

# Experimental Determination of Densities and Refractive Indices of the Ternary Mixture 2-Methyl-2-propanol + Cyclohexylamine + *n*-Heptane at $T = (303.15 \text{ to } 323.15) \text{ K}$

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The experimental data of the ternary 2-methyl-2-propanol + cyclohexylamine + *n*-heptane (densities  $\rho$  and refractive indices  $n_D$  data) and three binary systems: 2-methyl-2-propanol + cyclohexylamine ( $n_D$  data), 2-methyl-2-propanol + *n*-heptane ( $\rho$  and  $n_D$  data), and cyclohexylamine + *n*-heptane ( $n_D$  data) have been determined. Experimental  $\rho$  and  $n_D$  measurements have been performed simultaneously, at five temperatures, (303.15, 308.15, 313.15, 318.15 and 323.15) K, and atmospheric pressure, using an Anton Paar DMA 5000 digital vibrating-tube densimeter, with a refractometer Anton Paar RXA 156. Excess molar volumes  $V^E$  and deviations of refractive indices  $\Delta n_D$  were correlated by the Redlich–Kister equation for binary mixtures and the Nagata–Tamura equation for the ternary mixture.

## Introduction

This work is continuation of our systematic study of the volumetric properties of binaries and multicomponent mixtures containing butanol.<sup>1–5</sup> The systems of 1-butanol or 2-butanol with cyclohexylamine and *n*-heptane were recently investigated in our previous papers.<sup>4,5</sup>

In this work density  $\rho$  and refractive index  $n_D$  measurements of the ternary system 2-methyl-2-propanol + cyclohexylamine + *n*-heptane at temperatures of (303.15, 308.15, 313.15, 318.15 and 323.15) K and atmospheric pressure were presented. In addition, refractive index data for all binary constituents (2-methyl-2-propanol + cyclohexylamine, 2-methyl-2-propanol + *n*-heptane, and cyclohexylamine + *n*-heptane) and density data for the 2-methyl-2-propanol + *n*-heptane binary system are presented over the same temperature range. In our previous papers<sup>5,6</sup> the density data for the binary systems 2-methyl-2-propanol + cyclohexylamine<sup>6</sup> and cyclohexylamine + *n*-heptane<sup>5</sup> are given.

The excess molar volume  $V^E$  was calculated from the measured  $\rho$  data, while deviations of refractive indices  $\Delta n_D$  were calculated from the measured  $n_D$  data.

The Redlich–Kister<sup>7</sup> equation was used for the binary system, while the Nagata and Tamura<sup>8</sup> equation was used for ternary  $V^E$  and  $\Delta n_D$  data correlation.

To the best of our knowledge only  $\rho$  data for the 2-methyl-2-propanol + *n*-heptane system (at the temperature 313.15 K) were previously presented in the literature by the other authors.<sup>9</sup> The  $\rho$  and  $n_D$  experimental data are not available for the investigated ternary system, as well as for the other binary systems presented in this paper.

## Experimental Section

**Chemicals.** 2-Methyl-2-propanol and *n*-heptane (Fluka) were supplied with a mass purity of > 99.7 % and 99.5 %, respectively, while cyclohexylamine was a product of Merck

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**Table 1. Densities  $\rho$  and Refractive Indices  $n_D$  of the Pure Components at Temperature  $T$  and Atmospheric Pressure**

component	$T$ K	$\rho \cdot 10^{-3} / (\text{kg} \cdot \text{m}^{-3})$		$n_D$	
		exptl	lit.	exptl	lit.
2-methyl-2-propanol	303.15	0.77541	0.77541 <sup>a</sup>	1.3822	1.3825 <sup>b</sup>
	313.15			1.3767	1.376 <sup>c</sup>
cyclohexylamine	288.15	0.87129	0.87128 <sup>a,d</sup>		
	298.15			1.4564	1.4565 <sup>a</sup>
<i>n</i> -heptane	298.15	0.67949	0.67951 <sup>a,d</sup>	1.3852	1.38511 <sup>a</sup>

