NIST SPECIAL PUBLICATION **260-143**

**U. S. DEPARTMENT OF COMMERCE/Technology Administration**
**National Institute of Standards and Technology**

*Standard Reference Materials*®

# Certification of the Rheological Behavior of SRM 2490, Polyisobutylene Dissolved in 2,6,10,14-Tetramethylpentadecane

**Carl R. Schultheisz and Stefan D. Leigh**

NIST

*T*he National Institute of Standards and Technology was established in 1988 by Congress to ''assist industry in the development of technology . . . needed to improve product quality, to modernize manufacturing processes, to ensure product reliability . . . and to facilitate rapid commercialization . . . of products based on new scientific discoveries.''

NIST, originally founded as the National Bureau of Standards in 1901, works to strengthen U.S. industry's competitiveness; advance science and engineering; and improve public health, safety, and the environment. One of the agency's basic functions is to develop, maintain, and retain custody of the national standards of measurement, and provide the means and methods for comparing standards used in science, engineering, manufacturing, commerce, industry, and education with the standards adopted or recognized by the Federal Government.

As an agency of the U.S. Commerce Department's Technology Administration, NIST conducts basic and applied research in the physical sciences and engineering, and develops measurement techniques, test methods, standards, and related services. The Institute does generic and precompetitive work on new and advanced technologies. NIST's research facilities are located at Gaithersburg, MD 20899, and at Boulder, CO 80303. Major technical operating units and their principal activities are listed below. For more information contact the Publications and Program Inquiries Desk, 301-975-3058.

## Office of the Director
- National Quality Program
- International and Academic Affairs

## Technology Services
- Standards Services
- Technology Partnerships
- Measurement Services
- Information Services

## Advanced Technology Program
- Economic Assessment
- Information Technology and Applications
- Chemistry and Life Sciences
- Materials and Manufacturing Technology
- Electronics and Photonics Technology

## Manufacturing Extension Partnership Program
- Regional Programs
- National Programs
- Program Development

## Electronics and Electrical Engineering Laboratory
- Microelectronics
- Law Enforcement Standards
- Electricity
- Semiconductor Electronics
- Radio-Frequency Technology[1]
- Electromagnetic Technology[1]
- Optoelectronics[1]

## Materials Science and Engineering Laboratory
- Intelligent Processing of Materials
- Ceramics
- Materials Reliability[1]
- Polymers
- Metallurgy
- NIST Center for Neutron Research

## Chemical Science and Technology Laboratory
- Biotechnology
- Physical and Chemical Properties[2]
- Analytical Chemistry
- Process Measurements
- Surface and Microanalysis Science

## Physics Laboratory
- Electron and Optical Physics
- Atomic Physics
- Optical Technology
- Ionizing Radiation
- Time and Frequency[1]
- Quantum Physics[1]

## Manufacturing Engineering Laboratory
- Precision Engineering
- Automated Production Technology
- Intelligent Systems
- Fabrication Technology
- Manufacturing Systems Integration

## Building and Fire Research Laboratory
- Applied Economics
- Structures
- Building Materials
- Building Environment
- Fire Safety Engineering
- Fire Science

## Information Technology Laboratory
- Mathematical and Computational Sciences[2]
- Advanced Network Technologies
- Computer Security
- Information Access and User Interfaces
- High Performance Systems and Services
- Distributed Computing and Information Services
- Software Diagnostics and Conformance Testing
- Statistical Engineering

[1]At Boulder, CO 80303.
[2]Some elements at Boulder, CO.

# NIST Special Publication 260-143

*Standard Reference Materials*®

# Certification of the Rheological Behavior of SRM 2490, Polyisobutylene Dissolved in 2,6,10,14-Tetramethylpentadecane

Carl R. Schultheisz[1] and Stefan D. Leigh[2]

[1]Polymers Division, Materials Science and Engineering Laboratory
[2]Statistical Engineering Division, Information Technology Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-8544

**U.S. GOVERNMENT PRINTING OFFICE**
**WASHINGTON: 2002**

**Table of Contents**

## List of Tables

# Certification of the Rheological Behavior of SRM 2490, Polyisobutylene Dissolved in 2,6,10,14-Tetramethylpentadecane

Carl R. Schultheisz[1] and Stefan D. Leigh[2]
[1]Polymers Division
[2]Statistical Engineering Division
National Institute of Standards and Technology
Gaithersburg, MD 20899

## ABSTRACT

The certification of the rheological properties of Standard Reference Material® (SRM) 2490, a non-Newtonian fluid consisting of polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane, is described. The viscosity and the first normal stress difference were measured in steady shear at rates between 0.001 $s^{-1}$ and 100 $s^{-1}$ at 0 °C, 25 °C and 50 °C. The linear viscoelastic storage modulus G' and loss modulus G" were also measured in dynamic oscillatory measurements between 0.04 rad/s and 100 rad/s in the temperature range between 0 °C and 50 °C and master curves calculated using time temperature superposition.

## 1. Introduction

This report describes the certification of the rheological properties of Standard Reference Material® (SRM) 2490, a non-Newtonian fluid consisting of polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane. This fluid demonstrates shear thinning (decreasing viscosity with increasing shear rate) and development of normal stresses, which are rheological behaviors common to polymeric materials [1-6]. The viscosity and the first normal stress difference were measured in steady shear at shear rates between 0.001 s$^{-1}$ and 100 s$^{-1}$ at 0 °C, 25 °C and 50 °C. The linear viscoelastic storage modulus G' and loss modulus G" were also measured in dynamic oscillatory measurements between 0.04 rad/s and 100 rad/s in the temperature range between 0 °C and 50 °C, and master curves were calculated using time temperature superposition.

SRM 2490 replaces an earlier material, SRM 1490, which consisted of polyisobutylene dissolved in normal hexadecane. SRM 1490 was certified in 1977 [7], but discontinued in 1990 when deviations from the certified properties were identified. Among the possible reasons for the deviation from the certified properties were the packaging of the material and evaporation of the solvent. SRM 1490 was delivered in large (20 liter, 5 gallon) plastic containers, with material transferred to bottles for sale as needed; material taken from different large containers might have led to a difference in properties from one lot of bottles to the next. Similarly, evaporation of the solvent through the plastic containers over time could have led to changes in properties; in the measurements that showed a deviation, the viscosity was found to be higher than the certified value, which is consistent with solvent evaporation. In addition to these problems, the normal hexadecane used as a solvent also limited the useful temperature range of the material, since normal hexadecane crystallizes at 18 °C (64 °F) [8].

A polyisobutylene solution was still considered a good choice as an SRM, because the saturated hydrocarbon structure of polyisobutylene is chemically stable, and polyisobutylene poses no health risks. Also, polyisobutylene is widely available commercially and therefore cost effective. In addition, polyisobutylene solutions have been widely used as model non-Newtonian fluids, because they demonstrate significant shear thinning and relatively large normal stresses [9-13].

In an effort to overcome the difficulties associated with the normal hexadecane used as the solvent for SRM 1490, a new solvent was chosen for SRM 2490. The new solvent (2,6,10,14-tetramethylpentadecane [14]) also has a saturated hydrocarbon structure for chemical stability, but it was chosen to have a branched alkane structure to inhibit crystallization. Differential scanning calorimetry measurements show no evidence of crystallization or vitrification of this solvent down to temperatures as low as −60 °C. The new solvent also has a slightly higher molar mass than hexadecane, so its rate of evaporation is somewhat lower. This material is also commercially available from several sources, and its cost is comparable to that of normal hexadecane.

In an effort to eliminate possible problems of variations between large containers and/or differences arising from bottling at different times, all of the polymer solution was mixed in one large container and then bottled in 100 ml quantities at the same time.

The common name for normal hexadecane is *cetane*, which comes from the Latin word *Cetacea,*

the name for the order that contains whales and porpoises [15]. Cetane is found in sperm whale oil. The common name for 2,6,10,14-tetramethylpentadecane is *pristane*, which comes from *pristis*, the Latin word for shark [15]. Pristane is found in shark liver oil [14].

