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Density and Viscosity of Diisodecyl Phthalate $C_6H_4(COOC_{10}H_{21})_2$, with Nominal Viscosity at $T = 298$ K and $p = 0.1$ MPa of 87 mPa·s, at Temperatures from (298.15 to 423.15) K and Pressures up to 70 MPa

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The viscosity and density of diisodecyl phthalate (DIDP), with a nominal viscosity at $T = 298$ K and $p = 0.1$ MPa of 87 mPa·s, has been measured at temperatures from (298.15 to 423.15) K and pressures from (0.1 to 70) MPa. A vibrating wire viscometer, with a wire diameter of about 0.15 mm, was used for the viscosity measurements at pressures up to 70 MPa, and the results have an expanded uncertainty ($k = 2$), including the error arising from the pressure measurement, of between ± (2 and 2.5) %. The density was obtained from two vibrating tube densimeters, one for operation at $p \approx 0.1$ MPa with an expanded uncertainty ($k = 2$) of about ± 0.1 % and the other one that functioned at pressures up to 70 MPa, with an expanded uncertainty ($k = 2$) of about ± 0.3 %. Measurements of density and viscosity at $p = 0.1$ MPa were conducted with three samples of DIDP each with different purity stated by the supplier and as a function of water mass fraction in the range $(20 \text{ to } 417) \cdot 10^{-6}$. The values obtained agreed within the estimated expanded uncertainties of the measurements. One sample was from the same lot and purity as that used by both Caetano et al. (*J. Chem. Eng. Data* **2005**, *50*, 1875−1878) and Harris and Bair (*J. Chem. Eng. Data* **2007**, *52*, 272−278) for their measurements of viscosity and density. The measured viscosity and density are represented by interpolating expressions with differences between the experimental and calculated values that are comparable with the expanded ($k = 2$) uncertainties. The viscosities at $p = 0.1$ MPa agree with values reported in the literature within the combined estimated expanded ($k = 2$) uncertainties of the measurements while our densities differ by no more than ± 0.15 %. At $p > 0.1$ MPa the only other literature values are those reported by Harris and Bair. Deviations of their values from our smoothing equation increase with increasing pressure to be $< \pm 2$ % at $p < 1$ MPa and between (−9 to 11) % at $p = 50$ MPa; these differences are within 2.5 times the combined uncertainty.

Introduction

Viscometers that are used for performing accredited viscosity measurements must be calibrated through a chain of reference fluids to an international primary standard.[1] As an alternative, the International Association for Transport Properties, previously the Subcommittee on Transport Properties of Commission 1.2 on Thermodynamics of The International Union of Pure and Applied Chemistry (IUPAC), has considered the possibility of recommending other fluids as viscosity standards.[2] The viscosity of water at $T = 293.15$ K and $p = 0.101325$ MPa of (1.0016 ± 0.0017) mPa·s remains the only internationally accepted value.[3] In order to measure the viscosity of more viscous fluids, typically found in industrial applications, additional working reference fluids with higher viscosity are required. The viscosity of these reference fluids are determined by the so-called "step-up" procedure using a series of master capillary viscometers, along overlapping ranges, starting with water as the reference fluid. The measurements are made in National Standards Laboratories or other certified laboratories and result in a set of certified fluids for which the cited uncertainty in viscosity is about ± 0.35 %. Every sequential step in this procedure has an associated uncertainty, and this uncertainty is propagated with the increased number of comparisons.[1] In addition, these reference fluids have limited shelf-life because they are complex mixtures that oxidize and change composition with time.[1] Hence there has been a search for some time for reference fluids that are pure fluids that can be purchased from chemical suppliers.

The Bureau International des Poids et Measures (BIPM) has realized the need for alternative reference fluids and the Intentional Association of Transport Properties (IATP) has instigated a project on "Investigation of a New High-Viscosity Standard".[4] Diisodecyl phthalate (DIDP) has been proposed, by the members of IATP, as a suitable candidate in the viscosity range (100 to 200) mPa·s because it is a liquid over a wide range of temperatures, it is readily available (except in the United States), it has a low volatility, and it is nontoxic.[4] The purpose of this work is to provide reference quality data for the viscosity and density of DIDP over a wide temperature and pressure range on a number of commercial samples with different water content to determine its suitability as a reference material.

The vibrating wire viscometer was used in this work to measure the viscosity of DIDP because it can be operated over a wide range of viscosity, temperature, and pressure.[5−9]

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Measurements were made at temperatures from (298 to 423) K and pressures between (0.1 and 70) MPa, which partly satisfy the requirement, for example, of the petroleum industry for a calibrant at temperatures up to 473 K and pressures below 200 MPa.[7,8] The theory of the vibrating wire requires knowledge of the density of the fluid, so two vibrating tube densimeters have been used to measure the density of DIDP over the same pressure and temperature range.

Experimental Section

Three samples of diisodecyl phthalate, with the chemical formula $C_{28}H_{46}O_4$ and the CASRN 26761-40-0, were used for this work. The suppliers provided mass fraction chemical purities determined by GLC analysis for each sample. The suppliers and their cited purities for each sample are as follows: sample A was obtained from Fluka (lot no. 1228727) with a minimum stated mass fraction purity of 0.999; sample B was acquired from Merck KGaA (lot no. K22132622) with a nominal stated mass fraction purity of 0.998 that is same lot and purity as used by Caetano et al.[10] and Harris and Bair[11] for their measurements of viscosity; and, sample C also originated from Merck KGaA (lot number S4429432) with stated mass fraction purity of 0.99. Harris and Bair[11] have determined from ^{13}C NMR and GC−MS that the Merck KGaA samples of DIDP are isomeric mixtures. DIDP is partially hydrophilic; consequently, we also separately determined the mass fractions of water $w(H_2O)$ in each of the samples as determined by Karl Fischer titration using a Radiometer analytical titrator, TIM 550, with the following results: $w(H_2O, A) = 115\cdot10^{-6}$; $w(H_2O, B) = 417\cdot10^{-6}$; and, $w(H_2O, C) = 236\cdot10^{-6}$. The uncertainty in the water mass fraction was $\delta\{w(H_2O)\} \approx \pm\{0.1\cdot w(H_2O) + 5\cdot10^{-6}\}$. Aliquots of each sample were combined with 0.4 nm molecular sieves, previously dried at a temperature of about 500 K under vacuum, and left for a time > 14 d prior to use. The mass fraction of water in each of these dried samples were also determined by Karl Fischer titration to be as follows: $w(H_2O, A) = 20\cdot10^{-6}$; $w(H_2O, B) = 24\cdot10^{-6}$; and $w(H_2O, C) = 29\cdot10^{-6}$.

