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An Absolute Viscometer for Liquids: Measurement of the Viscosity of Water at $T = 298.15$ K and $p = 0.1$ MPa

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A vibrating-wire viscometer has been formed from cold-drawn centerless-ground tungsten wire of a nominal radius of 0.15 mm. The working equations for the vibrating-wire viscometer require values of the wire radius and internal damping. The radius of the wire was determined by contact micrometry combined with laser interferometry while the internal damping was obtained from measurements in vacuum. With these values, the vibrating-wire instrument provides an absolute measure of the viscosity of liquids. The apparatus has been used to measure the viscosity of water at a temperature of 293.15 K and a pressure of 101.325 kPa. The result obtained differs by less than 0.03 % from the International Organization for Standardization (ISO) recommendation (ISO/TR 3666, 1998) for the viscosity of water. The relative expanded ($k = 2$) uncertainty of the viscosity obtained from the vibrating-wire viscometer is ± 0.9 %, that is, 3.6 times greater than the relative uncertainty of the ISO recommendation. This work illustrates, for the first time, that a vibrating wire can be operated as an absolute viscometer for liquids.

Introduction

The vibrating-wire viscometer, formed from a tensioned wire clamped at both ends, when immersed in a fluid provides viscosity from the measurement of the width of the fundamental resonance using a hydrodynamic model.[1−5] These working equations require knowledge of the internal damping of the wire in vacuum, the radius and density of the wire, and the density of the fluid sample. The uncertainty of the measured viscosity is determined by the uncertainty of the wire radius[6] R (eq 9 of ref 7) and, depending on the magnitude of the viscosity to be measured, also the internal damping in vacuum Δ_0 (eq 3 of ref 7). If both R and Δ_0 were determined independently, the vibrating-wire viscometer would be an absolute viscometer.

In practice, the wire damping factor and radius are usually determined by measurements in vacuum and when the wire is immersed in a fluid for which both the viscosity and the density are known, respectively. Typically, the fluid used for the wire radius calibration is methylbenzene,[8,9] but as demonstrated by Caetano et al.,[28] water is preferred.

In this work, the wire radius was determined using contact micrometry combined with laser interferometry, and in the absence of an alternative approach, the value of Δ_0 was determined from the resonance line width in vacuum. The procedure used to determine Δ_0 means that all energy losses in the viscometer, including that within the wire, are determined. For measurements of the viscosity of liquids on the order of 1 mPa·s with an uncertainty of < ± 1 %, the working equations require the determination of Δ_0 with an uncertainty of < ± 10 %. The conjecture that a viscometer formed in this manner can be absolute requires validation through measurements with a fluid of independently known viscosity.

The International Organization for Standardization (ISO) recommended absolute standard for viscosity is water and is given the value $\eta(\mathrm{H_2O}, 293.15\ \mathrm{K}, 101.325\ \mathrm{kPa}) = (1.001_6 \pm 0.001_7)$ mPa·s.[10] This value is primarily based on the measurements of Swindells et al.[11] who employed a capillary method: the uncertainty associated with this value can be ignored when values from different laboratories are compared, but when a true value of viscosity is required, the relative uncertainty is probably greater than ± 0.25 %.[12] The variation of the viscosity of water at $p = 101.325$ kPa with temperature relative to the reference value has been discussed in the literature[13−18] and may be determined as a function of both temperature and pressure over a wide range from the correlation adopted by the International Association of Water and Steam reported by Huber et al.[19]

The viscosity of water obtained in this work at a temperature of 293.15 K and a pressure of 101.325 kPa differs by less than 0.03 % from the ISO recommended value[10] and demonstrates, for the first time, that a vibrating wire can be operated as an absolute viscometer for liquids.