## 2. Preparation, Bottling, and Sampling of SRM 2490

SRM 2490 consists of polyisobutylene (Aldrich Chemical, catalog number 18,146-3, CAS number 9003-27-4) dissolved in 2,6,10,14-tetramethylpentadecane (Aldrich Chemical, catalog number T2,280-2, CAS number 1921-70-6); the solution contains a mass fraction of 0.114 polyisobutylene. The supplier identifies the polyisobutylene as being stabilized with 500 ppm 2,6-di-*tert*-butyl-4-methylphenol, and gives the average Mv ca. 1,200,000; Mw ca. 1,000,000; Mn ca. 600,000 as measured by GPC/MALLS; the density of the polyisobutylene is given as 0.898 g/cm$^3$. The density of 2,6,10,14-tetramethylpentadecane is given by the supplier as 0.785 g/cm$^3$. The specification of a concentration having a mass fraction of 0.114 polyisobutylene was extrapolated from the viscosity of a solution prepared at NIST with a mass fraction of 0.10 polyisobutylene [12] and the concentration dependence of solutions of polyisobutylene dissolved in n-hexadecane [10]. The solution was intended to have a zero-shear-rate viscosity of 200 Pa·s at 25 °C. Subsequently, a solution having a mass fraction of 0.114 polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane prepared at NIST was found to have a zero-shear-rate viscosity of approximately 150 Pa·s at 25 °C [13].

Mixing and bottling of SRM 2490 was contracted out to Cannon Instrument Company. Quantities of polyisobutylene and 2,6,10,14-tetramethylpentadecane in the correct proportions were placed in four separate containers, and once the dissolution process was complete, the contents of the four containers were mixed together in a single container and then bottled. Initially, it was hoped that the dissolution process could proceed at room temperature with minimum agitation, in order to minimize degradation of the polyisobutylene [11]. The dissolution process was found to be very slow, however, so intermittent heating up to 50 °C and additional mechanical agitation were employed to speed up the process. Some degradation of the polyisobutylene may have occurred during mixing, as the zero-shear-rate viscosity of the resulting solution was approximately 100 Pa·s at 25 °C, as compared to the value of 150 Pa·s for the solution prepared at NIST. Some of that difference might also be attributed to different lots of the polyisobutylene. The solution was packaged in quantities of 100 ml in amber glass bottles with phenolic caps and conical polyethylene cap liners, and 439 bottles were delivered to NIST.

The bottles were numbered and filled sequentially. Ten bottles were taken as samples for homogeneity testing and for certification. One bottle was chosen randomly from each successive sequence of 45 bottles (1-45, 46-90, etc.). This procedure was intended to uncover any systematic variation in concentration that might have occurred in the course of the bottling process.

Before taking a sample from any bottle for testing in the rheometer, the bottle was turned end over end at a rate of 1 revolution per 10 min for a minimum of 30 min. For turning, the bottles were held with a three-jaw clamp attached to a small motor/gear assembly (McMaster -Carr). Turning the bottles was intended to ensure that the material within each bottle was homogeneous,

in case there was any settling due to gravity.

## 3. Testing

All rheological testing was carried out in a Rheometric Scientific, Inc. ARES controlled-strain rheometer. This rheometer employs a force rebalance transducer that measures both torque and normal force. The torque transducer has two ranges, a low range with higher sensitivity for torque with magnitude up to 0.02 N·m (200 $g_f$·cm, where $g_f$ indicates a unit of gram force, which is not an acceptable unit of the SI, but is commonly used), and a high range for torque with magnitude up to 0.2 N·m (2000 $g_f$·cm). The normal force transducer has a single range capable of measuring normal forces up to 20 N (2000 $g_f$) in magnitude. The rheometer transducer was calibrated each day before any testing. Calibration was accomplished following the manufacturer's instructions [16]; the procedure consists of hanging a known mass from a fixture mounted to the transducer to apply a known torque or normal force. The calibration masses were checked on routinely calibrated electronic balances at NIST (OHAUS GA200D for masses of 200 g or less, Mettler PE 3600 for masses greater than 200 g) and found to be within 0.02 % of the specified mass. The dimensions of the calibration fixture were also measured at NIST and found to be within 0.2 % of the specification. For dynamic measurements, the phase angle was also calibrated each day before any testing, again following the manufacturer's instructions.

Five sets of tests were conducted. The first set of tests investigated the homogeneity of the material delivered; the object of these tests was to compare the zero-shear-rate viscosity at 25 °C, which was used as an acceptance criterion. Four sets of certification tests were then performed: steady shear at 25 °C, 50 °C and 0 °C to determine the viscosity and first normal stress difference as functions of the shear rate, and dynamic tests over a range of frequency during temperature sweeps from 0 °C to 50 °C to determine the linear viscoelastic properties.

The steady shear tests were performed using cone and plate fixtures, and the dynamic tests were performed using parallel plate fixtures. The fixtures were cleaned with acetone before each experiment.

Fresh nitrile gloves were worn for cleaning the fixtures and for loading the samples. Gloves were worn both to prevent skin contact with the material and to prevent contaminating the fixtures or the samples with grease or dirt from the operator's hands. The material safety data sheet for 2,6,10,14-tetramethylpentadecane does not indicate any specific health hazards, but does suggest the possibility of skin irritation, as with most organic solvents. No unusual measures were taken for disposing of the samples after testing.

Temperature control in the ARES is maintained by a forced air system into an insulated chamber surrounding the test section; the air can be cooled with liquid nitrogen or heated with electrical resistance coils. The temperature control was calibrated before each series of certification tests using a NIST-calibrated thermistor [17]. The combined standard uncertainty in the temperature was estimated to be 0.1 °C.

3.1 Homogeneity Testing

The criterion for accepting the delivery of the bottled solution was that the zero-shear-rate

viscosity measured at 25 °C from a representative sample of the bottles should have a maximum spread of 4 % of the average zero-shear-rate viscosity. Because the time frame for homogeneity testing was limited, and because a smaller amount of data was required than for the steady shear certification tests, some of the parameters for the homogeneity tests were chosen differently than for the certification tests. The fixtures used were 50 mm diameter cone and plate, with a cone angle specified to be 0.0197 rad. The cone is truncated, and the distance between the point where the vertex of the cone should be and the plane of truncation is 0.048 mm. The dimensions of the fixtures were measured at NIST and found to agree with the manufacturer's specification within the uncertainties. The testing was performed at 25 °C, with the temperature control based on the temperature read by platinum resistance thermometers near the forced air inlets (ARES Control Mode 3). The tests were run using the Steady Rate Sweep template supplied with the ARES software, with the tests specified to start at a shear rate of $0.01 \text{ s}^{-1}$ and then at increasing rates, taking five points per decade up to a maximum of $100 \text{ s}^{-1}$. All of these tests were performed using the more sensitive low range of the torque transducer; the transient overshoot in the viscosity typically led to an overload in the torque at a shear rate of $16 \text{ s}^{-1}$. Measurements at each shear rate were made in both the clockwise and counterclockwise directions, with the shear applied for 100 s and then measurements of the torque and normal force averaged over the next 30 s. Before loading each sample, the cone and plate fixtures were installed and set to a nominal gap of 1 mm. The temperature control chamber was then closed and the fixtures brought to 25 °C for 30 min. The gap was then zeroed using the Autozero capability of the ARES (the fixtures brought together until they touched, to establish the baseline from which to set the gap to the proper dimension). The temperature control chamber was then opened, and the upper fixture was raised approximately 60 mm. As mentioned above, each bottle was turned end over end for at least 30 min before testing to thoroughly mix the material inside; a sample was then spooned onto the lower plate fixture, and the upper cone fixture was lowered to the specified offset of 0.048 mm. The sample was then trimmed flush with the edges of the fixtures, and the temperature control chamber closed. The specified Steady Rate Sweep test was then started; the test sequence included a 30 min hold at 25 °C to allow the temperature to reach steady state. Three samples were tested from each of the ten bottles; the thirty samples were tested in a random order. Typically, three samples were tested each day.

The testing indicated that the homogeneity of the material was within the specified acceptance criteria. Statistical tests using analysis of variance (ANOVA) showed that test-sequence effects were not statistically significant compared to the variability among bottles.

3.2 Steady Shear Testing for Certification

The steady shear testing for certification was similar to the tests for homogeneity, but there were some changes in the test procedure and parameters. A broader range of shear rates was employed, and the mode of temperature control was changed to use a platinum resistance thermometer that is in contact with the underside of the lower plate fixture (ARES control mode 2). Tests were carried out at 25 °C, 50 °C and 0 °C (in that order), and comparison with a NIST-calibrated thermistor was used to adjust the parameters in a look-up table for the temperature controller before beginning a series of tests at a new temperature.

