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Density of Diisodecyl Phthalate at Temperatures from (283.15 to 363.15) K and Pressures from (0.1 to 65) MPa

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Diisodecyl phthalate (DIDP) has recently been proposed to be an industrial reference fluid for moderately high viscosity at atmospheric pressure. Moreover, some results of the viscosity and also of the density of DIDP at high pressures have been reported recently. The main aim of the present work is to contribute with necessary data to enable the eventual proposal of DIDP to be a reference fluid for viscosity at high pressures. In this work, we describe measurements of the density of DIDP at 12 temperatures from (283.15 to 363.15) K and at pressures from (0.1 to 65) MPa. The measurements were performed using an Anton Paar GmbH model DMA HP vibrating tube densimeter with a reading device (model DMA 5000 unit). Prior to the DIDP measurements, the apparatus was calibrated with water and toluene. The sample of DIDP was obtained from Merck KGaA, with a minimum purity of 99.8 % by gas chromatography (GC). The results of the density of DIDP were correlated by a modified Tait equation. The relative root-mean-square deviation (rmsd) of the reference data from the correlation is ± 0.005 % with practically zero *bias*. The effects of the viscosity of the sample on the density measurements are discussed and taken into account in the estimated combined uncertainty of the present results. However, no corrections of the present measurements have been performed. The relative uncertainty of the present measurements is estimated to be less than ± 0.3 % at a 2σ level. This estimate is partly based on the tests performed with dichloromethane at temperatures within the range (283.15 to 333.15) K and pressures from (0.1 to 65) MPa. The present results are compared with literature density data of DIDP samples with purity equal to or greater than 99.8 % (GC), performed at either atmospheric pressure or pressures higher than 0.1 MPa. The literature data sets comprise measurements performed using vibrating tube instruments and pycnometry and results calculated from speed of sound measurements. The literature data were found to be, generally, in agreement with the present results, within their nominal relative uncertainty, except for one of the vibrating tube data sets that has a maximum relative deviation (−0.4 %), which is slightly larger than its claimed relative uncertainty.

Introduction

The viscosity of diisodecyl phthalate (DIDP) at atmospheric pressure has recently been studied aiming at its proposal as a reference fluid.[1−3] The final proposal of DIDP as an industrial reference fluid for viscosity at atmospheric pressure, with viscosity of about 125 mPa•s at 298.15 K,[4] was made under guidance of the International Association for the Transport Properties (http://transp.cheng.auth.gr/iatp.html), in the sequence of efforts started by the Bureau International de Poids et Measures (BIPM) to search for high viscosity reference fluids.[4−7] For that purpose, a significant number of articles have been published on the properties of DIDP at atmospheric pressure.[1,2,5,7−13] Moreover, some recent works have presented high pressure data for viscosity[5,14] and for both density and viscosity.[6,9−11,15] These studies widen the prospects for the use of DIDP as a viscosity standard, also at high pressures, which would satisfy in part the oil industry's need for reference values for viscosity.[16]

One limitation of DIDP, regarding its use as a reference liquid, lies in the possibility of thermal degradation, which has been reported by Fröba and Leipertz[7] to occur at temperatures above 373 K. This has led to a recommended practical upper temperature of 333 K for its manipulation and use as a reference fluid.[4] The present study presents results of the density of the DIDP up to 368 K, below the former temperature limit, although above the latter recommendation. No apparent changes, particularly in density, have been observed after submitting the sample to the highest temperature reached in the present measurements. A further problem regarding the utilization of DIDP as a reference fluid for viscosity is concerned with the fact that it is commercially available as a mixture of isomers. It should be pointed out, however, that this matter has been thoroughly discussed previously,[4] and the differences in the viscosity and the density found between samples of different sources with the same purity, as determined by gas chromatography, were within the nominal uncertainty of the respective measurements.[4]

Experimental Section

Fluid Samples. The diisodecyl phtalate (DIDP) sample was obtained from Merck KGaA, GR, acc. to DIN 752001, batch K22132622, with a minimum purity of 99.8 % by gas chromatography (GC), according to its certificate[17] of quality. It is noteworthy that this is the same source and nominal purity as the samples used previously by several authors to measure the viscosity[2,4,5,7,8,13,14] and also by Al Motari et al.[6] (sample B of ref

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6), Paredes et al.[15] (samples B and C of ref 15), and Peleties and co-workers[9−11] to measure the density and viscosity at high pressures.

The DIDP sample used for the density measurements was pretreated with 0.4 nm molecular sieves from Riedel-deHaën for water removal. The water content was monitored prior to and after the measurements using the Karl Fischer coulometric titration technique (Aquapal, model III, U.K.). Both results indicated a water mass fraction lower than $25 \cdot 10^{-6}$, meaning that no water contamination occurred during the measurements.

