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Vibrating Wire Viscometer with Nominal Wire Diameter of 0.15 mm: Measurement of the Viscosity of Two Certified Reference Fluids, with Nominal Viscosities at $T = 298$ K and $p = 0.1$ MPa of (16 and 29) mPa·s, at Temperatures between (298 and 353) K and Pressures below 55 MPa

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We have measured the viscosity of certified reference materials N10 and S20 with nominal viscosities at $T = 298$ K and $p = 0.1$ MPa of (16 and 29) mPa·s, respectively, at temperatures in the range (298 to 353) K and pressures between (0.1 and 55) MPa with a vibrating wire viscometer. This viscometer had a nominal wire diameter of 0.15 mm and provided viscosities with an estimated expanded ($k = 2$) uncertainty of ±2 % over the viscosity range (3 to 70) mPa·s. The latter value represents about half the upper operating viscosity of this vibrating wire that is about 150 mPa·s. The measured viscosity was compared with values predicted by interpolation expressions for N10 and S20 that represents the measured viscosities of different lots of the same fluids to within ±2 %. The results reported here have an average absolute deviation from those interpolation equations of about 2.4 % for N10 and 2.7 % for S20. These differences are within the combined estimated uncertainties of the measurements.

Introduction

The evaluation of the economics of a hydrocarbon bearing formation requires measurements of many physical properties including both the density and the viscosity of the reservoir hydrocarbon. Typically, recoverable reservoir liquids have viscosities between (1 and 100) mPa·s. Thus, fluids with known viscosities, that include this range, are required for laboratory evaluation of proposed measurement techniques and calibration of other viscometers as a function of both temperature and pressure.

In our previous paper,[1] we discussed the following: (1) the internationally agreed absolute standard for the viscosity of water at a temperature of 293.15 K and pressure of 101.325 kPa; (2) potential pure fluid reference materials all with viscosity less than 1 mPa·s; (3) preliminary measurements reported by Caetano et al.[2] of the viscosity of diisodecyl phthalate (with viscosities up to 121 mPa·s) and the proposal that this compound be a candidate for a moderate viscosity standard; (4) our proposition[1] that until measurements on either diisodecyl phthalate or another fluid as a function of temperature and pressure are complete, and the viscosity adopted by International agreement the continued use of other fluids, known as certified reference materials for viscosity, are required to calibrate and compare viscometers intended for measurements with liquids; and (5) described the methods required to provide certified values of these reference materials by comparison with the viscosity of water at $T = 293.15$ K and $p = 101.325$ kPa, either directly or indirectly, through a chain of intermediate reference liquids and a series of Master capillary viscometers that establish the kinematic viscosity by the so-called "step-up" procedure to ensure the smallest possible uncertainty in calibration.[3] In ref 1, the viscosity of certified reference materials N10 and S20 at temperature between (298 and 353) K at pressure below 55 MPa, for which neither the supplier nor the literature provides values, was determined over the range (1.8 to 76) mPa·s with a vibrating wire viscometer, with a nominal wire diameter of 0.1 mm; the density was also measured with an oscillating tube densimeter. These results were combined with the values of density and viscosity provided by the supplier at $p = 0.1$ MPa to obtain interpolation expressions for both properties with leading terms based on the cited values. In view of the reported international consistency of these fluids,[4] adopting this formalism might allow small differences between the cited values to be accommodated by solely substituting values obtained from either other lots of the same supplier or alternative supplier of certified reference material N10 and S20.

In this work, we have used a vibrating wire viscometer, with a wire diameter of 0.15 mm, to measure the viscosity of certified reference materials N10 and S20 obtained from batches of N10 and S20 that were different to those used for the results reported in ref 1. The measurements reported here were performed at temperature between (298 and 353) K at pressures below 55 MPa and covered the viscosity range (3 to 70) mPa·s; the latter represents about half the upper operating viscosity of about 150 mPa·s for our vibrating wire formed with a wire of nominal diameter 0.15 mm. The purpose of these measurements was to demonstrate that eq 9 of ref 1 can be used to predict the

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viscosity of N10 and S20 obtained from different lots with an uncertainty of less than ±5 %.

