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Density and Viscosity Measurements of Diethyl Adipate from (303 to 373) K and up to 20 MPa

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Density and viscosity results for diethyl adipate (DEA) along eight isotherms from (303.15 to 373.15) K and at pressures up to 20 MPa are presented. The measurements of both properties were performed simultaneously using a vibrating-wire instrument operated in the forced mode of oscillation. The overall uncertainties of these results are ± 2.0 % in viscosity and ± 0.2 % in density. The data obtained were correlated as functions of pressure and temperature. The maximum absolute deviation and the average absolute deviation of the measurement data from the correlation for viscosity are 1.2 % and 0.4 %, respectively, and for density are 0.06 % and 0.016 %.

Introduction

Automobiles are one of the major sources of air pollutants, producing carbon monoxide (CO), nitrogen oxides (NO_x), particulate matters (PM), and other harmful gases. However, it is difficult to simultaneously reduce NO_x and smoke in the traditional diesel engine. Practically, adding some oxygenated compounds into diesel fuel to reduce engine emissions without modifying the engine design seems to be more attractive. Diethyl adipate ($C_2H_5O(C{=}O)(CH_2)_4(C{=}O)OC_2H_5$) has a relatively high oxygen content and better intersolubility with diesel fuel at normal temperature and pressure. Thus, it is regarded as a promising oxygenated additive or potential alternative fuel for the future.[1,2] Unfortunately, only four literatures involved in the density and viscosity of diethyl adipate were found.[3–6] These studies were limited to a small temperature interval at $p = 0.1$ MPa. Until recently, the density of diethyl adipate was measured over a wide temperature range from (293.15 to 403.15) K and up to 140 MPa with a vibrating tube densimeter.[7] The pressure dependence of viscosity of this fluid was never studied. Therefore, the present work provides measurements of density and viscosity for diethyl adipate at pressures up to 20 MPa and over temperatures ranging from (303.15 to 373.15) K, attempting to fill this gap and help in the development of new clean alternative fuels.

Measurement Method and Equipment

Working Equations. The apparatus employed in this work was a vibrating-wire instrument measuring simultaneously the density and viscosity. The first vibrating-wire viscometer based on the Navier–Stokes equations was developed in 1963 by Tough et al.,[8] and the theory was improved in 1986 by Retsina.[9,10] Since then, many practical devices have been developed.[11–22] A comprehensive review of the literature on the vibrating wire instrument was reported by Kandil et al.[21] The instrument is composed of a thin metallic wire clamped at the upper end with a fixed support and a sinker suspended from the metallic wire, both immersed in fluid. The wire is placed in a magnetic field, and it is driven in steady state transverse oscillations by passing an alternating current. Our implementation was similar to that described by Audonnet et al.[18] and Pádua et al.;[22] therefore, only a brief description of the working equations was given here. The velocity of the wire in the fundamental mode of transverse oscillation is given by

$$\mathbf{u} = \frac{\partial \mathbf{y}}{\partial t} = \frac{F(z)e^{i\omega t}}{m_s[\omega(\beta' + 2\Delta_0) + i\omega(1 + \beta) + \omega_0^2/i\omega]} \tag{1}$$

Equation 1 can be rearranged into the form $\mathbf{u} = ue^{i(\omega t - \varphi)}$, the amplitude and phase of the complex velocity being given by

$$u = \frac{F}{m_s\{\omega^2(\beta' + 2\Delta_0)^2 + [\omega(1 + \beta) - \omega_0^2/\omega]^2\}^{1/2}} \tag{2}$$

$$\varphi = \tan^{-1}\frac{\omega^2(1 + \beta) - \omega_0^2}{\omega^2(\beta' + 2\Delta_0)} \tag{3}$$

where F describes the amplitude of the driving force per unit length, and the quantity $m_s = \rho_s \pi R^2$ is the mass per unit length of a wire with a circular cross-section of radius R having density ρ_s. The ω is angular frequency; ω_0 is natural frequency; Δ_0 is the internal damping coefficient of the wire; and β, β' are related to the density ρ and viscosity η of the fluid by the hydrodynamic theory.[18]

The characteristics of oscillation of the wire are obtained by means of an electromagnetic coupling.[23] Amplitude and phase data were collected in a range of about $f_r \pm 5g$, where g is the half-width of the resonance curve and f_r is the fundamental transverse resonance frequency. The density and viscosity of the fluid were determined by a directly simultaneous fit of the working equations to the entire curves of amplitude and phase.[18]

