

The Viscosity and Density of *n*-Dodecane and *n*-Octadecane at Pressures up to 200 MPa and Temperatures up to 473 K¹

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A vibrating-wire instrument for simultaneous measurement of the density and viscosity of liquids under conditions of high pressure is described. The instrument is capable of operation at temperatures between 298.15 and 473.15 K at pressures up to 200 MPa. Calibration was performed by means of measurements in vacuum, air, and toluene at 298.15 K. For *n*-dodecane measurements were made along eight isotherms between 298.15 and 473.15 K at pressures up to 200 MPa, while for *n*-octadecane measurements were measured along seven isotherms between 323.15 and 473.15 K at pressures up to 90 MPa. The estimated uncertainty of the results is 2% in viscosity and 0.2% in density. Comparisons with literature data are presented.

KEY WORDS: density; dodecane; high pressure; high temperature; octadecane; viscosity.

1. INTRODUCTION

In this paper, we describe a vibrating wire apparatus for the simultaneous measurement of viscosity and density and report results for *n*-dodecane and *n*-octadecane at pressures up to 200 MPa and in the temperature

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range of 298 to 473 K. This work is aimed at reducing gaps in the available viscosity data of hydrocarbons under the conditions of high temperature and pressure found in petroleum reservoirs for the purposes of testing predictive models [1]; we also measure density as this plays a key role in several predictive methods.

2. BACKGROUND

The origins of the vibrating wire technique can be traced back to early research on pendulums and the problem of why the period of oscillation increased with air density. As early as 1830, Bessel recognized that the buoyancy and inertial effects of air would increase the period of vibration. Stokes [2] recognized that the viscosity of the surrounding air also plays a role and, by applying the Navier–Stokes equations to the pendulum system, arrived at a full analytical solution for the pendulum motion. Stokes then applied the same technique to the problem of an infinitely long cylindrical rod oscillating in an infinite fluid, and this formed the hydrodynamic basis for vibrating wire viscometry.

A practical device based on the Navier–Stokes equations was developed in 1963 by Tough et al. [3] and the theory was re-visited in 1986 by Retsina et al. [4,5] who included the effect of the wire stiffness and obtained a solution appropriate to transient motion. Many practical devices, operating in either steady-state or transient modes, have since been developed [4–9].

3. WORKING EQUATIONS

Our implementation of the steady-state vibrating-wire technique is very similar to that described by Audonnet and Pádua [7] in which the wire is driven by a constant sinusoidal current of amplitude I and frequency f , and the emf $V(f)$ generated across the vibrating wire is measured with a two-phase lock-in amplifier. This measured emf generally comprises two terms: the first, $V_1(f)$, arises from the electrical impedance of the stationary wire; while the second, $V_2(f)$, arises from the motion of the wire. In the analysis, we express the complex function V_1 as

$$V_1(f) = a + \mathbf{i}b + \mathbf{i}cf, \quad (1)$$

where a , b , and c are real constants, and f is the driving frequency. The second contribution is given by [4,5]

$$V_2(f) = \frac{\Lambda f \mathbf{i}}{f_0^2 - (1 + \beta) f^2 + (\beta' + 2\Delta_0) f^2 \mathbf{i}}. \quad (2)$$

Here, f_0 is the “buoyancy corrected” fundamental transverse resonance frequency of the wire in vacuum which is related to the true vacuum resonance frequency $f_{0,\text{vac}}$, by

$$f_0^2 = f_{0,\text{vac}}^2 - \frac{gV_w\rho}{16\pi\rho_s R^2 L^2}. \quad (3)$$

Here, g is the acceleration of gravity, V_w is the volume of the weight, ρ is the density of the fluid, ρ_s is the density of the wire, R is the radius of the wire, and L is the half length of the wire. Typically, the true vacuum resonance frequency $f_{0,\text{vac}}$ and the internal damping term Δ_0 are determined in calibration measurements and f_0 is calculated for the state of interest from Eq. (3). The other parameters in Eq. (2) are an amplitude Λ , and two terms, β and β' , that are related to the density ρ and viscosity η of the fluid by the hydrodynamic theory [4,5].

