1.5283	-0.9126	0.4790	0.0056
313.15	-1.7343	0.7336	-0.4360	0.0809	0.0026
1,3-Dioxolane (1) + 2-Butanol (2)					
298.15	-3.9257	2.7796	-2.7713	1.8650	0.0099
313.15	-1.9697	1.2380	-1.1735	0.7938	0.0047
$T$ (K)	$B_1$	$B_2$	$B_3$	$\sigma(\Delta\eta)$ /(mPa·s)	
Cyclohexane (1) + 1,3-Dioxolane (2) + 2-Butanol (3)					
298.15	6.8245	-5.6395	-5.7200	0.0118	
313.15	3.6789	-3.4984	-3.3316	0.0077	

<sup>a</sup> Guzmán et al. (1999).

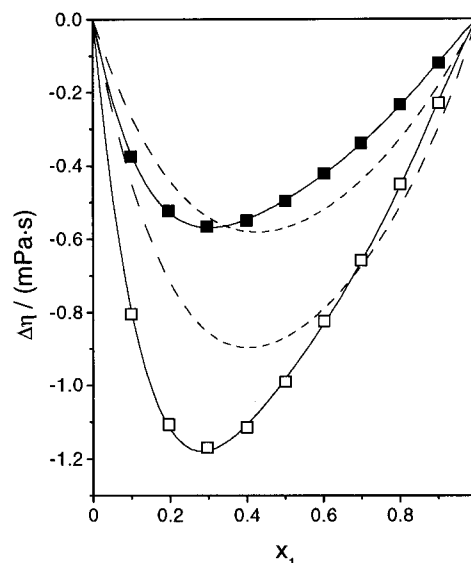


**Figure 1.** Viscosity deviations,  $\Delta\eta$ , for the binary mixture cyclohexane (1) + 2-butanol (2) at 298.15 K (○) and at 313.15 K (●) showing the Redlich–Kister correlation (continuous lines) and the Wu model (dashed lines).

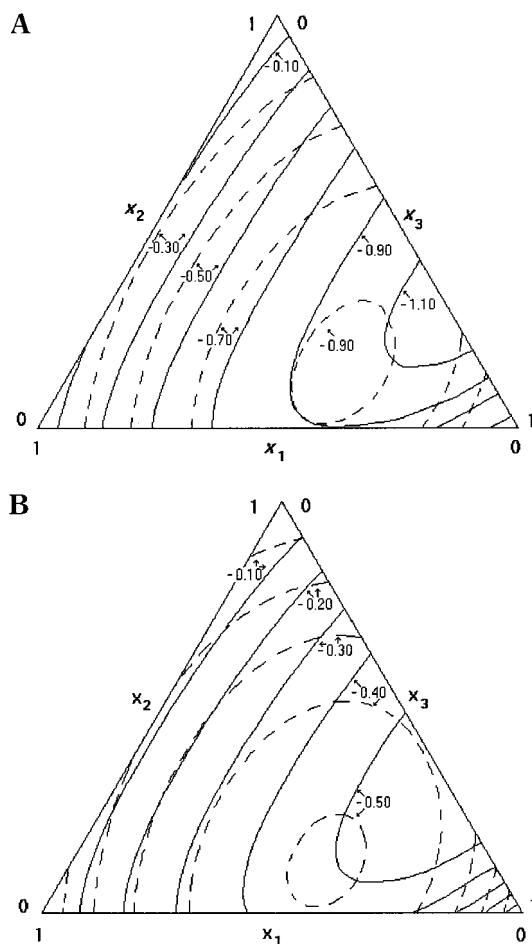
mixtures show more negative values of viscosity deviations than those with 1-butanol (see Gascón et al., 2000). For the mixture cyclohexane + 2-butanol, minimum values appear at  $x_{1\min} = 0.373$  with  $\Delta\eta_{\min} = -0.896$  mPa·s at 298.15 K and at  $x_{1\min} = 0.384$  with  $\Delta\eta_{\min} = -0.456$  mPa·s at 313.15 K, while for the other binary mixture, 1,3-dioxolane + 2-butanol, minimum values of  $\Delta\eta$  are  $x_{1\min} = 0.282$  with  $\Delta\eta_{\min} = -1.178$  mPa·s at 298.15 K and  $x_{1\min} = 0.301$  with  $\Delta\eta_{\min} = -0.568$  mPa·s at 313.15 K.

The ternary viscosity deviations are also negative over the whole composition range at both temperatures and, as for the binary mixtures, values increase with the temperature but the shape of tri-dimensional surface does not change. As with the binary mixtures, viscosity deviations of the ternary mixture are more negative than those of the corresponding mixture with 1-butanol at both temperatures.

The viscosity of a mixture depends on the molecular interactions between the components: mixtures with strong interactions between different molecules show positive viscosity deviations; while for mixtures without specific strong interactions, viscosity deviations are negative. In



**Figure 2.** Viscosity deviations,  $\Delta\eta$ , for the binary mixture 1,3-dioxolane (1) + 2-butanol (2) at 298.15 K (□) and at 313.15 K (■) showing the Redlich–Kister correlation (continuous lines) and the Wu model (dashed lines).



