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Density and Viscosity of Mixtures of *n*-Hexane and 1-Hexanol from 303 to 423 K up to 50 MPa

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Experimental results for the density and viscosity of *n*-hexane + 1-hexanol mixtures are reported at temperatures from 303 to 423 K and pressures up to 50 MPa. The binary mixture was studied at three compositions, and measurements on pure 1-hexanol are also reported. The two properties were measured simultaneously using a single vibrating-wire sensor. The present results for density have a precision of ±0.07% and an estimated uncertainty of ±0.3%. The viscosity measurements have a precision of ±1% and an estimated uncertainty of ±4%. Representations of the density and viscosity of the mixture as a function of temperature and pressure are proposed using correlation schemes.

KEY WORDS: density; 1-hexanol; high pressure; liquid; mixture; *n*-hexane; vibrating-wire sensor; viscosity.

1. INTRODUCTION

Mixtures of alkanes with alkanols are interesting for a number of scientific reasons, mostly connected to the formulation and testing of models to predict the properties of associating fluids, since the binary systems under study are composed of a non-associating molecule, the alkane, and an associating one, the alcohol. Thus, the behavior of the {*n*-hexane + 1-hexanol} mixture in terms of viscosity is asymmetric because of the different types of interaction between the component molecules. For example, at ambient temperature and atmospheric pressure, 1-hexanol is about one order of magnitude more viscous than *n*-hexane [1, 2]. However, for the volumetric properties, this mixture does not show large deviations from

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ideality. The excess volume at room temperature is smaller than $-0.3\ \mathrm{cm^3 \cdot mol^{-1}}$ [3]. In view of its behavior in terms of the two properties mentioned, this binary system may provide a good test for viscosity correlation models.

Experimental thermodynamic studies on binary mixtures of *n*-hexane and 1-hexanol have frequently been reported in the literature, most of them involving the volumetric properties [1–5]. But all the studies we know of were carried out at atmospheric pressure and in narrow ranges of temperature (298 to 323 K).

At high pressure and over wider temperature ranges, the density and viscosity are known for only the pure components. Pure *n*-hexane is well characterized in terms of its density [6, 7] and viscosity [8, 9]. For 1-hexanol at high pressure, three sets of measurements of density [10–12] and one set of measurements of viscosity [13] were found in the literature. One of our objectives is to complete the description of the density and viscosity of the mixtures of *n*-hexane and 1-hexanol by reporting original measurements up to high pressure over a wide temperature range.

2. EXPERIMENTAL METHOD

The density and viscosity of the fluids studied were measured simultaneously using the vibrating-wire method. The technique employed was described in detail in previous publications [14–19]. It is based on a vibrating-wire sensor, which is composed of a sinker suspended from a thin metallic wire, both of which are immersed in the sample of fluid. The oscillation characteristics of the wire are obtained by means of an electromagnetic coupling [18]. The response of the wire is related to the density of the fluid mainly through the buoyancy force acting upon the sinker, which changes the tension of the wire. The viscosity of the fluid affects the damping of the oscillations. The technique is based on a rigorous theoretical framework [14, 18, 20, 21] and thus, once all of the sensor parameters are known, either by independent means or from a single reference experiment, no extensive calibration procedures are required for measurements at high pressure or at temperatures away from ambient conditions. The particular instrument used in the present work has been validated by a series of measurements on pure liquids, published previously [19].

Pressure was measured with a TransInstruments manometer (BHL-4260-10) calibrated against a dead-weight gauge (Ruska Model 2435-930D) in our laboratory. The output signal of the manometer is read with a $6\frac{1}{2}$ digit multimeter (Keithley Model 2000). The pressure uncertainty is $\pm 0.1\%$ of the full scale. The temperature in the high-pressure vessel, which is surrounded by an isothermal shield in the interior of a vacuum chamber, is controlled to better than ±0.002 K. Two platinum resistance thermometers are used to measure the temperature of the pressure vessel at two different places. Three other thermometers monitor the temperature of the isothermal shield. One of the thermometers attached to the pressure vessel is read in the four-wire mode and was calibrated against a triple point of water cell, a cell of benzoic acid melting point (395.49 K), and against a secondary standard platinum resistance thermometer in the interval between the fixed points. All calibrations used facilities available in our laboratory. The resistances of the thermometers were measured using the same multimeter specified above. The maximum uncertainty in temperature is estimated to be ±0.02 K.