The toluene used in calibration was supplied by Sigma-Aldrich, Chromasolv Plus, to HPLC, minimum purity of 99.98 %, and the water was distilled and purified with the Millipore Milli-Q Plus system (resistivity $\geq$ 18.2 M$\Omega \cdot$cm). The dichloromethane used in the tests was supplied by Lab-Scan, Analytical Reagent (minimum mass purity of 99.8 %). It should be noted that dichloromethane is in the list of substances “reasonably anticipated to be human carcinogens” as indicated by the eleventh edition of the Report on Carcinogens (http://ntp.niehs.nih.gov/ntp/roc/eleventh/profiles/s066dich.pdf).

Equipment. The density measurements were performed with an Anton Paar DMA HP vibrating tube densimeter, using a model DMA 5000 unit as the reading device. The nominal repeatability of the density results is, at optimum conditions, $1 \cdot 10^{-5}$ g$\cdot$cm^{-3}. The measurement of the period of oscillation and the temperature control are built in the measuring system. The temperature was measured with a nominal uncertainty of ± 0.05 K. The pressure was created by a compressor (High Pressure Equipment, 60 000 psi) and measured using a Setra Systems Inc. transducer, model 280E (0 to 10 000) psig, with a nominal uncertainty of 0.11 % full scale.

The ancillary equipment, comprising the compressor, transducer, and pressure line, was kept in a wooden case to avoid direct contact with ambient air. The temperature of several components was controlled by the circulation of an external thermostatic fluid in plastic tubes coiled around the various pieces of equipment.

Densimeter Calibration and Tests. The calibration of the densimeter was carried out with water and toluene. The temperature ranges of the calibration were (278.15 to 368.15) K for water and (283.15 to 368.15) K for toluene. The corresponding pressure ranges were (0.1 to 70) MPa for both liquids. The reference data were taken at 13 and 12 equally spaced isotherms for water and toluene, respectively. A total number of 584 data points were used for the calibration. The reference density data used for water and toluene were obtained from Wagner and Pruss[18] and Assael et al.,[19] respectively. The calibration data were correlated by the equation[20]

$$\rho = \sum_{i,j,k} a_{ijk} P^i t^j p^k \tag{1}$$

where ρ is the reference density; P is the oscillation period; t is the temperature; p/MPa is the pressure; and a_{ijk} are fitting parameters, with $i = 0, 2$; $j = 0, 1, 2$; $k = 0, 1, 2$. The reference data have a relative root-mean-square deviation, *rmsd*, of 0.004 % and practically zero *bias*, according to the following definitions

$$\mathrm{rmsd} = \left[\frac{1}{N}\sum_i^N \left(\frac{\rho_i}{\rho_{\mathrm{calc},i}} - 1\right)^2\right]^{1/2} \tag{2}$$

$$bias = \frac{1}{N}\sum_i^N \left(\frac{\rho_i}{\rho_{\mathrm{calc},i}} - 1\right) \tag{3}$$

where ρ_i is the ith density datum; $\rho_{\mathrm{calc},i}$ is the density value calculated by means of eq 1 for the same temperature and pressure; and N is the number of data points used to calculate the fitting parameters. For the calibration, ρ_i stands for the ith reference density datum, and so forth.

The calibration was tested by measuring the density of dichloromethane along the ranges (283.15 to 333.15) K and (0.1 to 66) MPa and comparing with the reference data reported by Cibulka et al.[21] The rmsd and *bias* of the data are 0.07 % and 0.06 %, respectively. The maximum relative deviation found was 0.19 % and the minimum relative deviation was −0.04 %.

The relative uncertainty of the present measurements is estimated to be ± 0.3 % at a 2σ level. This estimate is partly based on the tests performed with dichloromethane. The other source of uncertainty taken into account stems from the influence of the viscosity of the samples on the density measurements, associated with the use of U-tube instruments. For the reasons explained in the following section, the present results have not been corrected for viscosity effects. The estimate of the contribution of that source of uncertainty was based on empirical accounts of the effect as described in the literature[15,16,22] and discussed below.

Effect of Viscosity on the Density Results. Some of the measurements of the density of DIDP at atmospheric pressure reported previously were performed with Anton Paar DMA 5000 vibrating U-tube densimeters,[2,7,8,16] possessing a built-in method for correction of viscosity effects on the density results.[20] However, the instrument used in the present measurements (Anton Paar DMA HP) does not possess a built-in correction for that effect. This is the usual case for density measurements at high pressures, using U-tube instruments.