Details of the vibrating wire viscometer, with a wire diameter of about 0.15 mm, the working equations, and the analysis procedure have been described previously.[6,9] The expanded uncertainty in the viscosity obtained is, based on our previous work,[6−9] ± 2 %. Over the temperature, pressure, and viscosity ranges of our measurements, the resonance frequency covered the range from (1.58 to 2.05) kHz and the resonance quality factor Q varied from 2.26 at $\eta(308.15\ \text{K}, 50\ \text{MPa}) \approx 135$ mPa·s to 38.5 at $\eta(423\ \text{K}, 0.1\ \text{MPa}) \approx 2$ mPa·s. Figure 1 shows Q^{-1}, which is essentially proportional to the fluid viscosity, obtained from our measurements along with the Q estimated from the working equations (eqs 3 to 9 of ref 5) as a function of viscosity for a wire of radius (0.05 and 0.75) mm formed from tungsten with density of 19 300 kg·m^{-3} tensioned between two rigid clamps so that the fundamental resonance frequency is 1.2 kHz. The Q was estimated from the complex voltage as a function of frequency by a subsequent analysis with the expression reported by Mehl.[12] Figure 1 also includes Q obtained when immersed in the certified reference material for viscosity S20 reported by Kandil et al.,[9] Lundstrom et al.,[7] and Sopkow et al.[8] with wires of radius about (0.05 and 0.075) mm. The measured Q are in reasonable agreement with those calculated from the theory. The differences are, as expected, because the measured resonance frequency is not a constant, it decreases with increasing viscosity, and the method used to estimate Q from the measured $\boldsymbol{V}(f)$ is not intended for low Q resonances.

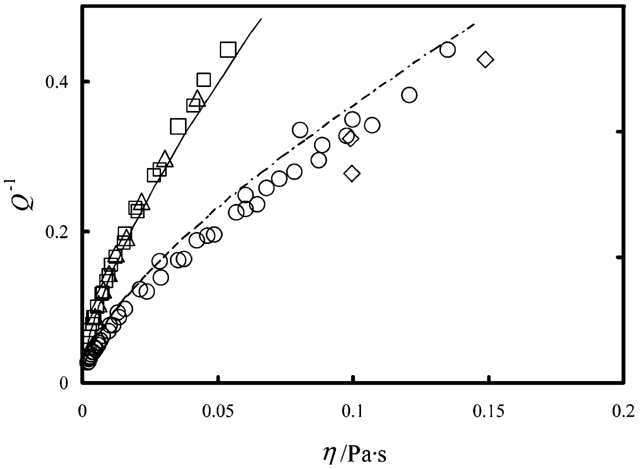

Figure 1. Resonance quality factor Q of a tensioned tungsten wire vibrating at a frequency $f_0 = 1.2$ kHz immersed in a fluid with $\rho = 850$ kg·m^{-3} as a function of viscosity η and wire radius R. —, estimated from the working equations (eqs 3 to 9 of ref 6) for $R = 0.05$ mm; – – –, estimated from the working equations (eqs 3 to 9 of ref 6) for $R = 0.075$ mm; ○, this work immersed in DIDP; △, ref 6 with $R \approx 0.0747$ mm for certified reference fluid N100 at temperatures between (301 and 313) K and $p = 0.1$ MPa; △, ref 7 with $R \approx 0.0479$ mm for certified reference fluid N10 at temperatures between (298 and 333) K and pressures in the range (0.1 to 55) MPa; □, ref 7 with $R \approx 0.0479$ mm for certified reference fluid S20 at temperatures between (298 to 393) K and pressures in the range (0.1 to 55) MPa.

Densities were measured with two Anton Paar vibrating tube densimeters: model DMA 602 H for pressures about 0.1 MPa and model DMA 512 P for high pressures in the range (0.1 to 70) MPa. Prior to commencing measurements, their constants were determined by calibration. For model DMA 512 P, the calibration was performed with water, for which the physical properties were taken from refs 1 and 13, and octane, where the density was taken from ref 14, at temperatures in the range of (298.15 to 432.15) K and pressures between (0.1 to 70) MPa. The calibration was verified with measurements of the density of methylbenzene, for which the density was obtained from ref 15, at temperatures from (298.15 to 423.15) K and pressures over the range (0.1 to 70) MPa. For model DMA 602 H, vacuum and water were used for the calibration from $T = (298.15$ to $363.15)$ K and for water at $p = 0.1$ MPa. The calibration parameters so determined were validated with measurements on methylbenzene. Based on a comparison of the measured and literature values of the third fluid (methylbenzene) over the temperature and pressure range of the calibration, we determined an expanded uncertainty of ± 0.3 % and ± 0.1 % for the model 512 P and model DMA 602 H densimeters, respectively. The working equations for the vibrating wire viscometer require values of density with an uncertainty of ± 2 % to yield an uncertainty of ± 1 % in viscosity. The measured densities were corrected for the observed effect of viscosity discussed elsewhere[7] although only at the highest viscosity studied was the correction barely significant, reaching about 0.05 %.

