# Operation of a Vibrating Wire Viscometer with a Wire Radius of 0.207 mm in a Fluid with Nominal Viscosity, at T = 289.1 K, of 1.581 Pa·s at Temperatures between (289.1 and 420.7) K and a Pressure of 0.1 MPa

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A viscometer formed of a tungsten wire of radius about 0.2 mm has been evaluated with measurements of the viscosity of certified reference material for viscosity N350 at viscosities up to 1.581 Pa·s, at temperatures between (289.1 and 420.7) K and a pressure of 0.1 MPa. The results differed from the cited values by less than  $\pm$  10 %.

## Introduction

Recently, Harrison et al.<sup>1</sup> used a vibrating wire of radius  $\approx 0.125$  mm to measure the viscosity of certified reference fluid for viscosity S60 at temperatures between (273 and 373) K, over which the certified values of viscosity covered the range (0.0062 to 0.652) Pa·s. These measurements extended the operating range of the vibrating wire by more than a factor of 3 over those reported by Kandil et al.<sup>2</sup> for a wire of nominal radius  $\approx 0.075$  mm. Harrison et al.<sup>1</sup> used the working equations of the vibrating wire to estimate the quality factor of the fundamental resonance as a function of the wire radius and viscosity of the fluid in which the wire was immersed. These calculations were in good agreement with the Q reported by Lundstrom et al.<sup>3</sup>, Sopkow et al.,<sup>4</sup> and Kandil et al.<sup>2</sup> and suggest a vibrating wire could be used to measure the viscosity up to 1 Pa·s. In this paper, we demonstrate the vibrating wire of radius  $\approx 0.2$  mm can be used to measure the viscosity up to  $\approx 1.6$  Pa·s. As a comparison, we mention that Caetano et al.<sup>5</sup> used a vibrating wire viscometer with a wire of radius 0.1997 mm, about the same radius as we used, that was determined by calibration with water at T = 293.15 K and p = 101.325 kPa. Caetano et al.<sup>5</sup> used the instrument to determine the viscosity of several fluids with viscosities in the range (0.5 to 135) mPa·s and stated the expanded (k = 2) uncertainty in their measurements was less than 0.6 % over this range.

## **Apparatus and Experimental Procedures**

*Vibrating Wire Viscometer.* The vibrating wire viscometer and pressure vessel used for these measurements have been described by Lundstrom et al.,<sup>3</sup> and the thermostat, thermometer, and pressure gauge were the same as those reported by Harrison et al.<sup>1</sup> Only the significant differences between this viscometer and that of refs 1 and 3 are described here.

Measurements in air at  $p \approx 0.1$  MPa were used to obtain a value for  $\Delta_0$  (eq 8 of ref 1). Because the damping in N350 is comparatively large, the difference between that determined in air and in a vacuum is insignificant for our purpose, whereas

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measurements in methylbenzene at a temperature of 298.15 K and a pressure of 0.1 MPa were used to obtain the radius using the viscosity ( $\eta \approx 0.57$  mPa·s) and density of methylbenzene obtained from Assael et al.;<sup>6</sup> a negligible difference in radius resulted if the viscosity of methylbenzene was taken from the assessment reported by Santos et al.<sup>7</sup> These measurements gave a radius of 0.2076 mm and  $\Delta_0 = 3.68 \cdot 10^{-4}$ . The internal damping should be obtained in the absence of external damping from measurements in a vacuum. However, when a viscometer is used to determine the viscosity of liquids ( $\eta > 1$  mPa·s), the difference between  $\Delta_0$  obtained from measurements in air and those in a vacuum gives an insignificant difference in the measured viscosity. The wire was tensioned between two clamps, with the procedure described in ref 3, using a mass of 1.2 kg to provide a resonance frequency of about 950 Hz. The temperature of the outer surface of the pressure vessel was determined with a K-type thermocouple with an uncertainty of  $\pm$  0.1 K. The largest source of uncertainty in our values of viscosity arises from the uncertainty in temperature. The estimated standard uncertainty in the measurements of viscosity with a vibrating wire, excluding the error in temperature measurements, is  $\pm 1 \%$ .<sup>2-4</sup>