The need to correct density measurements performed by vibrating U-tube densimeters, to account for effects due to significant differences between the viscosity of the samples and the viscosity of the calibrating fluids, has been discussed by several authors.[15,16,22−24] A theoretical description of the effect can be found in Retsina et al.[25] Those authors studied the mechanical problem of the oscillations of a tube filled with liquid, surrounded by a vacuum. The situation is paralleled with that of vibrating tube densimeters, and the authors show that the density of the fluid can be obtained from measurements of the resonant frequencies of the tube filled with liquid and under vacuum, if the internal damping coefficient, Δ_0, is known. The latter is determined by the material and the dimensions of the tube and also by the properties of the liquid sample contained in it. A quantitative theoretical description of the effect of the viscosity on the density measurements would, therefore, require the development of relation between the viscosity of the sample and the internal damping of the particular tube used for the density measurements. However, no theoretical approach of the subject, leading to a quantitative relation of that kind, could be found in the literature.

The empirical approach has been referenced as a possibility by several authors,[15,22−24] and some of them have used information provided by the supplier of the densimeter, namely, by Anton Paar GmbH on the models (DMA 521 P[6] and DMA HPM[15]).

Anton Paar GmbH supplied[26] a graphical description of that effect for the model of densimeter used in the present work (model DMA HP), including a few points at various viscosities. There are, however, two main difficulties regarding the correction of the present measurements using such an approach. The first impediment lies in the lack of viscosity data in part of the range of the present measurements, namely, at, simultaneously, pressures above 0.1 MPa and temperatures below 293.15 K. Second, it should be pointed out that the sets of high pressure viscosity data that could be found in the literature[5,6,14,15] at temperatures above 293.15 K evidence a mutual significant disagreement. In particular, the deviations between the data sets obtained by Harris and Bair[5] and Al Motari et al.,[6] as pointed out by the latter authors, attain the order of 10 % which is significantly larger than the estimated uncertainties of the measurements. It is the view of the present authors that the current situation recommends publication of the raw data, without any attempt to correct for the viscosity effect. It is, therefore, hoped that the present density data may be corrected after an assessment of the viscosity data on DIDP, over their measurement temperature and pressure ranges. Simultaneously, it is also hoped that a theoretically based approach to correct for viscosity effects on the density results of vibrating tube instruments may be developed in the near future.

For the reasons mentioned above and for the purpose of establishing an upper limit of the contribution of viscosity effects to the combined uncertainty of the present results, only an estimate of the order of magnitude of those corrections is provided. For the purpose of estimating the uncertainty introduced for not correcting the effect of viscosity on the present density measurements, the correlation equation for the high pressure viscosity data as a function of temperature and pressure published by Harris and Bair[5] has been used. The estimated relative value for that correction for the present results varies from −0.01 %, at the highest temperatures, to −0.12 %, at $T = 283.15$ K and 65 MPa, where the latter value has been obtained by extrapolation of the viscosity data.[5] It must be stressed that the highest correction factors occur at the lowest isotherm of the present measurements (283 K), which means that the viscosity data used for that estimate are extrapolated and, therefore, the corresponding uncertainty of the correction is indeed unknown.

A comparison of the present results obtained at 0.1 MPa with the density measurements previously reported[2] at the same pressure carried out with a model DMA 5000 Anton Paar apparatus, which possesses a built-in correction for the viscosity effect, is made in the following section. This provides an estimate of the error incurred by not performing the viscosity correction of those results. A further assessment of the same uncertainty concerning the whole set of the present data is provided by the comparison with the density measurements performed by Peleties et al.[10] by picnometry and calculated from speed of sound measurements, which are free of viscosity effects.

Results

The results obtained for the density of DIDP along the temperature range (283.15 to 363.15) K and pressures (0.1 to 65) MPa are shown in Table 1.

The data were fitted to a modified Tait equation of the form

$$\rho = \rho_0(T)\left(1 - C \ln \frac{D(T) + p}{D(T) + p_0}\right)^{-1} \tag{4}$$

where $\rho_0(T)$ is the density at a reference pressure, $p_0 = 0.1$ MPa, and temperature T; and D and C are empirical parameters. The parameter C was taken to be temperature independent, and the parameter $D(T)$ was described by a polynomial of the form

$$D(T) = \sum_{i=0}^{3}\left[d_i\left(\frac{T}{T_c}\right)^i\right] \tag{5}$$

as suggested by Dymond and Malhotra.[27] The parameters d_i are listed in Table 2 and $C = 0.08710$.

The temperature dependence of ρ_0 was described by the equation

$$\rho_0(T) = \sum_{i=0}^{2} b_i T^i \tag{6}$$

with a rmsd = ±0.004 % and virtually zero *bias*. The values obtained for the parameters b_i are listed in Table 2.

The deviations of the measured data from the correlation eqs 4 to 6 are characterized by a rmsd of ± 0.005 % and virtually zero *bias*. These statistical parameters were obtained by eqs 2 and 3, with ρ_i standing for the *i*th experimental density datum and $\rho_{\mathrm{calc},i}$ the corresponding value calculated by eqs 4 to 6. The relative maximum deviation does not exceed ± 0.02 %.