Experimental Section

Apparatus and Experimental Procedures. The apparatus used for the measurements reported here was described in detail previously,[1] and it has been used with only minor modifications that are discussed here. The vibrating wire was formed from a ≈0.15 mm diameter tungsten 52 mm long that was drawn taut, in the holder shown in Figure 1 of ref 1, between two clamps. The wire used in this experiment was obtained from Nippon Tungsten, Fukuoka, Japan, with mass fraction purity greater than 0.9995. The wire was cold drawn and mechanically (center less) ground, and the manufacturer supplied a measured diameter of 0.14859 mm with an upper bound of 0.14984 mm and a lower bound of 0.14859 mm. When the outer surface was viewed, with a scanning electron microscope operated at a magnification of about 1000, it showed essentially a smooth outer surface. The wire holder differed from that shown in Figure 1 of ref 1 in that each metallic clamp had a pin that fitted within a slot at each end of the Shapal-M tube that prevented the clamps rotating relative to each other about the ceramic.

The magnetic field was provided by the same two magnets used in ref 1 that were fabricated from $(\mathrm{Nd, Pr, Dy})_2(\mathrm{Fe, Co})_{14}\mathrm{B}$ (known commonly as NdFeB) mounted opposite each other on a C-shaped clamp formed from carbon steel. At a temperature of 293 K, the measured magnet flux density between the magnets was about 0.38 T and the largest force applied, at the maximum current of ≈5 mA (= 5 V/1000 Ω) about 99 μN.

Prior to commencing measurements, the vibrating wire was aligned within the magnetic flux, so as to preferentially excite the highest orthogonal mode of the fundamental oscillation that arises from the noncircular cross-section of the wire, with the procedure described in ref 1.

For Newtonian fluids, the vibrating wire has exact working equations that have been fully described in refs 5 to 9. We have used the working equations as described in ref 1 without modification. The hydrodynamic model includes the resonance frequency in the absence of fluid and damping, the internal damping of the wire in a vacuum, the wire radius, and the wire density. When these parameters are known along with the density of the fluid sample, the viscosity of the fluid can be obtained from the width of the resonance curve. However, in practice the wire damping factor and radius cannot be determined with sufficient accuracy by independent methods, and those values are usually determined by calibration. To do this, measurements are made in both vacuum, which yields the damping factor Δ_0, and methylbenzene, for which the viscosity and density are known, to provide the wire radius, R.[2,6,10]

For each fluid temperature and pressure, the in-phase and quadrature voltages $\boldsymbol{V}$ (eq 1 of ref 1) measured at a series of discrete frequencies that included the motional emf $\boldsymbol{V}_2$ (eq 2 of ref 1) were determined with a lock-in amplifier over the frequency range ($f_r \pm 5g$), where g is half the resonance line width at a frequency corresponding to an amplitude determined by 0.707 times that of the maximum amplitude and f_r is the fundamental transverse resonance frequency. Prior to acquiring $\boldsymbol{V}$, the majority of the contribution arising from the drive voltage $\boldsymbol{V}_1$ was removed by setting the lock-in offset voltage at $f < (f_r - 5g)$. The measured $\boldsymbol{V}$ were then replicated by adjusting a, b, c, and d of (eq 2 of ref 1) that represent $\boldsymbol{V}_1$, the resonance frequency in a vacuum f_0 (eq 3 of ref 6), and the viscosity η (eq 9 of ref 6) using R and Δ_0 from the calibration and the appropriate density. This numerical procedure separates the complex voltages $\boldsymbol{V}_1$ and $\boldsymbol{V}_2$ utilizing the different frequency dependence of eq 2 and eq 3 of ref 6, respectively.

The sinusoidal voltage generated by the lock-in amplifier was varied between (5 and 5000) mV to maintain the amplitude of the wire motion less than 10 % of its radius. In a vacuum and air, a driving current of 5 μA was used while 5 mA was required when the wire was submerged in methylbenzene, N10, or S20. For these currents and environments, the resulting temperature rise in the wire over the 200 s data acquisition time contributed a negligible error in the measured viscosity.[1]

The internal damping factor was assumed to be $120 \cdot 10^{-6}$, equivalent to that reported by Kandil et al.[6] for a wire of nominal diameter 0.15 mm, supplied by Goodfellow, Cambridge, UK, and because the determination of the viscosity of liquids is insensitive to the value of Δ_0 there is no requirement to determine this parameter with high precision.[6,7] Measurements in methylbenzene at a temperature of 298.15 K and four pressures in the range (13.8 to 55.2) MPa were combined with the viscosity and density of methylbenzene determined from the correlation reported by Assael et al.[11] to obtain $\langle R \rangle = (74.302 \pm 0.175) \cdot 10^{-6}$ μm, and no systematic variations of R from $\langle R \rangle$ were observed with pressure. Our measurement of $\langle R \rangle$ differs from the value provided by the wire supplier by 0.009 %. The uncertainty in R is the major source of error in our measurements of viscosity, which have an estimated expanded ($k = 2$) uncertainty of ±2 % based on the work reported in refs 6 and 7.