Pure 1-hexanol and *n*-hexane were obtained from Fluka, purities of 99 and 99.5% in mass, respectively. The major impurity reported in *n*-hexane is water ($\leqslant 0.05\%$), and no additional information is given by Fluka concerning the impurities present in 1-hexanol. Both liquids were used without further purification, but the initial purities were checked by gas chromatography. The mixtures were prepared gravimetrically into a gas-tight syringe and introduced in the apparatus under vacuum. Our compositions are precise within ±0.001 in mole fraction. After each series of measurements on pure 1-hexanol as well as for the three mixtures, chromatographic analyses were performed to verify the initial compositions. These were confirmed to within the estimated uncertainty, and no impurities were detected.

3. RESULTS

Measurements were carried out along five isotherms, from atmospheric pressure up to 50 MPa. The experimental results are collected in Table I. Subscript 1 in the mole fractions refers to *n*-hexane.

The present values of density were correlated using the Tait equation, which represents the density as a function of pressure along each isotherm by two parameters, B and C.

$$\frac{\rho-\rho_0}{\rho}=C\log_{10}\frac{B+p}{B+p_0} \tag{1}$$

For the *n*-alkane family, parameter C usually takes values between 0.20 and 0.21 [6, 22] and can be assumed to be constant. For the present application, we took $C=0.200$ for all compositions. Parameter B depends on temperature as well as on composition. For each isotherm an anchor point (ρ_0, p_0) must also be given, since the Tait equation describes the compressibility. We have chosen as starting points for our correlated

Table I. Experimental Density and Viscosity of *n*-Hexane(1) + 1-Hexanol(2)

p (MPa)	ρ ($kg \cdot m^{-3}$)	η ($mPa \cdot s$)	p (MPa)	ρ ($kg \cdot m^{-3}$)	η ($mPa \cdot s$)
		$x_1 = 0.000$			
	$T = 303.15$ K			$T = 323.15$ K	
0.35	810.5	4.042	0.17	797.0	2.221
4.97	813.0	4.306	5.03	801.0	2.334
9.99	816.1	4.565	9.89	804.4	2.440
15.07	819.7	4.809	14.97	806.3	2.606
20.12	821.8	5.098	20.05	809.5	2.753
30.24	828.4	5.679	29.93	815.7	3.006
40.07	836.2	6.169	39.86	819.6	3.333
50.27	843.0	6.730	50.01	823.6	3.737
	$T = 353.15$ K			$T = 383.15$ K	
0.19	775.1	1.142	0.15	749.9	0.654
5.00	779.6	1.190	4.96	755.1	0.696
10.12	783.5	1.257	10.04	760.6	0.732
15.00	787.2	1.319	14.98	765.5	0.765
19.91	791.2	1.372	20.02	769.8	0.798
29.97	798.0	1.498	30.05	778.4	0.866
39.77	804.2	1.622	40.11	786.1	0.932
50.20	809.8	1.774	49.90	792.6	1.005
	$T = 423.15$ K				
0.21	715.0	0.359			
5.06	722.0	0.381			
10.00	728.4	0.400			
14.90	734.0	0.421			
20.11	739.7	0.442			
30.10	749.1	0.488			
40.31	757.8	0.530			
50.18	765.4	0.581			
		$x_1 = 0.250$			
	$T = 303.15$ K			$T = 323.15$ K	
0.09	773.2	1.906	0.10	757.9	1.211
4.88	777.1	1.990	5.06	762.1	1.277
10.15	781.3	2.101	9.93	765.9	1.344
15.27	784.4	2.211	14.93	770.1	1.405
20.53	789.8	2.304	20.35	774.5	1.474
30.08	792.7	2.620	30.28	782.7	1.604
40.08	799.6	2.842	40.19	788.8	1.734
49.91	806.3	3.051	50.32	794.2	1.893