Figure 1 shows a deviation plot of the experimental data from the correlation eqs 4 to 6.

Comparisons and Discussion

In this section, we restrict the comparisons of the present results with data obtained with DIDP samples having purity equal to or higher than 99.8 % by gas chromatography. Most of the published density data of DIDP have been measured at atmospheric pressure. Figure 2 shows the deviations of atmospheric pressure literature density data from the correlation eq 6 of the present results. The correlation previously proposed by Caetano et al.[4] has negative deviations from the present work ranging from −0.09 % to −0.04 %. That correlation includes data sets obtained by Fröba and Leipertz,[7,12] Bauer,[13] Harris and Bair,[5] Caetano et al.,[2,8] Peleties and Trusler,[9] and Peleties[11] with a rmsd of $4.9 \cdot 10^{-3}$ % and *bias* of $-0.2 \cdot 10^{-3}$ %. The agreement is therefore excellent, as the relative deviations do not exceed ± 0.10 %. Two of those data sets (refs 5 and 7) have temperature ranges that are wider than the one covered by the above correlation. For this reason, the deviations of those data from the correlation eq 6 are shown in Figure 2, along the temperature range of the present measurements. Those data[5,7] show negative relative deviations, the maximum being −0.02 % and the minimum −0.09 %, which show an excellent agreement with the present results.

Caetano et al.[3] have published new density data at two temperatures obtained with a sample of DIDP with a mass fraction of water of $107 \cdot 10^{-6}$. As stated in that article,[3] those data deviate less than 0.002 % from the results published in ref 2, obtained with a sample with a mass fraction of water of $20 \cdot 10^{-6}$, and therefore, their deviations are not shown in Figure 2.

Recently, Peleties et al.[10] published a set of density data obtained at atmospheric pressure by pycnometry. The relative deviations of their results from the present measurements are shown in Figure 2 and do not exceed −0.08 %.

Al Motari et al.[6] published several measurements of the density at atmospheric pressure, within the temperature range (283 to 368) K. Two of those values, obtained with two different models of Anton Paar vibrating-tube instruments, namely, DMA 512P and DMA 602H, concerning the density of the same sample (sample B in ref 6, with water mass fraction, $w(H_2O) = 417 \cdot 10^{-6}$) differ from each other by about 0.4 %. The measurements performed with the atmospheric pressure model DMA 602H, with a nominal expanded uncertainty ($k = 2$) of ± 0.1 %, agree with the correlation eq 6 well within the estimated uncertainty of the present results. The largest relative deviation of the results obtained by Al Motari et al.[6] with that instrument from our correlation is −0.16 %.

Paredes et al.[15] have measured two samples with the same purity (99.8 %), source (Merck KGaA, GR, acc. to DIN 752001), and lot but with a different batch number (sample B and C in ref 15). The density of both samples at atmospheric

Table 1. Experimental Measurements of Density, ρ, of DIDP Along the Isotherms T as a Function of Pressure, p

