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Simultaneous Measurement of the Density and Viscosity of Compressed Liquid Toluene

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A vibrating-wire densimeter described previously has been used to perform simultaneous measurements of the density and viscosity of toluene at temperatures from 222 to 348 K and pressures up to 80 MPa. The density measurements are essentially based on the hydrostatic weighing principle, using a vibrating-wire device operated in forced mode of oscillation, as a sensor of the apparent weight of a cylindrical sinker immersed in the test fluid. The resonance characteristics for the transverse oscillations of the wire, which is also immersed in the fluid, are described by a rigorous theoretical model, which includes both the buoyancy and the hydrodynamic effects, owing to the presence of the fluid, on the wire motion. It is thus possible, from the working equations, to determine simultaneously, both the density and the viscosity of the fluid from the analysis of the resonance curve of the wire oscillation, the density being related essentially to the position of the maximum and the viscosity to its width. New results of measurements of the density and viscosity of toluene in the compressed liquid region are presented, and compared with literature data. The density results extend over a temperature range 222 K $\leqslant T \leqslant$ 348 K, and pressures up to 80 MPa. The viscosity results cover a temperature range of 248 K $\leqslant T \leqslant$ 348 K and pressures up to 80 MPa. The uncertainty of the present density data is estimated to be within $\pm 0.1\%$ at temperatures 298 K $\leqslant T \leqslant$ 350 K, and $\pm 0.15\%$ at 222 K $\leqslant T \leqslant$ 273 K. The corresponding overall uncertainty of the viscosity measurements is estimated to be $\pm 2\%$ for temperatures 298 K $\leqslant T \leqslant$ 350 K, and $\pm 3\%$ for 248 K $\leqslant T \leqslant$ 273 K.

KEY WORDS: density; high pressure; toluene; vibrating-wire, viscosity.

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1. INTRODUCTION

Toluene is often regarded as a possible standard reference substance for several thermophysical properties, such as thermal conductivity [1] and viscosity [2]. However, some discrepancies between the results of different authors can be found in the literature, in particular, for its viscosity in the compressed liquid region. Moreover, viscosity data below room temperature, and, particularly, at pressures above atmospheric are scarce. On the other hand, a standard reference liquid for transport properties must have accurate density data available over the temperature and pressure ranges of its application. Cibulka and Takagi [3] have recently summarised the available toluene density data in the compressed liquid region. From the work of those authors the scarcity of direct experimental measurements in the low temperature region is apparent.

The present simultaneous measurements of the density and viscosity of toluene have been carried out at temperatures from 222 to 348 K and pressures from 0.1 to 80 MPa. The measurements were performed using a vibrating-wire technique in the forced oscillation mode of operation, which enables the simultaneous measurement of the density and viscosity of fluids over wide ranges of temperature and pressure. The method has been presented and validated elsewhere [4]. The instrument based on this concept has previously been used to study 2,2,4-trimethylpentane [5], 1,1,1,2-tetrafluoroethane (HFC 134a) [6], and cyclohexane [4]. The applicability of the apparatus has subsequently been extended in order to study the viscosity of mixtures of poly(ethyleneglycols) saturated with supercritical carbon dioxide [7].