Working Equations. In this work, with a Newtonian fluid, the vibrating wire has exact working equations that have been fully described in the refs 1 to 5 and summarized by Kandil et al.[7]

Experimental Section

The apparatus consists of a vibrating-wire viscometer, formed from a wire-clamped at both ends, a pressure vessel, and magnets that were described in detail previously.[6,20] This viscometer has been used to measure the viscosity of certified reference fluids N10 and S20[6,20,21] and to study measurements of viscosity with flowing fluid,[22] compare both transient and steady-state measurement techniques,[23] and demonstrate mea-

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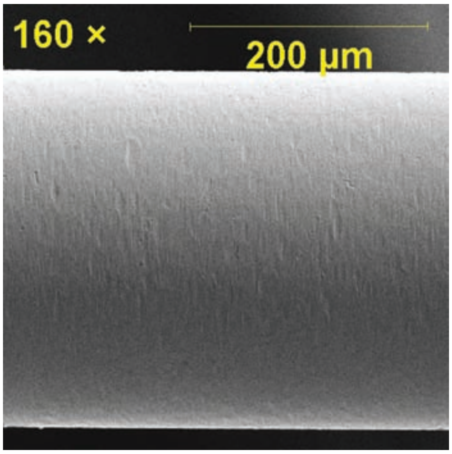

Figure 1. Outer surface of cold-drawn centerless ground tungsten wire of a nominal diameter of 0.3 mm used in this work, viewed with a scanning electron microscope at a magnification of 160.

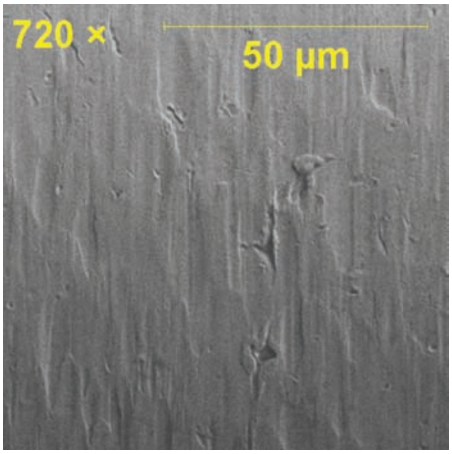

Figure 2. Outer surface of cold-drawn center-less ground tungsten wire of a nominal diameter of 0.3 mm used in this work, viewed with a scanning electron microscope at a magnification of 720.

surements with a wire of a diameter of about 0.2 mm at viscosities up to ≈ 1.6 Pa·s.[24]

In this work, a tungsten wire of nominal radius 0.15 mm of length 52 mm was drawn taut between two clamps to provide a fundamental resonance frequency of about 520 Hz in vacuum and 486 Hz when immersed in water.

The wire was obtained from Nippon Tungsten, Fukuoka, Japan and was cold-drawn and mechanically centerless ground; this wire fabrication method and source were used for the measurements reported in ref 21 where the wire diameter provided by the manufacturer was 0.148 59 mm and was found to differ by 0.009 % from the value obtained from measurements of the resonance line width when immersed in methylbenzene.

The radius of the tungsten wire was measured by the John Stoup of the Engineering Metrology Group, Precision Engineering Division, National Institute of Standards and Technology. The measurements used a contact micrometer combined with a laser displacement interferometer. Measurements were performed at 25 mm intervals along the wire of a nominal length of 300 mm at 250 axial positions to provide the artifact geometry and thus overcome the effects on the average $\langle R\rangle$ reported. The diameter was measured 1 750 times along the length of the wire. For the 52 mm long wire used in this work, the wire radius and expanded uncertainty are $R = (0.148\ 91_2 \pm 0.000\ 04_3)$ mm; for this wire $\delta R/\langle R\rangle = 2.9\cdot10^{-4}$, that is, a factor of about 45 lower than that determined from measurements with the same viscometer but different wire using methylbenzene for which $\delta R/\langle R\rangle = 1.3\cdot10^{-2}$.[6,20,21] We will discuss the effect of wire radius on the measurement of viscosity in the Results and Discussion.

The density of tungsten, required in the working equations, was determined gravimetrically, using a wire of length 0.3 m, to be $(19\ 300 \pm 5)$ kg·m^{-3} where the uncertainty is sufficient for our purpose.

The importance of the rugosity of the outer surface of the wire on the measurement of viscosity has been reported by Wilhelm et al.[25] for gases and by Ciotta and Trusler[26] in a systematic study with liquids. The outer surface of the wire used in this work was viewed with a scanning electron microscope and is shown in Figure 1 at a magnification of 160 and Figure 2 at a magnification of 720. Figures 1 and 2 can be compared with Figure 4 of ref 26 to illustrate the relatively smooth exterior surface of centerless ground wire relative to cold-drawn and gold plated wire.