Experimentally, one measures the real and imaginary components of V as a function of frequency f for frequencies in the neighborhood of f_0 in the fluid of interest. Then the parameters a, b, c, Λ, ρ , and η are fitted by $V(f) = V_1(f) + V_2(f)$ making use of Eqs. (1)–(3) together with the expression [4,5] relating β and β' to ρ and η . In our work, the lock-in amplifier was operated with offset real and imaginary voltage readings so as to permit measurements to be made over the most sensitive range possible. These offsets were absorbed into the parameters a and b .

Theoretical treatments of the vibrating wire have normally considered simple end conditions such as pinned, clamped, or free ends [4]. Typically, end-pinned conditions are assumed [10] but experimental vacuum frequencies are found to deviate from the values predicted under that assumption. For example, the vacuum resonance measured by Dix et al. [11] is greater by 0.5% than that calculated assuming end-pinned conditions. To reconcile this discrepancy, we write the true vacuum resonance frequency as

$$f_{0,\text{vac}} = \frac{1}{4}\kappa \left(\frac{\pi^2 E R^2 \kappa^2}{16\rho_s L^4} + \frac{gM_w}{\pi\rho_s R^2 L^2} \right)^{1/2}, \quad (4)$$

where E is Young’s modulus for the wire, M_w is the mass of the suspended weight, and κ is a parameter that reflects the end conditions. For end-pinned conditions, κ is exactly unity while, for end clamped conditions, $\kappa \approx 1.05$. In the present work, κ was adjusted to match an experimental measurement of the vacuum resonance frequency at one temperature.

4. INSTRUMENT

The vibrating wire viscometer has been designed to operate in the viscosity range $0.3\text{--}30\text{ mPa}\cdot\text{s}$ and at densities ranging from 600 to $1000\text{ kg}\cdot\text{m}^{-3}$. As shown in Fig. 1, the vibrating wire and magnetic assembly were housed in a commercially-available pressure vessel (HIP Model R1-10-30) rated for operation at 200 MPa. The key parameters of the apparatus are listed in Table I. The temperature of the fluid was inferred from the reading of a platinum resistance thermometer (PRT) which was mounted in a well in the cap of the pressure vessel. This thermometer was calibrated with an uncertainty of $\pm 0.01\text{ K}$. The pressure was measured in the external pipework by means of a Paroscientific transducer (Model 40K-110) with a full scale reading of 207 MPa and an uncertainty of $\pm 0.02\text{ MPa}$.

The pressure vessel was enclosed in a massive aluminium block thermostat and insulated from the environment with 40 mm thick Kaowool V.F. ceramic fibreboard. The temperature of the block was controlled in

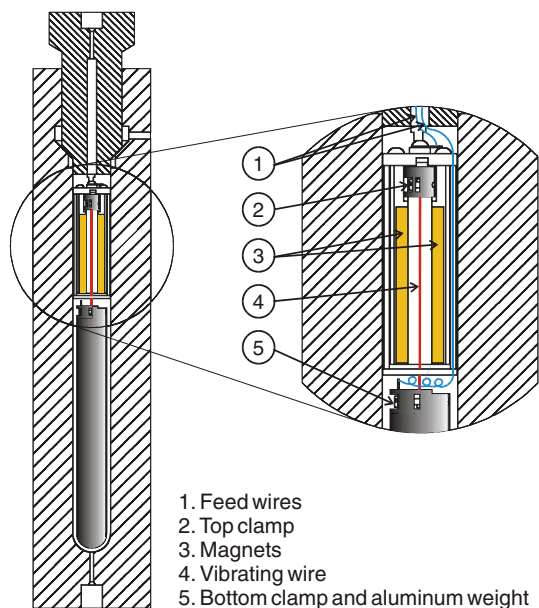


Fig. 1. Diagram of the vibrating wire apparatus.

three zones (top 1/5th, middle 3/5th, and lower 1/5th) by means of PID controllers (Eurotherm 2210e) operating with PRT sensors and a set of 10 cartridge heaters each rated at 110 W. This arrangement was able to keep the temperature stable and uniform to within ± 0.05 K over a period of days.

