



J. Serb. Chem. Soc. 79 (1) 77–87 (2014)
JSCS–4566

Densities, refractive indices and viscosities of the binary mixtures of dimethyl phthalate or dimethyl adipate with tetrahydrofuran

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(Received 7 April, revised 23 April 2013)

Abstract: Densities, refractive indices and viscosities of the binary mixtures of dimethyl phthalate (or dimethyl adipate) + tetrahydrofuran have been measured at eight temperatures (288.15 to 323.15 K) and atmospheric pressure. All measurements were performed using an Anton Paar DMA 5000 digital vibrating-tube densimeter, Anton Paar RXA 156 refractometer and Anton Paar SVM 3000/G2 digital Stabinger viscometer, respectively. From the experimental densities, refractive indices and viscosities, the excess molar volumes, V^E , deviations of refractive indices, Δn_D , and viscosity deviations, $\Delta \eta$, were calculated.

Keywords: experimental measurements; excess molar volumes; deviations of refractive indices; viscosity deviations; esters; ethers.

INTRODUCTION

This article is a continuation of previous research dealing with the experimental determination of volumetric and transport properties of binary and ternary mixtures containing different organic solvents, such as alcohols and aromatics, chlorinated aromatics, esters, ketones, chlorinated alkanes or amines.^{1–10} An intention of this study is to provide a set of volumetric and transport data in order to assess the influence of temperature and molecular structure on the behaviour of mixtures of esters (dimethyl phthalate or dimethyl adipate) and ethers (tetrahydrofuran). From the theoretical viewpoint, this is an important source of information for the characterization of interactions between components and is helpful in understanding the liquid state theory, as well. Besides, esters and ethers are widely used in a variety of industrial and consumer applications, and hence, knowledge of their physical properties is of great importance from a practical

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doi: 10.2298/JSC130407045K



point of view, too. Phthalates are widely used in the automobile, cable, medical equipment and the toy industries, as plasticizers and as performance enhancers in the manufacture of glues and paints. Dimethyl phthalate is used as an additive for plastics, which increases their flexibility, transparency and stability. Dimethyl adipate is an organic solvent for inks, coatings and adhesives, an emollient that can be used in the manufacture of agrochemicals, synthetic leather, paint strippers, plasticizers, or as a food additive and pigment dispersant. Tetrahydrofuran is primarily used as an industrial solvent for poly(vinyl chloride) and varnishes.

In the present work, measured densities, ρ , refractive indices, n_D , and viscosities, η , are reported for the two binary systems containing dimethyl phthalate (or dimethyl adipate) and tetrahydrofuran at eight temperatures (from 288.15 to 323.15 K) and atmospheric pressure. The excess molar volumes, V^E , deviations of the refractive indices, Δn_D , and viscosity deviations, $\Delta\eta$, of the investigated mixtures were calculated from the measured data.

To the best of our knowledge, ρ , n_D and η experimental data are not available for the investigated binary systems and no V^E , Δn_D and $\Delta\eta$ values were found in the currently published articles.

EXPERIMENTAL

Materials

All products were of high purity (mass fraction purity > 0.99) and used without further purification: dimethyl phthalate (>0.99) was supplied by Fluka, while dimethyl adipate (≥ 0.99) and tetrahydrofuran (min 0.995) were Merck products. Two different bottles of Merck-supplied high purity tetrahydrofuran (min 0.995) were used in the experimental work: one was used in the experiments with dimethyl phthalate and another in the experiments with dimethyl adipate. All the organic liquids were stored in brown glass bottles under an inert nitrogen atmosphere. Pure components were degassed in an ultrasonic bath shortly before sample preparation. Densities, refractive indices and viscosities of the pure components at the temperatures 293.15 and 298.15 K, together with the corresponding literature values,^{11–19} are listed in Table I.