2. EXPERIMENTAL

The apparatus and the measurement technique are described elsewhere [4–6, 8–10], and the equipment was used without significant changes for the present measurements. The principle of operation of the apparatus relies on the analysis of the velocity response of the forced transverse oscillations of a thin metallic wire, immersed in the test liquid, from which a cylindrical solid (sinker) is suspended. The frequency corresponding to the maximum velocity (the resonance condition) is determined essentially by the fluid density, and the width of the resonance peak by the fluid viscosity, according to a set of working equations [4, 8, 10], which includes the effects of the fluid dynamics on the wire oscillations [11]. Consequently, the analysis of the resonance characteristics for the transverse oscillations of the wire, enable the simultaneous measurement of the density and the viscosity of the test fluid. The design characteristics of the present apparatus have been chosen in order to optimise the accuracy of the density measurement [4]. Two of the parameters of the measuring cell, namely, the average radius of the wire and the volume of the sinker, are, for practical reasons, determined *in situ*. This is done by means of one single calibrating operation, whose general procedure has been described before [4–6, 8]. This determination consists of the analysis of the experimental resonance characteristics of the wire oscillations, both under vacuum, and immersed in a fluid, at a certain temperature and pressure (usually, 0.1 MPa), where both its density and viscosity are known from an independent source, or measured by an independent technique. For the present work, those parameters have been obtained from an analysis of the wire resonance characteristics, both under vacuum, and in toluene at 298 K and 0.1 MPa. The reference data used for this purpose were the viscosity measurements by Gonçalves et al. [2] and the density of the toluene sample as measured with a 60 ml picnometer, previously calibrated with water and mercury.

Temperature stability during a typical measurement was within ±0.005 Kand the uncertainty of the readings was ±0.01 K (ITS-90). The uncertainties of the pressure readings were ±0.03 MPa up to 35 MPa, and ±0.07 MPa up to 80 MPa.

The measurements were performed with HPLC-grade toluene, supplied by Sigma-Aldrich, with a stated purity of 99.8%. The samples were water dried over molecular sieves, and degassed by helium sparging prior to the measurements. The purity of the samples was checked by GLC before and after the measurements were carried out, and confirmed to be in excess of the nominal value.

The overall uncertainty of the present density measurements is estimated to be less than ±0.1% at temperatures $298\ \mathrm{K} \leqslant T \leqslant 350\ \mathrm{K}$, and ±0.15% at lower temperatures. The corresponding uncertainty of the viscosity measurements is estimated to be ±2% and ±3%, respectively.

3. RESULTS

The results obtained for the density, ρ, and viscosity, η, of toluene along six isotherms between 222 and 348 K, and pressures from 0.1 to 80 MPa, are shown in Table I. The results obtained for the viscosity of toluene at 222.51 K are estimated by the authors to have an uncertainty larger than that for other temperatures for a variety of reasons, including the extreme sensitivity of the viscosity to temperature at low temperatures. For that reason, we prefer to omit the data for this isotherm, rather than quote values with a larger, necessarily systematic, uncertainty. This problem will be the subject of further investigation.

Table I. Experimental Density and Viscosity of Toluene

p (MPa)	ρ (kg·m^{-3})	η (mPa·s)	p (MPa)	ρ (kg·m^{-3})	η (mPa·s)
	222.51 K			298.28 K	
0.1	932.29		0.1[a]	862.36[a]	0.5536[a]
1.1	933.07		2.1	864.28	0.5593
5.1	935.05		5.1	866.59	0.5709
7.6	936.18		7.6	868.55	0.5830
10.0	937.32		20.0	877.02	0.6432
15.0	939.82		24.3	879.86	0.6621
20.1	941.97		30.0	883.85	0.6885
25.1	944.44		39.8	890.14	0.7431
30.1	946.78		50.0	896.29	0.7877
39.8	951.01		60.0	901.36	0.8331
50.1	955.53		70.0	906.88	0.8879
60.3	959.51		80.1	912.21	0.9459
70.0	963.45				
80.2	967.23				
	248.32 K			323.12 K	
0.1	908.84	1.139	0.1	839.46	0.4207
1.1	909.46	1.146	2.6	841.76	0.4295
2.1	910.74	1.162	5.1	843.98	0.4383
5.0	911.81	1.180	7.5	846.03	0.4456
7.6	913.24	1.205	10.0	848.11	0.4552
10.4	914.76	1.236	15.3	852.56	0.4720
15.1	917.30	1.285	20.1	856.09	0.4912
20.1	919.94	1.338	30.0	863.38	0.5271
24.3	922.13	1.386	40.3	870.59	0.5648
29.9	924.87	1.453	50.2	877.06	0.6025
39.4	929.42	1.568	70.1	888.60	0.6813
49.8	934.41	1.693	80.3	894.01	0.7219
60.8	939.41	1.842			
70.0	943.35	1.974			
80.6	947.66	2.133			
	273.30 K			348.13 K	
0.1	886.07	0.7580	0.1	815.43	0.3326
1.1	886.71	0.7648	2.5	817.99	0.3392
2.0	887.37	0.7703	5.1	820.67	0.3465
5.0	889.33	0.7891	7.6	823.14	0.3539
7.6	891.03	0.8036	10.1	825.43	0.3611
11.5	893.58	0.8300	15.0	830.19	0.3763
15.2	895.87	0.8553	20.0	834.63	0.3901
20.0	898.81	0.8863	29.9	842.93	0.4183
25.2	901.87	0.9191	40.1	850.74	0.4485
30.0	904.57	0.9521	49.3	857.28	0.4763
40.3	910.33	1.0300	59.9	864.42	0.5086
49.5	915.04	1.0983	78.8	875.74	0.5672
59.9	920.23	1.1770			
70.5	925.18	1.2648			
80.1	929.67	1.3472			