5. CALIBRATION AND TESTING

Calibration of the apparatus was performed at 298.15 K by means of measurements in vacuum and in both ambient air and toluene at 0.1 MPa. The toluene was supplied by Riedel-de Haën with a stated mass-fraction purity of 99.9% and was used without further purification. The true vacuum resonance frequency, and hence the value of κ , was obtained from the measurement *in vacuo*. In principle, the internal damping Δ_0 can be obtained from the same measurement but the small-bore pressure tubing used made it difficult to achieve a sufficiently good vacuum in the cell. Accordingly, Δ_0 was obtained from a measurement in ambient air with due allowance for the viscosity and density of air [12]. The radius of the wire and the volume of the suspended mass were obtained from the measurement in toluene. The resulting cell parameters are given in Table I. It should be noted that, when measuring the resonance frequency in vacuum or air, the wire can easily be driven to nonlinear behavior. Small nonlinear

Table I. Parameters of the Measurement Cell

			Source
Cell Parameters			
Volume		55 cm ³	Measured
Tungsten Vibrating Wire			
Half-length	L	32.5 mm	Measured
Radius	R	49.129 μ m	Calibrated
Density	ρ_s	19300 kg·m ⁻³	From supplier
Young's modulus	E	411 GPa	From supplier
Internal damping	Δ_0	20×10^{-6}	Measured
Thermal expansion	α_s	4.5×10^{-6} K ⁻¹	From supplier
End condition perturbation	κ	1.02745	Calibrated
Aluminum Mass			
Mass	M_w	134.77 g	Measured
Volume	V_w	50.647 cm ³	Calibrated
Thermal expansion	α_w	23.6×10^{-6} K ⁻¹	[27]
Compressibility	β_w	1.32×10^{-11} Pa ⁻¹	[12]

behavior has also been noticed in toluene at 348 K with the apparent viscosity increasing linearly with drive voltage squared; similar behavior has been observed by Wilhelm et al. [9]. The use of a current of 0.9 mA produced a 1% increase in observed viscosity from that at currents below 0.1 mA.

To verify proper operation of the vibrating wire, a complete isotherm was measured after each calibration in toluene. The repeatability of successive viscosity measurements carried out at the same state point was typically $\pm 0.1\%$. Results obtained at different times with three different wires from the same batch are compared in Figs. 2 and 3. It can be seen that the repeatability in density was better than $\pm 0.1\%$ while that in viscosity was better than $\pm 1\%$. Some of the reported data were measured only a short time after the installation of a new wire. We now have evidence that the reproducibility improves somewhat if the wire is allowed to “relax” for a period of several days, preferably with temperature cycling. Also shown in Fig. 2 and 3 are literature data, and we note satisfactory agreement. The overall uncertainties of the present result are estimated to be $\pm 0.2\%$ in density and $\pm 2\%$ in viscosity.

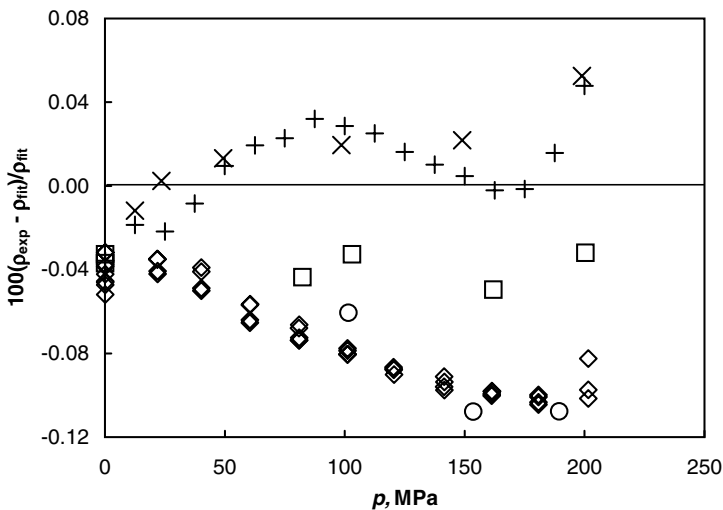


Fig. 2. Deviation in the density of toluene at 298.15 K from the correlation of Assael et al. [13]: (\diamond) this work (May 2002); (\square) this work (August 2002); (\circ) this work (February 2003); (\times) Harris et al. [14]; ($+$) Kashiwagi et al. [15].