<sup>a</sup> Riddick et al.<sup>10</sup> <sup>b</sup> Muñoz et al.<sup>11</sup> <sup>c</sup> Cataliotti et al.<sup>12</sup> <sup>d</sup> Timmermans.<sup>13</sup>

with a purity > 99 %. Chemicals were kept in brown bottles under inert nitrogen atmosphere and ultrasonically degassed just before a sample preparation. Table 1 lists the measured densities of 2-methyl-2-propanol, cyclohexylamine, and *n*-heptane along with the corresponding literature values, and they agree to within  $\pm 0.01 \text{ kg} \cdot \text{m}^{-3}$  in densities measurements and  $\pm 0.0002$  in refractive index measurements.<sup>10–13</sup>

**Measurements.** The densities  $\rho$  of the ternary and binary mixtures and corresponding pure substances were measured by means of an Anton Paar DMA 5000 digital vibrating U-tube densimeter. The temperature in the cell was regulated to  $\pm 0.001$  K with a built-in solid-state thermostat. The temperature in the cell was measured by means of two integrated Pt 100 platinum thermometers, and the temperature stability was better than  $\pm 0.002$  K. The refractive index was measured by an Anton Paar RXA 156 refractometer, and the temperature was controlled with an internal Peltier thermostat to  $\pm 0.03$  K.

To minimize evaporation of the volatile solvents and to avoid the errors in composition, all mixtures presented in this paper were prepared by mass using the cell and the procedure described previously.<sup>14,15</sup> A Mettler AG 204 balance with a precision of  $1 \cdot 10^{-4}$  g was used. The uncertainty of the mole fraction calculation was less than  $\pm 1 \cdot 10^{-4}$ . All molar quantities were based on the International Union of Pure and Applied Chemistry (IUPAC) relative atomic mass table. The experimental uncertainty in the density and refractive index is about  $\pm 1 \cdot 10^{-2} \text{ kg} \cdot \text{m}^{-3}$  and  $\pm 1 \cdot 10^{-4}$ , respectively, while the average

**Table 2. Parameters  $A_p$  of Equation 3 and the Corresponding rmsd  $\sigma$  for the Binary Mixtures at Temperature  $T$** 

	$T/K$	$A_0$	$A_1$	$A_2$	$A_3$	$A_4$	$\sigma \cdot 10^3$
2-Methyl-2-propanol (1) + Cyclohexylamine (2) <sup>a</sup>							
$V^E \cdot 10^6$ ( $\text{m}^3 \cdot \text{mol}^{-1}$ )	303.15	-4.101	-0.671	-0.025	-0.084		4
	308.15	-4.272	-0.790	-0.112	-0.092		4
	313.15	-4.448	-0.893	-0.178	-0.142		4
	318.15	-4.623	-1.016	-0.248	-0.135		4
	323.15	-4.797	-1.139	-0.280	-0.088		4
$\Delta n_D$	303.15	0.0311	0.0044	0.0011			0.04
	308.15	0.0315	0.0047	0.0018			0.04
	313.15	0.0319	0.0052	0.0017			0.04
	318.15	0.0324	0.0055	0.0015			0.03
	323.15	0.0330	0.0058	0.0017			0.03
2-Methyl-2-propanol (1) + <i>n</i> -Heptane (2)							
$V^E \cdot 10^6$ ( $\text{m}^3 \cdot \text{mol}^{-1}$ )	303.15	3.955	0.932	0.673	-1.618	0.651	4
	308.15	4.052	0.792	0.811	-1.664	0.818	5
	313.15	4.172	0.629	0.931	-1.739	0.981	5
	318.15	4.323	0.445	1.022	-1.838	1.181	6
	323.15	4.525	0.190	0.725	-1.939	1.922	8
$\Delta n_D$	303.15	-0.0133	-0.0032	-0.0032			0.02
	308.15	-0.0132	-0.0026	-0.0041			0.02
	313.15	-0.0136	-0.0025	-0.0046			0.02
	318.15	-0.0136	-0.0015	-0.0049			0.02
	323.15	-0.0136	-0.0006	-0.0066			0.03
Cyclohexylamine (1) + <i>n</i> -Heptane (2)							
$\Delta n_D$	303.15	-0.0218	0.0001	-0.0017			0.04
	308.15	-0.0218	-0.0001	-0.0015			0.04
	313.15	-0.0219	-0.0007	-0.0012			0.03
	318.15	-0.0220	-0.0008	-0.0012			0.03
	323.15	-0.0223	-0.0007	-0.0008			0.06

<sup>a</sup>Densities for (2-methyl-2-propanol + cyclohexylamine) are already reported,<sup>6</sup> but the data reduction was performed with the modified RK polynomial with temperature-dependent parameters. In this paper  $V^E$  data were used for new parameter generation using eq 3.

uncertainty in excess molar volume and refractive index deviation is estimated to be  $\pm 1 \cdot 10^{-9} \text{ m}^3 \cdot \text{mol}^{-1}$  and  $\pm 2 \cdot 10^{-4}$ , respectively.