TABLE I. Density, ρ , refractive index, n_D , and viscosity, η , values of the studied pure components

T / K	$\rho / 10^3 \text{ kg m}^{-3}$		n_D		$\eta / \text{mPa s}$	
	Exp.	Lit.	Exp.	Lit.	Exp.	Lit.
Tetrahydrofuran						
298.15	0.882330 ^a	0.882358 ¹¹	1.404984 ^a	1.40496 ¹³	0.48821 ^a	0.456 ¹²
	0.882402 ^b	0.882502 ¹²	1.404322 ^b		0.47984 ^b	0.460 ¹³
Dimethyl phthalate						
298.15	1.186933	1.18657 ¹⁴	1.513544	1.5137 ¹⁵ , 1.513 ¹⁶	13.847	13.76169 ¹⁴
Dimethyl adipate						
293.15	1.061928	1.06190 ¹⁷	–	–	3.2894	3.36 ¹⁹
298.15	–	–	1.426356	1.4215 ¹⁷ , 1.4283 ¹⁸	2.9101	2.98 ¹⁹

^aFirst bottle, system with dimethyl phthalate; ^bsecond bottle, system with dimethyl adipate

Apparatus and procedure

The densities of the binary mixtures, and corresponding pure substances were measured with an Anton Paar DMA 5000 digital vibrating U-tube densimeter having a stated accuracy $\pm 5 \times 10^{-3} \text{ kg m}^{-3}$. The temperature in the cell was measured by means of two integrated Pt 100 platinum thermometers with a stability of better than $\pm 0.002 \text{ K}$; the temperature was regulated to $\pm 0.001 \text{ K}$ with a built-in solid-state thermostat. The refractive indices were measured by an Anton Paar RXA 156 refractometer with a stated accuracy of $\pm 5 \times 10^{-5}$, and the temperature was controlled with an internal Peltier thermostat to $\pm 0.03 \text{ K}$. The viscosities were measured with an Anton Paar SVM 3000/G2 digital Stabinger viscometer, with a stated accuracy of $\pm 0.1 \%$ of the measurement value and the temperature was regulated with an uncertainty of $\pm 0.01 \text{ K}$ by a built in solid-state thermostat.

In order to minimize evaporation of the volatile solvents and to avoid errors in the compositions, all mixtures presented in this paper were prepared by mass using the cell and a previously described procedure.^{20,21} The masses were measured using a Mettler AG 204 balance, with a precision of $1 \times 10^{-7} \text{ kg}$. The uncertainty in the calculation of the mole fraction was less than $\pm 1 \times 10^{-4}$. All molar quantities were based on the IUPAC relative atomic mass table. The experimental uncertainty (from repeated measurements) in the density, refractive index and viscosity measurements were about $\pm 1 \times 10^{-2} \text{ kg m}^{-3}$, $\pm 1 \times 10^{-4}$ and $< 1.5 \%$, respectively, while the average uncertainty in the excess molar volume, refractive index deviation and viscosity deviation were estimated at $\pm 3 \times 10^{-9} \text{ m}^3 \text{ mol}^{-1}$, $\pm 2 \times 10^{-4}$ and better than $\pm 3 \times 10^{-3} \text{ mPa s}$, respectively.

RESULTS AND DISCUSSION

Excess molar volumes V^E were calculated from the density data by the equation:

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

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; ρ and ρ_i are the measured densities of the mixture and the pure component i , respectively.

The refractive index deviations, Δn_D , were calculated as follows:

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

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

The viscosity deviations, $\Delta \eta$, were calculated from the equation:

$$\Delta \eta = \eta - \sum_{i=1}^N x_i \eta_i \quad (3)$$

where η and η_i are the measured viscosities of the mixture and the pure component i , respectively.

The results of V^E , Δn_D and $\Delta\eta$ were correlated with the Redlich–Kister (RK) Equation:²²

$$Y = x_i x_j \sum_{p=0}^k A_p (2x_i - 1)^p \quad (4)$$

where Y denotes $V_{ij}^E / \text{m}^3 \cdot \text{mol}^{-1}$, Δn_D , or $\Delta\eta / \text{mPa} \cdot \text{s}$, A_p , are the adjustable parameters of the related property, and the number of adjustable parameters ($k + 1$) was determined using the F -test.²³

The results of ρ , V^E , n_D , Δn_D , η and $\Delta\eta$ for the investigated binaries, in the temperature range and over the entire concentration range, are summarized in Table S-1 of the Supplementary material to this paper. Table II lists the coefficients A_p for the V^E , Δn_D and $\Delta\eta$ at each temperature separately, and the corresponding root-mean-square deviations (σ) given by the equation:

$$\sigma = \left(\sum_{i=1}^m (Y_{\text{exp},i} - Y_{\text{cal},i})^2 / m \right)^{1/2} \quad (5)$$

where m is the number of experimental data points.