T/K	p/MPa	ρ/kg·m^{-3}	p/MPa	ρ/kg·m^{-3}
283.15	0.10	974.5	9.02	979.3
	0.20	974.5	10.03	979.8
	0.40	974.6	15.00	982.4
	0.60	974.7	19.97	985.0
	0.80	974.9	25.03	987.3
	1.01	975.0	29.98	989.8
	2.00	975.5	35.03	992.1
	3.02	976.0	40.00	994.5
	4.00	976.6	45.03	996.6
	5.00	977.1	49.98	998.8
	6.00	977.7	55.03	1000.9
	6.98	978.2	59.99	1002.6
	8.01	978.7	64.99	1005.1
288.15	0.10	970.7	9.00	975.5
	0.20	970.8	10.00	976.1
	0.42	970.9	14.99	978.7
	0.60	971.0	19.97	981.4
	0.79	971.1	25.00	983.8
	1.00	971.2	30.00	986.4
	1.99	971.7	34.99	988.8
	3.02	972.3	40.02	991.1
	4.01	972.9	45.00	993.3
	5.00	973.4	49.98	995.7
	6.01	974.0	54.98	997.7
	7.02	974.5	60.00	999.9
	7.98	975.0	65.00	1002.0
293.15	0.10	967.0	9.02	972.0
	0.20	967.1	10.01	972.6
	0.40	967.2	15.01	975.2
	0.63	967.3	20.00	977.9
	0.79	967.4	25.03	980.5
	1.00	967.5	30.02	983.0
	2.01	968.1	35.03	985.4
	3.02	968.7	40.01	987.8
	4.02	969.2	44.98	990.1
	4.97	969.8	50.02	992.4
	5.98	970.3	55.02	994.6
	7.02	970.9	60.02	996.8
	8.00	971.5	65.00	998.9
298.15	0.10	963.4	9.04	968.5
	0.20	963.4	10.03	969.0
	0.41	963.6	15.01	971.7
	0.62	963.7	19.99	974.4
	0.82	963.8	25.00	977.0
	1.00	963.9	30.02	979.6
	2.02	964.5	35.00	982.1
	3.04	965.0	40.02	984.5
	4.03	965.7	44.97	986.9
	4.98	966.2	49.99	989.2
	6.07	966.8	54.97	991.4
	7.02	967.3	60.00	993.7
	8.04	967.9	65.02	995.8
303.15	0.10	959.7	9.00	964.9
	0.20	959.8	10.02	965.5
	0.40	959.9	14.97	968.2
	0.58	960.0	20.01	971.0
	0.80	960.2	25.03	973.6
	1.00	960.3	30.02	976.3
	2.00	960.9	35.00	978.8
	2.99	961.4	40.00	981.2
	4.00	962.0	45.00	983.6
	5.00	962.6	50.01	986.0
	6.00	963.2	54.98	988.3
	7.00	963.8	60.00	990.5
	7.98	964.3	64.99	992.7
308.15	0.10	956.2	9.02	961.4
	0.20	956.2	10.00	962.0
	0.41	956.4	15.01	964.8
	0.61	956.5	19.99	967.6
	0.80	956.6	25.00	970.3
	1.00	956.7	30.01	973.0
	2.01	957.3	35.00	975.5
	3.02	957.9	39.99	978.0
	4.00	958.5	44.99	980.4
	5.00	959.1	50.01	982.9
	6.00	959.7	55.00	985.2
	7.02	960.3	60.00	987.4
	8.00	960.8	65.00	989.7
313.15	0.10	952.6	9.02	957.9
	0.20	952.6	10.01	958.5
	0.40	952.8	15.01	961.4
	0.62	952.9	20.00	964.2
	0.80	953.0	25.03	967.0
	1.03	953.1	30.00	969.7
	2.01	953.7	35.02	972.2
	3.00	954.3	40.02	974.8
	4.00	955.0	45.01	977.2
	5.02	955.5	49.97	979.7
	5.99	956.2	55.00	982.0
	7.03	956.7	59.98	984.4
	8.00	957.4	64.97	986.6
323.15	0.10	945.4	9.01	950.9
	0.21	945.5	10.02	951.6
	0.40	945.6	15.00	954.5
	0.60	945.7	20.01	957.5
	0.80	945.9	25.03	960.3
	1.00	946.0	30.01	963.2
	2.00	946.6	34.97	965.8
	3.03	947.2	40.00	968.5
	4.03	947.9	45.00	970.9
	5.02	948.5	49.98	973.5
	6.01	949.1	54.98	975.8
	7.00	949.7	60.00	978.3
	8.00	950.3	65.00	980.6
333.15	0.10	938.3	9.01	944.1
	0.20	938.4	10.03	944.8
	0.40	938.5	14.99	947.9
	0.63	938.7	20.00	951.0
	0.80	938.8	25.00	953.8
	1.00	938.9	30.02	956.7
	2.01	939.6	34.99	959.4
	3.00	940.2	39.97	962.2
	4.00	940.9	45.02	964.8
	5.00	941.5	50.00	967.4
	6.00	942.2	55.02	969.8
	7.03	942.8	60.00	972.3
	7.98	943.5	65.00	974.7
343.15	0.10	931.2	9.02	937.3
	0.22	931.3	9.98	937.9
	0.43	931.5	15.01	941.2
	0.61	931.6	19.99	944.3
	0.81	931.8	25.01	947.4
	1.00	931.9	29.98	950.4
	2.00	932.6	35.03	953.3
	3.00	933.3	40.03	956.1
	3.98	934.0	45.01	958.7
	5.01	934.6	50.05	961.4
	5.98	935.3	55.00	964.0
	7.00	936.0	60.00	966.5
	7.99	936.6	65.02	968.9
353.15	0.10	924.2	9.00	930.5
	0.20	924.3	10.01	931.2
	0.41	924.5	15.04	934.6
	0.63	924.6	20.00	937.9
	0.80	924.8	24.97	941.0
	1.01	924.9	29.98	944.1
	2.00	925.6	35.01	947.1
	3.00	926.3	40.00	950.0
	3.98	927.0	45.00	952.7
	4.99	927.7	49.99	955.5
	6.00	928.4	55.00	958.1
	7.00	929.1	60.01	960.7
	8.01	929.8	65.02	963.2
363.15	0.10	917.2	9.03	923.8
	0.20	917.3	10.00	924.5
	0.42	917.4	15.00	928.0
	0.60	917.6	19.99	931.4
	0.80	917.7	25.01	934.7
	0.99	917.9	30.02	937.9