## Results and Discussion

The excess molar volumes  $V^E$  were calculated from the equation:

$$V^E = \sum_{i=1}^N x_i M_i [(1/\rho) - (1/\rho_i)] \quad (1)$$

where  $N$  is the number of components;  $x_i$  is the mole fraction of component  $i$  in the mixture;  $M_i$  is its molecular weight;  $\rho$  and  $\rho_i$  are the measured densities of the mixture and the pure component  $i$ , respectively.

The refractive index deviations,  $\Delta n_D$ , were obtained from the expression:

$$\Delta n_D = n_D - \sum_{i=1}^N x_i n_{Di} \quad (2)$$

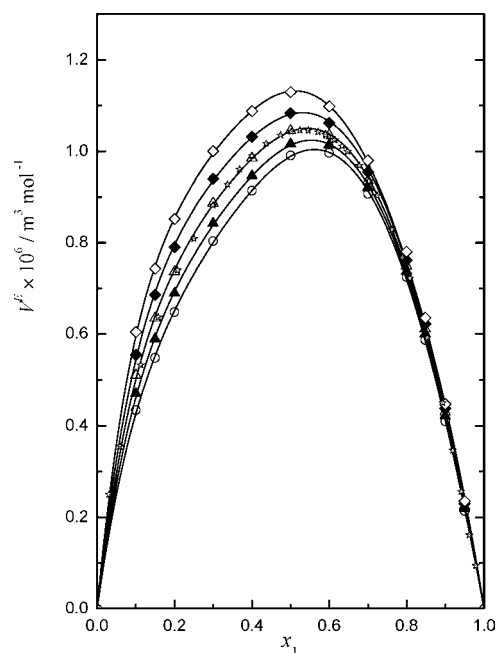
where  $n_D$  and  $n_{Di}$  are the measured refractive indices of the mixture and the pure component  $i$ , respectively.

The experimental volumetric results for the density data for the 2-methyl-2-propanol + cyclohexylamine<sup>6</sup> and cyclohexylamine + *n*-heptane<sup>5</sup> binary systems have been presented in our previous papers.