The results were also fitted and explained using the reduced excess molar volume, V^E/x_1x_2 , refractive index deviation, $\Delta n_D/x_1x_2$, and viscosity deviation, $\Delta\eta/x_1x_2$.

TABLE II. Parameters A_p of Eq. (4) and the corresponding root mean square deviations, σ

T / K	A_0	A_1	A_2	A_3	A_4	$\sigma \times 10^2$
Dimethyl phthalate (1) + tetrahydrofuran (2)						
$V^E / 10^{-6} \text{ m}^3 \text{ mol}^{-1}$						
288.15	-2.6986	0.3765	0.3855	1.0737	–	0.85
293.15	-2.7797	0.3896	0.4101	1.1170	–	0.87
298.15	-2.8622	0.4216	0.3845	1.1240	–	0.85
303.15	-2.9520	0.4544	0.3826	1.1361	–	0.85
308.15	-3.0495	0.4699	0.3800	1.1922	–	0.87
313.15	-3.1571	0.4934	0.3777	1.2369	–	0.89
318.15	-3.2404	0.6075	0.2676	1.0932	–	0.76
323.15	-3.3078	0.6088	0.3930	0.9423	–	0.79
Δn_D						
288.15	0.0849	-0.0325	0.0124	-0.0063	–	0.01
293.15	0.0853	-0.0327	0.0117	-0.0068	–	0.01
298.15	0.0855	-0.0327	0.0126	-0.0076	–	0.01
303.15	0.0861	-0.0329	0.0120	-0.0082	–	0.01
308.15	0.0862	-0.0313	0.0123	-0.0128	–	0.01
313.15	0.0858	-0.0313	0.0132	-0.0124	–	0.01
318.15	0.0863	-0.0308	0.0128	-0.0132	–	0.01
323.15	0.0861	-0.0309	0.0155	-0.0152	–	0.02

TABLE II. Continued

T / K	A_0	A_1	A_2	A_3	A_4	$\sigma \times 10^2$
Dimethyl phthalate (1) + tetrahydrofuran (2)						
$\Delta\eta / \text{mPa s}$						
288.15	-33.4291	-20.0319	-11.3009	-4.4937	-	2.43
293.15	-23.9657	-13.2136	-6.6039	-2.2368	-	2.10
298.15	-17.6840	-8.8378	-3.8201	-1.2562	-	1.89
303.15	-13.3160	-6.1789	-2.4057	-0.4603	-	1.51
308.15	-10.2461	-4.3833	-1.4981	-0.0010	-	1.27
313.15	-8.0454	-2.9403	-0.6578	-0.7116	-	1.87
318.15	-6.3678	-2.2431	-0.6323	0.1581	-	0.89
323.15	-5.1077	-1.7111	-0.5757	0.5363	-	0.80
Dimethyl adipate (1) + tetrahydrofuran (2)						
$V^E / 10^{-6} \text{ m}^3 \text{ mol}^{-1}$						
288.15	-0.1986	0.1311	-0.1620	-0.0862	0.2422	0.02
293.15	-0.2335	0.1434	-0.1640	-0.0810	0.2390	0.01
298.15	-0.2725	0.1567	-0.1634	-0.0822	0.2297	0.01
303.15	-0.3152	0.1705	-0.1616	-0.0768	0.2215	0.01
308.15	-0.3593	0.1872	-0.1697	-0.0761	0.2196	0.02
313.15	-0.4086	0.2037	-0.1764	-0.0697	0.2204	0.02
318.15	-0.4612	0.2247	-0.1821	-0.0683	0.2178	0.02
323.15	-0.5190	0.2463	-0.1947	-0.0594	0.2206	0.03
Δn_D						
288.15	0.0153	-0.0057	0.0028	-0.0019	-	0.001
293.15	0.0158	-0.0060	0.0030	-0.0019	-	0.001
298.15	0.0163	-0.0060	0.0030	-0.0023	-	0.001
303.15	0.0169	-0.0068	0.0027	-0.0010	-	0.001
308.15	0.0174	-0.0066	0.0026	-0.0017	-	0.001
313.15	0.0178	-0.0067	0.0026	-0.0020	-	0.001
318.15	0.0180	-0.0069	0.0028	-0.0020	-	0.001
323.15	0.0184	-0.0067	0.0026	-0.0022	-	0.001
$\Delta\eta / \text{mPa s}$						
288.15	-1.7896	-0.0179	0.0909	-0.0164	-0.0088	0.08
293.15	-1.4060	0.0285	0.0519	-0.0027	0.0264	0.04
298.15	-1.1035	0.0621	0.0004	-0.0073	0.0962	0.04
303.15	-0.8827	0.0868	-0.0111	-0.0347	0.0494	0.14
308.15	-0.6952	0.1021	-0.0925	-0.0198	0.1344	0.15
313.15	-0.5431	0.1447	-0.0766	-0.0850	0.0353	0.14
318.15	-0.4063	0.0614	-0.3521	0.0455	0.3789	0.12
323.15	-0.2925	0.1094	0.0610	-0.2807	0.0478	0.10