Temperatures of the densimeters were measured with industrial grade platinum resistance thermometers of nominal resistance 100 Ω that had been calibrated against a standard platinum thermometer of nominal resistance 25 Ω, which itself had been calibrated on ITS-90. The 25 Ω thermometer was used for the measurements of viscosity. Thus, for the density measurements the uncertainty in temperature was estimated to be ± 0.02 K while for viscosity measurement it was estimated at ± 0.01 K.

Pressure was generated with a hydraulic pump and measured in the pressure range (5 to 70) MPa, with a dial gauge (Heise,

Table 1. Densities ρ Obtained with the Vibrating Tube Densimeter (DMA 512 P) along with Viscosities η Determined with a Vibrating Wire Viscometer for Diisodecyl Phthalate Sample B with $w(H_2O) = 417 \cdot 10^{-6}$ at Temperature T and Pressure $p > 0.1$ MPa with Expanded Uncertainties (Confidence Interval 0.95)

T/K	p/MPa	ρ/kg·m^{-3}	η/mPa·s	T/K	p/MPa	ρ/kg·m^{-3}	η/mPa·s	T/K	p/MPa	ρ/kg·m^{-3}	η/mPa·s
298.15	5	962.8 ± 2.9		313.15	50		100.1 ± 2.1	373.15	30	929.0 ± 2.8	7.84 ± 0.17
298.15	10	965.6 ± 2.9	107.3 ± 2.4	323.15	5	945.4 ± 2.8		373.15	50	941.0 ± 2.8	10.33 ± 0.22
298.15	20	970.8 ± 2.9		323.15	10	948.5 ± 2.8	29.24 ± 0.73	373.15	70	952.1 ± 2.9	13.45 ± 0.28
298.15	30	976.0 ± 2.9		323.15	20	954.6 ± 2.9	35.63 ± 0.83	398.15	5	893.4 ± 2.7	
298.15	50	985.5 ± 3.0		323.15	30	960.4 ± 2.9	42.46 ± 0.95	398.15	10	898.8 ± 2.7	3.601 ± 0.079
298.15	70	995.2 ± 3.0		323.15	50	970.7 ± 2.9	60.4 ± 1.3	398.15	20	906.6 ± 2.7	4.120 ± 0.089
303.15	10		78.5 ± 1.9	323.15	70	980.1 ± 2.9	80.6 ± 1.7	398.15	30	913.5 ± 2.7	4.741 ± 0.101
303.15	20		98.0 ± 2.2	348.15	5	927.9 ± 2.8		398.15	50	926.0 ± 2.8	5.98 ± 0.12
303.15	30		120.9 ± 2.6	348.15	10	931.3 ± 2.8	11.55 ± 0.27	398.15	70	938.9 ± 2.8	7.64 ± 0.16
308.15	10		60.3 ± 1.5	348.15	20	938.0 ± 2.8	13.81 ± 0.31	423.15	5	877.5 ± 2.6	
308.15	20		73.1 ± 1.7	348.15	30	944.1 ± 2.8	15.93 ± 0.35	423.15	10	881.6 ± 2.6	2.401 ± 0.052
308.15	30		89.0 ± 2.0	348.15	50	955.3 ± 2.9	21.50 ± 0.45	423.15	20	890.4 ± 2.7	2.707 ± 0.058
308.15	50		135.4 ± 2.9	348.15	70	965.7 ± 2.9	28.86 ± 0.59	423.15	30	898.6 ± 2.7	3.001 ± 0.063
313.15	10		46.5 ± 1.1	373.15	5	911.1 ± 2.7		423.15	50	912.6 ± 2.7	3.857 ± 0.080
313.15	20		57.0 ± 1.3	373.15	10	914.8 ± 2.7	5.92 ± 0.13	423.15	70	926.7 ± 2.8	4.826 ± 0.099
313.15	30		68.1 ± 1.5	373.15	20	922.1 ± 2.8	6.96 ± 0.15				

Table 2. Experimental Densities ρ Obtained with a Vibrating Tube Densimeter (DMA 512 P) along with Viscosities of Sample B with $w(H_2O) = 417 \cdot 10^{-6}$ at Temperature T and $p = 0.1$ MPa with Expanded Uncertainties (Confidence Interval 0.95)

T/K	ρ/kg·m^{-3}	η/mPa·s
298.15	960.1 ± 2.9	87.5 ± 1.8
303.15		64.6 ± 1.3
308.15		48.99 ± 0.98
313.15		37.74 ± 0.76
323.15	941.6 ± 2.8	23.85 ± 0.48
348.15	924.3 ± 2.8	9.72 ± 0.19
373.15	907.0 ± 2.7	5.03 ± 0.10
398.15		3.043 ± 0.061
423.15		2.052 ± 0.041

Table 3. Densities ρ of Samples A, B, and C Obtained with a Vibrating Tube Densimeter (DMA 602 H) along with Viscosities η of Diisodecyl Phthalate with Water Mass Fraction $w(H_2O)$ at Temperatures T and $p = 0.1$ MPa with Expanded Uncertainties (Confidence Interval 0.95)

sample	$10^6\,w(H_2O)$	T/K	ρ/kg·m^{-3}	η/mPa·s
A	20	298.15	963.66 ± 0.97	87.2 ± 1.7
		323.15	945.62 ± 0.95	23.85 ± 0.48
		348.15	927.67 ± 0.94	9.71 ± 0.19
		363.15	916.97 ± 0.93	6.39 ± 0.13
A	115	298.15	963.63 ± 0.97	
		323.15	945.55 ± 0.95	
		348.15	927.66 ± 0.94	
		363.15	916.89 ± 0.93	
B	417	298.15	963.81 ± 0.97	
		323.15	945.64 ± 0.95	
		348.15	927.68 ± 0.94	
		363.15	916.95 ± 0.93	
B	24	298.15	963.85 ± 0.97	
		323.15	945.66 ± 0.95	
		348.15	927.74 ± 0.94	
		363.15	917.07 ± 0.93	
C	236	298.15	964.99 ± 0.97	
		323.15	946.41 ± 0.95	
		348.15	928.51 ± 0.94	
		363.15	917.80 ± 0.93	
C	29	298.15	964.61 ± 0.97	87.3 ± 1.7
		323.15	946.14 ± 0.95	23.84 ± 0.48
		348.15	928.23 ± 0.94	9.71 ± 0.19
		363.15	917.53 ± 0.93	6.39 ± 0.13

