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DETERMINING MOTOR INERTIA OF A STRESS-CONTROLLED RHEOMETER

Sarah A. Klemuk¹ and Ingo R. Titze^{1,2}

¹National Center for Voice and Speech at the Department of Communication Sciences and Disorders, The University of Iowa

²The Denver Center for the Performing Arts

Synopsis

Viscoelastic measurements made with a stress-controlled rheometer are affected by system inertia. Of all contributors to system inertia, motor inertia is the largest. Its value is usually determined empirically and precision is rarely if ever specified. Inertia uncertainty has negligible effects on rheologic measurements below the coupled motor/plate/sample resonant frequency. But above the resonant frequency, G' values of soft viscoelastic materials such as dispersions, gels, biomaterials, and non-Newtonian polymers, err quadratically due to inertia uncertainty. In the present investigation, valid rheologic measurements were achieved near and above the coupled resonant frequency for a non-Newtonian reference material. At these elevated frequencies, accuracy in motor inertia is critical. Here we compare two methods for determining motor-inertia accurately. For the first (commercially-used) phase method, frequency responses of standard fluids were measured. Phase between G' and G'' was analyzed at 5–70 Hz for motor inertia values of 50–150% of the manufacturer's nominal value. For a newly-devised two-plate method (10 mm and 60 mm parallel plates), dynamic measurements of a non-Newtonian standard were collected. Using a linear equation of motion with inertia, viscosity, and elasticity coefficients, G' expressions for both plates were equated and motor inertia was determined to be accurate (by comparison to the phase method) with a precision of $\pm 3\%$. The newly developed two-plate method had advantages of expressly eliminating dependence on gap, was explicitly derived from basic principles, quantified the error, and required fewer experiments than the commercially used phase method.

I. INTRODUCTION

System inertia affects measurements made with a stress controlled rheometer [Ferry (1980); Krieger (1990); Macosko (1994)]. Erroneous thixotropic behavior of a Newtonian oil and non-Newtonian materials were attributed to system inertia for short 60-sec torque ramp times [Krieger (1990)]. Similar results were attributed to a combination of (1) torque response times during ramping and (2) system inertia, the two of which had varying degrees of influence depending on the viscosity of the Newtonian fluid [Baravian and Quemada (1998a)]. Coupling effects between system inertia and material properties of weak viscoelastic solids have also been shown [Baravian *et al.* (2007); Baravian and Quemada (1998b); Titze *et al.* (2004)]. From vibration theory, the impedance below resonance is governed by the elastic properties of the system and impedance above resonance is governed by the inertial properties of the system [Fletcher and Rossing (1998)]. Strain per unit of torque γ^*/T^* is derived from momentum balance, Rabinowitsch and Weisenberg's constitutive equation, and Boltzmann's superposition equation (see appendix for details):

$$\frac{\gamma^*}{T^*} = \frac{1.5}{G' \pi R^3} \frac{1}{1 - (\omega/\omega_0)^2 + iG''/G'} \quad (1)$$

The undamped resonant frequency of the system is

$$\omega_0 = \sqrt{\frac{K}{I}}, \quad (2)$$

where system stiffness is K , and system inertia is I . Under oscillatory conditions and taking into account the shear elastic modulus, ω_0 becomes

$$\omega_0 = \sqrt{\frac{\pi G' R^4}{2hI}} \quad (3)$$

where R is the sample radius and h is the gap [Titze *et al.* (2004)]. Data from Titze *et al.* showed that γ^* / T^* was highly sensitive to G' below the resonance peak, indicating that G' can be determined with accuracy in this region. Response γ^* / T^* remained sensitive to G' at the resonant frequency and up to 2 times the resonant frequency, but response curves eventually converged for varying values of G' or G''/G' , indicating that the response was no longer controlled by material properties, but rather by system inertia.

To confirm this effect, rheologic measurements were made in our lab of a PDMS Newtonian fluid (polydimethylsiloxane) (Brookfield Engineering Laboratories, Inc., Middleboro, MA) and non-Newtonian reference material SRM-2490 (polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane) (National Institute of Standards and Technology, Gaithersburg, MD) [Schultheisz and Leigh (2002)]. Data were collected at 0.1 – 150 Hz with a 40 mm parallel plate, and a 0.1 mm gap. Applied stress was 8 Pa at 25° C and applied strain was 0.2 at 20° C for the PDMS and SRM-2490 respectively. Raw data were Fourier transformed to give input torque T and resulting angular displacement θ , where θ was converted to shear strain using geometry constants of gap and $\frac{3}{4}$ radius. As predicted by Eqs. (1) and (2), the resonant frequency of PDMS was near zero (Fig. 1). Response magnitude theoretically went to infinity because elasticity of the fluid was near zero. The response curve of SRM-2490 was similar to the Newtonian fluid in the low frequency region, where its loss tangent G''/G' is 25 at 0.0063 Hz, but as the loss tangent approached and dropped below 1, material response declined and then rose to a resonant peak at 62 Hz (Fig. 1). Beyond this frequency, the system was inertially driven.