Table I. *(Continued)*

p (MPa)	ρ ($\mathrm{kg \cdot m^{-3}}$)	η ($\mathrm{mPa \cdot s}$)	p (MPa)	ρ ($\mathrm{kg \cdot m^{-3}}$)	η ($\mathrm{mPa \cdot s}$)
		$x_1 = 0.250$			
	$T = 353.15$ K			$T = 383.15$ K	
0.49	732.9	0.689	0.51	707.6	0.429
4.95	737.4	0.731	4.96	713.2	0.452
9.89	742.6	0.773	10.02	719.1	0.478
14.81	747.4	0.814	15.05	724.4	0.503
19.87	752.2	0.855	20.18	729.5	0.527
29.98	760.7	0.926	29.98	737.9	0.582
40.03	768.2	1.000	39.97	746.0	0.644
49.85	777.6	1.066	50.30	754.0	0.700
	$T = 423.15$ K				
0.54	668.7	0.260			
5.11	676.9	0.279			
10.05	685.0	0.295			
14.69	691.7	0.312			
20.00	699.0	0.327			
30.10	710.2	0.360			
39.73	719.3	0.395			
50.01	728.8	0.426			
		$x_1 = 0.399$			
	$T = 303.15$ K			$T = 323.15$ K	
0.12	747.2	1.127	0.13	731.7	0.766
5.05	750.9	1.188	4.95	736.3	0.809
10.05	754.4	1.262	9.97	740.8	0.849
14.91	757.5	1.334	14.93	745.0	0.890
19.84	760.9	1.404	20.20	749.1	0.938
30.01	766.7	1.579	30.14	755.7	1.034
40.03	772.6	1.750	40.01	762.5	1.123
50.63	778.6	1.926	49.94	767.8	1.232
	$T = 353.15$ K			$T = 383.15$ K	
0.13	705.5	0.477	0.54	678.3	0.323
4.99	711.0	0.512	4.93	684.8	0.342
9.89	716.3	0.543	9.94	691.6	0.362
14.90	721.9	0.570	14.99	697.6	0.382
20.01	726.9	0.595	20.00	703.4	0.400
30.21	735.6	0.651	30.10	713.1	0.442
39.96	743.3	0.702	40.08	721.4	0.489
50.08	750.6	0.758	49.98	729.8	0.531

Table I. *(Continued)*

p (MPa)	ρ ($kg \cdot m^{-3}$)	η ($mPa \cdot s$)	p (MPa)	ρ ($kg \cdot m^{-3}$)	η ($mPa \cdot s$)
		$x_1 = 0.399$			
	$T = 423.15$ K				
0.76	637.7	0.202			
5.02	646.5	0.218			
9.99	655.4	0.235			
15.02	663.7	0.251			
20.13	671.2	0.267			
29.92	683.9	0.292			
40.00	694.6	0.321			
50.48	704.4	0.350			
		$x_1 = 0.599$			
	$T = 303.15$ K			$T = 323.15$ K	
0.27	713.7	0.639	0.51	697.3	0.484
4.88	717.8	0.675	5.07	701.9	0.510
10.02	722.2	0.713	10.00	707.0	0.535
15.10	726.1	0.753	15.07	711.6	0.566
20.21	729.8	0.798	20.05	716.0	0.592
30.24	736.4	0.877	30.19	724.3	0.645
40.42	746.8	0.930	40.20	731.5	0.707
49.69	751.8	1.014	50.36	738.4	0.761
	$T = 353.15$ K			$T = 383.15$ K	
0.53	670.6	0.326	0.55	641.9	0.233
5.12	676.5	0.346	4.88	649.0	0.249
9.95	682.4	0.366	9.74	656.4	0.265
14.83	687.5	0.389	14.98	663.6	0.282
20.07	692.9	0.413	20.17	670.2	0.297
30.15	702.7	0.457	29.94	681.0	0.327
40.12	711.5	0.493	40.15	690.6	0.358
50.15	719.4	0.527	50.42	699.1	0.389
	$T = 423.15$ K				
0.88	599.4	0.155			
5.03	610.0	0.168			
9.94	620.4	0.182			
14.78	629.4	0.195			
19.88	637.8	0.208			
29.91	652.2	0.232			
39.94	664.2	0.253			
50.04	674.4	0.277			