The temperature of the vibrating wire viscometer was determined with a platinum resistance thermometer with a nominal resistance of 100 Ω which, when compared against a standard 25 Ω platinum resistance thermometer that had been calibrated on the ITS-90, was found to have an uncertainty of less than ±0.01 K.

Pressure was generated with a positive displacement pump and measured, in the range (0 to 55) MPa, with a transducer (Honeywell Sensotec, USA, model THE/0743-11TJA, serial number 833326 with a maximum operating pressure of 69 MPa) that when calibrated against a dead weight gauge with a precision of better than ±0.001 MPa, was found to have an uncertainty $\delta p/\mathrm{MPa} = \pm(0.001p + 0.028)$.

Materials and Measurements. Three fluids were used for the measurements. Methylbenzene from Merck BDH Ltd had a mass fraction greater than 0.99917 and was used to determine the wire radius as described in the preceding section. The two certified reference materials for viscosity N10 and S20 {with nominal viscosities of (16 and 29) mPa·s, respectively at $T = 298$ K and $p = 0.1$ MPa} were obtained from Cannon Instruments, USA, with assigned lot numbers 4201 and 4301, respectively. The supplier measured the kinematic viscosity for both N10 and S20 at temperatures between (293 and 373) K using long capillary Master viscometers according to ASTM D 2164. The supplier also provided the density measured in accordance with ASTM D 1480. The uncertainty in the kinematic viscosity was ±0.25 % relative to water, and the uncertainty in the density was ±0.02 %.

For N10 measurements were performed at three temperatures of (293, 313, and 353) K while for S20 measurements were performed at two temperatures of (313 and 353) K and for both fluids at pressures between 0.2 MPa and 55 MPa. The supplier cited values of both density and viscosity at a pressure of 0.1 MPa and 7 temperatures of (293.15, 298.15, 310.93, 313.15, 323.15, 372.04, and 373.15)

Table 1. Viscosity η and Density ρ Provided by the Manufacturer for Certified Reference Materials N10 and S20 at Temperature T and p = 0.1 MPa

fluid	T/K	η/mPa·s	ρ/kg·m^{-3}
N10	293.15	20.05	887.4
	313.15	8.740	884.1
S20	313.15	15.35	851.4

Table 2. Viscosity η Obtained from Vibrating Wire Viscometer for Certified Reference Materials N10 and S20 at Temperatures T and Pressures p with Expanded Uncertainties[a]

fluid	T/K	p/MPa	η/mPa·s
N10	293.15 ± 0.01	0.222 ± 0.056	19.48 ± 0.39
		13.785 ± 0.084	27.19 ± 0.56
		27.54 ± 0.11	37.75 ± 0.78
		41.37 ± 0.14	51.1 ± 1.1
		55.16 ± 0.17	69.5 ± 1.5
	313.15 ± 0.01	0.280 ± 0.057	8.65 ± 0.17
		13.793 ± 0.084	11.17 ± 0.23
		27.54 ± 0.11	15.24 ± 0.31
		41.37 ± 0.14	19.83 ± 0.42
		55.15 ± 0.17	26.16 ± 0.55
	353.15 ± 0.01	0.297 ± 0.057	2.988 ± 0.060
		13.788 ± 0.084	3.752 ± 0.076
		27.57 ± 0.11	4.657 ± 0.095
		41.39 ± 0.14	5.73 ± 0.12
		55.15 ± 0.17	6.97 ± 0.14
S20	313.15 ± 0.01	13.813 ± 0.084	20.07 ± 0.41
		27.58 ± 0.11	26.38 ± 0.54
		41.38 ± 0.14	34.06 ± 0.71
		55.16 ± 0.17	43.29 ± 0.91
	353.15 ± 0.01	13.781 ± 0.084	6.02 ± 0.12
		27.58 ± 0.11	7.43 ± 0.15
		41.37 ± 0.14	9.07 ± 0.19
		55.16 ± 0.17	11.07 ± 0.23

[a] Confidence interval of 0.95.

K. Our measurements of viscosity at temperatures of 293.15 K and 313.15 K corresponded to temperatures at which certified reference values were provided by the supplier and are listed in Table 1 along with the cited densities. In the analyses to obtain viscosity for N10 and S20 we have used the densities predicted from eqs 7 and 8 of ref 1, albeit these equations were derived for lots different from those used for the measurements reported here. The additional uncertainty in viscosity arising from this assumption will be discussed below.