These results were consistent with other studies. System inertia was used to control free oscillations in creep mode [Baravian and Quemada (1998b)], and material stress of a weak gel was more than 3 times greater than the applied stress at a coupling frequency of 8 Hz [Baravian *et al.* (2007)]. The coupled system response, operating in a stress control mode, exceeded the yield stress, after which data were considered unreliable. Accurate measurements are still possible beyond resonance, however, if two criteria are satisfied: (1) rotational torque is delivered in a strain control mode or with stress adjustments that maintain linear viscoelastic strains even at resonance, and (2) accurate system inertia is known with quantified uncertainty.

Primary contributors to system inertia are the moving plate in contact with the sample and the motor assembly. Plate inertia is calculated using direct micrometer measurements and knowledge of the material density. The motor assembly inertia value is not calculated from

basic principles because it includes electromagnetic inertia and is unique to each instrument. Technicians usually use empirical methods to specify a value, but it may be prone to error depending on the empirical method used, the diligence of the technician, available time, or some combination. Sources of uncertainty in viscoelastic measurements, such as torque, phase, plate radius, temperature, and sample to sample differences have been considered, but system inertia has not [Schultheisz and Leigh (2002)].

Uncertainty in motor inertia generates quadratic errors in G' above the resonant frequency. G' values of the PDMS and SRM-2490 were calculated with Eqs. (34) and (35) (found in the appendix) using data shown in Fig. 1, geometry constants, raw phase values, and the nominal motor inertia (as determined by a manufacturer technician). When inertia values were increased or decreased by 10% in the calculation, quadratic deviation in PDMS G' values (away from near zero) was evident at 7 Hz (Fig. 2). For SRM-2490, deviation in G' values began near the resonant frequency of 62 Hz. Both G' and G'' data continued along a predictable trajectory up to 104 Hz (Fig. 3). The question is which G' data set is most accurate. Rheologic data are clearly possible beyond the coupled resonant frequency, but the accuracy of the rheology is linked to how well the motor inertia value is known.

The aim of the present investigation was to compare a new method to a traditional method for determining motor inertia in terms of accuracy and precision. Accuracy is the extent to which the measured value is close to the actual value, and precision is the degree to which the measurement is reproducible.

II. METHODS

A. Materials and instrumentation

Fluids used for instrument calibration and motor inertia determination were two PDMS Newtonian fluids (Brookfield Engineering Laboratories, Inc., Middleboro, MA), with certified viscosities of $975 \text{ cP} \pm 1\%$ and $11800 \text{ cP} \pm 1\%$ at 25°C ., and non-Newtonian reference material SRM-2490 (National Institute of Standards and Technology, Gaithersburg, MD). A Bohlin CVO-120 stress-controlled rheometer (Malvern Instruments Ltd, Worcestershire, UK) was used for all rheologic testing. Temperature of the rheometer base plate was regulated by a Neslab RTE-111 water-circulating, temperature control unit (Thermo Electron Corporation, Waltham, MA). A stainless steel thermal cover was used to maintain the set temperature surrounding the sample.

Parallel plate attachments varied across experiments. A 45 mm diameter plate (PP45) was used for gap calibration, a 40 mm diameter plate (PP40) was used for the phase method, and a polycarbonate 60 mm diameter plate (PP60) and a 10 mm diameter plate (PP10) were used for the two-plate method. The largest plate was made of polycarbonate in order to reduce plate inertia. Three different radial measurements and three different thickness measurements R_i and L_i were made in triplicate on every plate using a micrometer of accuracy 0.05 mm. These dimensions, along with density ρ for the material were used to calculate the inertia value for each plate. The formula for inertia of a cylinder was used, summing the inertias of the three cylinders that comprised any given plate.

B. Instrument calibration and sample preparation

For each experiment a plate was zeroed to the base plate at ambient temperature. The plate was then raised, covered with a stainless steel cover, left to equilibrate to the base plate temperature for 10 minutes, and then re-zeroed. Standard sample insertion was followed; the sample completely filled the gap and the vertical sides of the sample matched the perimeter of the upper plate. The sample was then re-covered, and testing began 5–10 minutes later.