Table II. Coefficients of the Tait Equation

T (K)	B (MPa)	σ_ρ (%)	B (MPa)	σ_ρ (%)	B (MPa)	σ_ρ (%)	B (MPa)	σ_ρ (%)
	$x_1 = 0.000$		$x_1 = 0.250$		$x_1 = 0.399$		$x_1 = 0.599$	
303.15	96.181	0.17	84.334	0.12	85.851	0.02	63.944	0.15
323.15	104.852	0.11	71.379	0.07	67.508	0.06	54.964	0.01
353.15	76.550	0.04	54.623	0.10	49.980	0.03	42.000	0.05
383.15	57.297	0.02	47.790	0.03	38.820	0.05	31.017	0.03
423.15	43.298	0.06	30.386	0.06	24.276	0.03	18.012	0.02
σ_ρ (%)		0.09		0.08		0.04		0.07

isotherms the densities measured at the lowest pressure for each temperature, taken from Table I. The correlation using Eq. (1) with values for the parameters given in Table II describes our entire set of experimental densities with an overall standard deviation of ±0.07%. Partial standard deviations taken for each temperature and composition are also given in Table II. A monotonic behavior of the parameter B with temperature is observed in all cases except for pure 1-hexanol at the lowest two isotherms. This anomaly is interpreted as a consequence of the reduced sensitivity of the instrument in this region, due to the high viscosity of the alcohol. It coincides with the largest standard deviations obtained in the fits to the isotherms.

For pure 1-hexanol at high pressure, comparisons with literature values of density are possible. In Fig. 1 we show a deviation plot of the present values, and of experimental data from several references, from the density correlation proposed by Cibulka and Ziková [23]. The maximum deviations observed are between −0.4 and +0.2%, which is comparable with the mutual uncertainties.

Comparisons of our densities for the binary system with literature values are possible only at atmospheric pressure, since no data were found in the literature for the density of the mixture at high pressure. In Fig. 2 we show a deviation plot of the present densities as a function of composition. The baseline is given by interpolation between the pure-substance densities as calculated from the correlations of Cibulka and co-workers [7, 23], neglecting the volume of mixing, i.e., assuming $V^E = 0$. Two sets of experimental data from the literature are also shown. According to the literature sources, the volume of mixing attains a maximum of about $-0.3\ \mathrm{cm}^3 \cdot \mathrm{mol}^{-1}$ in the temperature range 288 to 323 K, at compositions

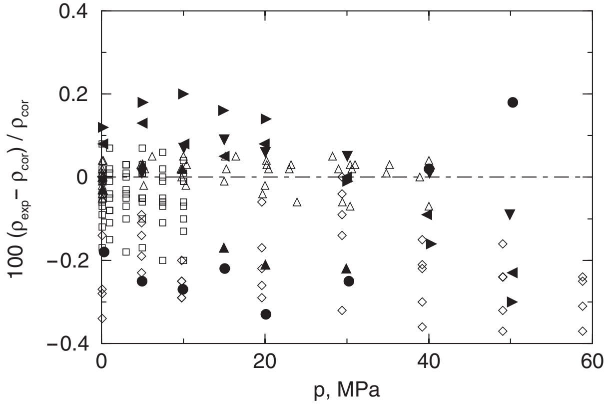

Fig. 1. Deviation plot of density of pure 1-hexanol. The baseline represents the correlation of Cibulka and Ziková [23]. (●) 303.15 K, (▲) 323.15 K, (◄) 353.15 K, (▼) 383.15 K, (►) 423.15 K. Three sets of experimental points from the literature are also shown: (□) Garg et al. [11], (◇) Gylmanov et al. [10], and (△) Matsuo and Makita [12].