### 3.2.1 Testing at 25 °C

The fixtures were again 50 mm diameter, 0.0197 rad cone and plate, with the cone truncated at 0.048 mm from its vertex. The tests were run using the Steady Rate Sweep template supplied with the ARES software, taking five points per decade. Tests were begun using the more sensitive low range of the torque transducer, sweeping from an initial shear rate of 0.001 $s^{-1}$ up to a shear rate of 10 $s^{-1}$. The instrument was then switched to use the high range of the torque transducer, and a rate sweep from 10 $s^{-1}$ to 100 $s^{-1}$ was performed. (The switch was necessary, because the torque at the higher shear rates was sufficient to overload the low range of the transducer.) Measurements at each shear rate were made in both the clockwise and counterclockwise directions, with the shear applied for 30 s and then measurements of the torque and normal force averaged over the next 30 s. Before loading each sample, the cone and plate fixtures were installed and set to a nominal gap of 1 mm. The temperature control chamber was then closed and the fixtures brought to 25 °C for 30 min. The gap was then zeroed using the Autozero capability of the ARES (the fixtures brought together until they touched, to establish the baseline from which to set the gap to the proper dimension). The temperature control chamber was then opened, and the upper fixture was raised approximately 60 mm. Each bottle was turned end over end for at least 30 min before testing to thoroughly mix the material inside; a sample was then spooned onto the lower plate fixture, and the upper cone fixture was lowered to a position of 0.046 mm. The sample was then trimmed flush with the edges of the fixtures, and the cone was repositioned to the specified gap of 0.048 mm. This procedure was used in an effort to compensate for the expansion of the sample when changing from room temperature to the test temperature of 25 °C, in order to achieve the appropriate spherical sample geometry. The temperature control chamber was closed, and the specified Steady Rate Sweep test was started; the test sequence included a 30 min hold at 25 °C to allow the temperature to reach steady state. Two samples were tested from each of the ten bottles; the twenty samples were tested in a random order.

In reducing the data, the viscosity measurements taken at 10 $s^{-1}$ using the low transducer range and the high transducer range were averaged, although typically the difference was much smaller than the uncertainty of the measurements. Averaging measurements made using rotations in both directions eliminates any problems of an offset in the zero position for the viscosity. The normal force measurement, however, is sensitive to the zero position and is strongly affected by temperature fluctuations that cause transient volumetric changes. The zero position of the normal force measurements was adjusted by averaging the first six measurements and subtracting that value from all subsequent measurements. The normal force in that range (0.001 $s^{-1}$ to 0.01 $s^{-1}$) is well below the sensitivity of the transducer and so can be taken as zero. In changing the transducer from the low torque range to the high torque range, the difference between the two normal force measurements (taken at the same shear rate of 10 $s^{-1}$) was then subtracted from the subsequent measurements taken with the transducer using the high torque range.

The total time for a single test was approximately 90 min, including the 30 min delay to ensure thermal steady state before measurements were begun. A possible source of error in the measurement is evaporation of the solvent during the experiment caused by the forced air

temperature control system. To investigate whether evaporation of the solvent caused a significant change in the sample over the duration of the test, two experiments were performed using 90 min delays inside the temperature chamber before measurements were begun. These tests indicated that the effects of solvent evaporation are not significant compared to the uncertainties in the measurements for tests lasting 90 min inside the chamber at 25 °C. Two additional tests using delays inside the temperature chamber of 6 h before testing showed a relative increase in the viscosity over the tests with 30 min delays of approximately 1 %. A simple linear model is assumed for the relative change in the viscosity with time at a rate of 1 % for an increased delay of 330 min. This assumption leads to the incorporation of a component of relative standard uncertainty of 0.1 % at the first shear rate of 0.001 s$^{-1}$, with the relative standard uncertainty increasing linearly with time to 0.3 % at the end of the test. The effects of evaporation on the measurements of the first normal stress difference were not significant compared to the uncertainties for either the 90 min delay tests or the 6 h delay tests. Since the pressure in the fluid increases toward the center of the cone and plate fixture [1-6], the first normal stress difference measurement might be less sensitive to effects at the edge such as solvent evaporation. The measurement of the first normal stress difference is apparently more sensitive to characteristics of the instrument, such as temperature fluctuations or transducer response than to the evaporation of the solvent. For the first normal stress difference, it is assumed that the effects of evaporation are similar to those found for the viscosity, incorporating a component of relative standard uncertainty of 0.1 % at the first shear rate of 0.001 s$^{-1}$, with the relative standard uncertainty increasing linearly with time to 0.3 % at the end of the test.

### 3.2.2 Testing at 50 °C

The fixtures were again 50 mm diameter, 0.0197 rad cone and plate, with the cone truncated at 0.048 mm from its vertex. The tests were run using the Steady Rate Sweep template supplied with the ARES software, taking five points per decade. Tests were begun using the more sensitive low range of the torque transducer, sweeping from an initial shear rate of 0.001 s$^{-1}$ up to a shear rate of 25.12 s$^{-1}$. The instrument was then switched to use the high range of the torque transducer, and a rate sweep from 25.12 s$^{-1}$ to 100 s$^{-1}$ was performed. (The switch was necessary, because the torque at the higher shear rates was sufficient to overload the low range of the transducer.) Measurements at each shear rate were made in both the clockwise and counterclockwise directions, with the shear applied for 20 s and then measurements of the torque and normal force averaged over the next 30 s. Before loading each sample, the cone and plate fixtures were installed and set to a nominal gap of 1 mm. The temperature control chamber was then closed and the fixtures brought to 50 °C for 30 min. The gap was then zeroed using the Autozero capability of the ARES (the fixtures brought together until they touched, to establish the baseline from which to set the gap to the proper dimension). The temperature control chamber was then opened, and the upper fixture was raised approximately 60 mm. Each bottle was turned end over end for at least 30 min before testing to thoroughly mix the material inside; a sample was then spooned onto the lower plate fixture, and the upper cone fixture was lowered to a position of 0.020 mm. The sample was then trimmed flush with the edges of the fixtures, and the cone was repositioned to the specified gap of 0.048 mm. This procedure was used in an effort to compensate for the expansion of the sample when changing from room temperature to the test temperature of 50 °C, in order to achieve the appropriate spherical sample geometry. The temperature control chamber was closed, and the specified Steady Rate Sweep test was started;

the test sequence included a 30 min hold at 50 °C to allow the temperature to reach steady state. Two samples were tested from each of the ten bottles; the twenty samples were tested in a random order.

In reducing the data, the viscosity measurements taken at 25.12 s$^{-1}$ using the low transducer range and the high transducer range were averaged, although typically the difference was much smaller than the uncertainty of the measurements. Averaging measurements made using rotations in both directions eliminates any problems of an offset in the zero position for the viscosity. The normal force measurement, however, is sensitive to the zero position and is strongly affected by temperature fluctuations that cause transient volumetric changes. The zero position of the normal force measurements was adjusted by averaging the first six measurements and subtracting that value from all subsequent measurements. The normal force in that range (0.001 s$^{-1}$ to 0.01 s$^{-1}$) is well below the sensitivity of the transducer and so can be taken as zero. In changing the transducer from the low torque range to the high torque range, the difference between the two normal force measurements (taken at the same shear rate of 25.12 s$^{-1}$) was then subtracted from the subsequent measurements taken with the transducer using the high torque range.

The total time for a single test was approximately 80 min, including the 30 min delay to ensure thermal steady state before measurements were begun. A possible source of error in the measurement is evaporation of the solvent during the experiment caused by the forced air temperature control system. To investigate whether evaporation of the solvent caused a significant change in the sample over the duration of the test, three experiments were performed using 80 min delays inside the temperature chamber before measurements were begun, and three experiments were performed using 160 min delays. The results of the 80 min delay tests show a viscosity increase of approximately 1 % over the tests with 30 min delays, and the 160 min delay tests show a viscosity increase of approximately 4 %. A simple linear model is again assumed for the relative change in the viscosity with time at the initial rate of 1 % for an additional delay of 50 min. This assumption leads to the incorporation of a component of relative standard uncertainty of 0.6 % at the first shear rate of 0.001 s$^{-1}$, with the relative standard uncertainty increasing linearly with time to 1.6 % at the end of the test. Even with the higher rate of evaporation at this temperature, the effects of evaporation on the measurements of the first normal stress difference were not significant compared to the uncertainties for either the 80 min delay tests or the 160 min delay tests. Some additional tests were performed using a series of rate sweep tests on the same specimen, and the effect of evaporation on the viscosity was consistent with the earlier tests with longer delay times. However, the results for the first normal stress difference were still inconclusive. Again, since the pressure in the fluid increases toward the center of the cone and plate fixture [1-6], the first normal stress difference measurement might be less sensitive to effects at the edge such as solvent evaporation. The measurement of the first normal stress difference is apparently more sensitive to characteristics of the instrument, such as temperature fluctuations or transducer response than to the evaporation of the solvent. For the first normal stress difference, we assume that the effects of evaporation are similar to those found for the viscosity, incorporating a component of relative standard uncertainty of 0.6 % at the first shear rate of 0.001 s$^{-1}$, with the relative standard uncertainty increasing linearly with time to 1.6 % at the end of the test.