The amplitude of shear stress applied to a sample was chosen either to match protocols from previous publications or by performing an amplitude sweep at various frequencies. Amplitudes that produced no change in G' or G'' indicated that linear viscoelastic theory was satisfied.

Errors in maintaining a uniform gap were diminished by checking the parallel orientation around the plate perimeter, checking the trueness of the base and the plate attachment and by calibrating the gap with a Newtonian standard. Trueness of plates used for uniform gap evaluation was found to be 3–10 μm by The University of Iowa Medical Instruments staff. The gap was calibrated using the PP45 plate and the 975 cP PDMS at 25° C. The gap offset was adjusted until viscosity accuracy was within 3% and variability of viscosity measurements at the two gaps of 1.00 mm and 0.100 mm were a minimum (3%).

The gap loading criterion was satisfied for all frequencies over 50 Hz and for each material in the present investigation. According to Schrag [(1977)], the ratio of the wavelength λ_s propagating vertically through the sample thickness h should be greater than 40 to eliminate sample inertia effects:

$$\lambda_s/h > 40 \quad (4)$$

Ferry [(1980)] restated the criterion in terms of the sample viscosity η , the sample density ρ , and the frequency at which the sample is being sheared:

$$h \ll \sqrt{\frac{2\pi\eta}{\rho f}}. \quad (5)$$

The density of SRM-2490 was 775–795 kg/m^3 and the density of the PDMS fluids was 940 kg/m^3 , as reported in the material data safety sheet for each material. The right-hand side of Eq. (5) was calculated and then divided by 40 to satisfy Eq. (4). For all data reported, the gap loading condition was met or was exceeded.

C. Phase method

Phase measurements between G'' and G' of the 975 cP PDMS and the 11880 cP PDMS were collected at four frequencies using the PP40 plate at a 0.100 mm gap. The frequencies were 5, 12.1, 29, and 70 Hz, and the temperature was 25° C. Oscillatory stress was set to 30 Pa and 300 Pa for the low and high viscosity fluid, respectively, based on results from amplitude sweeps at 0.1, 1, 10, and 63 Hz. Motor inertia was then varied by 50%–150% of the nominal motor inertia value.

D. Two-plate method

1. Amplitude sweeps—Amplitude sweeps at several frequencies were performed on SRM-2490 in stress-control mode. Twenty shear stress amplitudes were applied in a linear distribution from 1 to 100 Pa at 0.1, 1.0, 10.0, and 63.4 Hz. The resulting stresses that generated a flat viscoelastic response were 0.5 Pa for 0.01–0.252 Hz and 5.2 Pa for 0.4–150 Hz when using the PP10 and 22 Pa for all frequencies when using the PP60 plate, both at 0.2 mm gaps.

2. Phase correction—Titze et al (2004) showed that reported phase measurements exceeded the theoretical limit of $-\pi$, using a stress-controlled rheometer, a parallel plate attachment, and SRM-2490 at frequencies greater than 50 Hz. The excessive phase delays were presumably due to electromagnetic inertia of the rheometer motor. A linear phase correction was then

proposed and was used in the present investigation. To summarize, Eq. (1) was used to solve for the phase angle ϕ at an angular frequency x times the resonant frequency:

$$\omega_{hi} = x\omega_0 \quad (6)$$

$$\widehat{\phi}(\omega_{hi}) = \frac{\tan^{-1} \frac{G''_0}{G'_0}}{|1 - x^2|} - \pi \quad (7)$$

Eq. (7) uses values of G' and G'' at resonance, G'_0 and G''_0 respectively, because both values are measured with great accuracy at this frequency and are presumed to show little change within one frequency decade. The slope between Eq. (7) and the raw phase $\phi_{raw}(\omega_{hi})$ at the same high frequency was calculated

$$\alpha = \frac{\widehat{\phi}(\omega_{hi}) - \phi_{raw}(\omega_{hi})}{\omega_{hi}}, \quad (8)$$

and a linear phase correction was constructed:

$$\phi(\omega) = \phi_{raw}(\omega) + \alpha\omega. \quad (9)$$

Titze *et al* (2004) demonstrated that this linear phase correction, anchoring the phase at 2.2 times the resonant frequency, improved viscoelastic measurements of SRM-2490 at frequencies above resonance.