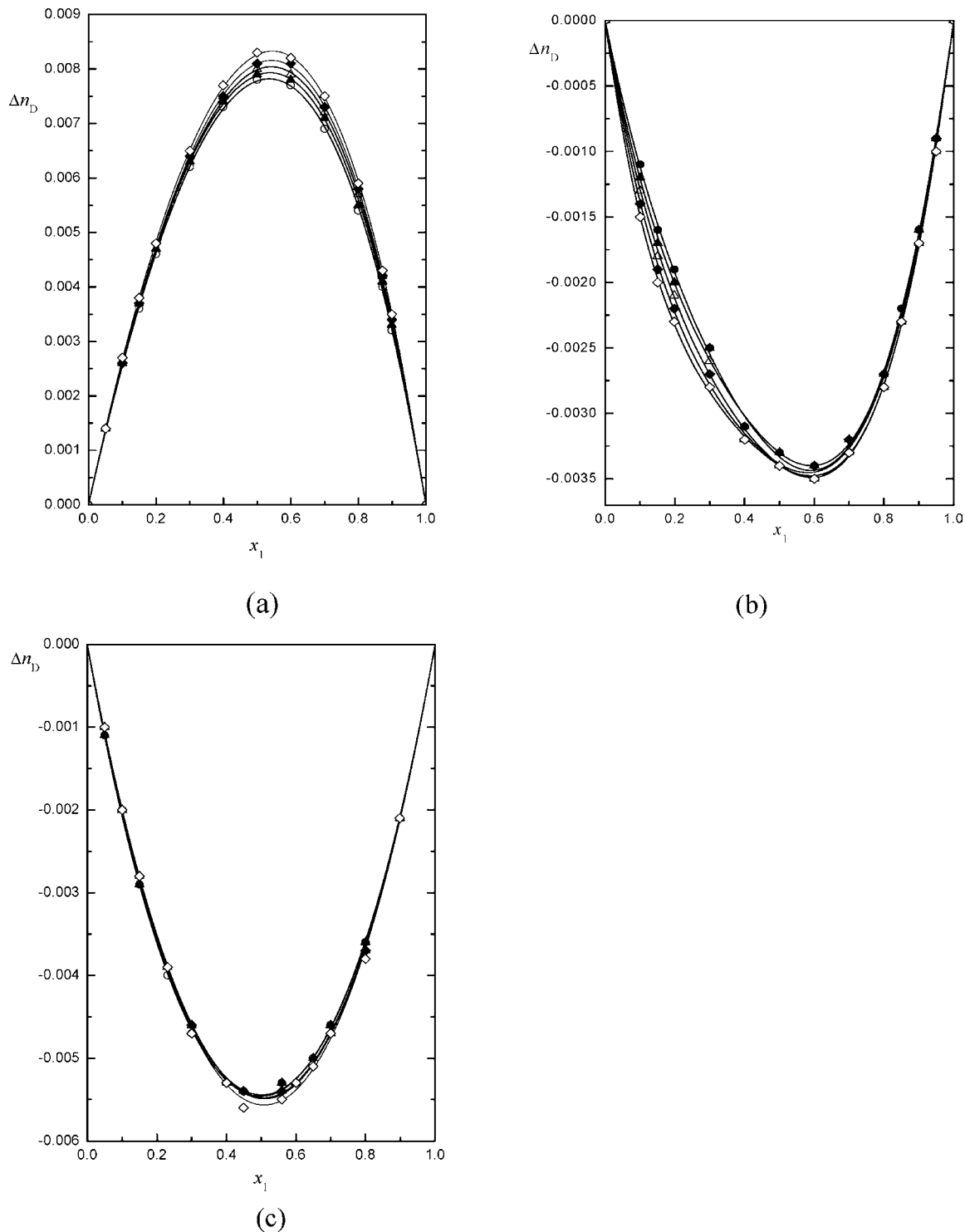
Data for the binary mixtures were correlated with the Redlich-Kister (RK) equation:<sup>7</sup>

$$Y^E = x_i x_j \sum_{p=0}^k A_p (2x_i - 1)^p \quad (3)$$

where  $Y^E$  denotes  $V_{ij}^E/10^{-6} \text{ m}^3 \cdot \text{mol}^{-1}$  or  $\Delta n_D$ ,  $A_p$  are the adjustable parameters of the related property, and the number of adjustable parameters ( $k + 1$ ) has been determined using the  $F$ -test.



**Figure 1.** Experimental  $V^E$  data for the system 2-methyl-2-propanol (1) + *n*-heptane (2). Symbols refer to experimental points at:  $\circ$ , 303.15 K;  $\blacktriangle$ , 308.15 K;  $\triangle$ , 313.15 K;  $\blacklozenge$ , 318.15 K;  $\diamond$ , 323.15 K;  $\star$ , literature data,<sup>9</sup> at 313.15 K. Lines present the results calculated by eq 3.



**Figure 2.**  $\Delta n_D$  data for the binary systems: (a) 2-methyl-2-propanol (1) + cyclohexylamine (2), (b) 2-methyl-2-propanol (1) + *n*-heptane (2), and (c) cyclohexylamine (1) + *n*-heptane (2). Symbols refer to experimental points at:  $\circ$ , 303.15 K;  $\blacktriangle$ , 308.15 K;  $\triangle$ , 313.15 K;  $\blacklozenge$ , 318.15 K;  $\diamond$ , 323.15 K. Lines present the results calculated by eq 3.

Adjustable parameters of the fits for the  $V^E$  and  $\Delta n_D$  at each temperature separately and the corresponding root-mean-square deviations (rmsd's) defined by the equation:

$$\sigma = \left( \sum_{i=1}^m (Y_{\text{exp},i}^E - Y_{\text{cal},i}^E)^2 / m \right)^{1/2} \quad (4)$$

are given in Table 2. In eq 4  $m$  is the number of experimental data points.