The force applied to the wire of length 52 mm is determined by the magnetic field intensity of about 0.4 T, which is perpendicular to the wire axis, and the ac current. Owing to the stiffness of a wire (rod) of nominal diameter of 0.2 mm immersed in fluid of viscosity 1.6 Pa·s, a current up to 0.35 A was required to excite the resonance, producing a maximum force of 0.007 N. The current was obtained by amplifying the signal from the lockin amplifier with a power amplifier (Krohn-Hite, USA, model 7500). The tungsten wire  $\{c_p(W, 298 \text{ K}) \approx$ 133 J·kg<sup>-1</sup>·K<sup>-1</sup> and  $\rho(W, 298 \text{ K}) \approx 19300 \text{ kg·m}^{-3}$ }, of diameter 0.4 mm and mass  $126 \cdot 10^{-6}$  kg, has a resistance of the order 1  $\Omega$ , and when immersed in N350 { $c_p(298 \text{ K}) \approx 2$ kJ·kg<sup>-1</sup>·K<sup>-1</sup>,  $\rho(298 \text{ K}) \approx 840 \text{ kg} \cdot \text{m}^{-3}$ , and  $\kappa \approx 120 \text{ mW} \cdot \text{m}^{-1} \cdot$  $K^{-1}$ }, with a current of 0.35 A, over the 200 s acquisition time the fluid temperature was estimated to rise by about 0.07 K {assuming all electrical energy dissipated into the fluid volume enclosed by the wire holder (about  $1.1 \cdot 10^{-6} \text{ m}^3$ ). This temperature change is less than the uncertainty of our temper-

Table 1. Mean Experimental Viscosity,  $\langle \eta(\text{exptl}) \rangle$ , of Certified Reference Fluid for Viscosity N350 Determined with a Vibrating Wire Viscometer of Nominal Radius 0.2 mm from *N* Observations with Standard Deviation of the Mean and Certified Viscosities,  $\eta(\text{cert})$ , as a Function of Temperature, *T*, at a Pressure of 0.1 MPa

| Т               |    | $<\eta(\text{exptl})>$ | $\eta(\text{cert})$ |
|-----------------|----|------------------------|---------------------|
| K               | Ν  | mPa•s                  | mPa•s               |
| $420.7\pm0.1$   | 19 | $6.302 \pm 0.138$      | 6.3288              |
| $376.7 \pm 0.1$ | 14 | $17.194 \pm 0.355$     | 18.062              |
| $365.1 \pm 0.1$ | 13 | $24.734 \pm 0.190$     | 25.893              |
| $362.2 \pm 0.1$ | 13 | $27.117 \pm 0.276$     | 28.536              |
| $359.2 \pm 0.1$ | 16 | $29.856 \pm 0.284$     | 31.658              |
| $355.3\pm0.1$   | 15 | $34.728 \pm 0.153$     | 36.424              |
| $352.5\pm0.1$   | 10 | $39.129 \pm 0.133$     | 40.438              |
| $345.5\pm0.1$   | 13 | $50.200 \pm 0.707$     | 53.317              |
| $338.5\pm0.1$   | 19 | $68.427 \pm 0.957$     | 71.978              |
| $335.5\pm0.1$   | 17 | $78.0975 \pm 0.477$    | 82.509              |
| $332.85\pm0.1$  | 15 | $94.233 \pm 0.851$     | 93.479              |
| $326.7 \pm 0.1$ | 14 | $122.498 \pm 0.733$    | 126.95              |
| $319.7 \pm 0.1$ | 15 | $178.728 \pm 1.018$    | 185.37              |
| $313.1 \pm 0.1$ | 11 | $280.317 \pm 2.756$    | 273.75              |
| $309.4\pm0.2$   | 15 | $356.958 \pm 11.938$   | 345.92              |
| $306.7\pm0.2$   | 16 | $395.922 \pm 16.861$   | 413.43              |
| $303.0\pm0.2$   | 15 | $511.400 \pm 3.954$    | 533.63              |
| $299.7\pm0.3$   | 14 | $653.638 \pm 6.759$    | 677.64              |
| $297.8\pm0.3$   | 35 | $731.590 \pm 20.043$   | 781.52              |
| $295.4\pm0.3$   | 20 | $951.385 \pm 53.794$   | 941.02              |
| $293.9\pm0.3$   | 12 | $1094.141 \pm 24.985$  | 1060.3              |
| $293.3\pm0.3$   | 10 | $1107.040 \pm 44.709$  | 1112.9              |
| $292.5\pm0.3$   | 10 | $1178.732 \pm 43.243$  | 1188                |
| $290.7\pm0.3$   | 30 | $1379.724 \pm 30.869$  | 1379.7              |
| $289.1\pm0.3$   | 18 | $1477.329 \pm 46.999$  | 1581.1              |

ature measurement. The error in viscosity resulting from a temperature uncertainty of  $\pm 0.3$  K at T = 289.1 K, the worst case, where  $\eta = 1.581$  Pa·s and  $d\eta/dT \approx -0.135$  Pa·s·K<sup>-1</sup>, would be  $\approx 2.58$  %. However, a more typical uncertainty while at higher temperatures would be  $\pm 0.1$  K corresponding to an error in viscosity of 0.031 %.