[a] Results obtained independently, as described in the text, and used to calculate the cell calibration parameters.

Table II. Coefficients of Eq. (1)

T (K)	ρ_0 (kg·m^{-3})	C	D (MPa)	σ (%)
222.505	932.29	0.09033	156.56	0.01
248.318	908.84	0.09033	140.16	0.01
273.298	886.07	0.09033	117.51	0.01
298.282	862.36	0.09033	96.33	0.02
323.124	839.46	0.09033	82.84	0.01
348.128	815.43	0.09033	68.51	0.01

3.1. Density

The density of toluene along each isotherm was correlated using the modified Tait equation [12]:

$$\rho = \rho_0(T)\left(1 - C\ln\frac{D(T)+p}{D(T)+p_0}\right)^{-1} \tag{1}$$

where $\rho_0(T)$ is the density at a reference pressure, $p_0 = 0.1$ MPa, and temperature T; D and C are empirical parameters. Values for these parameters obtained by fitting the data along each isotherm are shown in Table II, together with the respective standard deviations. The densities at 0.1 MPa, $\rho_0(T)$, were obtained by carrying out small extrapolations from the present experimental results. For the purposes of interpolation, the entire set of density data of Table I may be described by means of Eq. (1), the parameter C is assumed to be temperature independent, and D is a function of temperature [13].

The $\rho_0(T)$ values in Table I are described by the equation:

$$\rho_0(T) = b_0 + b_1 T + b_2 T^2 \tag{2}$$

with a standard deviation of ± 0.02%. The values obtained for the parameters are listed in Table III. The quantity $D(T)$ may be described as a function of temperature by the equation [8, 14]:

$$D(T) = -p_c + \sum_i \left(d_i\left(1 - \frac{T}{T_c}\right)^i\right) \tag{3}$$

with the parameters d_i listed in Table III, and the critical constants, $T_c = 591.8$ K and $p_c = 4.1$ MPa [15]. The (p, ρ, T) surface defined by Eqs. (1)–(3), with the parameters listed in Table III, and $C = 0.09033$, describe the whole

Table III. Coefficients of Eqs. (2) and (3)

i	b_i ($\mathrm{kg \cdot m^{-3} \cdot K^{-i}}$)	d_i (MPa)
0	1121.387	–
1	−0.7988	113.28
2	−0.00023	77.34
3	–	5.49
4	–	445.96

set of the present results with a standard deviation of ±0.02%. The deviations do not exceed ±0.06%. Deviations of the present density results and of literature data from the correlation Eqs. (1)–(3), with the parameter values given in Tables II and III, are shown in Fig. 1.