Apparatus. A vibrating-wire instrument was developed by our group recently for simultaneous measurements of the density and viscosity of liquid. The vibrating-wire assembly was shown in Figure 1. The nominal radius of the wire used in this experiment was about 0.05 mm, obtained from Goodfellow (Cambridge, U.K.) with a mass fraction purity greater than 99.95 %. The upper end of the wire was kept in place by a chuck, insulated from the support plate by PTFE washers. An aluminum

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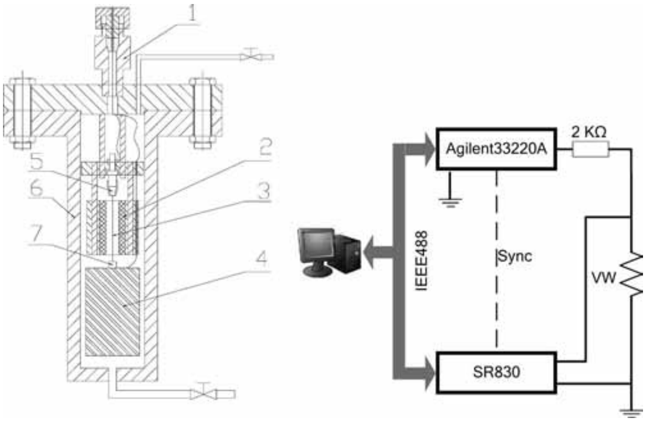

Figure 1. Diagram of the vibrating-wire apparatus. (1) Feedthrough; (2) Magnets; (3) Tungsten wire; (4) Weight; (5) Upper clamp; (6) Pressure vessel; (7) Bottom clamp.

weight attached to its lower end kept the wire under tension in the vertical position. A pair of samarium−cobalt magnets (supplied by Xi'an Siqiang Technology Corporation, China) was mounted onto a cage. The magnetic induction in the center of the gap between the magnetic poles, where the wire was located, was about 0.3 T. The cell was mounted inside a custom-made stainless steel vessel. The electrical connections of the vibrating wire were made using four copper leads, two for each end of the wire, which passed through the cap of the vessel by means of a custom-made feedthrough.

A sinusoidal voltage generated by a function generator (Agilent 33220A) was passed through a resistance of about 2 kΩ connected in series to the tungsten wire. The amplitude and phase of the signal were detected with the lock-in amplifier (Stanford SR830) over a frequency range. Such a typical resonance curve was shown in Figure 2 together with calculations using the theoretical equations.

The apparatus was immersed completely in a thermostatic bath (Fluke 7037). The temperature stability and uniformity of the bath were better than ± 2 mK. The total uncertainty of temperature was less than ± 10 mK. Pressure was measured, in the range of (0 to 20) MPa, with a transducer (Micro Sensor Co., Ltd., model MPM480, with a maximum operating pressure of 40 MPa). The accuracy of the transducer was ± 0.25 % FS, and the uncertainty of the pressure was ± 0.1 MPa.

Experimental Section

Fluid Samples. The toluene was supplied by TEDIA Company, USA (minimum mass fraction purity of 99.8 %), while diethyl adipate was provided by Sinopharm Chemical Reagent Co., Ltd., China (nominal mass fraction purity of 99.0 %). The fluid samples were introduced in the measuring cell without any further treatment through a reciprocating plunger pulsation-free pump (2PB05, Beijing Xingda Technology and Development Corporation).

Calibration and Testing. The vibrating-wire instrument, in principle, requires no calibration. However, some physical properties of the wire and weight cannot be determined to sufficient accuracy by independent methods.[22] In practice, those properties are usually determined by calibration. To do this, calibration of the apparatus was performed at 298.15 K by means of measurements in vacuum and toluene at 0.1 MPa. The density and viscosity reference data for toluene were taken from Assael et al.[24] The parameters of the cell were given in Table 1. To test the apparatus, a set of data listed in Table 2 were measured. Taking all factors into account, the uncertainties of the measurements were estimated to be ± 2.0 % in viscosity and ± 0.2 % in density.