### 3.2.3 Testing at 0 °C

The fixtures were again 50 mm diameter, 0.0197 rad cone and plate, with the cone truncated at 0.048 mm from its vertex. The tests were run using the Steady Rate Sweep template supplied with the ARES software, taking five points per decade. Tests were begun using the more sensitive low range of the torque transducer, sweeping from an initial shear rate of $0.001 \text{ s}^{-1}$ to a shear rate of $3.98 \text{ s}^{-1}$. The instrument was then switched to use the high range of the torque transducer, and a rate sweep from $3.98 \text{ s}^{-1}$ to $100 \text{ s}^{-1}$ was performed. (The switch was necessary, because the torque at the higher shear rates was sufficient to overload the low range of the transducer.) Measurements at each shear rate were made in both the clockwise and counterclockwise directions, with the shear applied for 40 s and then measurements of the torque and normal force averaged over the next 30 s. Before loading each sample, the cone and plate fixtures were installed and set to a nominal gap of 1 mm, and then the temperature control chamber was closed and the fixtures brought to 0 °C for 30 min. Temperatures below room temperature are maintained by evaporated liquid nitrogen into the temperature control chamber. The gap was then zeroed using the Autozero capability of the ARES (the fixtures brought together until they touched, to establish the baseline from which to set the gap to the proper dimension). The temperature control chamber was then opened, and the upper fixture was raised approximately 60 mm. The tool temperature was allowed to reach 15 °C before loading the sample to allow the moisture that condensed on the tool when exposed to the atmosphere to evaporate. Each bottle was turned end over end for at least 30 min before testing to thoroughly mix the material inside; a sample was then spooned onto the lower plate fixture, and the upper cone fixture was lowered to a position of 0.090 mm. The sample was then trimmed flush with the edges of the fixtures. The temperature control chamber was closed, and the sample and fixtures cooled for 4 min. The cone was then repositioned to the specified gap of 0.048 mm. This procedure was used in an effort to compensate for the decrease in sample volume when changing from room temperature to the test temperature of 0 °C, in order to achieve the appropriate spherical sample geometry. The temperature control chamber was closed, and the specified Steady Rate Sweep test was started; the test sequence included a 30 min hold at 0 °C to allow the temperature to reach steady state. Two samples were tested from each of the ten bottles; the twenty samples were tested in a random order.

In reducing the data, the viscosity measurements taken at $3.98 \text{ s}^{-1}$ using the low transducer range and the high transducer range were averaged, although typically the difference was much smaller than the uncertainty of the measurements. Averaging measurements made using rotations in both directions eliminates any problems of an offset in the zero position for the viscosity. The normal force measurement, however, is sensitive to the zero position and is strongly affected by temperature fluctuations that cause transient volumetric changes. The zero position of the normal force measurements was adjusted by averaging the first six measurements and subtracting that value from all subsequent measurements. The normal force in that range ($0.001 \text{ s}^{-1}$ to $0.01 \text{ s}^{-1}$) is well below the sensitivity of the transducer and so can be taken as zero. In changing the transducer from the low torque range to the high torque range, the difference between the two normal force measurements (taken at the same shear rate of $3.98 \text{ s}^{-1}$) was then subtracted from the subsequent measurements taken with the transducer using the high torque range.

Because of the slow rate of change of the viscosity demonstrated by the evaporation tests at 25 °C, no evaporation tests were performed at 0 °C. An estimate of the effect of evaporation on the viscosity and first normal stress difference has been made using the measured rates of change in the viscosity at 25 °C and 50 °C coupled with an Arrhenius temperature dependence. This estimate leads to a component of relative standard uncertainty in both the viscosity and first normal stress difference of 0.01 % at the first shear rate of 0.001 s$^{-1}$, with the relative standard uncertainty increasing linearly with time to 0.03 % at the end of the test.

3.3 Dynamic Testing

The fixtures used for the dynamic testing were 50 mm diameter parallel plates with the nominal gap being 1 mm. The tests were run using the Dynamic Frequency/Temperature Sweep template supplied with the ARES software. Measurements were taken at 0 °C, 10 °C, 20 °C, 30 °C, 40 °C, and 50 °C; at each temperature the frequency of oscillation increased from 0.0398 rad/s to 100 rad/s, taking five points per decade. Tests were performed at a strain magnitude of 20 %, and all data was taken using the more sensitive low range of the torque transducer. Although the ARES does have some capability to adjust the position of the fixtures to compensate for thermal expansion, this feature is apparently not yet implemented for the Dynamic Frequency/Temperature Sweep test. Therefore, in an effort to minimize the effects of changes in the gap and changes in the sample volume because of thermal expansion of the tools and the sample itself, the gap was zeroed at 25 °C (the midpoint of the temperature range). The change in the gap caused by thermal expansion of the fixtures was measured to be 1.75 μm/°C (with a standard uncertainty of 0.1 μm/°C). Before loading each sample, the parallel plate fixtures were installed and set to a nominal gap of 1 mm, and then the temperature control chamber was closed and the fixtures brought to 25 °C for 30 min. The gap was then zeroed using the Autozero capability of the ARES (the fixtures brought together until they touched, to establish the baseline from which to calculate the gap). The temperature control chamber was then opened, and the upper fixture was raised approximately 60 mm. Each bottle was turned end over end for at least 30 min before testing to thoroughly mix the material inside; a sample was then spooned onto the lower plate fixture, and the upper plate fixture was lowered. The sample was centered in the fixtures by turning off the motor and rotating the lower plate while lowering the upper plate. The upper plate was lowered until the edges of the sample were flush with the edges of the fixtures at room temperature. The gap was then increased 0.01 mm in an effort to compensate for expansion of the tools and the sample for a temperature change from room temperature to 25 °C. The temperature control chamber was closed with the temperature setpoint at 0 °C. The specified Dynamic Frequency/Temperature Sweep test was started; the test sequence included a 30 min hold at 0 °C to allow the temperature to reach steady state. In an effort to reduce the test time somewhat, the subsequent soak time following each 10 °C increment was shortened to 10 min. One sample was tested from each of the ten bottles; the ten samples were tested in a random order. The minimum gap used was 1.115 mm at 25 °C, and the maximum gap used was 1.435 mm at 25 °C.

In reducing the data for G' and G", a correction to the gap was made to account for thermal expansion of the fixtures. Since the fixtures expand with increasing temperature, the gap decreases with increasing temperature. The actual gap is therefore $h_0 - (1.75\ \mu m/°C)(T - 25\ °C)$, where $h_0$ is the nominal gap, and $T$ is the temperature at which the measurement is made. The

calculated shear moduli are linearly functions of the gap. Therefore, the corrections to the shear moduli at each temperature are given by

$$G'_{corrected} = \left[ \frac{h_0 - (1.75 \ \mu m/°C)(T - 25°C)}{h_0} \right] G'_{calculated}$$

$$G''_{corrected} = \left[ \frac{h_0 - (1.75 \ \mu m/°C)(T - 25°C)}{h_0} \right] G''_{calculated}$$

(1)

### 4. Analysis of Sources of Uncertainty

Uncertainties from each of the sources that are identified are combined through the mathematical formula for the propagation of uncertainties [18]. For a quantity $y$ that is a function of a number of independent quantities $x_i$, with $y = f(x_i)$, the combined standard uncertainty in $y$ (symbol $u_c(y)$) is calculated from the standard uncertainty in each $x_i$ (symbol $u(x_i)$) as

$$u_c^2(y) = \sum_i \left(\frac{\partial f}{\partial x_i}\right)^2 u^2(x_i) \tag{2}$$

For input quantities that are not independent, equation (2) would contain terms involving the covariances, but we will assume that the input variables are independent of one another. An alternative way to express equation (2) is that the combined standard uncertainty in $y$ is the summation in quadrature of the components of the standard uncertainty in $y$ arising from each source $x_i$,

$$u_c^2(y) = \sum_i u^2(y, x_i) \tag{3}$$

In equation (3), $u(y, x_i)$ is the component of standard uncertainty in $y$ arising from source $x_i$, which includes Type A uncertainties determined through statistical analysis of the data and Type B uncertainties determined from the formula for the propagation of uncertainty and/or determined by any other means [18].