3.2. Viscosity

The viscosity of toluene was correlated with the density using a scheme based on the hard-sphere theory of transport properties of dense fluids, previously applied to *n*-alkanes [16], and aromatic hydrocarbons [17]. This scheme has been applied to other liquids, e.g., HFC-134a and 2,2,4-trimethylpentane [5, 6]. A reduced, dimensionless viscosity, η', is defined as

$$\eta' = 9.118 \times 10^{7} \eta V^{2/3} / (MRT)^{1/2} \tag{4}$$

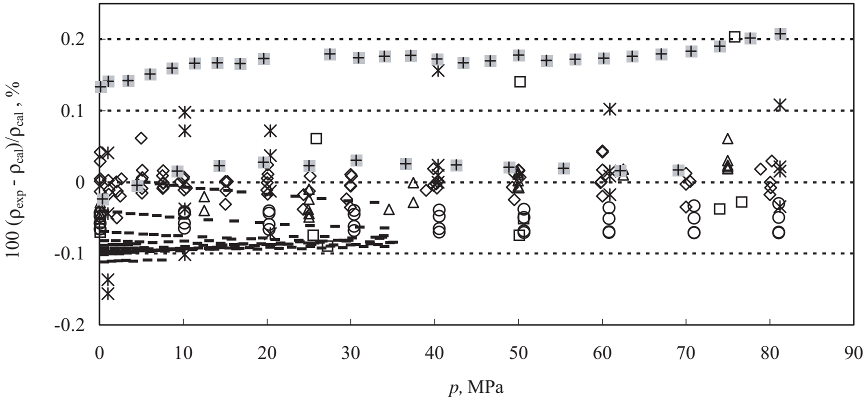

Fig. 1. Deviations of the present density results and of literature data from the correlation Eqs. (1)–(3), with the parameter values given in Tables II and III. (◇) Present work; (▬) Magee and Bruno [20]; (○) Muringer et al. [21]; (∗) Mopsik [22]; (⊞) Taravillo et al. [23]; (□) Dymond et al. [24]; (△) Kashiwagi et al. [30].

Table IV. Characteristic Volumes, V_0

T (K)	$10^6 V_0$ ($\mathrm{m^3 \cdot mol^{-1}}$)
248.318	92.00
273.298	90.97
298.282	89.62
323.124	88.57
348.128	87.63

where M is the molar mass, R is the gas constant, T is the temperature, and V is the molar volume, all quantities being given in SI units. For a hard-sphere system, η' depends just on V/V_0, where V_0 is a characteristic molar volume, which is weakly temperature dependent, reflecting the finite steepness of the repulsive potential for real molecules. Accordingly, plots of η' versus $\ln(V/V_0)$ for different isotherms should be superimposable one on another by a shift along the $\ln(V/V)_0$ axis. Thus, the ratio $V_0(T)/V_0(T_r)$ has been obtained for each temperature by superposition of the curve η' for each isotherm, on a reference isotherm, $\eta'(T_r)$. As a first approximation of V_0, the value reported by Oliveira and Wakeham [18] at 303.15 K was used. The values obtained for $V_0(T)$ are listed in Table IV. They are described, with a standard deviation, $\sigma = 0.01\%$, by the following equation:

$$V_0 \times 10^6/(\mathrm{m^3 \cdot mol^{-1}}) = 109.594 - 0.090081T/K + 7.75 \times 10^{-5}(T/K)^2 \tag{5}$$

Subsequently, all the viscosity results shown in Table I are represented by the equation:

$$\ln \eta' = \sum_{i=0}^{N} a_i \left(\frac{V_0}{V - V_0} \right)^{-i} \tag{6}$$

with the coefficients a_i listed in Table V. The deviations of the present results from this correlation scheme are shown in the deviation plot of Fig. 2.