Stratford, CT, model CM12524) with a resolution of 0.1 MPa and uncertainty specified as ± 0.25 MPa for the measurements of viscosity while another dial gauge (Heise, Stamford, CT, model 18357) was used for the density measurements with an uncertainty of ± 0.1 MPa. This uncertainty of each gauge was confirmed by calibration against a force balance dead weight gauge (Desgranges et Huot model 21000) with an uncertainty of ± 0.05 MPa.

Results and Discussion

The viscosity and density of DIDP for sample B at $w(H_2O) = 417 \cdot 10^{-6}$ and $p > 0.1$ MPa are given in Table 1 while those for $p = 0.1$ MPa are listed in Table 2; at $T = 298$ K and $p > 10$ MPa, the $Q < 2$ and measurements of viscosity were not performed. This sample with this water content was used for the majority of measurements as this is the most readily available commercial sample and there would be considerable practical advantages if it could be used without drying. Additional density measurements were performed at a pressure of 0.1 MPa with the DMA 602 H densimeter on sample B with water mass fraction $w(H_2O) = 24 \cdot 10^{-6}$, on sample A with $w(H_2O) = 20 \cdot 10^{-6}$ and $w(H_2O) = 115 \cdot 10^{-6}$, and on sample C with $w(H_2O) = 29 \cdot 10^{-6}$ and $w(H_2O) = 236 \cdot 10^{-6}$. The viscosity was measured for sample A with $w(H_2O) = 20 \cdot 10^{-6}$ and sample C with $w(H_2O) = 29 \cdot 10^{-6}$. These values are listed in Table 3. Small corrections have been applied to the reported viscosity and density to reduce all values to the stated temperature for each isotherm.

The uncertainties, listed in Tables 1 to 3, are at a confidence interval of 0.95 ($k = 2$) and were obtained by combining in quadrature uncertainties arising from the instrument, $d\eta/dT$ and $d\eta/dp$ for viscosity and $d\rho/dT$ with $d\rho/dp$ for density. For both viscosity and density the major source of uncertainty (by at least factor of 5) arises from the uncertainty of the viscometer and densimeter calibration. For viscosity, the expanded uncertainty in the vibrating wire viscometer is, based on the work reported in refs 6 to 9, assumed to be ± 2 %, while for density the expanded uncertainty was ± 0.1 % for the DMA 602 H operated at a $p \approx 0.1$ MPa and ±0.3 % for the 512 P at $p > 0.1$ MPa; the uncertainties for both densimeters are as determined from the measurements performed to verify the calibration. The next most significant and quantifiable contribution to the uncertainties arises from $d\eta/dp$ for viscosity and $d\rho/dp$ for density. These derivatives were estimated from a combination of our measured $\eta(p)_T$ with $\delta p = 0.25$ MPa and $\rho(p)_T$ with $\delta p = 0.1$ MPa. The contribution to $\delta\eta$ from δp lies between (0.07 and 0.55) % and decreased with increasing temperature while the $\delta\rho$ from δp was at least a factor of 50 less than for viscosity and never exceeded more than 0.01 %. The contribution to the uncertainty from either $d\eta/dT$ or $d\rho/dT$ was estimated from a combination of $\rho(T)_p$ and $\delta T = 0.02$ K and $\eta(T)_p$ and $\delta T = 0.01$ K. The contribution to $\delta\eta$ from δT was never more than about 0.01 % and for $\delta\rho$ from δT was about a factor of 10 lower. The required

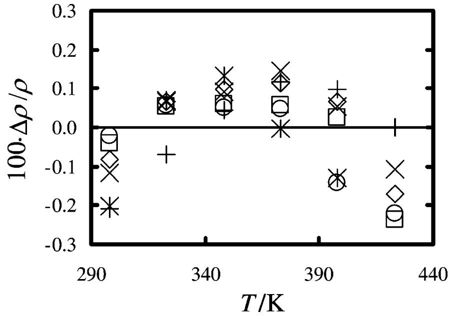

Figure 2. Fractional deviation $\Delta\rho/\rho = \{\rho(\text{expt}) - \rho(\text{calc})\}/\rho(\text{calc})$ of the experimental density $\rho(\text{expt})$ of DIDP of Table 1 from $\rho(\text{calc})$ of eqs 1 to 3 for sample B with $w(H_2O) = 417\cdot10^{-6}$ as a function of temperature T. ○, $p = 5$ MPa; □, $p = 10$ MPa; ◇, $p = 20$ MPa; ×, $p = 30$ MPa; ∗, $p = 50$ MPa; +, $p = 70$ MPa. The scale of the ordinate axis is equal the expanded uncertainty in ρ of ± 0.3 %.

derivatives were estimated from a preliminary analysis of the results. The densities obtained from the two vibrating tube densimeters at the overlapping pressure agreed within the combined expanded uncertainty.