Table 1. Continued

T / K	p / MPa	ρ / kg·m^{-3}	p / MPa	ρ / kg·m^{-3}
	2.00	918.6	34.98	940.9
	2.98	919.4	39.99	943.9
	3.96	920.1	44.98	946.8
	4.97	920.9	49.99	949.6
	5.99	921.6	55.00	952.3
	7.02	922.3	60.04	955.0
	7.98	923.0	64.99	957.6

Table 2. Fitting Parameters of Equations 5 and 6

i	b_i / kg·m^{-3}	d_i / MPa
0	981.729	407.68
1	−0.7383	−654.53
2	2.40·10^{-4}	−42.313
3	−	399.58

pressure was measured with a DMA 5000 Anton Paar instrument. Figure 2 shows the deviations of the results obtained by Paredes et al.[15] with sample B from the correlation of the present results. Their relative maximum deviation is 0.13 %, and the rmsd is 0.066 %. The deviations of the results obtained by those authors with sample C agree with those of sample B within experimental uncertainty and are not shown in Figure 2 for the sake of clarity. Paredes at al.[15] have also measured the density of DIDP at atmospheric pressure with an Anton Paar SVM3000 rotational viscometer. Those authors claim that all their atmospheric pressure density measurements agree within the uncertainty of the results obtained with their high-pressure model HPM Anton Paar densimeter.

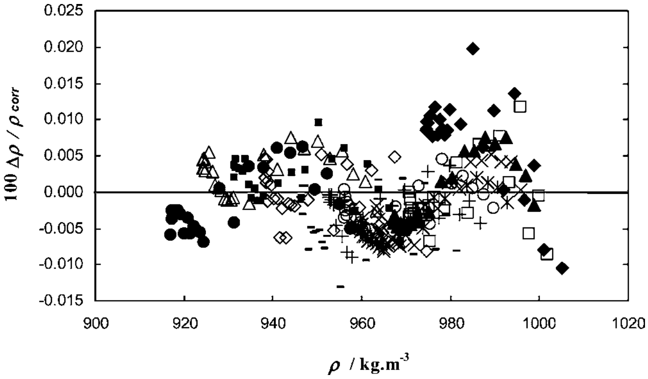

Figure 1. Relative deviations, $\Delta\rho = \rho_{\text{exp}} - \rho_{\text{corr}}$, of the present density measurements of DIDP, ρ_{exp}, from the correlation eqs 4 to 6, ρ_{corr}, as a function of density: ◆, 283 K; □, 288 K; ×, 298 K; $*$, 303 K; ○, 308 K; +, 313 K; −, 323 K; ◇, 333 K; ■, 343 K; △, 355 K; ●, 363 K.

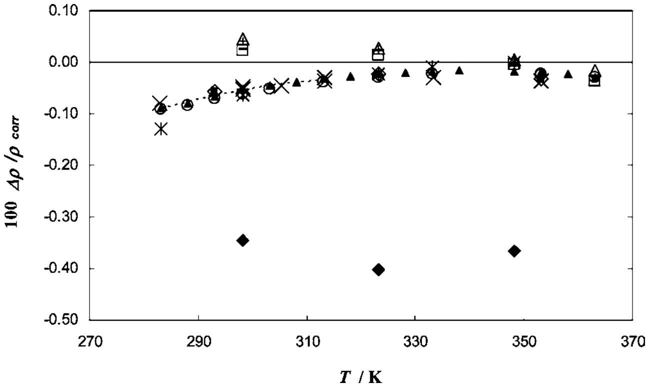

Figure 2. Relative deviations, $\Delta\rho = \rho_{\text{lit}} - \rho_{\text{corr}}$, of literature density data, ρ_{lit}, at 0.1 MPa from the correlation eq 6, ρ_{corr}, along the temperature range of the present measurements: Al Motari et al.[6] [(Anton Paar DMA 602 H): −, sample A, $w(H_2O) = 20\cdot10^{-6}$; □, sample A, $w(H_2O) = 115\cdot10^{-6}$; +, sample B, $w(H_2O) = 417\cdot10^{-6}$; △, sample B, $w(H_2O) = 24\cdot10^{-6}$]; Al Motari et al.[6] [(Anton Paar DMA 512P): ◆, sample B, $w(H_2O) = 417\cdot10^{-6}$]; Caetano et al.[4] [(various methods): - - - (correlation of experimental data sets from several authors)]; Paredes et al.[15] [(Anton Paar DMA 5000): $*$, sample B]; Harris and Bair[5] [(Anton Paar DMA 5000): ○, sample B]; Fröba and Leipertz[7] [(Surface Light Scattering):▲]; Peleties et a1[10] [(pycnometer): ×].