The dependence $V^E_{-x_1}$, Δn_{D-x_1} and $\Delta\eta_{-x_1}$, respectively, for the systems measured in this work are shown in Fig. 1.

The calculated $V^E_{/x_1x_2}$, $\Delta n_{D/x_1x_2}$ and $\Delta\eta_{/x_1x_2}$ values, respectively, are plotted as a function of the mole fraction of an ester in Fig. 2. The nonlinear curves obtained for all the investigated systems represent their evident non-ideal behaviour.

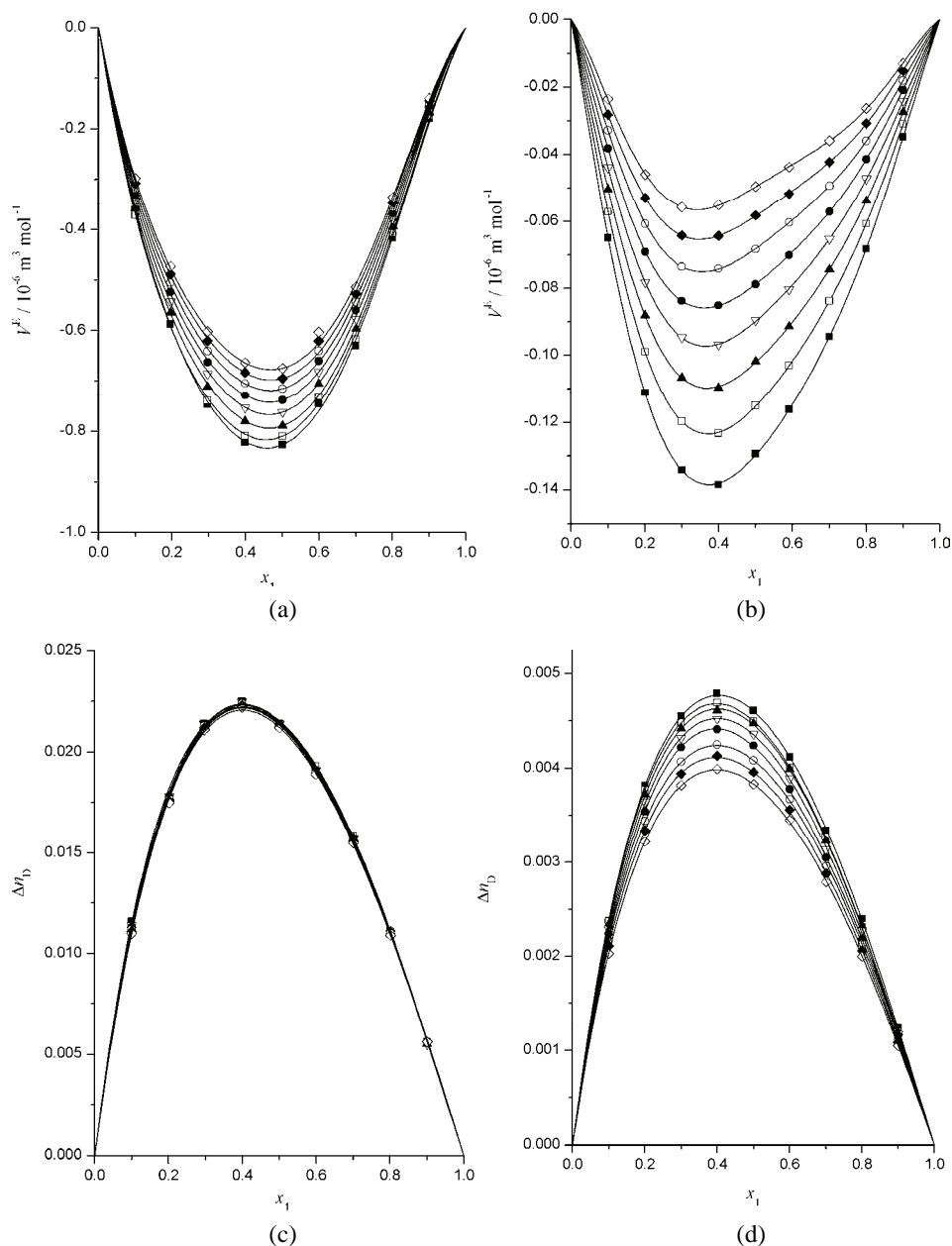


Fig. 1. Data for the binary systems: a) V^E values for dimethyl phthalate (1) + tetrahydrofuran (2), b) V^E values for dimethyl adipate (1) + tetrahydrofuran (2), c) Δn_D values for dimethyl phthalate (1) + tetrahydrofuran (2) and d) Δn_D values for dimethyl adipate (1) + tetrahydrofuran (2). The symbols refer to experimental data points at: \diamond , 288.15; \blacklozenge , 293.15; \circ , 298.15; \bullet , 303.15; ∇ , 308.15; \blacktriangle , 313.15; \square , 318.15; \blacksquare , 323.15 K. The lines present the results calculated by Eq. (4) with the parameters presented in Table II.

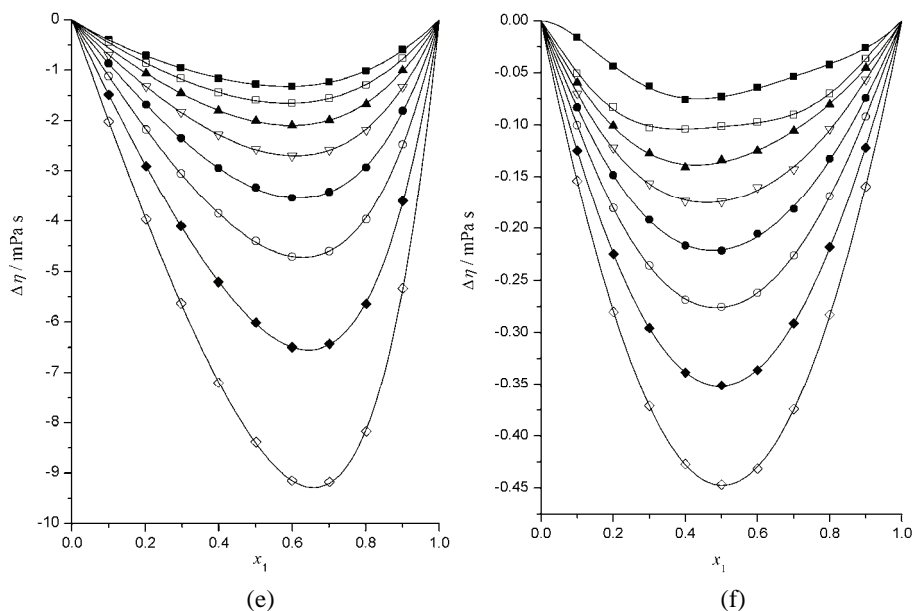


Fig. 1. (Continued). e) $\Delta\eta$ values for dimethyl phthalate (1) + tetrahydrofuran (2) and f) dimethyl adipate (1) + tetrahydrofuran (2).