3. Frequency sweeps—Delivered torque, resulting angular displacement, and phase were recorded for SRM-2490 using the PP60 and the PP10 plates. Data were collected at 32 frequencies logarithmically spaced between 0.01 and 150 Hz. The resonant frequency was located, and raw phase measurements were linearly corrected using Eqs. (6)–(9). G' values were calculated using Eq. (34). A single inertia value was calculated at each frequency by equating the large sample G'_L to the small sample G'_s :

$$G'_L = G'_s \quad (10)$$

$$\frac{2h}{\pi R_L^4} \left(\frac{M_L \cos \phi_L + (I_{motor} + I_L) \omega^2 \theta_L}{\theta_L} \right) = \frac{2h}{\pi R_s^4} \left(\frac{M_s \cos \phi_s + (I_{motor} + I_s) \omega^2 \theta_s}{\theta_s} \right) \quad (11)$$

Solving for motor inertia gave

$$I_{motor} = \frac{1}{R_s^4 - R_L^4} \left[\frac{R_L^4}{\omega^2} \frac{M_s \cos \phi_s}{\theta_s} - \frac{R_s^4}{\omega^2} \frac{M_L \cos \phi_L}{\theta_L} + R_L^4 I_s - R_s^4 I_L \right]. \quad (12)$$

4. Error determination—Error determination of the motor inertia, based on Eq. (12), was carried out using the error propagation equation, also known as the absolute error [Bevington and Robinson (1992)]

$$\sigma_x^2 = \sigma_u^2 \left(\frac{\partial x}{\partial u} \right)^2 + \sigma_v^2 \left(\frac{\partial x}{\partial v} \right)^2 + \sigma_w^2 \left(\frac{\partial x}{\partial w} \right)^2 + \dots \quad (13)$$

In using this equation, it was assumed that all input parameter errors were independent and relatively small so that second order derivatives in the Taylor expansion were negligible. A total of 18 variables make up Eq. (12), 9 related each to small plate and large plate measurements. They are: torque, angular displacement, phase, three radial dimensions of the plate, and three length or thickness dimensions of the plate. The plate dimensions come from explicitly calculating the plate inertia values; error in material density was not considered. Errors in each parameter were known through triplicate measurements either on the rheometer or with a micrometer, and partial derivatives were calculated. The relative error was calculated by weighting the absolute error with the mean inertia value squared and the coefficient of variation was computed by taking the square root of the relative error and multiplying by 100. The relative error, written out, then, was

$$\left(\frac{\sigma_{I_{motor}}}{I_{motor}} \right)^2 = \left(\frac{1}{I_{motor}} \right)^2 \left[\begin{aligned} & \left(\frac{\partial I_{motor}}{\partial M_L} \sigma_{T_L} \right)^2 + \left(\frac{\partial I_{motor}}{\partial \theta_L} \sigma_{\theta_L} \right)^2 + \left(\frac{\partial I_{motor}}{\partial \phi_L} \sigma_{\phi_L} \right)^2 + \\ & \left(\frac{\partial I_{motor}}{\partial M_S} \sigma_{T_S} \right)^2 + \left(\frac{\partial I_{motor}}{\partial \theta_S} \sigma_{\theta_S} \right)^2 + \left(\frac{\partial I_{motor}}{\partial \phi_S} \sigma_{\phi_S} \right)^2 + \\ & \left(\frac{\partial I_{motor}}{\partial R_L} \sigma_{T_L} \right)^2 + \left(\frac{\partial I_{motor}}{\partial R_{L1}} \sigma_{R_{L1}} \right)^2 + \left(\frac{\partial I_{motor}}{\partial R_{L2}} \sigma_{R_{L2}} \right)^2 + \\ & \left(\frac{\partial I_{motor}}{\partial R_S} \sigma_{R_S} \right)^2 + \left(\frac{\partial I_{motor}}{\partial R_{S1}} \sigma_{R_{S1}} \right)^2 + \left(\frac{\partial I_{motor}}{\partial R_{S2}} \sigma_{R_{S2}} \right)^2 + \\ & \left(\frac{\partial I_{motor}}{\partial t_L} \sigma_{t_L} \right)^2 + \left(\frac{\partial I_{motor}}{\partial t_{L1}} \sigma_{t_{L1}} \right)^2 + \left(\frac{\partial I_{motor}}{\partial t_{L2}} \sigma_{t_{L2}} \right)^2 + \\ & \left(\frac{\partial I_{motor}}{\partial t_S} \sigma_{t_S} \right)^2 + \left(\frac{\partial I_{motor}}{\partial t_{S1}} \sigma_{t_{S1}} \right)^2 + \left(\frac{\partial I_{motor}}{\partial t_{S2}} \sigma_{t_{S2}} \right)^2 + \end{aligned} \right] \quad (14)$$