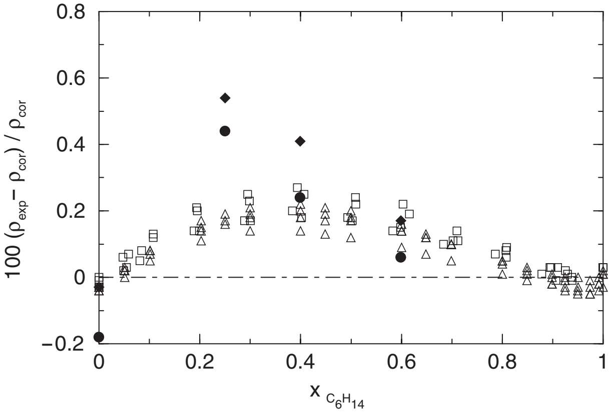

Fig. 2. Deviation plot for density of the binary system $\{x$ n-hexane $+ (1-x)$ 1-hexanol$\}$ at atmospheric pressure. The baseline represents the interpolation between the pure-substance densities as calculated from the correlations of Cibulka and co-workers [7, 23], neglecting the volume of mixing, i.e., assuming $V^E = 0$. Present results are shown at the lowest pressures: (●) 303.15 K, (♦) 323.15 K. Two sets of data from the literature at atmospheric pressure are also shown: (□) Cominges et al. [5] and (△) Heintz et al. [3].

around $x_1 = 0.4$. This behavior is evident in Fig. 2. Our results at the composition of $x_1 = 0.250$ exhibit somewhat larger deviations than at the remaining compositions, but globally the agreement of the present results with those of the literature lies within $\pm 0.2\%$.

From comparisons of the density of 1-hexanol at high pressure and of the mixture at atmospheric pressure, we conclude that the uncertainty of the present measurements is $\pm 0.3\%$. This value is slightly above the uncertainty claimed for previous measurements on *n*-pentane [19], which was $\pm 0.2\%$. We attribute this increase in uncertainty to the high viscosity of 1-hexanol at the lowest temperatures and highest pressures of the present study, which affects the sensitivity of the instrument.

The viscosity of the liquid mixtures was correlated using a model based on the theory of transport for the hard-sphere fluid. Assael and Dymond [8, 9] proposed a generalized framework that renders this model applicable to the *n*-alkane family for the purpose of predicting the three transport coefficients: viscosity, thermal conductivity, and self-diffusion coefficient. The model has also been applied to *n*-alkanols [9, 24]. According to this model a reduced, dimensionless viscosity, η^*, is defined as

$$\eta^* = 6.035 \times 10^8 \frac{V^{2/3}}{(MRT)^{1/2}} R_\eta \eta \tag{2}$$

where M is the molar mass, R is the gas constant, T is the temperature, and V is the molar volume. The parameter R_η normally has a fixed value for each substance. For *n*-alkanes this parameter is state-independent and shows a smooth behavior along the homologous series [8, 9]. For alkanols up to 1-pentanol, Assael and Dymond [24] obtained a better representation of viscosity if R_η is also temperature dependent. For 1-hexanol and higher alcohols, this parameter was held fixed. Here we considered R_η to be independent of the thermodynamic state and adjusted its value for each composition only.

The reduced viscosity defined in Eq. (2) should be a function of the ratio of the molar volume to a temperature-dependent characteristic volume, $V_0(T)$, only. This characteristic volume generally decreases with increasing temperature, which can be interpreted as accounting for the finite steepness of the repulsive part of the intermolecular potential. A universal curve of reduced viscosity as a function of density and characteristic volume has been established for a large number of substances:

$$\ln \eta^* = \sum_{i=0}^{7} a_i \left(\frac{V_0(T)}{V}\right)^i \tag{3}$$

Table III. Coefficients for the Viscosity Correlation

x_1	R_η	$b_0 \times 10^4$ ($\mathrm{m^3 \cdot mol^{-1}}$)	$b_1 \times 10^8$ ($\mathrm{m^3 \cdot mol^{-1} \cdot K^{-1}}$)	σ_η(%)
0.0000	1.62	1.2885	−10.970	3.3
0.2501	1.61	1.2203	−10.004	3.8
0.3993	1.33	1.1683	−8.791	2.3
0.5991	1.19	1.0793	−6.809	2.4

We have therefore correlated our results of viscosity by superimposing our isotherms on this universal curve defined by the values for the coefficients a_i given by Dymond and Assael [8, 9]. The superposition process yields values for $V_0(x_1, T)$ and for $R_\eta(x_1)$. A linear temperature dependence of the characteristic volumes yields a correct fitting of our viscosities, and we adopted this simple scheme, although a fit using quadratic functions would improve slightly the standard deviations presented.

$$V_0(T) = b_0 + b_1 T \tag{4}$$

The values obtained for the parameters R_η, b_0, and b_1, together with the standard deviations for the correlations of the present viscosity results, lumped for each composition, are given in Table III. A deviation plot of the present measurements from their correlation is shown in Fig. 3.