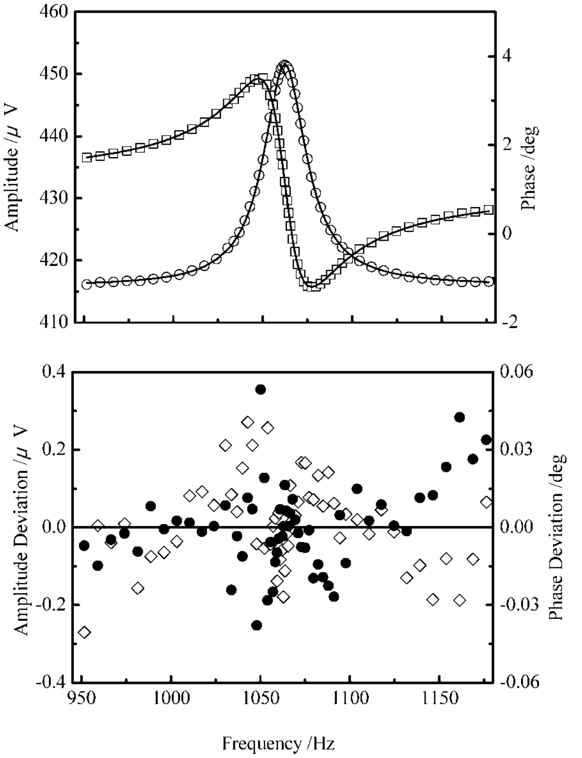

Figure 2. Experimental and theoretical resonance curves of amplitude and phase for toluene at 298.15 K and 0.1 MPa. Solid line, theory; ○, experimental amplitude; □, experimental phase; ◇, amplitude deviation between experiment and theory; ●, phase deviation between experiment and theory.

Table 1. Parameters of the Cell

			source
Vibrating Wire (Tungsten)			
Half-length	L/m	$24.55 \cdot 10^{-3}$	measured
Radius	R/m	$49.693 \cdot 10^{-6}$	calibrated
Density	ρ_s/kg·m^{-3}	$19.230 \cdot 10^{3}$	literature
Young's modulus	E/GPa	406	literature
Internal damping	Δ_0	$1.3082 \cdot 10^{-5}$	measured
Aluminum Mass			
Mass	M_w/kg	0.23244	measured
Density	ρ_w/kg·m^{-3}	2731.43	calibrated

Results and Discussion

Measurements of density and viscosity of diethyl adipate were carried out along eight isotherms from (303.15 to 373.15) K at pressures up to 20 MPa, and the results are listed in Table 3. All the experimental results were correlated with a Tait-type equation for density and a hard-sphere scheme for viscosity. To assess the performances of correlations, the average absolute deviation (AAD), maximum deviation (DM), and average deviation (Bias) are introduced.

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

$$\frac{\rho - \rho_0}{\rho} = C \log\left[\frac{B + p}{B + p_0}\right] \qquad (4)$$

where ρ_0, the density at the reference pressure ($p_0 = 0.1$ MPa), is given by $\rho_0 = A_0 + A_1T + A_2T^2 + A_3T^3$. The B is a quadratic

Table 2. Density ρ and Viscosity η of Toluene Measured with the Vibrating-Wire Sensor as a Function of Temperature T and Pressure p

T/K	p/MPa	ρ/kg·m^{-3}	η/mPa·s	100 $\Delta\rho/\rho^b$	100 $\Delta\eta/\eta^b$
298.15	0.10	862.500	0.555	0.00	0.00[a]
	2.30	864.389	0.569	−0.01	−0.59
	4.40	865.888	0.580	0.00	−0.91
	7.43	868.038	0.593	0.02	−0.77
	10.40	870.540	0.597	−0.02	0.89
	12.47	871.736	0.613	0.01	−0.21
	14.49	873.496	0.617	−0.03	0.59
323.15	0.12	840.467	0.425	−0.15	−0.73
	3.34	843.459	0.431	−0.16	0.39
	6.43	846.144	0.448	−0.16	−0.85
	9.60	848.793	0.461	−0.16	−1.28
	12.75	851.586	0.464	−0.18	0.42
	14.77	853.276	0.466	−0.19	1.50

[a] Calibration point. [b] $\Delta\rho/\rho = \{\rho(\text{calcd}) - \rho(\text{exptl})\}/\rho(\text{calcd})$, $\Delta\eta/\eta = \{\eta(\text{calcd}) - \eta(\text{exptl})\}/\eta(\text{calcd})$; ρ(calcd) and η(calcd) were calculated from the correlation of Assael et al.[24]

Table 3. Experimental Density ρ and Viscosity η of DEA Determined with the Vibrating-Wire Sensor as a Function of Temperature T and Pressure p