III. RESULTS AND DISCUSSION

A. Phase method

The criterion for an acceptable motor inertia value, as determined by the phase method, was that the phase between G'' and G' should be $88.5^\circ - 90^\circ$

$$\phi_{material} = \tan^{-1} \left(\frac{G''}{G'} \right). \quad (15)$$

The most accurate inertia value was identified when the criterion was satisfied for multiple frequencies. Phase measurements of the high viscosity PDMS at 5 Hz, did not constrain the motor inertia; the criterion was satisfied for 50% – 120% of the nominal value (Fig. 4, top). At higher frequencies, the phase criterion was nearly satisfied at 50% of the nominal motor inertia. This discrepant value compared to the nominal motor inertia raises questions about using a high viscosity fluid with the phase method. The gap loading criterion was satisfied so the resulting value was not attributed to sample inertia. Instead, it may be that for short time scales, this high viscosity Newtonian standard is actually viscoelastic and should not be used for motor inertia determination.

Motor inertia values were more predictably constrained using the lower viscosity Newtonian standard (Fig. 4, bottom). The 88.5° phase criterion was satisfied for motor inertia values 90% – 110%, 95% – 100%, and 97% – 100% of the nominal inertia at 5 Hz, 12.1 Hz, and 29 Hz respectively. Taking into account the phase trends at 70 Hz, the best motor inertia value is 97% of nominal motor inertia with uncertainty of $\pm 3\%$.

B. Two-Plate method

The two-plate method included frequency sweep measurements of SRM-2490 using a small and large plate (PP10 and PP60). Plate/sample radii, the only variation between the frequency sweeps, shifted the resonant frequency of the motor/plate/sample system response, calculated as strain per Pascal stress at the $\frac{3}{4}$ radius position (Fig. 5 top left). The PP10 system generated a resonant frequency at 1.6 Hz and the PP60 system generated a resonant frequency at 86 Hz. As an additional comparison, measurements were made using a PP45 parallel plate. Response was coincident with the PP10 and PP60 data at low frequencies, but resonance shifted to 63 Hz. As expected [Titze *et al.* (2004)], the larger radius compared to the PP10 resulted in higher resonance, and the slightly smaller radius compared to the PP60 shifted resonance lower.

Response magnitudes decreased more than 10 times from 0.01 to 1 Hz for all three plates. This was due to the diminishing loss tangent of SRM-2490 (25 down to 1). For PP10, response values above 1.6 Hz decreased even more rapidly, progressing to an inertially controlled system. Phase measurements shifted by 180 degrees between 1 and 8 Hz (shown in Fig. 5 bottom left), characterizing transition from material control to inertial control. When the sample no longer moved in synchrony with the torque input, phase leveled to a steady state condition. Additional phase decline after 16 Hz may indicate other resonances in the motor assembly. For the large plate systems, phase measurements continued to increase beyond 1.6 Hz where the loss tangent of SRM-2490 was less than one. Material properties continued to control system response but were eventually overcome by motor inertia beyond 100 Hz because of the weak elastic modulus of the material.

Impact of material stiffness on the two-plate method was tested using a stiffer NIST non-Newtonian fluid, SRM-2491, polydimethylsiloxane [Schultheisz *et al.* (2003)]. Its cross-over frequency (loss tangent ~ 1) is around 10 Hz at 20°C , and the material is much stiffer, with shear moduli approximately 25 times higher than SRM-2490 at 10 Hz. For this material, a resonant frequency was evident at 11.3 Hz when using the PP10 plate (Fig. 5 top right). No resonant frequency was detected when using the PP45 plate due to the larger plate and the high elastic modulus of the material. Phase measurements showed a trend towards an inertially controlled system when using the PP10 plate, but a steady state was not achieved (Fig. 5, bottom right). Rather, rheology of the material influenced response and phase for both the small and large plate systems throughout the tested frequency range. It was not expected, therefore, that a motor inertia value could be obtained without the sample contributing to the value.

Using four different combinations of plates and materials, motor inertia values were calculated with Eq. (12) (Fig. 6 top). Motor inertia values fell into three general regions along the frequency axis. Up to about 4 Hz, inertia values tended to decline, as did variability, this being the frequency range of viscoelastic liquid measurement and a transition into an inertially dominated system. Between 4 and 25 Hz, three out of the four combinations produced a leveling off of calculated motor inertia. The PP60/PP10 and PP45/PP10 combinations using SRM-2490 generated nearly identical motor inertia values. Values were higher for the PP45/PP10 combination using SRM-2491, because the material's elasticity was still influencing response measures.