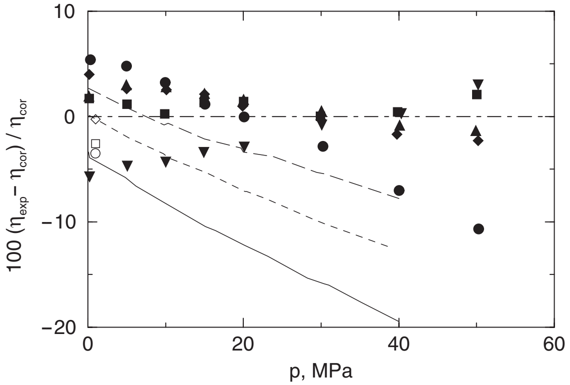

Fig. 3. Viscosity of 1-hexanol. Filled symbols represent our results: (●) 303.15 K, (■) 323.15 K, (♦) 353.15 K, (▲) 383.15 K, (▼) 423.15 K. Open symbols correspond to the experimental data of Chowdhury et al. [2]: (○) 303.15 K, (□) 313.15 K, (◇) 323.15 K. The lines correspond to the correlation of Matsuo and Makita [13]: (—) 298.15 K, (---) 323.15 K, (– – –) 348.15 K. The baseline represents the correlation of Assael and Dymond [8, 9].

The standard deviations reported in Table III are not identical to the precision of our measurements, which is close to ±1%. This is because the correlation scheme of Dymond and Assael imposes a number of constraints, such as the form of the universal curve and the parameter R_η independent of the thermodynamic state that restrict the flexibility of the model as a regression tool. The standard deviations shown in Table III are related to the agreement of the present data with the model and are similar to the deviations achieved when correlating other data for alkanols [24], which range from ±2.5 to ±5%.

From the composition dependence of the coefficients in Table III, and keeping in mind that for pure *n*-hexane the parameter $R_\eta = 1.18$ [8], it can be inferred that interpolation with composition is possible for the purpose of estimating the viscosity of the mixture. Such interpolations would produce estimates with an error of the order ±5% or slightly higher.

At high pressure, our measurements of viscosity of pure 1-hexanol can be compared with those of Matsuo and Makita [13], measured using a falling-body viscometer, and for which an uncertainty of ±5% is claimed. A deviation plot of the correlation equations proposed by these authors, from the correlation of the present data is also included in Fig. 3. The values of density used when applying Assael and Dymond's scheme to calculate the viscosity at the state points given by Matsuo and Makita [13] were those reported by these last authors in another publication [12], up to a maximum pressure of 40 MPa. When the results of these authors are compared to those from this work, systematic trends with pressure and temperature are evident; although both sets of measurement agree well at low pressure, at 40 MPa and at 298 K, deviations between the two sets of experimental points reach 10%, which is the limit of the mutual uncertainties. While it is true that the precision of our measurements is, at its worse, for pure 1-hexanol at the points of higher viscosity (low temperature, high pressure), we do not find a reason to attribute an uncertainty larger than 4% to our points. Previous experience using the vibrating-wire technique shows that, for viscosities of the order of 1 MPa · s, the vibrating-wire instrument yields an uncertainty of 2.5% [19]. Due to the simultaneous nature of the measurements of density and viscosity, consistency between the results for the two properties is enforced and an experimental anomaly in one should be reflected on the other property. Or in the present case, no systematic trend is apparent in our results of density of 1-hexanol up to high pressure, as can be concluded from Fig. 1.