4.1 Steady Shear Testing in the Cone and Plate Fixtures

There are a number of possible sources of uncertainty in the measurements of the viscosity and first normal stress difference. The Type A uncertainties associated with variability in the material and random influences on the test conditions were assessed through multiple measurements.

Those sources of uncertainty considered to be Type B are listed below.
1. Temperature
2. Transducer
3. Rotation rate
4. Geometry
   a. Gap
   b. Cone angle
   c. Cone/Plate diameter
   d. Cone truncation
   e. Tilt
   f. Concentricity
5. Solvent evaporation
6. Shear heating
7. Inertia-driven secondary flows
8. Edge effects
9. Surface tension

12

4.1.1 Uncertainty in the Independent Variables: Temperature and Shear Rate

The viscosity and first normal stress difference are tabulated as functions of temperature and shear rate. The standard uncertainty in the temperature is estimated to be 0.1 °C. The advantage of using the cone and plate is that the shear rate is approximately constant throughout the sample. Uncertainty in the shear rate arises from uncertainties in the rotation rate and in the geometry. The nominal shear rate $\dot{\gamma}_0$ in the cone and plate is given by

$$\dot{\gamma}_0 = \frac{\omega_0}{\tan \beta_0} \tag{4}$$

where $\omega_0$ is the specified rate of rotation of the plate, and $\beta_0$ is the nominal cone angle (0.0197 rad). The standard uncertainty in the rotation rate $u(\omega)$ is estimated to be $0.005\omega_0$, and the standard uncertainty in the cone angle $u(\beta)$ is estimated to be $10^{-4}$ rad, based on information from the instrument manufacturer. Measurement of the cone angle at NIST agreed with that given by the manufacturer within that uncertainty. However, it should be noted that Mackay and Dick [19] found much larger uncertainties in a round robin test measuring the geometry of a cone. In equation (4), it is assumed that the cone is perfect and aligned so that the tip would just touch the plate. However, to prevent friction between the fixtures, the tip of the cone is truncated, so there is a small region of decreased shear rate at the center of the fixtures. The manufacturer gives the height of the truncation as 48 μm. There is also uncertainty in the gap between the plate and the position of the tip of the cone. The standard uncertainty in the gap is estimated to be 2.65 μm. Uncertainty in the gap arises from uncertainties in the cone truncation, uncertainty in position at zero gap and at the specified setting, uncertainty in the flatness of the plate and profile of the cone, compliance of the transducer and thermal expansion of the transducer [16, 20-22]. Each of these seven influences was assigned a standard uncertainty of 1 μm. These components of uncertainty were then added in quadrature to calculate the combined standard uncertainty in the gap. Tilting of the axis of the cone with respect to the axis of the plate also introduces uncertainty; the standard uncertainty of the angle of tilt is estimated to be $2 \times 10^{-4}$ rad. The propagation of uncertainties in $\omega$ and $\beta$ into the uncertainty in $\dot{\gamma}$ can be calculated analytically from equation (4). However, uncertainties associated with the cone truncation, the gap and a tilt lead to effects that vary spatially. The uncertainty in the shear rate is therefore calculated as an average over the area of the plate. Incorporating the effects of the truncation, an offset $h_0$ from the intended gap and an angle of tilt $\phi$, the area-averaged shear rate is given by

$$\bar{\dot{\gamma}} = \frac{1}{\pi R^2} \left[ \int_0^{2\pi} \int_0^{r_1} \frac{\omega r^2}{h_0 + r_1 \tan \beta + \phi \sin \theta} dr d\theta + \int_0^{2\pi} \int_{r_1}^{R} \frac{\omega r^2}{h_0 + r \tan \beta + \phi \sin \theta} dr d\theta \right] \tag{5}$$

where $r_1 = 2.45$ mm is the radius of the truncated region, and $R = 25$ mm is the outer radius of the cone or plate. Markovitz et al. [23] examined the effects of geometry on Newtonian viscosity measurements; the terms describing the tilt in equation (5) follow their analysis. The uncertainty associated with each of these sources was calculated separately, using numerical integration [24]. If the angle of tilt is zero, equation (5) reduces to a one-dimensional integral over $r$; but for nonzero tilt the integral is fully two-dimensional and was calculated by first integrating over $r$ and then over $\theta$. Both the truncation and tilt introduce a bias, which is treated as a standard uncertainty [18]. All of these effects lead to an uncertainty in $\dot{\gamma}$ that is proportional to $\dot{\gamma}$, and the results are given in Table 1 below. It can be seen that the effects of the tilt are negligible.

The combined standard uncertainty is calculated by adding all of the components in quadrature [18].

| Table 1. Uncertainty in the Shear Rate | |
|---|---|
| Source of Uncertainty | Contribution to Standard Uncertainty in $\dot{\gamma}$ |
| Offset in Gap, $h_0$ | $0.010 \times \dot{\gamma}$ |
| Rotation Rate, $\omega$ | $0.005 \times \dot{\gamma}$ |
| Cone Angle, $\beta$ | $0.005 \times \dot{\gamma}$ |
| Cone Truncation | $0.003 \times \dot{\gamma}$ |
| Angle of Tilt, $\phi$ | $5 \times 10^{-5} \times \dot{\gamma}$ |
| Combined Standard Uncertainty | $0.013 \times \dot{\gamma}$ |

### 4.1.2 Uncertainties in the Viscosity and First Normal Stress Difference

The Type A uncertainties were assessed through multiple measurements (twenty samples at each temperature using two samples from each of ten randomly chosen bottles) and statistical analysis. Since the intention is to certify the *mean* values of the viscosity and first normal stress difference as functions of shear rate, the Type A uncertainty is calculated by dividing the standard deviation of the twenty measurements by the square root of the number of measurements, yielding the standard uncertainty of the mean. These components of uncertainty are given in Table 3 for the viscosity and Table 4 for the first normal stress difference.

The effects of the Type B sources of uncertainty listed above are calculated below. The effects of some of these influences will be calculated using functions fit to the experimental data for the viscosity and first normal stress difference. The viscosity $\eta(\dot{\gamma},T)$ has been fit to a Cross model [6, 25], with

$$\eta(\dot{\gamma},T) = \left( \frac{T\rho}{T_R \rho_R} \right) \left( \frac{\eta_R a(T)}{1 + (\xi_0 a(T)\dot{\gamma})^{1-n}} \right) \tag{6}$$

where $\dot{\gamma}$ is the shear rate, $T$ is the temperature, $\rho$ is the density at temperature $T$, $\eta_R$ is the zero-shear-rate viscosity at the reference temperature $T_R = 25$ °C, $\rho_R$ is the density at the reference temperature $T_R$, $\xi_0$ is a parameter that governs the transition from the Newtonian regime at low shear rates to the power law regime at high shear rates, $a(T)$ is the temperature shift factor (discussed below), and $n$ is the power at which the shear stress increases with shear rate. The density is approximated as a linear function of temperature, with $\rho(T) = \rho_R(1 - \alpha(T - T_R))$, where $\alpha$ is the volumetric coefficient of thermal expansion. The volumetric coefficient of thermal expansion is estimated to be $\alpha = 6 \times 10^{-4}$ cm$^3$/(cm$^3$ K) [26, 27]. The effect of the change in the density is small compared to the change in the temperature itself.