T/K	p/MPa	ρ/kg·m^{-3}	η/mPa·s
303.15	0.10	998.842	2.786
	4.52	1001.807	2.909
	9.61	1005.240	3.061
	14.70	1008.513	3.226
	19.79	1012.523	3.343
313.15	0.10	989.860	2.257
	4.52	993.535	2.335
	9.63	996.693	2.472
	14.75	1000.420	2.589
	19.89	1003.553	2.726
323.15	0.10	981.395	1.846
	4.52	984.720	1.932
	9.60	988.740	2.021
	14.69	992.273	2.126
	19.71	995.581	2.230
333.15	0.10	971.597	1.580
	4.52	975.516	1.642
	9.63	979.685	1.716
	14.71	983.483	1.802
	19.71	987.107	1.886
343.15	0.10	962.622	1.350
	4.51	966.544	1.407
	9.60	970.932	1.471
	14.70	975.031	1.542
	19.70	978.932	1.608
353.15	0.10	953.224	1.179
	4.52	957.353	1.227
	9.62	962.118	1.280
	14.71	966.479	1.338
	19.68	970.392	1.402
363.15	0.10	943.820	1.041
	4.52	948.203	1.078
	9.61	953.106	1.129
	14.70	957.919	1.174
	19.71	962.125	1.226
373.15	0.10	934.753	0.914
	4.52	939.138	0.956
	9.62	944.359	1.000
	14.68	948.986	1.046
	19.91	953.741	1.096

Table 4. Parameters for the Density Correlation of Equation 4

parameter	value	parameter	value
A_0	684.585	C	0.18
A_1	4.250	B_0	552.737
A_2	$-1.496\cdot10^{-2}$	B_1	−2.162
A_3	$1.439\cdot10^{-5}$	B_2	$2.312\cdot10^{-3}$
AAD/%	0.016		
DM/%	0.061		
Bias/%	−0.0001		

Table 5. Parameters for the Viscosity Correlation of Equations 5 to 7

T/K	10^6 V_0/m^3·mol^{-1}	parameter	value
303.15	148.09	a	212.752
313.15	146.82	b	−0.305225
323.15	145.60	c	$3.0256\cdot10^{-4}$
333.15	144.66	R_η	1.95
343.15	143.66	AAD/%	0.41
353.15	142.77	DM/%	1.17
363.15	141.85	Bias/%	−0.006
373.15	140.92		

function of temperature, with the form of $B = B_0 + B_1T + B_2T^2$, and the parameter C can usually be assumed to be constant, with the best-fit value of 0.18 found for diethyl adipate. The AAD, DM, and Bias, obtained from the Tait-type correlation of diethyl adipate, are listed in Table 4 together with the values of the parameters.

The viscosity of diethyl adipate was correlated using a model based on the theory of transport on the hard-sphere fluid. Assael and Dymond[25,26] proposed a generalized framework for the simultaneous correlation of self-diffusion, viscosity, and thermal conductivity of dense fluids. This correlation uses a reduced dimensionless viscosity, η^*, which, using base SI units, is defined by

$$\eta^* = 6.035\cdot10^8\left[\frac{1}{MRT}\right]^{1/2}\frac{\eta V^{2/3}}{R_\eta} \tag{5}$$

where η is the viscosity; M is the molar mass; T is the temperature; V is the molar volume; and R is the gas constant. The proportionality factor, R_η, accounts for molecular roughness and departure from molecular sphericity, independent of pressure and temperature. The reduced viscosity should be a function of the ratio of the molar volume to a temperature-dependent characteristic volume, V_0, only. This characteristic volume generally decreases linearly with increasing temperature. A universal curve of reduced viscosity as a function of density and characteristic volume has been established for a large number of substances

$$\log(\eta^*) = \sum_{i=0}^{7} a_i\left(\frac{V_0}{V}\right)^i \tag{6}$$

We have, therefore, correlated our results of viscosity by superimposing our isotherms on this universal curve using the values for the coefficients a_i given by Assael and Dymond[25,26] and obtained the V_0 for each temperature, listed in Table 5. The best-fit value we found for the fixed coefficient R_η is 1.95. The values of V_0 in Table 5 were represented by the empirical relation

$$V_0\cdot10^6/(\text{m}^3\cdot\text{mol}^{-1}) = a + b(T/\text{K}) + c(T/\text{K})^2 \tag{7}$$

The obtained parameters and the results for the hard-sphere scheme are presented in Table 5.