In Figure 1 the experimental  $V^E$  data for the binary system 2-methyl-2-propanol + *n*-heptane are plotted for all investigated temperatures, while Figure 2 presents experimental  $\Delta n_D$  data for the binaries 2-methyl-2-propanol + cyclohexylamine (Figure 2a), 2-methyl-2-propanol + *n*-heptane (Figure 2b), and cyclohexylamine + *n*-heptane (Figure 2c).

A comparison of our experimental results of  $V^E$  for the system 2-methyl-2-propanol + *n*-heptane at 313.15 K with the data

**Table 3.** Parameters  $B_p$  of Equation 5 and Corresponding  $\text{rmsd } \sigma$  for 2-Methyl-2-propanol (1) + Cyclohexylamine (2) +  $n$ -Heptane (3) at Temperature  $T$  and Atmospheric Pressure

$T/K$	303.15	308.15	313.15	318.15	323.15
	$V^E$				
$B_0$	$-9.794 \cdot 10^{-3}$	$-1.062 \cdot 10^{-2}$	$-9.375 \cdot 10^{-3}$	$-9.221 \cdot 10^{-3}$	$-8.845 \cdot 10^{-3}$
$B_1$	$-8.396 \cdot 10^{-4}$	$-6.140 \cdot 10^{-3}$	$2.344 \cdot 10^{-3}$	$1.911 \cdot 10^{-2}$	$3.905 \cdot 10^{-3}$
$B_2$	$-1.961 \cdot 10^{-2}$	$-2.424 \cdot 10^{-2}$	$-1.957 \cdot 10^{-2}$	$-1.872 \cdot 10^{-2}$	$-1.803 \cdot 10^{-2}$
$B_3$	$-2.092 \cdot 10^{-2}$	$-8.526 \cdot 10^{-3}$	$-3.012 \cdot 10^{-2}$	$-2.902 \cdot 10^{-2}$	$-3.294 \cdot 10^{-2}$
$B_4$	$1.304 \cdot 10^{-2}$	$2.295 \cdot 10^{-2}$	$1.390 \cdot 10^{-2}$	$1.185 \cdot 10^{-2}$	$1.190 \cdot 10^{-2}$
$B_5$	$7.701 \cdot 10^{-4}$	$5.019 \cdot 10^{-3}$	$-3.729 \cdot 10^{-3}$	$-1.953 \cdot 10^{-3}$	$-4.254 \cdot 10^{-3}$
$B_6$	$2.158 \cdot 10^{-2}$	$1.288 \cdot 10^{-2}$	$2.769 \cdot 10^{-2}$	$2.707 \cdot 10^{-2}$	$2.858 \cdot 10^{-2}$
$B_7$	$1.966 \cdot 10^{-3}$	$-4.696 \cdot 10^{-3}$	$1.887 \cdot 10^{-3}$	$3.319 \cdot 10^{-3}$	$2.898 \cdot 10^{-3}$
$B_8$	$2.511 \cdot 10^{-2}$	$2.187 \cdot 10^{-2}$	$3.639 \cdot 10^{-2}$	$3.254 \cdot 10^{-2}$	$3.554 \cdot 10^{-2}$
$\sigma/10^{-6} \text{ m}^3 \cdot \text{mol}^{-1}$	0.013	0.013	0.012	0.011	0.011
	$\Delta n_D$				
$B_0$	$4.810 \cdot 10^{-5}$	$4.027 \cdot 10^{-5}$	$3.411 \cdot 10^{-5}$	$3.343 \cdot 10^{-5}$	$3.125 \cdot 10^{-5}$
$B_1$	$-7.056 \cdot 10^{-3}$	$-1.238 \cdot 10^{-4}$	$-1.1263 \cdot 10^{-4}$	$-1.113 \cdot 10^{-4}$	$-6.052 \cdot 10^{-5}$
$B_2$	$2.473 \cdot 10^{-4}$	$2.366 \cdot 10^{-4}$	$1.983 \cdot 10^{-4}$	$1.888 \cdot 10^{-4}$	$1.454 \cdot 10^{-4}$
$B_3$	$2.693 \cdot 10^{-4}$	$3.805 \cdot 10^{-4}$	$3.366 \cdot 10^{-4}$	$3.392 \cdot 10^{-4}$	$2.306 \cdot 10^{-4}$
$B_4$	$-4.184 \cdot 10^{-4}$	$-4.488 \cdot 10^{-4}$	$-3.865 \cdot 10^{-4}$	$-3.469 \cdot 10^{-4}$	$-2.482 \cdot 10^{-4}$
$B_5$	$1.476 \cdot 10^{-5}$	$1.274 \cdot 10^{-4}$	$1.253 \cdot 10^{-4}$	$1.165 \cdot 10^{-4}$	$7.208 \cdot 10^{-5}$
$B_6$	$-1.989 \cdot 10^{-4}$	$-2.570 \cdot 10^{-4}$	$-2.268 \cdot 10^{-4}$	$-2.186 \cdot 10^{-4}$	$-1.538 \cdot 10^{-4}$
$B_7$	$2.483 \cdot 10^{-4}$	$2.843 \cdot 10^{-4}$	$2.515 \cdot 10^{-4}$	$2.170 \cdot 10^{-4}$	$1.547 \cdot 10^{-4}$
$B_8$	$-1.234 \cdot 10^{-4}$	$-2.992 \cdot 10^{-4}$	$-2.704 \cdot 10^{-4}$	$-2.688 \cdot 10^{-4}$	$-1.986 \cdot 10^{-4}$
$\sigma/10^{-3}$	0.1	0.1	0.1	0.1	0.1