The temperature dependence is primarily governed by the shift factor $a(T)$, which is equal to the ratio of the zero-shear-rate viscosity at temperature $T$ divided by the zero-shear-rate viscosity at the reference temperature $T_R = 25$ °C. The shift factor has been fit with a function of the WLF type [6, 25], giving

$$a(T) = \exp\left(\frac{-C_1(T-T_R)}{C_2+T-T_R}\right) \tag{7}$$

The first normal stress difference $N_1(\dot{\gamma},T)$ was also fit to an empirical model similar to the Cross model, but with an additional term in the denominator (using the same temperature shift factor $a(T)$ calculated for the viscosity):

$$N_1(\dot{\gamma},T) = \left(\frac{T\rho}{T_R\rho_R}\right)\left(\frac{\psi_R(a(T)\dot{\gamma})^2}{1+\xi_1 a(T)\dot{\gamma}+(\xi_2 a(T)\dot{\gamma})^p}\right) \tag{8}$$

In equation (8), $T$ is the temperature, $\psi_R$ is the zero-shear-rate first normal stress coefficient at the reference temperature $T_R = 25$ °C, $\rho$ is the density at temperature $T$, $\rho_R$ is the density at the reference temperature $T_R$; $\xi_1$, $\xi_2$ and $p$ are parameters fit to the data. Values for the parameters in equations (6) through (8) are given in Table 2.

| Table 2. Parameters for $\eta(\dot{\gamma},T)$, $a(T)$ and $N_1(\dot{\gamma},T)$ in the models found in equations (6) through (8). | | |
|---|---|---|
| Parameter | Value | Standard Uncertainty |
| $\eta_R$ | 100.2 Pa·s | 0.6 Pa·s |
| $\xi_0$ | 0.234 s | 0.004 s |
| $n$ | 0.195 | 0.004 |
| | | |
| $C_1$ | 7.23 | 0.24 |
| $C_2$ | 150 °C | 5 °C |
| | | |
| $\psi_R$ | 129 Pa·s$^2$ | 5 Pa·s$^2$ |
| $\xi_1$ | 1.69 s | 0.13 s |
| $\xi_2$ | 0.247 s | 0.026 s |
| $p$ | 1.67 | 0.047 |

The measured viscosity $\eta$ is calculated from the twisting moment $M$ applied to the transducer as

$$\eta = \frac{3\tan\beta_0}{2\pi R_0^3 \omega_0} M \tag{9}$$

where $R_0 = 25$ mm is the nominal radius of both cone and plate, $\omega_0$ is the nominal rotation rate and $\beta_0 = 0.0197$ rad is the nominal cone angle. These nominal values are taken to be constants used in the calculation, with the effects of the uncertainties in $R$, $\omega$ and $\beta$ incorporated through their effect on the moment $M$, which is given by the integral over the area of the plate of the radius times the shear stress (the viscosity multiplying the shear rate):

$$M = \int_0^{2\pi} \int_0^R \eta(\dot{\gamma}, T)\dot{\gamma}(r,\theta)r^2\,dr\,d\theta \qquad (10)$$

Markovitz *et al.* [23] assessed the effects of geometric imperfections for Newtonian fluids in closed form, and we have adapted their analysis to the non-Newtonian case. This analysis employs the assumption that the shear rate $\dot{\gamma}$ is given by relative angular velocity of the plate with respect to the cone divided by the gap between them. If the cone and plate are well aligned (concentric and not tilted), the shear rate at a radius $r$ is approximated as

$$\dot{\gamma} = \frac{\omega r}{h_0 + r\tan\beta} \qquad (11)$$

where $\omega$ is the rate of rotation of the plate, $h_0$ is an offset spacing between the cone and the plate, and $\beta$ is the cone angle. Ideally, the offset $h_0$ would be zero, in which case the shear rate is independent of the radius $r$. To achieve the condition $h_0 = 0$ and a constant shear rate throughout most of the sample, the cone is truncated so that the tip of the cone will not touch the plate and cause friction. Approximately 50 μm is truncated from the cone, so the radius of the truncated region is approximately 2.5 mm. The truncation allows the cone to lie above or below its intended position, so that $h_0$ can be either positive or negative. Within the truncated area, $\beta = 0$, and the shear rate is a linear function of $r$, as is the case with parallel plate fixtures. To assess the effects of the cone truncation or nonzero $h_0$, equation (10) is evaluated numerically [24], using the viscosity model in equation (6). In general, geometric imperfections affect the moment in the Newtonian case more strongly than in the non-Newtonian case, because with shear thinning, an error that causes an increase in the shear rate is offset somewhat by a decrease in the viscosity.

The uncertainties in the first normal stress difference are addressed in a similar manner, with $N_1$ determined from the axial force $F$ applied to the transducer.

$$N_1 = \frac{2}{\pi R_0^2}F \qquad (12)$$

Again, $R_0$ is taken to be a constant, and the effects of the uncertainties in each parameter on $N_1$ are calculated through their effect on $F$, which is calculated through an integral over the area of the plate similar to that in equation (10). Marsh and Pearson [28] have analyzed the axial force generated in the event that a perfect cone is offset from the plate by an amount $h_0$. The force in that case is given by

$$F = \int_0^{2\pi} \int_0^R \frac{(N_1 - N_2)}{2}r\,dr\,d\theta + \int_0^{2\pi} \int_0^R \frac{N_2}{2}\left[\frac{\tan\beta}{h_0 + r\tan\beta}\right]r^2\,dr\,d\theta \qquad (13)$$

where $N_2$ is the second normal stress difference. The argument for both $N_1$ and $N_2$ in equation (13) is the shear rate $\dot{\gamma}$ given by equation (11). When $h_0 = 0$, equation (13) reduces to the result expected for the cone and plate, in which case the terms containing $N_2$ cancel:

$$F = \int_0^{2\pi} \int_0^R \frac{N_1}{2}r\,dr\,d\theta$$
$$= \frac{\pi R^2 N_1}{2} \qquad (14)$$

For parallel plates, where $\beta = 0$, equation (13) reduces to

$$F = \int_0^{2\pi} \int_0^R \frac{(N_1 - N_2)}{2} r \, dr \, d\theta \tag{15}$$

To assess the effects of the cone truncation on $N_1$, one could assume that equation (15) holds over the truncated area, while the integral in equation (14) holds over the rest of plate (with the radius of the truncated region as the lower limit in the integration instead of 0). Alternatively, Marsh and Pearson [28] also give an analytical expression for the partial derivative of $F$ with respect to $h_0$, evaluated at $h_0 = 0$,

$$\frac{\partial F}{\partial h_0} = \frac{-\pi R}{\tan \beta} \left( \dot\gamma_0 \frac{\partial N_1}{\partial \dot\gamma_0} + N_2 \right) \tag{16}$$

where $\dot\gamma_0 = \omega / \tan \beta$ is the nominal shear rate. Tanner [3] gives an equivalent expression in a different form. Note that $N_2$ is typically opposite in sign and smaller in magnitude than $N_1$. Since $N_1$ is a positive, increasing function of $\gamma$, a conservative estimate of $\partial F / \partial h_0$ in equation (16) is obtained by taking $N_2 = 0$. Similarly, a conservative estimate of the effect of the cone truncation is obtained by taking $N_2 = -N_1$ in equation (15) applied to the area of the truncation. Alternatively, $N_2$ can be calculated from measurements made using parallel plates along with the $N_1$ data measured with the cone and plate fixtures [1-6].

### 4.1.2.1 Uncertainty $\eta$ and $N_1$ Arising from Uncertainty in Temperature

The effects of uncertainty in the temperature can be calculated directly from equations (6) through (8) with the parameters in Table 1. For the viscosity, the component of uncertainty associated with uncertainty in the temperature is given by

$$u(\eta, T) = \frac{\partial \eta}{\partial T} u(T)$$
$$= \eta(\dot\gamma, T) \left\{ \frac{1}{T} - \frac{\alpha}{1 - \alpha(T - T_R)} + \left[ \frac{1 + n(\xi_0 a(T)\dot\gamma)^{1-n}}{1 + (\xi_0 a(T)\dot\gamma)^{1-n}} \right] \left( \frac{-C_1 C_2}{(C_2 + T - T_R)^2} \right) \right\} u(T) \tag{17}$$

For the first normal stress difference, the component of uncertainty associated with uncertainty in the temperature is given by

$$u(N_1, T) = \frac{\partial N_1}{\partial T} u(T)$$
$$= N_1(\dot\gamma, T) \left\{ \frac{1}{T} - \frac{\alpha}{1 - \alpha(T - T_R)} + \left[ \frac{2 + \xi_1 a(T)\dot\gamma + (2 - p)(\xi_2 a(T)\dot\gamma)^p}{1 + \xi_1 a(T)\dot\gamma + (\xi_2 a(T)\dot\gamma)^p} \right] \left( \frac{-C_1 C_2}{(C_2 + T - T_R)^2} \right) \right\} u(T) \tag{18}$$

The standard uncertainty in the temperature $u(T)$ is estimated to be 0.1 °C. The components of uncertainty in viscosity and first normal stress difference associated with temperature are given in Tables 3 and 4. Note that these uncertainties represent the effects of temperature on the material properties. Temperature fluctuations also affect the measurement of the normal force directly, because the subsequent thermal expansion and contraction of the sample and the fixtures introduce an axial force on the transducer. These temperature fluctuations are accounted for in the Type A uncertainty calculated from the scatter in the data over repeated tests. The temperature fluctuations in the rheometer are much smaller when heating above the ambient temperature than when cooling below the ambient temperature, because the power to the electric heating system can be varied, whereas the cooling is achieved by the flow of cold air that is either on or off. There is a larger scatter in the first normal stress difference at 0 °C compared to the

scatter at 25 °C or 50 °C, which is particularly noticeable at low levels of $N_1$. The scatter in the viscosity data does not show the same dependence on temperature because the thermal fluctuations do not directly affect the measurement of the moment.