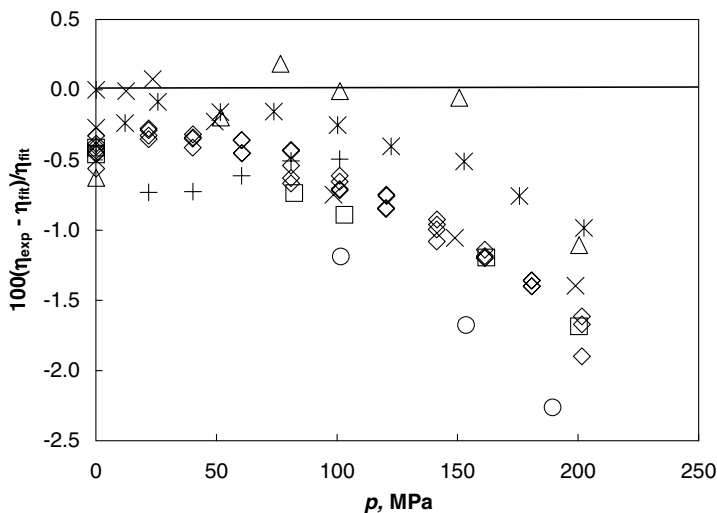


Fig. 3. Deviations in the viscosity of toluene at 298.15 K from the correlation of Assael et al. [13]: (\diamond) This work (May 2002); (\square) this work (August 2002); (\circ) this work (February 2003); (Δ) Dymond et al. [16]; (\times) Harris et al. [14]; ($*$) Harris [17]; ($+$) Kashiwagi and Makita [18].

6. RESULTS

n-dodecane and *n*-octadecane, both of specified mass-fraction purity >99%, were supplied by Aldrich and used without further purification. The viscosity and density of *n*-dodecane were measured along eight isotherms between 298.15 and 473.15 K at pressures up to 200 MPa, and the results are presented in Table II. The viscosity and density of *n*-octadecane were measured along seven isotherms between 323.15 and 473.15 K at pressures up to 90 MPa, and the results are presented in Table III. Higher pressures were not studied due to freezing of *n*-octadecane in the pressure transducer (which was maintained at approximately 320 K). Small adjustments, never more than 0.2% in viscosity and 0.01% in density, have been applied so as to reduce all tabulated results to the stated nominal isotherm temperatures (Figs. 4 and 5).

For purposes of interpolation, the density data have been fitted by a Tait-type equation,

$$\rho = \rho_0 \left\{ 1 - C \log_{10} \left(\frac{B + p}{B + p_0} \right) \right\}^{-1}, \quad (5)$$

where $p_0 = 0.1$ MPa, C is a constant, and ρ_0 and B are given as follows:

Table II. Viscosity and Density of *n*-Dodecane

p (MPa)	ρ (kg·m ⁻³)	η (mPa·s)	p (MPa)	ρ (kg·m ⁻³)	η (mPa·s)	p (MPa)	ρ (kg·m ⁻³)	η (mPa·s)
$T = 298.15$ K								
0.1	746.0	1.344	101.54	799.1	3.750	119.98	806.6	4.378
41.62	771.5	2.139	121.71	806.1	4.497	80.09	790.5	3.102
81.91	790.8	3.154	152.18	817.3	5.728	0.1	746.1	1.343
$T = 323.15$ K								
0.1	727.4	0.911	121.70	794.5	2.762	161.33	809.1	3.677
42.21	756.8	1.422	160.48	808.9	3.627	81.35	778.1	2.007
81.44	777.3	2.012	191.67	818.8	4.502	0.1	727.5	0.910
$T = 348.15$ K								
0.1	708.9	0.659	120.49	782.3	1.886	120.15	782.9	1.896
41.45	741.1	1.006	160.27	797.7	2.452	80.66	764.5	1.411
81.85	764.4	1.428	191.19	808.2	2.974	0.1	708.9	0.659
$T = 373.15$ K								
0.1	689.8	0.503	120.44	770.9	1.406	120.26	771.2	1.405
41.30	726.7	0.777	161.34	787.7	1.809	80.76	751.8	1.073
80.20	750.9	1.069	191.74	798.7	2.149	0.1	690.0	0.503
$T = 398.15$ K								
0.1	670.8	0.401	121.42	759.9	1.123	81.06	739.0	0.858
40.82	712.1	0.623	159.09	777.2	1.395	0.1	670.9	0.401
80.51	738.7	0.855						
$T = 423.15$ K								
0.1	651.1	0.324	121.56	749.4	0.915	81.03	727.1	0.704
40.74	698.1	0.512	163.26	768.2	1.158	0.1	651.1	0.323
81.22	727.3	0.706						
$T = 448.15$ K								
0.1	630.3	0.264	120.65	738.7	0.763	80.81	715.4	0.591
40.72	683.9	0.429	162.00	758.3	0.966	0.1	630.4	0.264
80.14	714.8	0.588						
$T = 473.15$ K								
0.1	608.9	0.218	121.41	729.0	0.657	80.16	703.7	0.506
40.44	670.2	0.367	160.94	748.6	0.813	0.1	609.0	0.218
81.10	704.3	0.510						