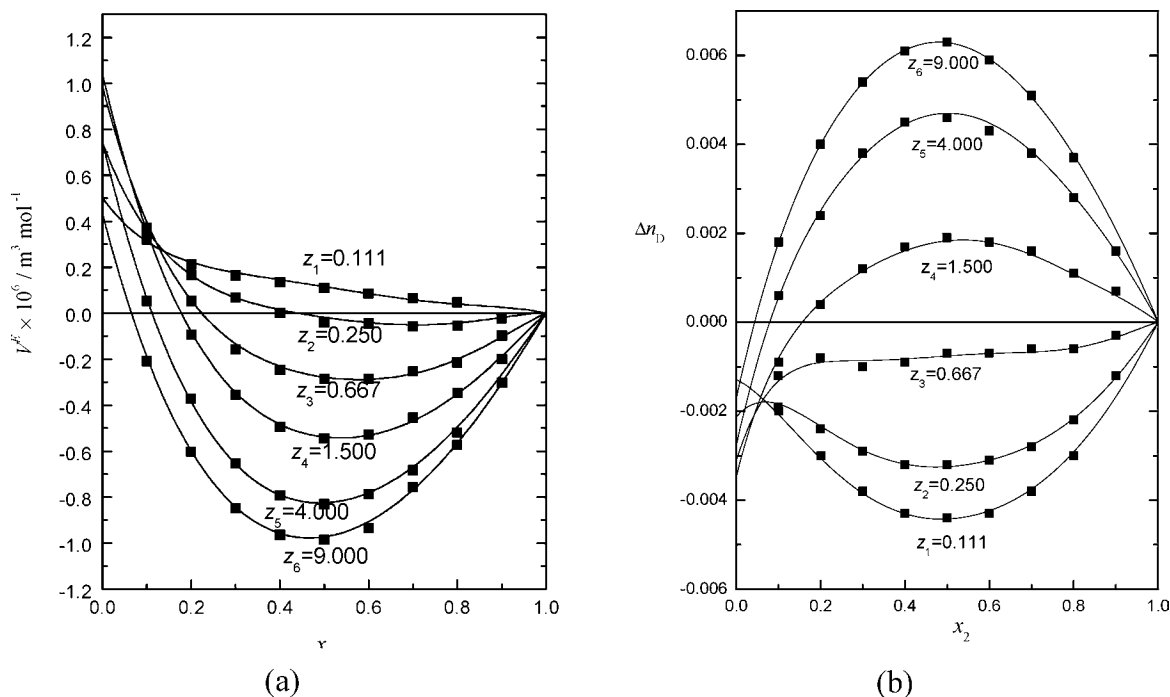
reported previously shows that our  $V^E$  data are in very good agreement with the literature data<sup>9</sup> (less than 0.1 % at  $x_1 = 0.5$ ).