Table V. Coefficients of Eq. (6)

i	a_i
0	4.51831
1	−24.2499
2	90.3580
3	−204.095
4	191.886

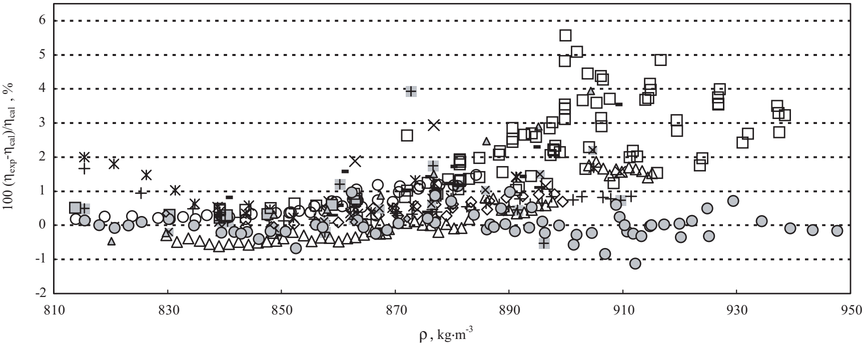

Fig. 2. Deviations of the present viscosity results and of literature data from the correlation Eqs (4)–(6), with the parameters given in Table V. (●) Present work; (▨) Gonçalves et al. [2]; (▬) Harris et al. [13]; (∗) Oliveira and Wakeman [18];(■) Santos and Nieto de Castro [25]; (△) Assael et al. [26]; (□) Harris [27]; (⊠) Assael et al. [28]; (▲) Kaiser et al [29]; (×) Dymond et al. [31]; (○) Krall et al. [32]; (◇) Assael et al. [33]; (⊞) Dymond et al. [34]; (◇) Dymond and Robertson [35];(+) Kashiwagi and Makita [36].

Equation (6) describes the complete set of the present results with a maximum deviation of ±1.1% and a standard deviation of ±0.4%.

4. COMPARISONS

4.1. Comparison of the Density Results with Literature Data

The thermodynamic properties of toluene have been reviewed by Goodwin [19] who proposed an equation of state for toluene from 178 to 800 K and pressures up to 1000 bar. However, according to Magee and Bruno [20], that equation of state is unable to describe the (p, ρ, T) surface of toluene at temperatures around and below 270 K, with an accuracy commensurate with the experimental uncertainties of the measurements. Recently, Cibulka and Takagi [3] have extensively reviewed the literature density data on compressed liquid toluene, and have proposed a correlation scheme for data evaluation. The present results deviate from the latter by a maximum of ±0.12%, and the average deviation is 0.02_3%, using the present values for the reference density at 0.1 MPa, given in Table I, which is the preferred procedure followed by those authors [3]. If, however, the correlation suggested by Cibulka and Takagi for the density of toluene at 0.1 MPa (vd. Table VI of Ref. 3) is used, the maximum and average deviations found are 0.15% and 0.08%, respectively. Nevertheless, the deviations encountered are believed to be within the uncertainty of the correlation scheme proposed by those authors.

Table VI. Literature Toluene Density Data

First author	Ref.	Method	T (K)	p (MPa)	Uncertainty (%)	Deviation limits[a] (%)	Average deviation[b] (%)
Present work		Vibrating-wire	222–348	0.1–80	±0.1 (0.15)[c]	−0.05; 0.06	0.01
Magee (1996)	[20]	Isochoric	180–400	0.1–35	±0.05	−0.11; 0.0	0.08
Taravillo (1994)	[23]	Expansion method	223–303	0.1–110	d	−0.02; 0.21	0.12
Dymond (1988)	[24]	Metal bellows	298–373	0.1–400	±0.1	−0.09; 0.20	0.08
Muringer (1985)	[21]	Sound velocity	180–320	0.1–260	±0.2	−0.07; 0.11	0.05
Kashiwagi (1982)	[30]	High-pressure burette	273–348	0.1–250	±0.1	−0.05; 0.06	0.03
Mopsik (1969)	[22]	Metal bellows	223–298	1–203	–	−0.16; 0.16	0.06

[a] The deviation limits shown are the minimum and maximum deviations of each data set from Eqs. (1)–(3), within the temperature and pressure ranges of the present results.

[b] Average of the absolute value of the deviations from Eqs. (1)–(3), within the temperature and pressure ranges of the present results.

[c] The uncertainty at $T \leqslant 273$ K is shown within parentheses.

[d] The authors claim a maximum uncertainty of 0.003 $\mathrm{mol \cdot dm^{-3}}$.