For density, the deviations of the present measurements and the results of other authors from our correlation were shown in Figure 3. The AAD, DM, and Bias of the present results from the correlation are 0.016 %, 0.06 %, and −0.0001 %, respectively. The data at 293.15 K and 0.1 MPa reported by Kirbaslar et al.[5] agree with eq 4 with a relative deviation of −0.05 %. Ren et al.[6] measured the density of DEA at 301.15 K and 0.1 MPa, which shows a deviation of 0.15 % from this work. Vogel[4] reported density values for this diester at 0.1 MPa and at (293.15, 314.85, 334.55, and 353.75) K. The absolute deviations are all within 0.2 % except for the result at 353.75 K, which amount

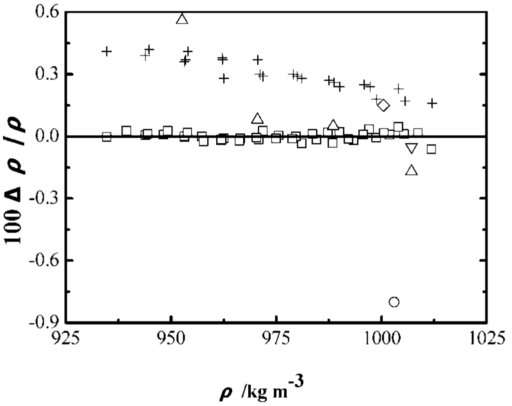

Figure 3. Deviation, $\Delta\rho/\rho = \{\rho(\text{calcd}) - \rho(\text{exptl})\}/\rho(\text{calcd})$, of the present density measurements of diethyl adipate (Table 3), ρ(exptl), from the correlation of the present experimental results given by eq 4, ρ(calcd), as a function of density along the isotherms. □, this work; ○, Bried;[3] △, Vogel;[4] ▽, Kirbaslar;[5] ◇, Ren;[6] +, Comuñas.[7]

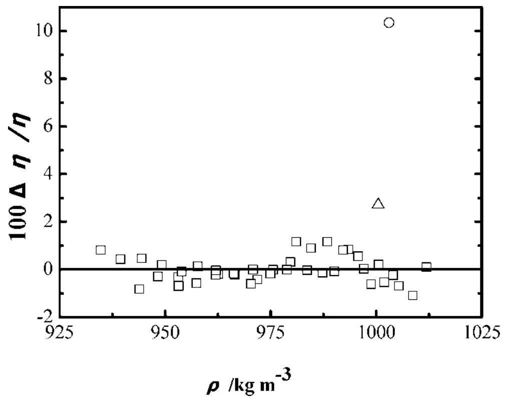

Figure 4. Deviation, $\Delta\eta/\eta = \{\eta(\text{calcd}) - \eta(\text{exptl})\}/\eta(\text{calcd})$, of the present viscosity measurements of diethyl adipate (Table 3), η(exptl), from the correlation of the present experimental results given by eqs 5 to 7, η(calcd), as a function of density along the isotherms. □, this work; ○, Bried;[3] △, Ren.[6]

to 0.56 %. Bried et al.[3] reported the density of diethyl adipate at 298.15 K and at 0.1 MPa, with a deviation of −0.8 % from eq 4. Recently, Comuñas et al.[7] measured the density of diethyl adipate over a temperature range from (293.15 to 403.15) K and up to 140 MPa with a vibrating tube densimeter. Comparisons were made at the overlapped ranges of temperature and pressure. Those values are smaller than the results of this work, and the differences vary from 0.16 % at 303.15 K to 0.42 % at 373.15 K.

Concerning the viscosity of diethyl adipate, only two data were found in the literature, and the deviations were shown in Figure 4. The AAD, DM, and Bias of the present results from the correlation are 0.4 %, 1.2 %, and −0.006 %, respectively. Ren et al.[6] also reported the viscosity of DEA at 301.15 K and 0.1 MPa, with a deviation of 2.7 % from this work. The viscosity of this fluid at 298.15 K at atmospheric pressure was reported by Bried et al.,[3] and the deviation from the correlations of this work amounts to 10.4 %. To the authors' knowledge, no results of viscosity at elevated pressure have been found for diethyl adipate.

Conclusions

The density and viscosity measurements of the present work performed on liquid diethyl adipate with a vibrating-wire sensor are generally characterized by uncertainties of ± 0.2 % and ± 2.0 %, respectively. The experiments were performed over temperatures ranging from (303.15 to 373.15) K along eight isotherms and at pressures up to 20 MPa. The experimental results were fitted successfully with a Tait-type equation for density and a correlation scheme based on a hard-sphere model for viscosity.

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