4.1.2.2 Uncertainty in $\eta$ and $N_1$ Arising from Uncertainty in Transducer Readings

The effects of uncertainties in the transducer readings can be calculated directly from equations (9) and (12). For the viscosity,

$$u(\eta, M) = \frac{3 \tan \beta_0}{2\pi R_0^3 \omega_0} u(M) \tag{19}$$

while for the first normal stress difference,

$$u(N_1, F) = \frac{2}{\pi R_0^2} u(F) \tag{20}$$

The standard uncertainty in the moment is estimated to be $u(M) = 10^{-7}$ N·m + 0.002$M$, while the standard uncertainty in the axial force measurement is estimated to be $u(F) = 8\times10^{-4}$ N + 0.002$F$. These estimates are based on information from the instrument manufacturer and the variability observed in the data. The components of uncertainty in viscosity and first normal stress difference associated with the transducer are given in Tables 3 and 4.

4.1.2.3 Uncertainty in $\eta$ and $N_1$ Arising from Uncertainty in Rotation Rate

Uncertainty in the rotation rate affects both the shear rate $\dot{\gamma}$ and the viscosity in the calculation of the moment $M$ in equation (10). This effect can be calculated using the chain rule to take the derivative of $M$ with respect to $\dot{\gamma}$ in equation (10), and then take the derivative of $\dot{\gamma}$ in equation (11) with respect to $\omega$, evaluated at $h_0 = 0$. Using the model in equation (6) for the viscosity, the component of uncertainty associated with uncertainty in the rotation rate is given by

$$u(\eta, \omega) = \frac{\eta(\dot{\gamma}, T)}{\omega_0} \left[ \frac{1 + n(\xi_0 a(T)\dot{\gamma})^{1-n}}{1 + (\xi_0 a(T)\dot{\gamma})^{1-n}} \right] u(\omega) \tag{21}$$

The component of uncertainty in the first normal stress difference associated with uncertainty in the rotation rate can be calculated directyl from the model in equation (8), with

$$u(N_1, \omega) = \frac{N_1(\dot{\gamma}, T)}{\omega_0} \left[ \frac{2 + \xi_1 a(T)\dot{\gamma} + (2 - p)(\xi_2 a(T)\dot{\gamma})^p}{1 + \xi_1 a(T)\dot{\gamma} + (\xi_2 a(T)\dot{\gamma})^p} \right] u(\omega) \tag{22}$$

The standard uncertainty in the rotation rate $u(\omega)$ is estimated to be $0.005\omega_0$, based on information from the instrument manufacturer. The components of uncertainty in viscosity and first normal stress difference associated with rotation rate are given in Tables 3 and 4.

4.1.2.4 Uncertainty in $\eta$ and $N_1$ Arising from Uncertainties Associated with Geometry

4.1.2.4.1 Gap

The effects of uncertainty in the gap were calculated using numerical solution of equations (10) and (13) for the truncated cone geometry with $h_0 = 1$ µm and $h_0 = -1$ µm in equation (11) outside the area of the truncation. The partial derivatives of $\eta$ and $N_1$ with respect to $h_0$ were then

evaluated at $h_0 = 0$ for use in equation (2) with the standard uncertainty in the gap estimated to be 2.65 μm. Uncertainty in the gap arises from uncertainty in the cone truncation, uncertainty in the position at zero gap and at the specified setting, uncertainty in the flatness of the plate and profile of the cone, compliance of the transducer and thermal expansion of the transducer [16, 20-22]. Each of these seven influences was assigned a standard uncertainty of 1 μm. These components of uncertainty were then added in quadrature to calculate the combined standard uncertainty in the gap of 2.6 μm. Equation (16) has been used to estimate the uncertainty in $N_1$, with $N_2 = 0$. The components of uncertainty in viscosity and first normal stress difference associated with the gap are given in Tables 3 and 4.

### 4.1.2.4.2 Cone Angle

Effects of uncertainty in the cone angle are evaluated similarly to the effects of uncertainty in the rotation rate. Using the model in equations (6) for the viscosity, the component of uncertainty associated with uncertainty in the cone angle is given by

$$u(\eta, \beta) = \frac{-\eta(\dot{\gamma}, T)}{\cos^2 \beta_0 \tan \beta_0} \left[ \frac{1 + n(\xi_0 a(T)\dot{\gamma})^{1-n}}{1 + (\xi_0 a(T)\dot{\gamma})^{1-n}} \right] u(\beta) \tag{23}$$

Using the model in equation (8) for the first normal stress difference, the component of uncertainty associated with uncertainty in the cone angle is given by

$$u(N_1, \beta) = \frac{-N_1(\dot{\gamma}, T)}{\cos^2 \beta_0 \tan \beta_0} \left[ \frac{2 + \xi_1 a(T)\dot{\gamma} + (2 - p)(\xi_2 a(T)\dot{\gamma})^p}{1 + \xi_1 a(T)\dot{\gamma} + (\xi_2 a(T)\dot{\gamma})^p} \right] u(\beta) \tag{24}$$

Since $\beta_0 = 0.0197$ rad, $\cos^2 \beta_0 \approx 1$ and $\tan \beta_0 \approx \beta_0$. The standard uncertainty in the cone angle $u(\beta)$ is estimated to be $10^{-4}$ rad, based on information from the instrument manufacturer. Measurement of the cone angle at NIST agreed with that given by the manufacturer within that uncertainty. However, it should be noted that Mackay and Dick [19] found much larger uncertainties in a round robin test measuring the geometry of a cone. The components of uncertainty in viscosity and first normal stress difference associated with cone angle are given in Tables 3 and 4.

### 4.1.2.4.3 Cone/Plate Diameter

For the viscosity, the effects of uncertainties in the diameter of the cone and/or plate are given by

$$u(\eta, R) = \frac{3\eta(\dot{\gamma}, T)}{R_0} u(R) \tag{25}$$

where $R_0$ is the specified cone/plate diameter, which is 25 mm. For the first normal stress difference, the effects of uncertainties in the diameter of the cone and/or plate are given by

$$u(N_1, R) = \frac{2N_1(\dot{\gamma}, T)}{R_0} u(R) \tag{26}$$

The standard uncertainty in the radius of the cone or the plate is estimated to be 0.0025 mm. The combined standard uncertainty calculated by adding these two components in quadrature is $u(R) = 0.0035$ mm. The components of uncertainty in viscosity and first normal stress difference associated with cone and/or plate diameter are given in Tables 3 and 4.

#### 4.1.2.4.4 Cone Truncation

The truncation of the cone leads to a region where the shear rate is lower than the nominal shear rate of $\dot{\gamma}_0 = \omega / \tan \beta$, which leads to a decrease in the moment and a decrease in the calculated viscosity. The cone truncation therefore introduces a bias. This bias is expressed in the form of a standard uncertainty to be added in quadrature with the other components. For the viscosity, the relative uncertainty associated with the cone truncation is proportional to the cube of the ratio of the diameter of the truncated region to the diameter of the cone/plate; in this case, that ratio is approximately 1/10, so the relative uncertainty in the viscosity is on the order of $10^{-3}$. The component of uncertainty in the viscosity arising from cone truncation has been evaluated numerically using the model in equation (6), and the results are given in Table 3.

For the first normal stress difference, the region of decreased shear rate and the change in the geometry in the truncated region also leads to a bias in the calculated $N_1$. In this case, the relative standard uncertainty associated with the cone truncation is proportional to the square of the ratio of the diameter of the truncated region to the diameter of the cone/plate, so the relative standard uncertainty in $N_1$ is on the order of $10^{-2}$. The component of uncertainty in the viscosity arising from cone truncation has been evaluated numerically using the model in equation (8), and the results are given in Table 4.