Although several sets of liquid toluene (p, ρ, T) data can be found in the literature, the published density data at temperatures below 273 K consist mainly of those calculated from speed-of-sound measurements by Muringer et al. [21], and those published by Mopsik [22] and Magee and Bruno [20]. Another data set (two isotherms, at 223.16 and 303.14 K, and pressures up to 106 MPa) has been published [23] with the purpose of checking the consistency of the calibration of an expansion apparatus, which used literature toluene data as a reference. A further data set referred to by Cibulka and Takagi [3], which was subsequently discarded by those authors in their data analysis, is not used in the comparisons below.

Table VI summarizes the toluene literature density data considered for comparison with the present results. In that table, the source, method employed, temperature and pressure ranges, and quoted uncertainty, are shown for each of the data sets. The last two columns on the right present, respectively, the average deviation and the deviation limits of the data in each set from the correlation of the present results (Eqs. (1)–(3)), along the overlapping temperature and pressure ranges. Figure 1 shows the deviation plot of those sets of liquid toluene density data from the same correlation. A general good agreement is observed between the literature data and the present measurements, most of the deviations being well within the combined uncertainties of the measurement techniques. At the highest temperatures the maximum deviation (0.2%) is found for the datum obtained by Dymond et al. [24] at 323 K, at a pressure of ca. 76 MPa. At temperatures below 273 K the largest discrepancies are found for the data published by Taravillo et al. [23] at 223 K, which show systematic positive deviations from the present results, but do not exceed 0.21%.

4.2. Comparison of the Viscosity Results with the Literature Data

Table VII summarizes the toluene literature viscosity data considered for comparison with the present results. In that table, the source, the method employed, the temperature and pressure ranges, the quoted uncertainty, and the average and the upper and lower limits of the data deviations from the correlation of the present results (Eqs. (4)–(6), and Table V), for each data set are shown. In Fig. 2, a deviation plot of the literature data, described in Table VII, from the correlation of the present data, over the temperature and pressure ranges of the present results, is displayed as a function of the density. For this purpose, the density has been recalculated using Eqs. (1)–(3) for each literature datum. Most of the literature data concern measurements at, and above, room temperature. At temperatures above 273 K, all the deviations are less than the combined nominal uncertainties of the experimental methods, but it should be noted that two points

Table VII. Literature Toluene Viscosity Data

First author	Ref.	Method	T (K)	P (MPa)	Uncertainty (%)	Deviation limits[a] (%)	Average deviation[b] (%)
Present work		Vibrating-wire	248–348	0.1–80	$\pm 2\ (\pm 3)^c$	−1.1; 1.0	0.25
Assael (2000)	[28]	Vibrating-wire	213–323	0.1	$\pm 0.5^d$	−0.2; 2.2	0.63
Harris (2000)	[27]	Falling body	255–323	0.1–400	±1	0.2; 5.6	2.39
Assael (1999)	[26]	Vibrating-wire	217–370	0.1–30	±0.5	−0.6; 1.9	0.59
Harris (1997)	[13]	Falling body	298–323	0.1–375	±2	−0.3; 1.2	0.66
Dymond (1995)	[31]	Falling body	299–323	0.1–500	$\pm 3\ (\pm 5)^c$	0.1; 2.9	1.32
Santos (1993)	[25]	Quartz-crystal	298–373	0.1–200	±0.5	0.2; 3.5	1.22
Krall (1992)	[32]	Oscillating-disk	298–425	0.1–30	±0.5	0.2; 1.5	0.60
Oliveira (1992)	[18]	Vibrating-wire	303–348	0.1–250	±0.5	0.2; 2.0	0.86
Assael (1991)	[33]	Vibrating-wire	303–323	0.1–80	±0.5	-0.0_5; 0.9	0.44
Dymond (1991)	[34]	Falling body	298–373	0.1–500	±4	−0.5; 3.9	0.91
Kaiser (1991)	[29]	Capillary	220–348	0.1	$\pm 1.0\ (\pm 1.5)^c$	−0.5; 3.9	1.34
Gonçalves (1987)	[2]	Capillary	297–350	0.1	±0.5	0.3 ;0.8	0.47
Dymond (1985)	[35]	Capillary	293–353	0.1	±0.5	0.0_1; 0.4	0.16
Kashiwagi (1982)	[36]	Torsional crystal	298–348	0.1–110	±2	0.1; 1.7	0.51