It should be useful to compare presented results of  $V^E$  with our previous results for binary systems alcohols (1-butanol or 2-butanol) + (cyclohexylamine or  $n$ -heptane).<sup>4–6</sup> The negative trend of  $V^E$  values for alcohol + cyclohexylamine mixtures is in the order 1-butanol > 2-methyl-2-propanol > 2-butanol where interactions of tertiary alcohol molecules are stronger than those of secondary alcohol molecules in mixtures with an amine. A very low temperature influence on  $V^E$  is present in all considered systems. Positive values of  $V^E$  for the system cyclohexylamine +  $n$ -heptane are a

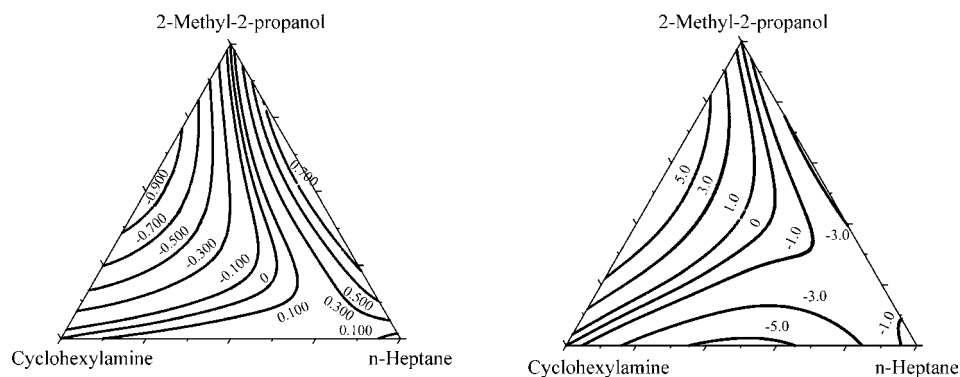
consequence of the H-bonded structure of amine. Stronger amine–amine interactions at higher temperatures probably effect decreasing  $V^E$  values.

The negative trend of  $V^E$  values for alcohol +  $n$ -heptane mixtures is in the following order: 2-methyl-2-propanol > 2-butanol > 1-butanol, indicating that steric hindrance was the largest in the mixture with tertiary alcohol. Interstitial accommodation between molecules is more difficult to occur in mixtures with tertiary alcohol, as a consequence of the chain length and the position of OH group; therefore, the temperature influence is also higher.

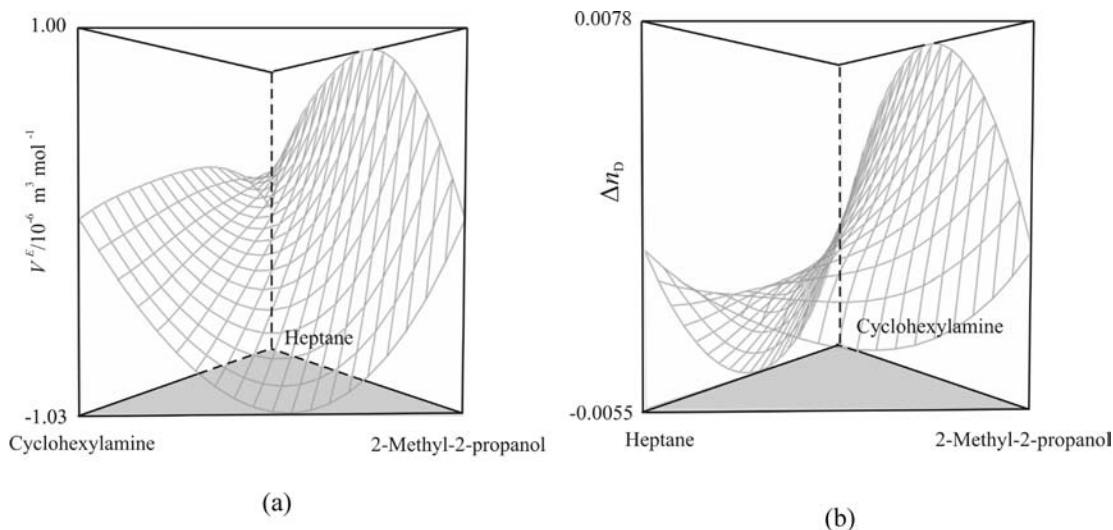
The ternary  $V^E$  and  $\Delta n_D$  data were correlated by Nagata and Tamura<sup>8</sup> equation:



**Figure 3.** (a) Excess molar volume  $V^E$  at 303.15 K and (b) deviations of refractive index  $\Delta n_D$  at 313.15 K for the ternary system 2-methyl-2-propanol (1) + cyclohexylamine (2) +  $n$ -heptane (3), along the curves of constant ratio  $z = x_1/x_3$  as a function of the cyclohexylamine composition. Symbols represent the experimental points. Solid curves were calculated by eq 5.