Concerning the viscosity of the binary mixture at high pressure, no other data were found in the literature. Comparisons are restricted to the region close to atmospheric pressure with two sets of results [1, 2], as plotted in Fig. 4. The data published by Franjo et al. [1] at 298.15 K

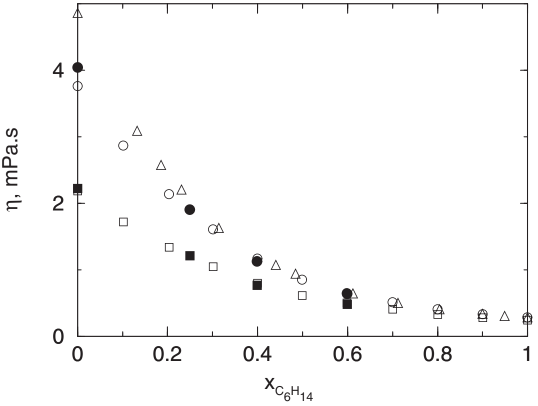

Fig. 4. Comparison of the viscosity of the binary system $\{x$ *n*-hexane$+(1-x)$ 1-hexanol$\}$. Present results at the lowest pressures: (●) 303.15 K; (■) 323.15 K. Values from the literature at atmospheric pressure: Franjo et al. [1] (△) 298.15 K; Chowdhury et al. [2] (○) 303.15 K, (□) 323.15 K.

cannot be compared directly with ours, since the temperatures do not coincide, but from Fig. 4 the two sets appear to be consistent. An immediate comparison is possible between some of our results and some from Chowdhuri et al. [2], at 303.15 and 323.15 K. Looking at these two temperatures for all compositions, only for pure 1-hexanol at 303.15 K does there seem to be a noticeable discrepancy, good agreement being observed for all other points. Based also on this comparison, we reiterate the estimation of ±4% for the uncertainty of the present viscosities.

4. CONCLUSION

The density and viscosity of pure 1-hexanol and of three binary liquid mixtures of *n*-hexane with 1-hexanol were studied as a function of temperature and pressure. The two properties were measured simultaneously for each composition, at five temperatures, with eight pressure points along each isotherm. For the mixture at pressures above atmospheric and temperatures above 323.15 K, the present data are the first as far as we could find in the literature.

The liquid system $\{n$-hexane$+$1-hexanol$\}$ shows small deviations from ideality in volumetric properties, but a marked behavior in terms of viscosity. It does not follow a simple Grunberg-Nissan mixing rule, showing deviations which are asymmetric relative to the equimolar composition, as

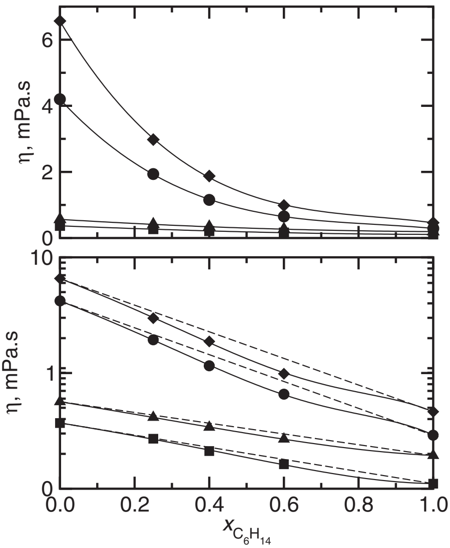

Fig. 5. Viscosity of the binary system $\{x$ *n*-hexane $+ (1-x)$ 1-hexanol$\}$. Only our experimental data at the lowest and highest temperature are presented, at two pressures of 5 and 50 MPa: (●) 303.15 K, 5 MPa, (◆) 303.15 K, 50 MPa, (■) 423.15 K, 5 MPa, (▲) 423.15 K, 50 MPa. The lower semilogarithmic plot shows the deviations from the relation $\ln\eta = x_1 \ln\eta_1 + x_2 \ln\eta_2$.

can be seen in Fig. 5. The correlation of the viscosity of the mixture using the model proposed by Assael and Dymond can be considered only as a partial success, since the representation of this property is not yet at the level of the experimental uncertainty.

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