[a] The deviation limits shown are the minimum and maximum deviations of each data set from Eqs. (4)–(6), within the temperature and pressure ranges of the present results.

[b] Average of the absolute value of the deviations from Eqs. (4)–(6), within the temperature and pressure ranges of the present results.

[c] Higher uncertainty limits at the lowest temperatures or highest pressures are shown within parentheses.

[d] The authors claim a higher, unspecified, uncertainty at low temperatures.

of the data set published by Santos and Nieto de Castro [25], at 298 K, deviate by 3.5%.

At low temperatures the available literature data are scarce, especially at pressures above 0.1 MPa. Recently, however, there has been an effort to provide experimental results for these thermodynamic conditions. Assael et al. [26] published a set of measurements obtained with a free-decay vibrating-wire technique, over a temperature range $217 \leqslant T \leqslant 323$ K, and pressures up to 30 MPa. For the overlapping ranges of temperature and pressure, very good agreement is observed with the present results, the deviations being well within the combined uncertainty limits of the experimental measurement techniques. The results published by Harris, obtained with a falling-body apparatus [27], span a smaller temperature range ($255 \text{ K} \leqslant T \leqslant 323$ K), but cover pressures up to 400 MPa. The deviations of those data from the present results show a systematic pattern, all being positive, and increasing with decreasing temperature. The higher deviations are thus found at 255 K, one datum attaining + 5.6%, which is clearly outside the mutual uncertainties of the measurements, considering the nominal uncertainty claimed by that author of ±1% [27].

Four data sets, containing results below 273 K, at a pressure of 0.1 MPa, have been found in the literature. The data recently published by Assael et al. [28] show very good agreement with the present results down to about 280 K. At lower temperatures, the deviations of their measurements from the present results increase with decreasing temperature, but they do not exceed 2.2% at ca. 250 K, and are thus within the uncertainty limits of the present results at low temperatures. The measurements reported by Kaiser et al. [29] deviate by less than ±0.5% near room temperature and above, but the deviations systematically increase with decreasing temperature, reaching a maximum of 3.9% at about 250 K.

In conclusion, very good agreement is observed between the published data and the present measurements, at, and above, room temperature, but some significant discrepancies are observed at low temperatures between the various data sets.

5. CONCLUSIONS

New results for the density and viscosity of compressed liquid toluene are presented. The measurements cover a pressure range from 0.1 to 80 MPa. The density and viscosity results cover the temperature ranges, $222 \text{ K} \leqslant T \leqslant 348$ K, and $248 \leqslant T \leqslant 348$ K, respectively. The density results show good agreement with literature data, including the lowest temperatures, where the direct density measurements at pressures higher than 35 MPa are very scarce (one data set published in 1969 [22] and one isotherm at 223 K [23] could be found in the literature). The present work provides new data on the viscosity of liquid toluene at pressures above atmospheric, covering temperatures below room temperature—a region where the scarcity of literature data has been felt, particularly, regarding the use of toluene as a standard reference fluid. A comparison of the present viscosity results with the available literature data shows an agreement, which is, in general, commensurate with the combined uncertainties of the experimental methods at, or above room temperature. At lower temperatures, however, important discrepancies are found between the available viscosity data sets. At the lowest temperature of the present results (248 K), the deviations of the literature data sets range from −1% to +5.6%. It should be pointed out, however, the good agreement found between recent, precise, measurements, obtained using a free-decay vibrating-wire technique [26, 28], and the present results.

The authors expect that the present work may contribute to the establishment of toluene as a reference substance for viscosity.

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