To represent the densities ρ, the modified Tait equation[16] was used in the form

$$\{\{\rho(p) - \rho_r(p_r)\}\}/\{\rho(p)\} = C\,\lg\{\{(B + p)\}/\{(B + p_r)\}\} \quad (1)$$

where B is a function of temperature given by

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

In eq 1, $p_r = 0.1$ MPa and $\rho_r(p_r)$ is the density listed in Table 2 that was represented by

$$\rho_r/\text{kg}\cdot\text{m}^{-3} = \sum_{i=0}^{2} A_i(T/\text{K})^i \quad (3)$$

with a standard deviation of the mean $100\cdot\sigma(<\rho>)/\rho = \pm\ 0.015$ when the parameters were $A_0 = 1228.371$, $A_1 = -1.05498$, and $A_2 = 5.20\cdot10^{-4}$. The $\rho_r(p_r)$ obtained from eq 3 were combined with the densities of Table 1, both obtained for sample B with $w(H_2O) = 417\cdot10^{-6}$, to determine the adjustable parameters of eqs 1 and 2 with the results $b_0 = 295.7572$ MPa, $b_1 = -0.299083$ MPa, $b_2 = -0.000343$ MPa, and $C = 0.111608$ that represented the data with $100\cdot\sigma(<\rho>)/\rho = \pm\ 0.1$. The densities from Tables 1 are shown relative to the smoothing eqs 1 to 3 in Figure 2 where the scale of the ordinate axis is equal to the expanded uncertainty given in Table 1. All differences shown in Figure 2 are within the expanded uncertainty of our measurements of ± 0.3 % for the densimeter designated DMA 512 P. Our measurements of density at $p >$ 0.1 MPa of Table 1 were used, prior to publication, by Harris and Bair[11] to assist with determining the parameters of a Hayward-type[16] equation for the pressure and temperature dependence of the density that was used to determine the viscosity from their falling sinker viscometer.

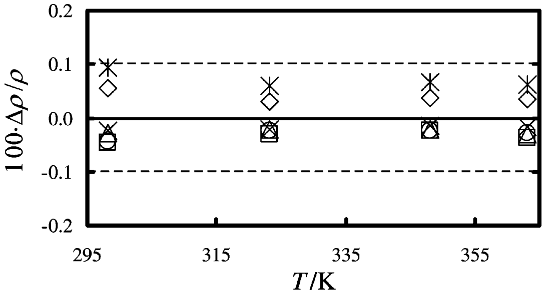

Figure 3. Fractional deviation $\Delta\rho/\rho = \{\rho(\text{expt}) - \rho(\text{calc})\}/\rho(\text{calc})$ of the experimental density $\rho(\text{expt})$ for DIDP of Table 3 from $\rho(\text{calc})$ of eq 3 at $p = 0.1$ MPa as a function of temperature T. ○, this work, sample A with $w(H_2O) = 20\cdot10^{-6}$; □, this work, sample A with $w(H_2O) = 115\cdot10^{-6}$; △, this work, sample B with $w(H_2O) = 417\cdot10^{-6}$; ×, this work, sample B with $w(H_2O) = 24\cdot10^{-6}$; ∗, this work, sample C with $w(H_2O) = 236\cdot10^{-6}$; ◇, this work, sample C with $w(H_2O) = 29\cdot10^{-6}$. The dashed lines are the expanded uncertainties in our measurements obtained with DMA 602 H.

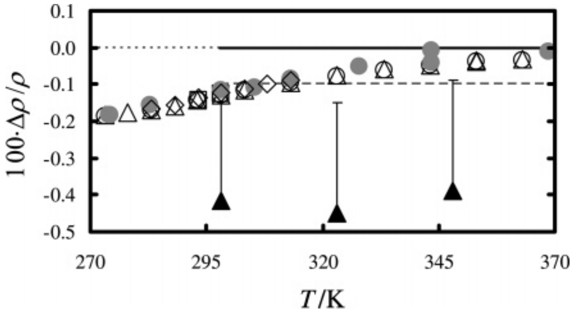

Figure 4. Fractional deviation $\Delta\rho/\rho = \{\rho(\text{expt}) - \rho(\text{calc})\}/\rho(\text{calc})$ of the experimental density $\rho(\text{expt})$ for DIDP from $\rho(\text{calc})$ of eq 3 at $p = 0.1$ MPa with the coefficients obtained from the results listed in Table 3 as a function of temperature T. ▲, this work, sample B with $w(H_2O) = 417\cdot10^{-6}$ obtained with the DMA 512 P with expanded uncertainty of ± 0.3 % for this instrument; ◇, ref 10, sample with purity of 0.995 and $w(H_2O) = 20\cdot10^{-6}$; □, ref 17, sample with purity of 0.998 and $w(H_2O) = 107\cdot10^{-6}$; ○, ref 11, sample with purity of 0.99 dried over 0.3 nm molecular sieve with unspecified $w(H_2O)$; △, ref 11, sample with purity of 0.998 dried over 0.3 nm molecular sieve with unspecified $w(H_2O)$; and gray circle, ref 18, sample with purity of 0.998. The dashed line at −0.1 is the expanded uncertainty in our measurements obtained with the DMA 602 H while that at 0 indicates an extrapolation of eq 3 to temperatures below our measurements to which the parameters of eq 3 were adjusted.

The densities ρ given in Table 3, obtained from DMA 602 H, for $w(H_2O)$ in the range (20 to 417)$\cdot10^{-6}$ and for samples of stated purity of 0.99, 0.998, and 0.999 were also fit to eq 3 with $100\cdot\sigma(<\rho>)/\rho = \pm0.04$ when the parameters were adjusted to $A_0 = 1201.056$, $A_1 = -0.856$ and $A_2 = 2.04\cdot10^{-4}$. The densities of Table 3 are shown relative to eq 3 with these parameters in Figure 3 and all lie within the expanded uncertainty of ±0.1 %. However, the densities obtained for sample C, with the lowest cited mass fraction purity of 0.99 used in this work, and both $w(H_2O)$ of (24 and 236)$\cdot10^{-6}$, are about 0.1 % above the values obtained for samples A and B. Based on our measurements we conclude, at the expanded uncertainties of our measurements, that there is no measurable difference between the densities of different purities of DIDP and $w(H_2O)$ in the range of (20 to 417)$\cdot10^{-6}$.