Properties of SRM-2490 allow the material to completely decouple from the PP10 system and to fully couple to the PP45 or PP60 systems in the 4 – 25 Hz frequency range. The material

contribution could therefore be subtracted in Eq. (12). The corresponding coefficients of variation, calculated with Eq. (14), also plateau in this second frequency region, with the SRM-2490 PP60/PP10 system having the lowest variability. When SRM-2490 responses using the PP60/PP45 were used to calculate motor inertia, anomalous negative values were obtained in the 4 – 25 Hz range and variability exceeded 200%. With these geometries, the resonant frequencies are too close together and the system is dominated by the material properties for both the large and the ‘small’ PP45 plate, giving erroneous motor inertia values.

The third frequency range, 25 – 150 Hz, generated similarly low motor inertia values regardless of the plate or material system, but variability was unpredictable. The variability suggested that the motor/plate/sample system in this frequency range is less stable and should therefore be avoided for motor inertia determination.

To better understand the key contributors to the inertia curve in Fig. 6, the terms in Eq. (12) were broken up. The first term on the right hand side, $1/(R_s^4 - R_L^4)$ is large and negative: $-1.24 \times 10^6 \text{ m}^{-4}$. The next two terms are dynamic and contain the inverse response of each plate coupled to the opposite plate’s radius:

$$\text{dynamic term} = \frac{R_L^4 T_s \cos \phi_s}{\omega^2 \theta_s} - \frac{R_s^4 T_L \cos \phi_L}{\omega^2 \theta_L} \quad (16)$$

It is the behavior of the dynamic term that coincides with the steady state condition around 10 Hz. For frequencies below 1 Hz, the dynamic term is changing, and considerable variability is present (Fig. 7). A steady state is achieved between 6.4 and 11 Hz, after which data become unstable. Within the steady state frequency range, the dynamic term is $1.24 \times 10^{-11} \pm 1.73 \times 10^{-13} \text{ kg m}^6$, a 1.4% variability across 4 frequencies and 3 samples. Instability on either side of 6.4 – 11 Hz region is evidenced in Fig. 6 (bottom) where coefficients of variation became very large. The final two terms in Eq. (12) are constants and together are $3.2 \times 10^{-14} \text{ kg m}^6$, making their contribution negligible. In summary, the leveling around 10 Hz, a frequency nearly equidistant between the two resonant peaks of the PP10 and PP60 SRM-2490 data sets, indicates that a stable, inertially driven system exists from which a motor inertia value can be calculated with accuracy and precision. The final motor inertia value, taken at 8.6 Hz, was $1.544 \times 10^{-5} \pm 4.5 \times 10^{-7} \text{ kg m}^2$, with a 2.9% variability based on the Taylor Series error.

IV. SUMMARY AND CONCLUSION

Viscoelastic measurements near or above the coupled resonant frequency are obtainable but are greatly affected by motor inertia. Error in the inertia value generates quadratic error in G' measurements. The purpose of the present investigation was to determine an accurate motor inertia value with quantified precision. Of the two motor-inertia-determining methods tested and the four materials used, motor inertia values for two conditions were closely matched. Results from the traditional phase method using a 975 cP Newtonian standard gave an inertia value of $1.558 \times 10^{-5} \text{ kg m}^2 \pm 3\%$. Results from the two-plate method using NIST SRM-2490 non-Newtonian standard gave a motor inertia value of $1.544 \times 10^{-5} \text{ kg m}^2 \pm 3\%$. The phase and two-plate methods produced values within 1% of each other.

The new two-plate method for determining motor inertia has several advantages over the traditional phase method. Mathematical derivation for the two-plate method is clearly laid out, all parameters and errors are directly measured, and variability is analyzed in two ways: (1) a Taylor Series expansion and (2) analysis of the dynamic term Eq. (16) across 4 frequencies. The largest contribution to motor inertia variability in the two-plate method was the phase angle. For all frequencies 11 Hz and lower, phase angle accounted for 53–99% of the

uncertainty. This raises questions about error associated with the phase method if phase is the sole determiner of inertia.

Another disadvantage of the phase method is the number of experiments required to obtain a value within $\pm 3\%$. Approximately 12 experiments, with three replicates each, were performed using a Newtonian standard material at 5 Hz, 12 Hz, 29 Hz, and 70 Hz to get the precision desired. This amounts to several hours of testing. A quick traditional phase method test may result in a motor inertia value $\pm 10\%$, variability that is problematic when seeking accurate rheological measurements at higher frequencies. The two-plate method, using NIST SRM-2490, gave nearly the same results as the in-depth phase method, thus ensuring accuracy. The precision was clearly defined and evaluated in two ways, and only two frequency sweeps with three replicates were required.