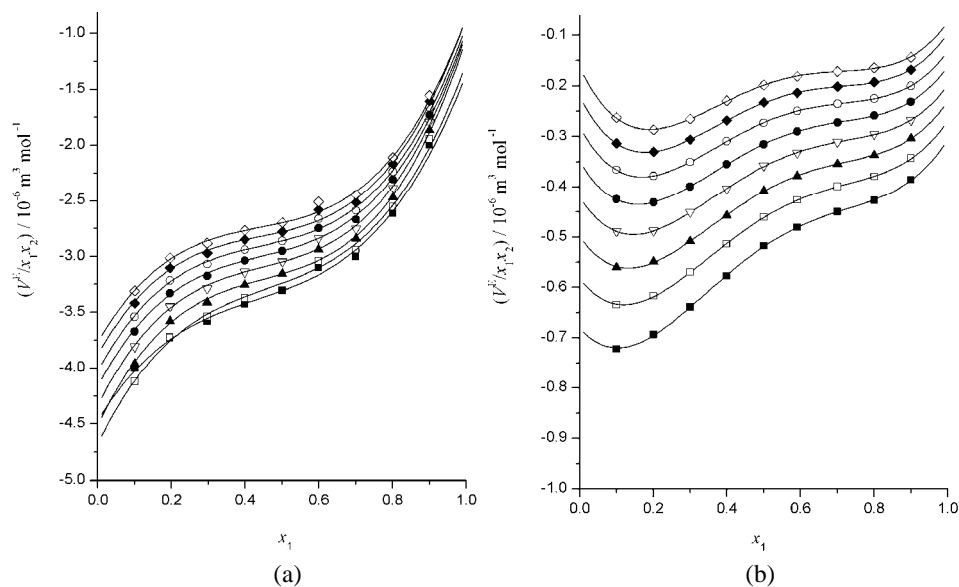


Fig. 2. Data for the binary systems: a) values of (V^E/x_1x_2) for dimethyl phthalate (1) + tetrahydrofuran (2) and b) dimethyl adipate (1) + tetrahydrofuran (2). The symbols refer to experimental data points at: \diamond , 288.15; \blacklozenge , 293.15; \circ , 298.15; \bullet , 303.15; ∇ , 308.15; \blacktriangle , 313.15; \square , 318.15; \blacksquare , 323.15 K. The lines present the results calculated by Eq. (4) with the parameters presented in Table II.