The data acquisition scheme has been described elsewhere.[6] However, a lock-in time constant of 1 s, rather than the 0.3 s typically used previously,[6,7,20,22,24] was chosen to increase the signal-to-noise ratio. The thermostat, thermometer, and pressure gauge used for the measurements have been described by Goodwin et al.[27] The methods used to measure temperature and pressure are summarized here.

The temperature of the bath fluid was determined on the International Temperature Scale of 1990 (ITS-90) using a long-stem platinum resistance thermometer (Isotech model 909B) that gave a resolution and absolute uncertainty of ± 1 mK, at each temperature, specified by ITS-90. During the time required to measure the resonance frequency (about 360 s) the temperature of the bath fluid varied, in the worst case, by about $\delta T = 3$ mK; for water at $T \approx 293$ K and $p \approx 0.1$ MPa, for which the $(\partial\eta/\partial T)_p \approx -0.024$ mPa·s·K^{-1}, this δT gives rise to $|\delta\eta| = 7.2\cdot10^{-5}$ mPa·s, which is insignificant for this work.[19]

Pressures of about 0.1 MPa were measured using a resonating quartz barometer (Paroscientific 740-16B with a maximum operating pressure of 0.11 MPa) with an uncertainty cited by the supplier of 0.008 % of full scale that corresponds to $\delta p = 8.8$ Pa; for water at $T \approx 293$ K and $p \approx 0.1$ MPa $(\partial\eta/\partial p)_T \approx -3\cdot10^{-4}$ mPa·s·MPa^{-1} and δp results in $|\delta\eta| = 3\cdot10^{-9}$ mPa·s.[19]

Deionized, distilled water with an electrical conductivity of about $6\cdot10^{-6}$ S·m^{-1} was degassed by boiling prior to use in the viscometer.

The Δ_0 was determined from measurements at a pressure $p < 1$ Pa with the result $\Delta_0 = (8.33 \pm 0.24)\cdot10^{-4}$, where the standard uncertainty is cited. The Δ_0 was also determined after the measurements with water and found to differ insignificantly from this value. However, $\Delta_0 = 8.33\cdot10^{-4}$ is about 2.5 times greater than the value estimated from an empirical representation of Δ_0, reported previously for different wires and viscometers,[6,7,20,22,24] as a function of R; $\Delta_0 = 16.9\cdot10^{-4}\cdot(R/\text{mm})$ for which the fractional standard deviation of the fit was $\sigma(\Delta_0)/\langle\Delta_0\rangle = 0.245$. There are two plausible explanations for the larger than anticipated Δ_0. The first arises from within the wire holder (shown in Figure 1 of ref 6) that was formed from nine parts: four of K-Monel for the two clamps that were each held together with two austenitic stainless steel 304 screws and one Shapal-M tube to provide electrical isolation and mechanical support between the clamps. The other arises from motion of the wire holder within the pressure vessel, shown in Figure 2 of ref 6. In the absence of measurements to identify the source of the larger than anticipated Δ_0, no specific explanation can be provided for the additional loss.

The working equations return the ratio of density to viscosity and require the density of the fluid to determine viscosity. A relative uncertainty of ± 0.1 % in fluid density yields a relative uncertainty of ± 0.05 % in the viscosity determined from the vibrating wire.[28] In this case, the density of water was obtained by combination of the measured temperature and pressure with the equation of state of Wagner and Pruss[29] for which the relative uncertainty in density was estimated to be ± 0.0001 %; this uncertainty contributes insignificantly to the uncertainty of the viscosity obtained in this work. The equation of state[29] was coded within the National Institute of Standards and Technology, Standard Reference Database 23 Version 8.1, commonly known by the acronym REFPROP.[30]