$$\rho_0/(\text{kg} \cdot \text{m}^{-3}) = \sum_{i=0}^2 a_i (T/K)^i, \quad (6)$$

$$B/\text{MPa} = \sum_{i=0}^2 b_i (T/K)^i \quad (7)$$

The viscosity has been represented by a hard-sphere scheme employed by Assael et al. [13] for toluene in which η is given by

Table III. Viscosity and Density of *n*-Octadecane

p (MPa)	ρ ($\text{kg}\cdot\text{m}^{-3}$)	η ($\text{mPa}\cdot\text{s}$)	p (MPa)	ρ ($\text{kg}\cdot\text{m}^{-3}$)	η ($\text{mPa}\cdot\text{s}$)	p (MPa)	ρ ($\text{kg}\cdot\text{m}^{-3}$)	η ($\text{mPa}\cdot\text{s}$)
$T = 323.15 \text{ K}$								
0.1	762.4	2.460	62.23	799.0	5.033	40.83	788.7	4.028
20.78	776.6	3.196	91.96	812.3	6.728	0.1	763.2	2.466
40.30	787.7	3.999						
$T = 348.15 \text{ K}$								
0.1	744.6	1.595	61.09	783.7	3.092	40.89	773.1	2.538
21.60	760.6	2.065	90.77	797.9	4.035	0.1	744.7	1.597
42.09	773.4	2.566						
$T = 373.15 \text{ K}$								
0.1	726.2	1.123	62.81	771.1	2.167	40.90	758.1	1.770
22.69	745.3	1.467	92.20	786.2	2.766	0.1	726.2	1.123
43.30	759.5	1.813						
$T = 398.15 \text{ K}$								
0.1	709.2	0.834	61.49	757.8	1.584	40.58	744.4	1.310
22.51	730.5	1.088	82.39	769.7	1.874	0.1	709.3	0.834
42.40	745.6	1.332						
$T = 423.15 \text{ K}$								
0.1	691.8	0.646	62.55	746.1	1.241	40.26	730.7	1.017
21.63	715.0	0.841	83.87	758.9	1.469	0.1	691.8	0.646
42.32	732.2	1.035						
$T = 448.15 \text{ K}$								
0.1	674.2	0.516	60.40	732.8	0.980	39.89	717.3	0.819
21.36	700.3	0.676	83.21	747.5	1.170	0.1	674.2	0.516
41.34	718.5	0.829						
$T = 473.15 \text{ K}$								
0.1	656.2	0.420	60.71	721.7	0.816	40.75	705.5	0.685
21.15	685.6	0.558	82.47	736.6	0.965	0.1	656.2	0.420
41.31	705.8	0.689						

$$\eta/(\text{Pa}\cdot\text{s}) = \eta^* \left[4.778 \times 10^{-9} \left\{ V/(\text{m}^3 \cdot \text{mol}^{-1}) \right\}^{-2/3} \times \left\{ M/(\text{kg} \cdot \text{mol}^{-1}) \right\}^{1/2} \{ T/\text{K} \}^{1/2} \right], \quad (8)$$

where V is the molar volume, M is the molar mass, and η^* is a dimensionless viscosity given by