The $\rho(p = 0.1\text{ MPa})$ reported by other workers[10,11,17,18] are shown as deviations from eq 3 with parameters adjusted to the densities obtained with the DMA 602 H in Figure 4 that has an ordinate axis compressed by a factor of 1.5 as compared with that of Figure 3. In the overlapping temperature range, the literature values are all systematically below those obtained from the DMA 602 H but within the combined uncertainties. In particular, the values reported by Caetano et al.,[10,17] which were also obtained with a vibrating tube densimeter, differ from our densities by no more than −0.15 %, which is within the uncertainty of our measurements at a confidence interval of 0.995. The densities obtained with the DMA 512 P at $p = 0.1$ MPa and listed in Table 2, also shown in Figure 4, lie about 0.4 % below those obtained from the DMA 601 H but within the combined expanded uncertainties.

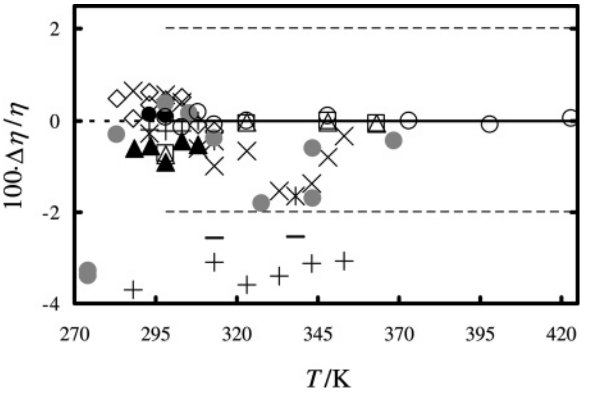

Figure 5. Relative differences $\Delta\eta/\eta = \{\eta(\text{expt}) - \eta(\text{calc})\}/\eta(\text{calc})$ of the experimentally determined viscosity $\eta(\text{expt})$ at $p = 0.1$ MPa for different DIDP purity and water mass fraction w, from the value obtained from eq 4 $\eta(\text{calc})$ as a function of temperature T. △, this work, sample A with $w(H_2O) = 20\cdot10^{-6}$; ○, this work, sample B with $w(H_2O) = 417\cdot10^{-6}$; □, this work, sample C with $w(H_2O) = 29\cdot10^{-6}$; ▲, ref 4, sample with purity of 0.995 and $w(H_2O) = 20\cdot10^{-6}$; ◇, ref 10, sample with purity of 0.998 with $w(H_2O) = 20\cdot10^{-6}$; ●, ref 17, sample with purity of 0.998 and $w(H_2O) = 107\cdot10^{-6}$; +, ref 11, sample with purity of 0.99 and unspecified $w(H_2O)$ obtained with the Canberra viscometer; ×, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer; ∗, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer; —, ref 11, obtained from ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer; and gray circle, ref 18, sample with purity of 0.998. The dashed lines at ± 2 are the expanded uncertainties in our measurements while those at 0 indicate an extrapolation of eq 4 to temperatures below our measurements to which the parameters of eq 4 were adjusted.

Using the rule of Vogel,[19] the viscosity $\eta(p = 0.1\text{ MPa})$ listed in Tables 2 and 3 were represented by

$$\eta_0/\text{mPa}\cdot\text{s} = \exp[e + f/\{g + (T/\text{K})\}] \qquad (4)$$

with $100\cdot\sigma(<\eta>)/\eta = 0.15$ when the constants were adjusted to be $e = -2.6164$, $f = 787.0715$, and $g = -187.1792$. The relative deviations of the measurements from eq 4, shown in Figure 5, are all within the estimated expanded uncertainty. The $\eta(p = 0.1\text{ MPa})$ reported by Caetano et al.,[4,10,17] with a vibrating wire and an expanded uncertainty of about ± 1 %, and by Harris and Bair[11] with falling sinker viscometers with an expanded uncertainty of between ± (2 and 4) % are shown in Figure 5 at temperatures that overlap our measurements. The sample used in refs 10, 11, and 17 were from the same lot as our sample B, albeit of different water mass fraction. The viscosity from refs 10, 11, and 17 agree with eq 4 within the combined uncertainty albeit with two measurement sets reported by Harris and Bair[11] systematically low by between (2.5 and 3) % but within the combined uncertainty. Nevertheless, the agreement between the results obtained from different techniques on samples of different purity and water mass fraction that varied from $(20\text{ to }417)\cdot10^{-6}$ is considered important for the selection of DIDP as a reference material for viscosity.

For DIDP sample B with $w(H_2O) = 417\cdot10^{-6}$, the $\eta(T, p)$ with $p > 0.1$ MPa of Table 1 were fit by the empirical Vogel−Fulcher−Tammann (VFT) equation:[19−21]

$$\eta(T,p)/\text{mPa}\cdot\text{s} = \exp\left\{a + b(p/\text{MPa}) + \frac{c + d(p/\text{MPa}) + e(p/\text{MPa})^2}{(T/\text{K}) - T_0}\right\} \qquad (5)$$

with $100\cdot\sigma(<\eta>)/\eta = \pm 1$ %, and the six parameters obtained are listed in Table 4. The viscosity listed in Table 1 for sample B with $w(H_2O) = 417\cdot10^{-6}$ are shown as a function of η in Figure 6 as relative deviations from eq 5. All the measurements lie within the estimated expanded uncertainty of between ± (2 and 2.5) % except for $\eta(308.15\text{ K}, 50\text{ MPa}) \approx 135$ mPa·s when the difference is +2.8 % and the estimated expanded uncertainty was ± 2.1 %.