Extending frequency-dependent measurement of soft biological tissues, gels, dispersions, and non-Newtonian standards are important for complete material characterization, and in particular for characterizing voice, hearing, and skin tissues – organs known to experience high frequency vibration exposure. With the advent of piezoelectric technology, accurate data overlap in the mid-frequencies 1 – 100 Hz, as measured by a stress controlled rheometer and by a rotational vibrator, will also ensure accurate rheologic characterization of soft viscoelastic solids into the kHz range [Kirschenmann and Pechhold (2002)].

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APPENDIX: THEORETICAL ANALYSIS OF RHEOMETER/SAMPLE SYSTEM

Key mathematical formulations of linear viscoelastic measurement are revisited to show connections between sample and system momentum conservation, constitutive equations, and inertia. Traditional and valid assumptions about the material are that it is an incompressible material undergoing sinusoidal deformation in the θ direction and in the z plane, where ambient pressure is constant and gravity forces are negligible [Macosko (1994)]. The equation of motion for this material is

$$\rho \left(\frac{\partial v_\theta}{\partial t} \right) = \partial_z \tau_{\theta z} \quad (17)$$

The tangential velocity component in the θ direction is related to both the angular velocity and the shear rate by assuming a small angle approximation. Acceleration is then written as:

$$\dot{v}_\theta = \ddot{\theta} r = z \ddot{\gamma} \quad (18)$$

Inserting strain acceleration in place of the velocity acceleration, integrating both sides, and restating in terms of the sample inertia I_S gives

$$\tau_{z\theta} = \frac{I_S}{\pi R^3} \ddot{\theta} \quad (19)$$

It is useful to write Eq. (19) in terms of torque experienced by the sample rather than stress. To accomplish this, integration over differential torque, in terms of shear stress, is performed

$$\begin{aligned} M &= \int F dr = 2\pi \int_0^R r^2 \tau_{z\theta}(\gamma, t) dr \\ &= \frac{2}{3} \pi R^3 \tau_{z\theta} \end{aligned} \quad (20)$$

The resulting statement of momentum conservation for the sample is

$$M = \frac{2}{3} I_s \ddot{\theta} \quad (21)$$

The general constitutive equation for shear stress in a parallel plate drag flow rheometer follows that of the Rabinowitsch and Weissenberg equation [Macosko (1994); Soskey and Winter (1984); Steffe (1996); Tanner and Walters (1998); Yoshimura and Prudhomme (1988)]:

$$G(t, \gamma_R) = \frac{M(t, \gamma_R)}{2\pi R^3 \gamma_R} \left(3 + \frac{\partial \ln \left[\frac{2M(t, \gamma_R)}{\pi R^3} \right]}{\partial \ln \gamma_R} \right) \quad (22)$$

Applied torque is directly proportional to strain in the linear viscoelastic region so that the derivative is equal to 1 and the relaxation modulus is only a function of time:

$$G(t) = \frac{2M(t)}{\pi R^3 \gamma_R} \quad (23)$$

Eq. (23) is then written in the form of Boltzmann's superposition equation by considering differential elements of dM and $d\gamma_R$, making a change of variable from $d\gamma_R$ to dt , and writing torque in terms of shear stress via Eq. (20) $M = 2/3 \pi R^3 \tau_{z\theta}$.

The result is

$$\tau_{z\theta} = 0.75 \int G(t) \dot{\gamma}_R dt \quad (24)$$

Notice that Eq. (24) has a factor of 0.75, because we used the linear viscoelastic limit of the Rabinowitsch expression. The linear function is convolved, and a small angle approximation is made in order to express sinusoidal angular displacement and velocity

$$\tau_{z\theta} = \frac{0.75R}{h} \int_{-\infty}^{t'} G(t-t') \dot{\theta}_R(t') dt' \quad (25)$$

A change of variable $s = t - t'$, use of the trigonometric identity $\cos(\omega t - \omega s) = \sin \omega t \sin \omega s + \cos \omega t \cos \omega s$, and using the abbreviated standard variables of G' and G'' [Ferry (1980)], gives Boltzmann's superposition equation, having gotten there via Rabinowitsch's equation

$$\tau_{z\theta} = \frac{0.75R}{h} \left\{ \theta_R G' + \dot{\theta}_R G'' / \omega \right\} \quad (26)$$

The final constitutive equation for the material is written in terms of torque rather than stress within the sample

$$M = \frac{\pi R^4}{2h} \left\{ \theta_R G' + \dot{\theta}_R G'' / \omega \right\} \quad (27)$$