Results and Discussion

The viscosity of N10 and S20 obtained with the vibrating wire viscometer at temperatures between (298 and 353) K at a pressure up to 55 MPa are listed in Table 2. Small corrections have been applied to the reported viscosity to reduce all values to the stated temperature for each isotherm; the correction was estimated from eq 9 of ref 1 with the coefficients of Table 6 of ref 1. The uncertainties, listed in Table 2, are at a confidence interval of 0.95 (k = 2) and were obtained by combining in quadrature uncertainties arising from the uncertainty of the instrument with dη/dT and dη/dp. The major source of uncertainty (by at least factor of 5) arises from the uncertainty of the vibrating wire viscometer the standard uncertainty (k = 1) that is, based on the work reported in refs 6 and 7, assumed to be ±1 %. The next most significant and quantifiable contribution to the uncertainties arises from $(\partial\eta/\partial p)_T$. These derivatives were estimated from a combination of eq 9 of ref 1 with the coefficients of Table 6 of ref 1 and the δp listed in Table 2. For N10 the contribution to $\delta\eta$ from δp was always less than 0.2 mPa·s (about 0.35 %) that decreased with increasing temperature. For certified reference specimen S20, the $\delta\eta$ arising from δp was less than 0.14 mPa·s (about 0.25 %). The contribution to the uncertainty from $(\partial\eta/\partial T)_p$ was estimated from a from eq 9 of ref 1 with the coefficients of Table 6 of ref 1 combined with the δT listed in Table 2. For N10, the contribution to $\delta\eta$ from δT was less than 0.009 mPa·s contributing between (0.01 and 0.08) % while for S20 $\delta\eta$ arising from δT was below 0.02 mPa s and also contributed between (0.01 and 0.03) %. Clearly, for our measurements the uncertainty with which the pressure is measured is more significant for N10 and S20 than the uncertainty of temperature. In the absence of a chemical analysis for these fluids, the contribution to the uncertainty arising from the uncertainty in composition was assumed to be 0.

The N10 and S20 used for the measurements reported here were obtained from lots that were different from those used for the measurements reported in ref 1; in ref 1 N10 was from lot 3201 while S20 from lot 3401; for the measurements reported here N10 was taken from lot 4301 and S20 was from lot 4201. For N10 the viscosities cited by the supplier for lot 4301 lie less than 1 % above those cited for lot 3201 while the densities cited by the supplier for lot 4301 lie about 0.6 % above those cited for 3201. For S20, the viscosities cited for lot 4201 are no more than 0.4 % above those cited for lot 3401 while the densities cited for lot 4201 are less than 0.2 % higher than those cited for lot 3401. The differences in viscosity cited by the supplier for these two lots are less than the expanded uncertainties assigned in both the measurements reported in ref 1 and those discussed here. These differences are also less than the average percentage deviation of the measurements reported in ref 1 from eq 9 of ref 1 that never exceeded 0.6 %. For the purpose of the current evaluation these variations in viscosity are insignificant. Thus the coefficients of the empirical expression eq 9 of ref 1 were not modified by inclusion of the variation of viscosity at a pressure of 0.1 MPa arising from different lots that would have contributed to the first term of eq 9 of ref 1, which was the rule of Vogel.[12]

For N10 the results at temperatures of (293 and 313) K can be compared directly with previous measurements reported in ref 1 while at T = 353 K the comparison is with an extrapolation of eq 9 of ref 1. For S20 the measurements listed in Table 2 at temperatures of (313 and 353) K are in the range of the results reported previously and also of the correlation eq 9 of ref 1.

The viscosities from Tables 1 and 2 are shown in Figure 1 for N10 and in Figure 2 for S20 with solid symbols relative to the values obtained from eq 9 of ref 1 for certified reference materials N10 and S20. The error bars represent the expanded uncertainty of the measurements. Figures 1 and 2 also show the viscosities reported in ref 1 that were used to adjust the coefficients of eq 9 of ref 1 along with dashed lines that represent the expanded uncertainty of the measurements of ref 1. At p = 0.1 MPa the viscosities of Table 1 for both N10 and S20 at temperatures of (293 and 313) K and 313 K, respectively, are (as shown in Figures 1 and 2) in good agreement with the smoothing eq 9 of ref 1 deviating by less than 1.2 % for N10 and 0.5 % for S20 that is within the combined uncertainty arising from the difference between the lots and uncertainty of the supplier's cited measurements for the reference fluids of ±0.25 %. At pressures greater than 0.1 MPa, the results for N10 agree with eq 9 of ref 1 within ±4 % while for S20 our viscosities are consistent with eq 9 within ±4.5 %. For both N10 and S20 these differences are about a factor of 2