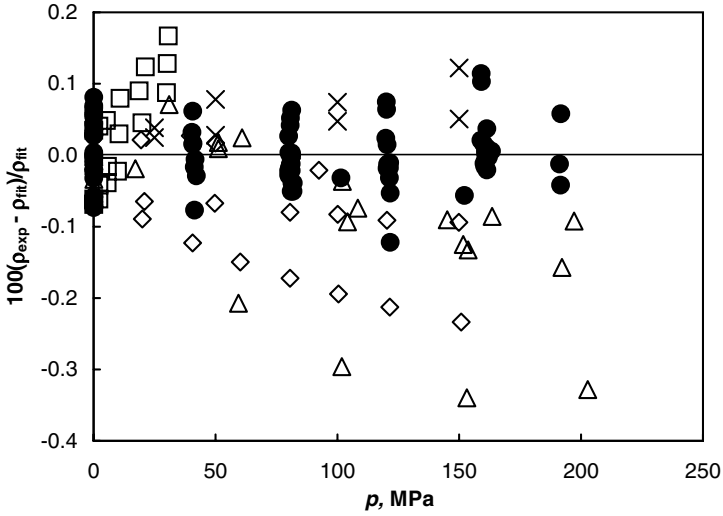


Fig. 4. Deviations of the density of *n*-dodecane from Eq. (5): (●) this work; (Δ) Dymond et al. [19]; (×) Eastal and Woolf [21]; (□) Rousseaux et al. [22]; (◊) Tanaka et al. [20].

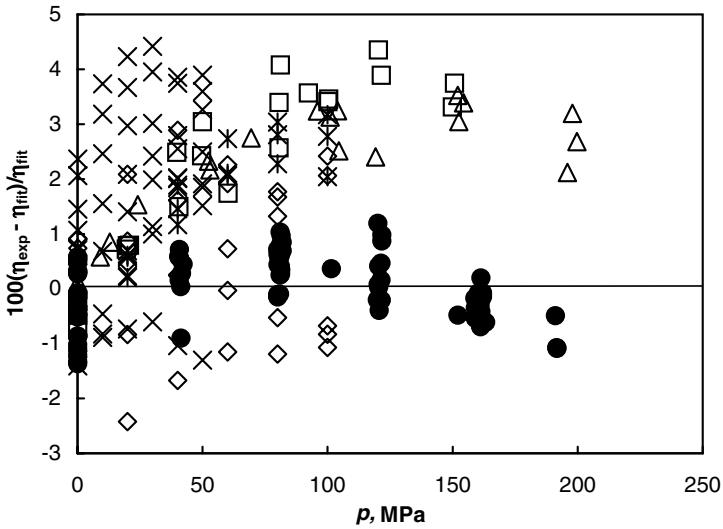


Fig. 5. Deviations of the viscosity of *n*-dodecane from Eq. (8): (●) this work; (◊) Ducoulombier et al. [23]; (Δ) Dymond et al. [19]; (*) Kashiwagi and Makita [18]; (×) Stephan and Lucas [24]; (□) Tanaka et al. [20].

$$\frac{1}{\eta^*} = \sum_{i=0}^3 d_i (V/V_0)^i. \quad (9)$$

In Eq. (9), V_0 is a temperature-dependent molar core volume given by

$$V_0/(10^{-6}\text{m}^3 \cdot \text{mol}^{-1}) = \sum_{i=0}^3 e_i (T/\text{K})^i. \quad (10)$$

Eqs. (5)–(10) form self-consistent representations for viscosity and density as functions of temperature and pressure. The values of the parameters determined for *n*-dodecane and *n*-octadecane are given in Table IV. For *n*-dodecane, the viscosity is represented with a maximum absolute deviation of 1.4% and an average absolute deviation of 0.5% while, for *n*-octadecane, the maximum absolute deviation is 0.8% and the average absolute deviation is 0.2%.

In the case of *n*-dodecane viscosity, the present results agree with other published data [18–20,23,24] within their mutual uncertainty of order $\pm 4\%$. Nevertheless, at higher pressures, above 50 MPa, a systematic trend can be observed whereby the viscosity values measured by Dymond

Table IV. Parameters of Eqs. (5)–(10) for *n*-Dodecane and *n*-Octadecane

Coefficient	<i>n</i> -Dodecane	<i>n</i> -Octadecane
a_0	921.984	1010.07
a_1	−0.47516	−0.80587
a_2	$−3.922 \times 10^{-4}$	1.2463×10^{-4}
b_0	345.1	388.09
b_1	−1.1458	−1.2761
b_2	0.9837×10^{-3}	1.1143×10^{-3}
C	0.2072	0.2021
d_0	0.321621	0.2237588
d_1	−0.4803715	−0.371348
d_2	0.222206	0.188779
d_3	$−2.964626 \times 10^{-2}$	$−2.67796 \times 10^{-2}$
e_0	191.54	285.892
e_1	−0.441338	−0.403541
e_2	8.98744×10^{-4}	6.10544×10^{-4}
e_3	$−6.7792 \times 10^{-7}$	$−3.792 \times 10^{-7}$
M (kg · mol ^{−1})	0.17034	0.254504
T_{\min} (K)	298.15	323.15
T_{\max} (K)	473.15	473.15