The working equations used to determine the viscosity from measurements of the resonance bandwidth have been discussed in detail elsewhere<sup>1–3,8,9</sup> and were used without modification. The viscosity reported is an average obtained from between 10 and 35 measurements.

*Materials.* The certified reference fluid for viscosity N350 was supplied by Cannon from lot number 06201. In our temperature range of (289.1 to 420.7) K at a pressure of 0.1 MPa, the cited viscosity varied from (0.0063 to 1.581) Pa·s (the latter is about 2800 times the viscosity used to obtain the wire radius); Cannon also provided certified values of the mass density. The wire radius was determined with methylbenzene (obtained from Aldrich Chromasolv, HPLC) with a mole fraction purity >0.999.

### **Results and Discussion**

The average viscosities,  $\langle \eta \rangle$ , obtained from *N* measurements at a pressure of 0.1 MPa as a function of temperature for N350 are listed in Table 1, along with the suppliers' cited values, and shown in Figure 1 as relative deviations from the suppliers' values. In Table 1, the uncertainties for our values are one standard deviation of *N* measurements. The peak-to-peak differences shown in Figure 1 are  $\langle \pm 10 \rangle$  with an average difference over our temperature range of  $-2.19 \rangle$  (suggesting a slight systematic offset) that is entirely consistent with our cited uncertainty in temperature; an uncertainty of  $\pm 10 \rangle$  in viscosity is acceptable for the evaluation and production of petroleum reserves.<sup>10</sup> The average of the absolute value of the differences is 3.42  $\rangle$ . The fluid density used in the working equations to obtain viscosity was provided by the supplier of



**Figure 1.** Relative deviation,  $\Delta \eta / \eta = \{\eta(\text{expt}) - \eta(\text{cert})\}/\eta(\text{cert})$ , of the viscosity of certified reference fluid N350 determined with the vibrating wire viscometer with nominal wire radius of 0.2 mm plotted against  $\eta(\text{cert})$ . The error bars correspond to one standard deviation of *N* observations at each temperature.



**Figure 2.** Relative deviation,  $\Delta \eta/\eta = {\eta(\text{expt}) - \eta(\text{cert})}/{\eta(\text{cert})}$ , of the viscosity of certified reference fluid N350 determined with the vibrating wire viscometer with nominal wire radius of 0.2 mm plotted against  $\eta(\text{cert})$  for viscosity  $\eta < 0.2$  Pa·s. The error bars correspond to one standard deviation of *N* observations at each temperature.



**Figure 3.** Resonance quality factor, Q, for the vibrating wire viscometer with nominal wire radius of 0.2 mm immersed in certified reference fluid for viscosity N350 as a function of viscosity  $\eta$ (cert). The solid line is an empirical representation of the results  $Q = 177.25\eta^{-0.6712}$ .

N350 with an uncertainty that introduces a negligible additional error in our estimates of viscosity. The measured viscosity covers the range from (0.0063 to 1.581) Pa·s, a range that covers the viscosity of conventional and a good fraction of the so-called heavy oil.

On an abscissa expanded by a factor of 7.5, Figure 2 shows the fractional differences between our experimental viscosities and the cited values at  $\eta < 0.2$  Pa·s. In the viscosity range (6.3 to 100) mPa·s, the differences are biased to about -3.8 %, and that may arise because as the wire radius increases the sensitivity to viscosity decreases. As expected, increasing the nominal wire radius from (0.075 to 0.2) mm, a factor of 2.7, increased the upper operating viscosity from (0.2 to 1.6) Pa·s, a factor of 7.85, while still operating at a viscosity of 0.57 mPa·s.

In Figure 3, the quality factor, Q, of the fundamental mode is shown as a function of viscosity using the measurement procedure described in ref 1. At  $\eta = 0.2$  Pa·s, we found Q = 5.8 which is in reasonable agreement with the value of 6.6 obtained from Figure 5 of ref 1 for a wire of radius 0.2 mm. The difference between these two values probably arises from the difference in resonance frequency resulting from the different wire tensions.

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