Properties of the motor/plate assembly and the sample are included in a complete force balance equation. This leads to formulas for G' and G'' that were used for all rheologic calculations made in the present investigation, excluding calculations made by the rheometer software. Total torque generated by the sample includes components of the material constitutive equation Eq. (27) and the material equation of motion Eq. (21):

$$M_{total} = \frac{2}{3} I_s \ddot{\theta}_R + \frac{\pi R^4}{2h} \left\{ \theta_R G'_s + \dot{\theta}_R \eta_s \right\} \quad (28)$$

where $\eta_s = G''/\omega$. The total torque generated by the motor/plate assembly is

$$M_{total} = K_m \theta + D_m \dot{\theta} + I_m \ddot{\theta} \quad (29)$$

Combining Eqs. (28) and (29) gives

$$M_{Total} = M_{total} + M_{motor} = \left(\frac{\pi R^4}{2h} G'_s \theta_R + K_m \theta_R \right) + \left(\frac{\pi R^4}{2h} \eta_s \dot{\theta}_R + D_m \dot{\theta}_R \right) + \left(I_m \ddot{\theta}_R + \frac{2}{3} I_s \ddot{\theta}_R \right) \quad (30)$$

Specifications for the air bearing of the motor/plate assembly state that stiffness and damping are negligible, causing Eq. (30) to reduce to

$$M_{Total} = \left(\frac{\pi R^4}{2h} G'_s \right) \theta_R + \left(\frac{\pi R^4}{2h} \eta_s \right) \dot{\theta}_R + \left(I_m + \frac{2}{3} I_s \right) \ddot{\theta}_R \quad (31)$$

The solution to this linear differential equation is written for a sinusoidal torque and a responding sinusoidal deformation in complex notation:

$$M_0 e^{i\phi} = \theta_0 \left[\left(\frac{\pi R^4}{2h} G' \right) + \left(\frac{\pi R^4}{2h} \eta_s \right) i\omega - \left(I_m + \frac{2}{3} I_s \right) \omega^2 \right] \quad (32)$$

The magnitude of the torque and angular displacement are M_0 and θ_0 , respectively. Then we use the relationship between exponential complex notation and trigonometric complex notation $e^{i\phi} = \cos \phi + i \sin \phi$ to write

$$\frac{M_0}{\theta_0} (\cos \phi + i \sin \phi) = \left(\frac{\pi R^4}{2h} G' \right) + \left(\frac{\pi R^4}{2h} \eta_s \right) i\omega - \left(I_m + \frac{2}{3} I_s \right) \omega^2 \quad (33)$$

The real part of Eq. (33) contains the sample elastic modulus, the sample inertia, and the motor inertia, and the imaginary part of Eq. (33) contains the viscous modulus of the sample. Solving for the material properties gives

$$G'_s = \frac{2h}{\pi R^4} \left[\frac{M_0}{\theta_0} \cos \phi + \left(I_m + \frac{2}{3} I_s \right) \omega^2 \right] \quad (34)$$

$$G''_s = \omega \eta_s = \frac{2h}{\pi R^4} \frac{M_0}{\theta_0} \sin \phi \quad (35)$$

These final expressions for G' and G'' were derived from basic principles of momentum conservation. A set of assumptions, appropriate to the geometry and the type of stresses in the system, reduced the equation of motion to two terms. Then a constitutive equation was worked out from fundamental tenants of rheology theory (i.e., Rabinowitsch and Boltzmann), terms were manipulated, and the final expressions for G' and G'' were the result.

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FIG. 1.

Comparison of system response across frequency for 975 cP PDMS Newtonian fluid and SRM-2490 non-Newtonian fluid using a PP40 plate and 0.1 mm gap

FIG. 2.
Deviations of near-zero G' values of a Newtonian standard due to motor inertia variation.

FIG. 3. Elastic and viscous modulus measurements of SRM-2490 at 20° C, and deviations of expected elastic moduli due to motor inertia uncertainty

FIG. 4.

Phase measurements between G'' and G' as a function of changing motor inertia value at four frequencies for 11880 cP (top) and 975 cP (bottom) Brookfield Newtonian PDMS

FIG. 5. Motor/plate/sample response (top left) and linearly corrected phase (bottom left) using non-Newtonian SRM-2490 with a PP10 stainless steel parallel plate, a PP60 polycarbonate parallel plate, and a PP45 titanium parallel plate. Motor/plate/sample response (top right) and linearly corrected phase (bottom right) using non-Newtonian SRM-2491 with a PP10 and a PP45 parallel plate

FIG. 6. Calculated motor inertia (Eq. (12)) (top) and corresponding coefficient of variation using (Eq. (14)) (bottom) using SRM-2490 data collected with a PP60/PP10 combination, a PP45/PP10 combination, a PP60/PP45 combination, and SRM-2491 data collected with a PP45/PP10 combination

FIG. 7.
Absolute value of Eq. (16), which is the dynamic portion of Eq. (12), as a function of frequency