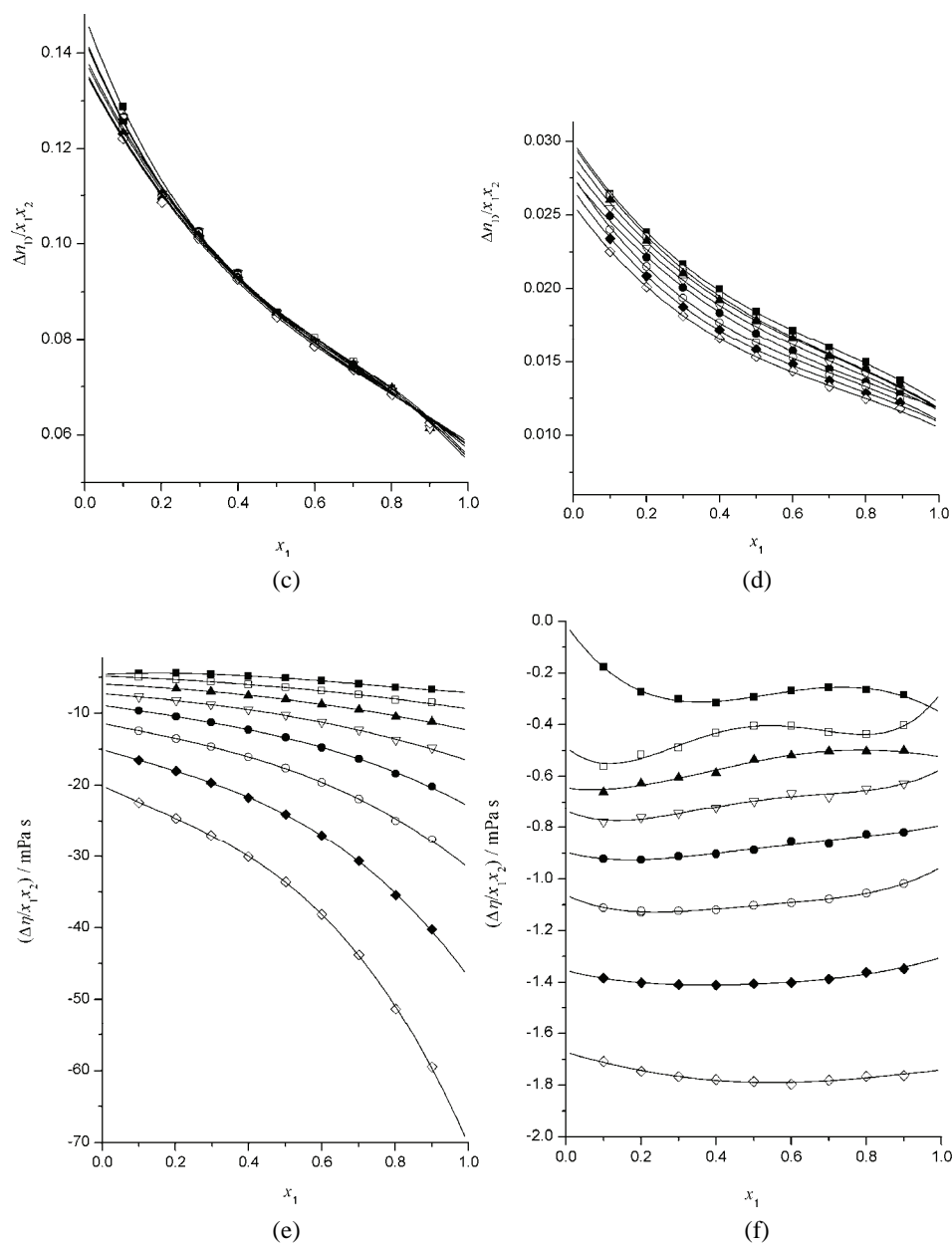


Fig.2. (Continued). c) Values of $(\Delta n_D/x_1x_2)$ for dimethyl phthalate (1) + tetrahydrofuran (2), d) values of $(\Delta n_D/x_1x_2)$ for dimethyl adipate (1) + tetrahydrofuran (2), e) values of $(\Delta \eta/x_1x_2)$ for dimethyl phthalate (1) + tetrahydrofuran (2), and f) values of $(\Delta \eta/x_1x_2)$ for dimethyl adipate (1) + tetrahydrofuran (2).