**Figure 3.** Isolines at constant  $\Delta\eta$  for the ternary system cyclohexane (1) + 1,3-dioxolane (2) + 2-butanol (3) correlated with Cibulka's equation (continuous lines) and predicted with the Wu-UNIFAC model with  $A = 1$  (dashed lines): (a) at 298.15 K; (b) at 313.15 K.

our mixtures two effects contribute to reduce the viscosity during the mixing process (i.e. cause negative values of viscosity deviation of the mixture): the breaking of hydro-

**Table 5. Root Mean Square Deviation (RMSD<sub>r</sub>) of the Wu Model for the Binary and Ternary Systems at the Temperatures 298.15 and 313.15 K**

T/K	RMSD <sub>r</sub> (%)			
	ASOG		UNIFAC	
	A = 1	A = 2.45	A = 1	A = 2.45
	Cyclohexane (1) + 2-Butanol (2)			
298.15	6.28	17.12	5.15	17.95
313.15	8.85	12.22	40.29	12.79
	1,3-Dioxolane (1) + 2-Butanol (2)			
298.15	65.69	54.88	17.68	40.04
313.15	46.52	39.57	43.75	12.29
	Cyclohexane (1) + 1,3-Dioxolane (2) + 2-Butanol (3)			
298.15	29.76	35.81	11.96	24.03
313.15	19.74	25.71	10.81	16.40

gen bonding of 2-butanol and the breaking of dipole–dipole interactions of 1,3-dioxolane. On the other hand the OH–O interactions increase the viscosity, but the effect is not as important as the breaking of self-interactions. Consequently viscosity deviations of all our mixtures are negative, which means that our systems flow more easily than predicted from the pure liquids.

**Viscosity Predictions.** Wu (1986) used the following modified viscosity equation of Eyring et al. (1941) to predict the viscosity of liquid mixtures:

$$\eta = \frac{hN}{V} \exp \left[ \frac{\left( \sum_i x_i G_i^* \right) - \frac{G^E}{A}}{RT} \right] \quad (5)$$

where  $h$  is Planck's constant,  $N$  is Avogadro's number,  $V$  is the molar volume of the liquid mixture,  $G_i^*$  is the Gibbs energy of activation of viscous flow of pure liquid  $i$ ,  $G^E$  is the Gibbs excess energy,  $A$  is an empirical factor which can be  $A = 1$  or  $A = 2.45$  according to the original work of Wu (1986),  $T$  is the absolute temperature, and  $R$  is the gas constant.  $G_i^*$  can be obtained from the corresponding viscosity and molar volume of the pure components using eq 6 with  $x_i = 1$  and  $G^E = 0$ .

In the Wu model  $G^E$  can be estimated from different group contribution methods. For this paper we have used the UNIFAC and ASOG parameters proposed by Gmehling et al. (1993) and Tochigi et al. (1990), respectively.

Table 5 shows the root-mean-square-deviations relative (RMSD<sub>r</sub>) values for the binary and ternary mixtures at both temperatures, defined by

$$\text{RMSD}_r = \left[ \frac{1}{m} \sum \left( \frac{\eta_{\text{calc}} - \eta}{\eta} \right)^2 \right]^{1/2} \quad (6)$$

where  $m$  is the number of experimental points,  $\eta_{\text{calc}}$  and  $\eta$  are calculated and experimental absolute viscosity, respectively.

In Table 5 we can see that the accuracy of viscosity predictions are very different for each system and temperature. The better results for the binary system cyclohexane + 2-butanol at 298.15 K are obtained with UNIFAC model using a value of parameter  $A = 1$ , while for the same mixture at 313.15 K ASOG predictions are much better, especially when a value of the parameter  $A = 1$  is used. Whereas for the other binary system, 1,3-dioxolane + 2-butanol, the better predictions are obtained with the UNIFAC model with  $A = 1$  at 298.15 K and  $A = 2.45$  at 313.15 K. We have plotted the better predictions for these systems in Figures 1 and 2 with the experimental results.

For the ternary system cyclohexane + 1,3-dioxolane + 2-butanol the UNIFAC model with  $A = 1$  gives the better prediction. We have plotted these predictions in Figures 3a and 3b. The ASOG model predicts correctly the sign of viscosity deviations but values are not so good as the UNIFAC model.

## Conclusions

Experimental absolute viscosities for the binary mixtures cyclohexane + 2-butanol and 1,3-dioxolane + 2-butanol and for the ternary mixture cyclohexane + 1,3-dioxolane + 2-butanol have been measured at atmospheric pressure at 298.15 K and 313.15 K.

Viscosity deviations have been calculated and fitted to the Redlich–Kister (binary mixtures) and Cibulka equations (ternary mixture).

The behavior of these mixtures has been discussed in terms of molecular interactions where the main effect is the breaking of self-interactions. We also have compared results with those of a previous article in which we presented viscosities of the ternary mixture cyclohexane + 1,3-dioxolane + 1-butanol.

The Wu (1986) model has been used to predict viscosity deviations of binary and ternary mixtures and viscosity predictions have been compared with experimental results.

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