Results and Discussion

The viscosity of water was determined with the vibrating-wire viscometer three times each with a different water sample, and the results obtained for $\eta(\mathrm{H_2O}, 293.15\ \mathrm{K}, 101.325\ \mathrm{kPa})$ are as follows: (1) $(1.001_6 \pm 0.009_0)$ mPa·s; (2) $(1.004_9 \pm 0.009_0)$ mPa·s; and (3) $(0.999_2 \pm 0.008_9)$ mPa·s. The average value of these three measurements is $\langle\eta\rangle(\mathrm{H_2O}, 293.15\ \mathrm{K}, 101.325\ \mathrm{kPa}) = (1.001_9 \pm 0.009_0)$ mPa·s. Small corrections have been applied, which are less than the expanded uncertainty, to reduce the reported viscosity to the stated temperature and pressure using the correlation reported by Huber et al.[19] and implemented in REFPROP.[30] The uncertainties, at a confidence interval of 0.95 ($k = 2$), were obtained by combining in quadrature estimated standard relative uncertainties arising from the following sources: (1) the fit to the measured voltages V (eqs 1, 2, and 4 of ref 7) over the fundamental resonance, which contributed ± 0.4 % to $100\cdot\delta\eta/\eta$; (2) the error from Δ_0 that contributed to the relative uncertainty in η of < 0.1 %; and (3) $\mathrm{d}\eta/\mathrm{d}T$ that contributed ± 0.002 % to the relative uncertainty in η because for water at $T \approx 293$ K and $p \approx 0.1$ MPa $(\partial\eta/\partial T)_p \approx -0.024\ \mathrm{mPa\cdot s\cdot K^{-1}}$.[19] Neither the contribution to $\delta\eta$ arising from $\mathrm{d}\eta/\mathrm{d}p$ nor $\delta R/R$ were included because these were considered negligible; for water at $T \approx 293$ K and $p \approx 0.1$ MPa $(\partial\eta/\partial p)_T \approx -3\cdot10^{-4}\ \mathrm{mPa\cdot s\cdot MPa^{-1}}$.[19]

The expanded ($k = 2$) relative uncertainty in our measurement of the viscosity of water is ± 0.9 %, that is, about 3.6 times that of the accepted true value reported in ref 10; it is perhaps fortuitous that $\langle\eta\rangle$ differs from the accepted value by less than 0.03 %. However, this work does illustrate that a vibrating-wire viscometer can be operated as an absolute instrument for liquids with an expanded relative uncertainty of about ± 0.9 %.

There are numerous potential improvements that could be made to the apparatus that might decrease the uncertainty in the measured viscosity, and these include the method used to clamp the wire and the location of the viscometer within the pressure vessel; both are described in ref 6. We will now discuss the wire radius that determines the operating viscosity range of the viscometer and the effect of radius on the uncertainty of the measured viscosity.

The inverse of the resonance quality factor Q (defined in refs 6 and 7) as a function of viscosity provides a means of selecting a wire radius for a particular viscosity range: as R increases, so does the upper operating viscosity as shown in Figure 1 of ref 31. For tungsten wire of a density of 19 300 $\mathrm{kg\cdot m^{-3}}$ with a resonance frequency of 650 Hz immersed in a fluid of a density of 1 000 $\mathrm{kg\cdot m^{-3}}$, Q can be estimated as a function of viscosity from the working equations. For a fluid with a viscosity of 1 mPa·s, a wire of radius 0.05 mm has an estimated quality factor of 29, while for a wire of radius 0.15 mm the quality factor is calculated to be 93; $Q = 2$ is obtained for $R = 0.05$ mm at $\eta = 66$ mPa·s, while for $R = 0.15$ mm the $Q = 2$ occurs at $\eta = 567$ mPa·s. Increasing R by a factor of 3 increases the operating viscosity range at a Q by a factor of about 8.6.

The sensitivity of the vibrating wire to viscosity is proportional to the product $Q\cdot\mathrm{d}Q^{-1}/\mathrm{d}\eta$. Decreasing R from (0.15 to 0.05) mm increases $Q\cdot\mathrm{d}Q^{-1}/\mathrm{d}\eta$ by about 8 %. On the sole basis of this estimate, we conclude that decreasing the wire radius to 0.05 mm will reduce the expanded uncertainty of the measurement by ± 0.2 % to about ± 0.7 %; further measurements will be performed with wires of nominal radii of (0.05 and 0.1) mm to evaluate this hypothesis.

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