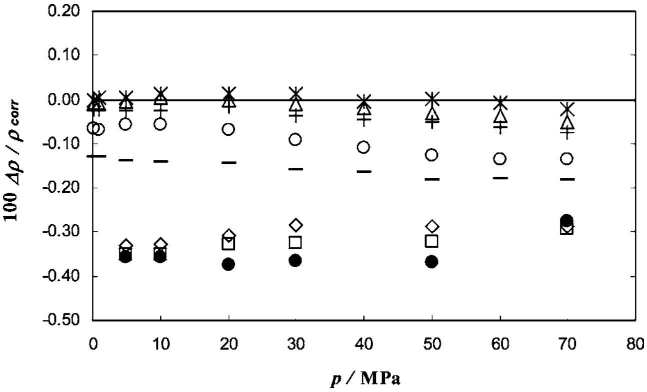

Figure 3. Relative deviations, $\Delta\rho = \rho_{\text{lit}} - \rho_{\text{corr}}$, of literature density data at pressures higher than 0.1 MPa, ρ_{lit}, for DIDP, performed with vibrating-tube densimeters and corrected by the respective authors for viscosity effects, from the correlation eqs 4 to 6, ρ_{corr}, along the pressure range of the present measurements: Al Motari et al.[6] [(measurements with Anton Paar DMA 512P), sample B: ●, 298 K: □, 323 K; ◇, 348 K]; Paredes et al.[15] [(measurements with Anton Paar DMA HPM), sample B: −, 283 K; ○, 298 K; +, 323 K; △, 333 K; $*$, 348 K].

Recently, density measurements performed at high pressures have been published. The deviations of the density measurements of DIDP at pressures above 0.1 MPa performed by Al Motari et al.[6] and Paredes et al.,[15] using Anton Paar vibrating U-tube densimeters, from the correlation eqs 4 to 6, with the parameters given in Table 2 of the present work, are shown in Figure 3. Al Motari et al.[6] reported density measurements performed on a DIDP sample of the same lot and nominal purity of the sample used in the present work, along three isotherms within the temperature range of the present measurements. Those authors have used an Anton Paar DMA 512P vibrating U-tube densimeter with an expanded uncertainty of about ± 0.3 % to measure the density from (298.15 to 423.15) K and up to 70 MPa. All their data show a systematic negative deviation from the correlation equation of the present measurements. The larger relative deviation does not exceed 0.4 %, which is slightly higher than the reported uncertainty of the measurements, namely, ± 0.3 %. The deviations do not show any significant trend with varying temperature or pressure. The density results obtained by Al Motari et al.[6] have been corrected for the viscosity effect, using the equation indicated by Lundstrum et al.[16]

Paredes et al.[15] have measured the density of three different samples with two different purity grades of DIDP from (283.15 to 398.15) K and pressures up to 60 MPa. The experimental measurements have been performed with an Anton Paar DMA HPM vibrating-tube densimeter. Paredes et al.[15] have applied a correction to their raw data due to the effect of viscosity, using an equation provided by Anton Paar GmbH. The rmsd observed between the two series of density measurements of DIDP with samples of 99.8 % purity by GC (samples B and C of ref 15) performed by Paredes et al.[15] from the correlation eqs 4 to 6 of the present work is 0.082 %. The relative deviations are negative for the lowest temperatures and slightly positive for the highest temperatures (−0.19 % to 0.025 %), all being within the uncertainty of the present measurements.

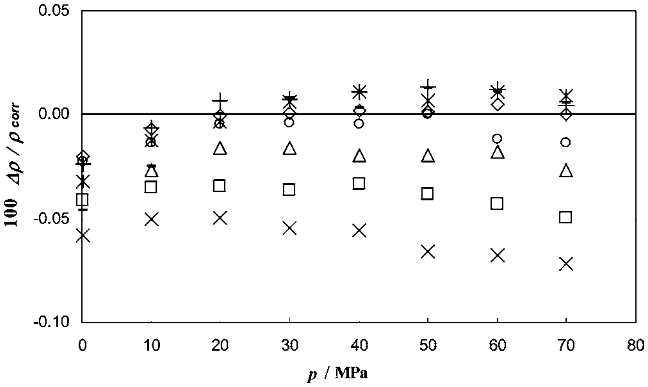

Figure 4. Relative deviations, $\Delta\rho = \rho_s - \rho_{corr}$, of the density data of DIDP calculated from speed-of-sound measurements by Peleties et al.,[10] ρ_s, from the correlation eqs 4 to 6, ρ_{corr}, at pressures higher than 0.1 MPa, at several temperatures, within the temperature range of the present measurements, as a function of pressure: ×, 293 K; □, 303 K; Δ, 313 K; ○, 323 K; ◇, 333 K; +, 343 K; ∗, 353 K; −, 363 K.