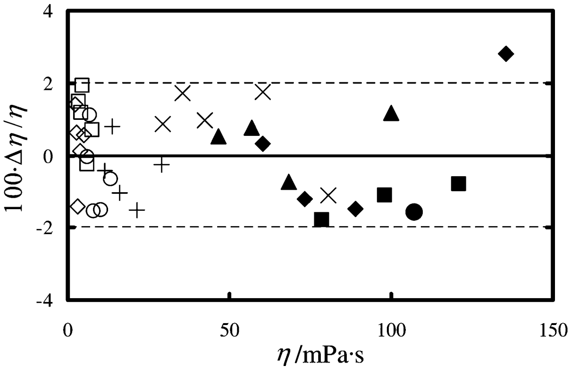

Figure 6. Relative deviations $\Delta\eta/\eta = \{\eta(\text{expt}) - \eta(\text{calc})\}/\eta(\text{calc})$ of the experimentally determined viscosity $\eta(\text{expt})$ for sample B (of purity 0.995) and $w(H_2O) = 417\cdot10^{-6}$ from the value $\eta(\text{calc})$ obtained from eq 5 with coefficients of Table 4 as a function viscosity η. ●, $T = 298$ K; ■, $T = 303$ K; ◆, $T = 308$ K; ▲, $T = 313$ K; ×, $T = 325$ K; +, $T = 348$ K; ○, $T = 373$ K; □, $T = 398$ K; ◇, $T = 423$ K. The dashed lines are the expanded uncertainties in our measurements.

Table 4. Coefficients of Equation 5 for the Viscosity of Sample B of Diisodecyl Phthalate Listed in Table 1 with $w(H_2O) = 417\cdot10^{-6}$

a	b	c	d	e	T_0
−2.61259	0.00429	792.27258	2.14952	−0.00469	186.15040

The only measurements of viscosity of DIDP as a function of pressure, of which we are aware, are those reported by Harris and Bair[11] from work with three falling body viscometers: (1) designated in ref 11 as the Canberra viscometer with an expanded uncertainty of ± 2 %; (2) the so-called Atlanta alpha-viscometer in ref 11 with an upper operating pressure of 450 MPa and an estimated uncertainty of ± 3 % at an unspecified confidence interval; and (3) the Atlanta viscometer given the acronym MHP in ref 11 capable of measurements at pressures up to 1 GP with an estimated uncertainty of ± 4 %. Two sinkers of nominal diameters (6.0 and 6.3) mm were used with the Canberra viscometer. The actual diameters of the sinkers were determined by calibration. For the nominal 6.0 mm diameter sinker, the effective area was determined from measurements at viscosities from (0.3 to 201) mPa·s with water, methylbenzene, octane, and certified reference fluids for viscosity while for the nominal 6.3 mm diameter sinker the calibration was performed at viscosities between (51 and 2 966) mPa·s with certified reference fluids for viscosity.[22−25] The calibrations described in refs 22 to 25 were at $T = 298.15$ K and $p \approx 0.1$ MPa at viscosities (and Reynolds number) that include those of DIDP; the thermal expansion and compressibility of the sinker were included in the analysis. At a viscosity, the results reported by Harris and Bair[11] from all three viscometers agree within their estimated uncertainties. In particular the measurements that overlap our range from the Canberra and Atlanta alpha-viscometers are compared with eq 5 in Figure 7 as a function of pressure. One measurement obtained from the Canberra viscometer, at $T = 313.15$ K and $p = 50.6$ MPa, lies 10.4 % above eq 5, about 2.5 times the combined uncertainty. The viscosity reported from the Atlanta alpha-viscometer at pressures of (25 and 50) MPa at three temperatures of (313.15, 338.15, and 373.15) K from two different DIDP samples lie within ± 9 % of eq 5 that is about 2 times the combined uncertainty; if the uncertainty cited in ref 11 for the Atlanta alpha-viscometer

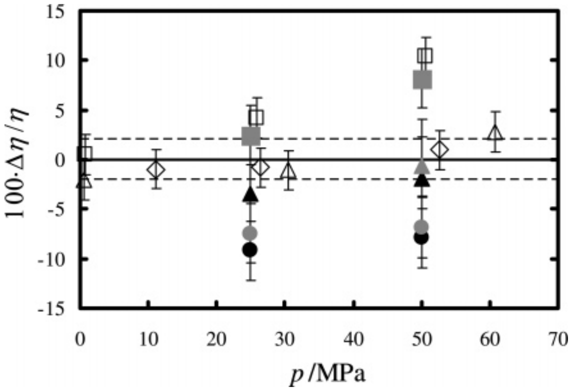

Figure 7. Relative deviations $\Delta\eta/\eta = \{\eta(\text{expt}) - \eta(\text{calc})\}/\eta(\text{calc})$ of the experimentally determined viscosity η(expt) from the value η(calc) obtained from eq 5 with coefficients of Table 4 as a function of pressure p at temperatures and pressures that overlap ours. □, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer at $T = 313$ K; △, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer at $T = 338$ K; ◇, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer at $T = 348$ K; ■, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 313$ K; ▲, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 338$ K; ●, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 373$ K; gray filled square, ref 11, ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 313$ K; gray filled triangle, ref 11, ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 338$ K; gray filled circle, ref 11, ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 373$ K. The dashed lines are the expanded uncertainties in our measurements and the error bars are those cited in ref 11.