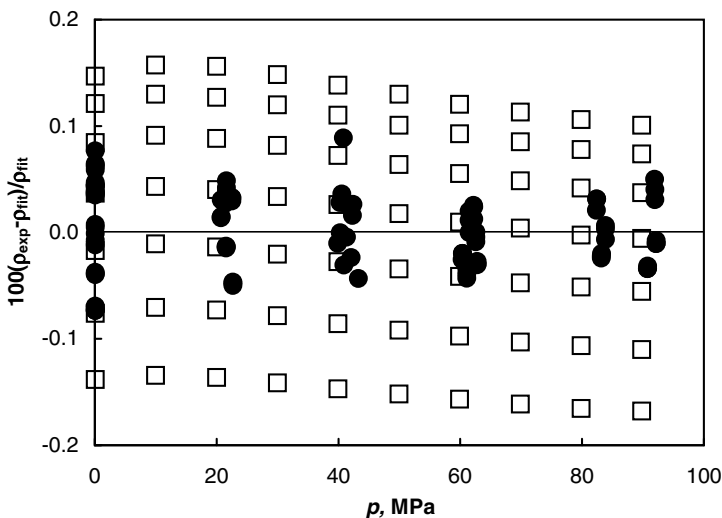


Fig. 6. Deviations of the density of *n*-octadecane from Eq. (5): (●) this work; (□) Dutour et al. [26].

et al. [19] and Tanaka et al. [20] are systematically 3–4% higher than the values reported in this work.

The results for *n*-octadecane are compared with the limited literature data available in Figs. 6 and 7. The density agrees reasonably well with values determined from sound-speed measurements by Dutour et al. [26] although the deviations show a marked temperature dependence with errors ranging from around -0.15% at 323.15 K to around $+0.15\%$ at 383.15 K. In the case of viscosity, the results of Ducolombier et al. [23] and Hogenboom et al. [25] agree with this work at low pressures but show positive deviations of up to 6% at higher pressures. It is interesting to note that, for *n*-dodecane, the results of Ducolombier et al. were in agreement with this work to $\pm 3\%$.

7. CONCLUSIONS

The results obtained for toluene demonstrate reliable operation of the new instrument and support an estimated uncertainty of $\pm 2\%$ in viscosity and $\pm 0.2\%$ in density. New results are reported for *n*-dodecane and *n*-octadecane. Measurements on other pure liquids and binary mixtures, especially those of interest to the petroleum industry, will be reported in the near future.

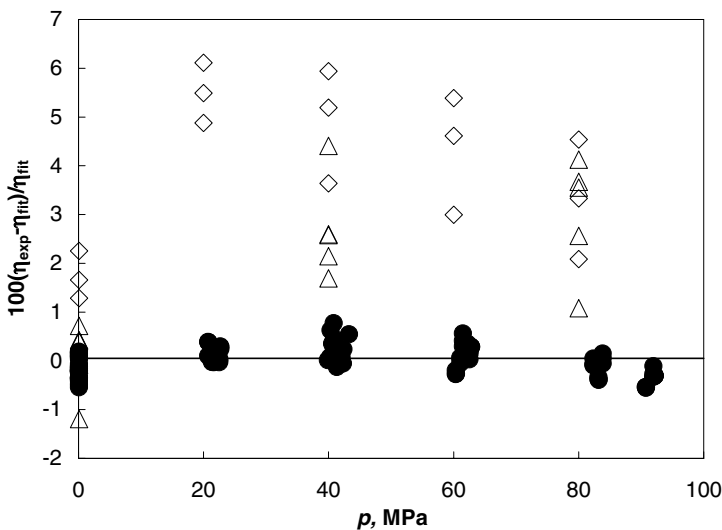


Fig. 7. Deviations of the viscosity of *n*-octadecane from Eq. (8); (●) this work; (◊) Ducoulombier et al. [23]; (Δ) Hogenboom et al. [25].

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