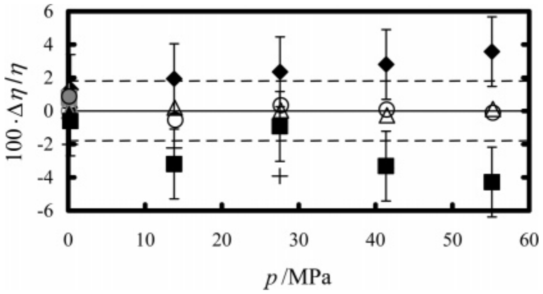

Figure 1. Fractional deviation $\Delta\eta/\eta = \{\eta(\text{exptl}) - \eta(\text{calcd})\}/\eta(\text{calcd})$ of the viscosity of either Table 1 or Table 2 η(exptl) from the value obtained from combination of eq 9 with coefficients listed in Table 6 of ref 1 η(calcd) for certified reference material N10. The error bars assigned to the results are the expanded uncertainty of Table 2 while the dashed lines are the expanded uncertainty of Table 4 of ref 1. +, $T = 293.15$ K from Table 2; gray +, value cited by supplier at $T = 293.15$ K from Table 1; ○, $T = 298$ K from Table 4 of ref 1; gray ○, value cited by supplier at $T = 298$ K from Table 3 of ref 1; ■, $T = 313$ K from Table 2; gray ■, value cited by supplier at $T = 313$ K from Table 1; △, $T = 333$ K from Table 4 of ref 1; gray △, eq 5 of ref 1 with coefficients of Table 1 of ref 1 at $T = 333$ K from Table 3 of ref 1; and ◆, $T = 353$ K from Table 2.

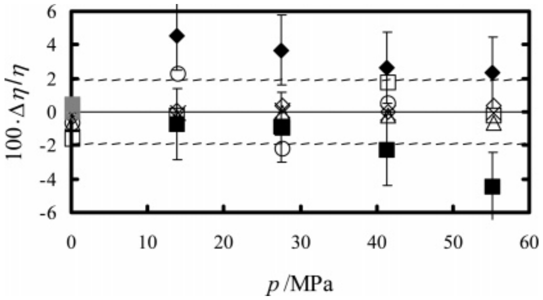

Figure 2. Fractional deviation $\Delta\eta/\eta = \{\eta(\text{exptl}) - \eta(\text{calcd})\}/\eta(\text{calcd})$ of the viscosity of either Table 1 or Table 2 η(exptl) from the value obtained from combination of eqs 9 with coefficients listed in Table 6 of ref 1 η(calcd) for certified reference material S20. The error bars assigned to the results are the expanded uncertainty of Table 2 while the dashed lines are the expanded uncertainty of Table 4 of ref 1.○, $T = 298$ K from Table 4 of ref 1; gray ○, value cited by supplier at $T = 298$ K from Table 3 of ref 1; ■; $T = 313$ K from Table 2; gray ■, value cited by supplier at $T = 313$ K from Table 1; □, $T = 313$ K from Table 4 of ref 1; gray □, value cited by supplier at $T = 313$ K from Table 3 of ref 1; △, $T = 333$ K from Table 4 of ref 1; gray △, eq 5 of ref 1 with coefficients of Table 1 at $T = 333$ K from Table 3 of ref 1; ◆, $T = 353$ K from Table 2; ◇, $T = 353$ K from Table 4 of ref 1; gray ◆, eq 5 of ref 1 with coefficients of Table 1 of ref 1 at $T = 353$ K from Table 3 of ref 1; ×, $T = 393$ K from Table 4 of ref 1; and gray ×, eq 5 of ref 1 with coefficients of Table 1 of ref 1 at $T = 393$ K from Table 3 of ref 1.

greater than the expanded uncertainty of either series of measurements but are within the combined uncertainty of both measurements excluding the known, but less than, 1 % differences between the viscosities of the lots.

The differences in the densities of the different lots either cited by the supplier or obtained from the modified Tait equation[13] representation of the results of ref 1 (eq 7 and eq 8 of ref 1 with coefficients of Table 5 of ref 1) are less than 0.6 % that is about 3 times the expanded uncertainty of 0.2 % for the measurements reported in ref 1; eq 7 and eq 8 represented the densities of ref 1 with an average percentage deviation that never exceeded 0.04 %. The up to ±0.6 % additional uncertainty in the density propagates to an uncertainty of less than ±0.3 % in the viscosity determined from the vibrating wire, and we concluded that this upper bound on the uncertainty of the density used in the analysis should not introduce any additional and significant error into the values of viscosity when compared with the assigned expanded uncertainty of the viscosity measurements of ±2 %.[14]

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