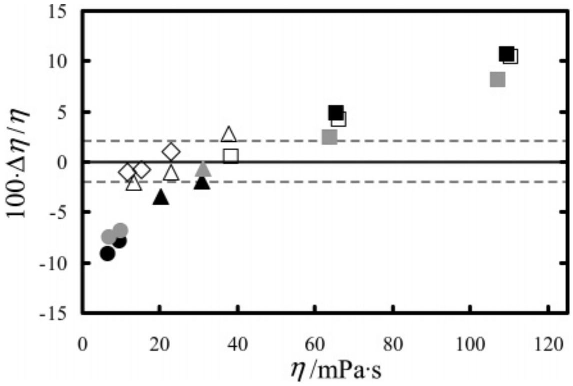

Figure 8. Relative deviations $\Delta\eta/\eta = \{\eta(\text{expt}) - \eta(\text{calc})\}/\eta(\text{calc})$ of the experimentally determined viscosity η(expt) from the value η(calc) obtained from eq 5 with coefficients of Table 4 as a function of viscosity η at temperatures and pressures that overlap ours. □, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer at $T = 313$ K; △, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer at $T = 338$ K; ◇, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Canberra viscometer at $T = 348$ K; ■, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 313$ K; ▲, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 338$ K; ●, ref 11, sample with purity of 0.998 and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 373$ K; gray filled square, ref 11, ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 313$ K; gray filled triangle, ref 11, ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 338$ K; gray filled circle, ref 11, ABCR GmbH sample and unspecified $w(H_2O)$ obtained with the Atlanta alpha-viscometer at $T = 373$ K. The dashed lines are the expanded uncertainties in our measurements.

was standard, then the expanded uncertainty ($k = 2$) would be ± 6 % and those measurements would then differ from eq 5 by about 1.1 times the combined uncertainty. Nevertheless, the deviations in Figure 7 increase with increasing pressure from $< \pm 2$ % at $p < 1$ MPa to be between (−9 to 11) % at $p = 50$ MPa. When the differences are plotted as a function of viscosity, as shown in Figure 8, the results from ref 10 deviate from eq 5 systematically from (−9 to 11) % and suggest a systematic error rather than an underestimated uncertainty. Unfortunately, no measurements have been performed by us to eliminate plausible causes for these differences, which include unaccounted for instrumental errors and variations in chemical composition of the fluid. However, we will comment on our observations based on our measurements with cyclopentane,[26] methylbenzene,[5] and four certified reference fluids for viscosity (N10,[6] N100,[6] S20,[9] and S60[27]). Both vibrating wire and falling body viscometers were used for the measurements of the viscosity of S20.[9]

For cyclopentane where the viscosity varied from (0.3 to 0.8) mPa·s, our results[26] obtained with the vibrating wire viscometer differed from those of Harris et al.[28] from measurements with a falling sinker viscometer, in the overlapping temperature and pressure range, by no more than ± 1.5 %, which is less than the expanded uncertainty in both sets of measurements. For methylbenzene, over the viscosity range of our measurements[6] of (0.3 to 0.6) mPa·s determined with the same vibrating wire as well as those by Harris et al.[29] agree within ± 2 % with the correlation of Assael et al.[15] However, the values reported in ref 29 showed the greatest deviation from ref 15.

For Cannon Instruments, USA, lot no. 5401 of certified reference material for viscosity S20 measurements were performed with both the vibrating wire viscometer used for this work and the Canberra falling body viscometer with sinkers of diameter (6.0 and 6.3) mm. For this fluid the vibrating wire measurements covered the viscosity range from (1.5 to 80) mPa·s while for the falling sinker the viscosity varied from (5.3 to 1231) mPa·s. In the overlapping viscosity range the results obtained from the vibrating wire and both sinker diameters differed by between (2 and 5.5) %. This difference increased with increasing viscosity but, in the worst case, was within 1.4 times the combined uncertainty. Based on these comparisons, we conclude that there may be an error that increases with increasing pressure that is larger for fluids of higher viscosity and might arise from a systematic error in either one or both of the experimental techniques.

Harris and Bair[11] observed differences in the viscosity of DIDP, obtained with falling body viscometers, from samples with purities of 0.99 and 0.998 that increased with increasing pressure. They speculated that these differences might have arisen from variations in the isomeric composition of DIDP. The viscosity reported for S20 sourced from different lots and suppliers varies by about ± 5 %.[7−9] S20 is a mixture of unspecified hydrocarbons, the exact constituents and composition are proprietary, and presumably differs from lot to lot and from supplier to supplier. This fluid is required to have a nominal viscosity at $T = 298$ K and $p = 0.1$ MPa of 29 mPa·s and a specified temperature dependence within the cited uncertainty of the certified values. We conclude that there are plausible variations in chemical composition that could give rise to the observed variations in viscosity.

We have used the vibrating wire viscometer at the lowest resonance quality factor of 2.3. Harrison et al.[25] have used a vibrating wire viscometer to measure the viscosity of certified reference material for viscosity S60 at temperatures between (273 and 373) K and a pressure of 0.1 MPa where the viscosity varies from (0.0062 to 0.652) Pa•s, which includes the viscosity range for DIDP, and the Q varied between (1.02 and 20.8). The viscosities determined in ref 27 differ by less than ± 3 % from the values obtained with capillary viscometers by the supplier and suggest that viscosities can be obtained from the vibrating wire viscometer operated at a Q of 2.3. Operation at higher Q has been confirmed in refs 6 and 26. The uncertainty cited by Harrison et al.[27] is about 1 % greater than ours, and this difference arises because in ref 27 $\delta T = \pm 0.3$ K and gave rise to an additional $\delta\eta$ of between (0.3 to 0.7) % that decreased with increasing temperature and decreasing viscosity.

Nevertheless, we conclude that DIDP can serve as an adequate calibrant of instruments intended to measure the viscosity at $p = 0.1$ MPa with an uncertainty of about ± 2 % and for $p > 0.1$ MPa within about ± 10 %; these uncertainty statements are equivalent to those reported for S20.[9] We also recommend further measurement and analysis of the differences observed in the viscosity obtained from the vibrating wire and falling sinker viscometers.

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