The compounds analyzed herein have good hydrogen bond abilities and polar nature. However, there is no possibility for hydrogen bonds in their mixtures since all analyzed compounds only act as good hydrogen bond acceptors and not proton donors. Tetrahydrofuran²⁴ has a dipole moment of 5.7×10^{-30} C m, whereas those of dimethyl phthalate²⁵ and dimethyl adipate²⁶ are somewhat higher 9.3×10^{-30} and 7.3×10^{-30} C m, respectively. Due to these distinguished polarities of the compounds, association through dipolar forces might be expected. As shown in Fig. 1a, both binaries are characterized by negative V^E values over the entire concentration range. The curve V^E-x_1 for the system dimethyl phthalate + tetrahydrofuran is symmetrical, while that for the system with dimethyl adipate is slightly asymmetrical and shifted towards lower mole fractions of the ester. Tetrahydrofuran is a saturated heterocyclic compound having the characteristics of an aliphatic ether with two free electron pairs on the oxygen atom. In a binary system with an ester, dipole-dipole interactions occur resulting in negative V^E values. The negative sign of V^E also indicates a net packing effect contributed by structural changes arising from interstitial accommodation. It is obvious from Fig. 1 that the contraction in volume for the system with dimethyl phthalate is almost an order of magnitude larger than that for the system with dimethyl adipate. It could be presumed that the dipole-dipole interactions between dimethyl phthalate and tetrahydrofuran are much stronger than the interactions between dimethyl adipate and tetrahydrofuran, due to the higher value of the dipole moment for dimethyl phthalate. With increasing temperature, the V^E values decrease due to the increased molecule activity.

Both binary systems are characterized by a slightly asymmetrical positive Δn_D-x_1 curve, shifted towards lower mole fractions of ester, over the entire concentration range (Fig. 1c and d). The refractive index deviations for the binary containing dimethyl phthalate are an order of magnitude higher than those recorded for the system with dimethyl adipate. The influence of temperature on Δn_D for the system containing dimethyl phthalate is almost negligible, while the Δn_D values increase with increasing temperature for the binary containing dimethyl adipate.

As it is shown in Fig. 1e and f, the $\Delta \eta-x_1$ values are negative for both systems over the entire concentration range, although symmetrical for the system with dimethyl adipate and asymmetrical and shifted towards the higher mole fraction of ester for the system containing dimethyl phthalate. Negative $\Delta \eta$ values mean that a mixture is more viscous than the pure substances. As in the case of the excess molar volumes and changes of refractive indices, deviations in viscosities are much more pronounced for the system with dimethyl phthalate, which confirms that the interactions between dimethyl phthalate and tetrahydrofuran are greatly stronger than those between dimethyl phthalate and tetrahydrofuran. Additionally, the influence of temperature is much more pronounced for

the system containing dimethyl phthalate. In both instances, the $\Delta\eta$ values become less negative with increasing temperature.

CONCLUSIONS

The densities, refractive indices and viscosities for the binary mixtures dimethyl phthalate (or dimethyl adipate) + tetrahydrofuran were measured at eight temperatures (288.15–323.15 K) at atmospheric pressure. The excess molar volumes, refractive index deviations and viscosity deviations were calculated from the experimental data. For both investigated systems, the excess molar volumes and viscosity deviations were negative, while the refractive index deviations were positive.

SUPPLEMENTARY MATERIAL

Densities, excess molar volumes, refractive indices, refractive index deviations, viscosities, and viscosity deviations, for the investigated binary mixtures at different temperatures and atmospheric pressure are available electronically from <http://www.shd.org.rs/JSCS/>, or from the corresponding author on request.

Acknowledgement. The authors gratefully acknowledge the financial support received from the Research Fund of the Ministry of Education, Science and Technological Development of the Republic of Serbia and the Faculty of Technology and Metallurgy, University of Belgrade (Project No 172063).

ИЗВОД

ГУСТИНЕ, ИНДЕКСИ РЕФРАКЦИЈЕ И ВИСКОЗНОСТИ БИНАРНИХ СМЕША ДИМЕТИЛФТАЛАТА ИЛИ ДИМЕТИЛАДИПАТА СА ТЕТРАГИДРОФУРАНОМ

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Густине, индекси рефракције и вискозности бинарних смеша диметилфталата (или диметиладипата) + тетраhydroфуран су мерене на осам температура (288,15–323,15 K) и на атмосферском притиску. Сва мерења су извршена на Anton Paar DMA 5000 дигиталном густинометру, односно Anton Paar RXA 156 рефрактометру и Anton Paar SVM 3000/G2 дигиталном вискозиметру. Из експерименталних вредности густина, индекса рефракције и вискозности израчунате су допунске моларне запремине, односно промене индекса рефракције и вискозности наведених смеша.

(Примљено 7. априла, ревидирано 23. априла 2013)

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