**Figure 4.** Curves of constant: (a)  $V^E/10^{-6} \text{ m}^3 \cdot \text{mol}^{-1}$  and (b)  $\Delta n_D/10^{-3}$  for the ternary system 2-methyl-2-propanol (1) + cyclohexylamine (2) + *n*-heptane (3) at 303.15 K; the lines present results calculated by eq 5.



**Figure 5.** Three-dimensional surface for the (a) excess molar volume  $V^E$  and (b) deviations of refractive index  $\Delta n_D$  for the ternary system 2-methyl-2-propanol (1) + cyclohexylamine (2) + *n*-heptane (3) at 303.15 K, generated by eq 5.

$$Y_{123}^E = Y_{12}^E + Y_{13}^E + Y_{23}^E + x_1 x_2 x_3 RT (B_0 - B_1 x_1 - B_2 x_2 - B_3 x_1^2 - B_4 x_2^2 - B_5 x_1 x_2 - B_6 x_1^3 - B_7 x_2^3 - B_8 x_1^2 x_2) \quad (5)$$

where  $Y_{123}^E$  denotes  $V_{123}^E/10^{-6} \text{ m}^3 \cdot \text{mol}^{-1}$  or  $\Delta n_{D,123}$ ,  $x_1$ ,  $x_2$ , and  $x_3$  are mole fractions of the ternary system;  $Y_{ij}^E$  represent the excess molar volumes ( $V_{12}^E$ ,  $V_{13}^E$ , and  $V_{23}^E$ ) or deviations of refractive indices ( $\Delta n_{D,12}$ ,  $\Delta n_{D,13}$ , and  $\Delta n_{D,23}$ ) calculated using eq 3, with ternary compositions  $x_i$  and  $x_j$ .  $B_0$ ,  $B_1$ , ..., to  $B_8$  are the adjustable parameters of ternary contribution obtained from ternary experimental  $V^E$  and  $\Delta n_D$  data. These fitted parameters of eq 5 along with the corresponding  $\sigma$ , calculated according to eq 4, are given in Table 3.

The experimental determinations of  $V^E$  and  $\Delta n_D$  for the ternary system were made following the lines of constant ratio  $z$  ( $= x_1/x_3$ ). Figure 3 represents experimental  $V^E$  at 303.15 K (Figure 3a) and  $\Delta n_D$  values (Figure 3b) at 313.15 K with the fitted lines of constant ( $x_1/x_3$ ) obtained using eq 5.

Figure 4 parts a and b shows the isolines of the  $V^E$  and  $\Delta n_D$  for the ternary system at 303.15 K, while Figure 5 presents the three-dimensional surface for the excess molar volume  $V^E$  (Figure 5a) and deviations of refractive index  $\Delta n_D$  (Figure 5b) for the ternary system 2-methyl-2-propanol (1) + cyclohexylamine (2) + *n*-heptane (3) at 303.15 K, generated by eq 5.

A comparison of ternary systems of alcohols (1-butanol, 2-butanol, or 2-methyl-2-propanol) with cyclohexylamine and *n*-heptane is in accordance to binary constituent behavior, as it is given previously.

#### Supporting Information Available:

Tables S1 to S3: the measured densities  $\rho$  with the corresponding values of  $V^E$ , for the system 2-methyl-2-propanol + *n*-heptane (Table S1); measured refractive indices  $n_D$  with the corresponding values of  $\Delta n_D$  for the 2-methyl-2-propanol + cyclohexylamine, 2-methyl-2-propanol + *n*-heptane, and cyclohexylamine + *n*-heptane binary systems (Table S2); the measured densities  $\rho$  and refractive indices  $n_D$  with the corresponding values of  $V^E$  and  $\Delta n_D$ , for the ternary 2-methyl-2-propanol + cyclohexylamine + *n*-heptane system (Table S3). This material is available free of charge via the Internet at <http://pubs.acs.org>.

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