Figure 4 shows the deviations of the DIDP density data, obtained by Peleties et al.[10] from their speed of sound measurements, from the present results described by correlation eqs 4 to 6 with parameters given in Table 2. Peleties et al.[10] have calculated DIDP densities from (298.15 to 423.15) K and up to 140 MPa, from speed of sound measurements. This method is free of viscosity effects on the results, and it is, therefore, interesting to compare with the above results. Figure 4 shows the deviation of the data obtained by Peleties et al.[10] from the correlation of the present results, eqs 4 to 6, as a function of pressure. The agreement is very good. The rmsd of their results from the correlation of the present work is 0.028 %, and the relative maximum and minimum deviations are 0.014 % and −0.072 %, respectively, well within the experimental uncertainty of the present measurements.

As a conclusion, the comparisons between the present experimental density data and those obtained by other authors, in the same temperature and pressure ranges of the present measurements, show in general a very good agreement. The only literature data showing absolute deviations larger than 0.2 % were obtained by Al Motari et al.[6] with an Anton Paar DMA 512P model. It is noteworthy that, at atmospheric pressure, the measurements carried out by those authors with model 512P differ from the values they have obtained,[6] using an Anton Paar DMA 602H model, by approximately 0.4 %. This suggests that the uncertainty of the density data obtained by those authors at high pressures, using the DMA 512P, may be higher than the uncertainties of the other data sets.

It must be pointed out that all the literature data are either corrected for viscosity effects by the respective authors or free of those effects, as is the case of the density data obtained from speed of sound measurements,[10] or by pycnometry.[9,10] It is noteworthy that the density data that are free of viscosity effects show relative deviations from the present results that in the modulus are smaller than 0.08 % (capillary measurements[10] at 282.95 K and at atmospheric pressure) and 0.07 % (from speed of sound measurements[10] at 293.15 K and 60 MPa). In particular, it is noticeable that the maximum deviation of the high pressure speed of sound results occurs at the lowest temperature measured by those authors (293.15 K), where the maximum estimated correction of viscosity effects for our results (at $p = 65$ MPa) does not exceed 0.11 %. It is therefore apparent that the absolute value of the observed deviation is smaller than the estimated correction of the viscosity effect on the measured density. Notwithstanding this, it is evident that most of the data points obtained by Peleties et al.[10] show negative deviations that become increasingly more negative as the temperature decreases. A qualitatively similar trend is observable with the results published by Paredes et al.,[15] whose raw data, obtained with a U-tube densimeter, have been corrected for viscosity effects. Despite all these deviations being smaller in absolute values than the uncertainty of the present results, they seem to indicate that a correction of the present density data may be justified in the future, when viscosity data are made available for the whole ranges of temperature and pressure of the present measurements, and the discrepancies between results from different authors may be resolved.

Conclusions

New density data are presented for DIDP at 12 temperatures from (283.15 to 326.15) K and pressures from (0.1 to 65) MPa. The results, obtained with an Anton Paar DMA HP U-tube densimeter, were not corrected for viscosity effects, and their relative uncertainty is estimated to be less than ± 0.3 %. Comparisons between the present experimental values and density results obtained by other authors were performed, showing in general a very good agreement. This work presents raw data for the density of DIDP without considering any empirical correction for the viscosity effect on the results since the literature viscosity data do not extend over the whole temperature and pressure ranges of the present measurements. Furthermore, the viscosity measurements of DIDP at high pressures which are available in the literature present some important deviations, significantly larger than the nominal uncertainties of the measurements. The comparisons of the measurements of the present work with the results obtained by other authors, at pressures above atmospheric, which have either been corrected for viscosity effects or are intrinsically free of those effects, are excellent—the deviations being well within the uncertainties of the measurements. Our own estimate of the correction for viscosity effects that may be affecting the present results ranges from (−0.012 % to −0.12 %), where the maximum correction was estimated using extrapolated literature viscosity data.[5]

All these observations indicate that a correction of the present density data may be justified in the future, when viscosity data are made available along the whole ranges of temperature and pressure, but the differences are expected, in general, to be of little significance.

The establishment of standard reference values for transport properties, in particular for viscosity, must have accurate density data available over the temperature and pressure ranges of interest. Furthermore, as suggested by Paredes et al.,[15] the recommendation of reference values for the viscosity of DIDP at high pressures could conveniently proceed following a scheme similar to the one used to build the correlation of the viscosity of toluene at high pressures, proposed by Assael et al.[19] in 2001. In particular, that correlation[19] is based on a hard-sphere scheme, whose utilization requires reliable density data along the same temperature and pressure ranges of the viscosity. We hope the present article might be a contribution toward the establishment of DIDP as a reference fluid for viscosity at high pressure.

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