#### 4.1.2.4.5 Tilt

An angle of tilt between the axis of the cone and the axis of the plate also introduces a bias in the viscosity and first normal stress difference. This angle is expressed as a standard uncertainty estimated to be $2 \times 10^{-4}$ rad, and the effects of such a tilt were determined by numerical calculation of equations (10) and (14) using the models in equations (6) and (8), along with the expression for the shear rate developed by Markovitz *et al.* [23]. In this case, the shear rate is a function of the angle $\theta$, so the integrals in equations (10) and (14) are fully two-dimensional; the numerical evaluation was broken into repeated one-dimensional integrals, first integrating over $r$ and then over $\theta$. The components of uncertainty in the viscosity and first normal stress difference arising from a tilt are given in Tables 3 and 4.

#### 4.1.2.4.6 Concentricity

An offset between the axis of the cone and the axis of the plate also introduces a bias in the viscosity and first normal stress difference. This offset is expressed as a standard uncertainty estimated to be 25 μm. The effects of such an offset were also estimated by numerical calculation of equations (10) and (14) using the models in equations (6) and (8). Markovitz *et al.* [23] give an expression for the shear rate as

$$\dot{\gamma}(r,\theta) = \frac{\omega r}{\tan \beta (r^2 + 2br\cos\theta + b^2)^{1/2}} \tag{27}$$

where $b$ is the offset between the axis of the cone and the axis of the plate. Again, the integrals in equations (10) and (14) are fully two-dimensional, and the numerical evaluation was broken into repeated one-dimensional integrals, first integrating over $r$ and then over $\theta$. In this case, the components of uncertainty in the viscosity and first normal stress difference arising from an offset between the axes of the cone and the plate are negligible compared to other components.

### 4.1.2.5 Uncertainty Associated with Solvent Evaporation

The uncertainties in the viscosity and first normal stress difference caused by solvent evaporation were estimated from experiments described in Section 3. The components of uncertainty in the viscosity and first normal stress difference arising from solvent evaporation are given in Tables 3 and 4.

### 4.1.2.6 Effects of Shear Heating

Energy dissipation through friction can lead to an increase in the temperature in the sample, which would decrease the viscosity and first normal stress difference. Bird, *et al*. [4] suggest that the maximum increase in the temperature caused by shear heating would be

$$\Delta T_{max} = \frac{\eta \omega^2 R^2}{8k} \tag{28}$$

where $k$ is the thermal conductivity of the fluid. The thermal conductivities of organic liquids are typically in the range from 0.1 W/(m·K) to 0.2 W/(m·K) [27, 29]. An estimate of the thermal conductivity of 2,6,10,14-tetramethylpentadecane can be made from its chemical structure [27], giving $k = 0.121$ W/(m·K) at 0 °C with a linear decrease to $k = 0.115$ W/(m·K) at 50 °C. It is reasonable to assume that the thermal conductivity of the solution is effectively the same as that of the solvent alone, but in any event, the thermal conductivity of polyisobutylene is similar to that of the solvent. Van Krevelen [28] gives the thermal conductivity of polyisobutylene as 0.130 W/(m·K). The temperature rise associated with shear heating is therefore calculated using equation (28) to be less than 0.025 °C over the entire range of conditions studied. Taking this value as a standard uncertainty in the temperature and adding it in quadrature with the standard uncertainty estimated for the temperature control of 0.1 °C, the combined standard uncertainty in the temperature is calculated to be 0.103 °C. The added effects of shear heating are therefore considered negligible.

### 4.1.2.7 Inertia-Driven Secondary Flows

The analysis of the cone and plate employs the assumption that the fluid travels in circular paths. Inertial effects can introduce instabilities that alter the assumed flow field and affect the resulting measurements. For the viscosity, these effects are proportional to the square of the Reynolds number Re [6], which is given by

$$Re = \frac{\rho \omega \beta^2 R^2}{\eta_0} \tag{29}$$

where $\rho$ is the fluid density and $\eta_0$ is the zero-shear-rate viscosity. The relative change in the moment associated with secondary flows is given [6] as

$$\frac{\Delta M}{M} = 6.1 \times 10^{-4} \, Re^2 \tag{30}$$

Since the Reynolds number is less than $10^{-4}$ for all the conditions tested, the effect of secondary flow on the moment (and therefore the viscosity measurement) is negligible.

For the first normal stress difference, inertia causes the fluid to try to flow out of the gap between

cone and plate, reducing the axial force. The change in the first normal stress difference is given [6] by

$$\Delta N_1 = -0.15 \rho \omega^2 R^2 \qquad (31)$$

This bias in the first normal stress difference is treated as a standard uncertainty to be added in quadrature with the other components of uncertainty. This component of uncertainty is given in Table 4. In the calculation, the density has been conservatively approximated as 1 g/cm$^3$.

## 4.1.2.8 Edge Effects

The conditions at the edge of the cone and plate can impact the measurements in several ways [6], but these effects are not easily quantifiable. Perhaps the most significant difficulty is that the fluid can escape from between the cone and plate. One indicator of loss of fluid would be a decrease in the moment with increasing shear rate. This decrease was only observed in three experiments at 0 °C in the step from a shear rate of 63 s$^{-1}$ to a shear rate of 100 s$^{-1}$. Those three measurements were discarded. The only other evidence of edge effects occurs at the three highest shear rates at all three temperatures, where there is an increase in the relative scatter of the viscosity data. For this reason, data at the three highest shear rates are provided as reference data only, since the sample geometry might not match our assumptions, and the uncertainty in the data cannot be completely quantified.

## 4.1.2.9 Surface Tension

Surface tension also affects the measurements [6], particularly the first normal stress difference. These effects have not been quantified, so no component of uncertainty has been assigned to the data.

| Table 3. Components of Standard Uncertainty in the Viscosity $\eta$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Shear Rate | Measured Viscosity | Standard Uncertainty (Type A) | $u(\eta,T)$ | $u(\eta,M)$ | $u(\eta,\omega)$ | $u(\eta,\beta)$ |
| °C | s$^{-1}$ | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s |
| | | | | | | | |
| 0.0 | 0.001000 | 382.900 | 1.176 | 2.604 | 3.822 | 1.910 | 1.939 |
| 0.0 | 0.001585 | 380.049 | 0.999 | 2.596 | 2.694 | 1.893 | 1.922 |
| 0.0 | 0.002512 | 382.381 | 0.946 | 2.584 | 1.982 | 1.901 | 1.931 |
| 0.0 | 0.003981 | 382.901 | 0.880 | 2.567 | 1.534 | 1.899 | 1.929 |
| 0.0 | 0.006310 | 383.973 | 0.826 | 2.543 | 1.252 | 1.898 | 1.927 |
| 0.0 | 0.01000 | 383.078 | 0.786 | 2.508 | 1.072 | 1.883 | 1.912 |
| 0.0 | 0.01585 | 382.897 | 0.680 | 2.460 | 0.959 | 1.868 | 1.897 |
| 0.0 | 0.02512 | 379.825 | 0.699 | 2.392 | 0.881 | 1.833 | 1.861 |
| 0.0 | 0.03981 | 375.059 | 0.629 | 2.299 | 0.827 | 1.782 | 1.809 |
| 0.0 | 0.06310 | 365.510 | 0.631 | 2.174 | 0.779 | 1.698 | 1.724 |
| 0.0 | 0.1000 | 350.006 | 0.551 | 2.011 | 0.731 | 1.576 | 1.601 |
| 0.0 | 0.1585 | 328.432 | 0.458 | 1.807 | 0.676 | 1.417 | 1.439 |
| 0.0 | 0.2512 | 300.793 | 0.409 | 1.565 | 0.614 | 1.225 | 1.244 |
| 0.0 | 0.3981 | 268.517 | 0.326 | 1.297 | 0.545 | 1.013 | 1.029 |
| 0.0 | 0.6310 | 232.977 | 0.260 | 1.021 | 0.471 | 0.799 | 0.811 |
| 0.0 | 1.000 | 196.534 | 0.211 | 0.762 | 0.396 | 0.600 | 0.609 |
| 0.0 | 1.585 | 161.096 | 0.155 | 0.540 | 0.324 | 0.430 | 0.437 |
| 0.0 | 2.512 | 128.413 | 0.105 | 0.366 | 0.258 | 0.296 | 0.301 |
| 0.0 | 3.981 | 99.449 | 0.075 | 0.239 | 0.200 | 0.197 | 0.200 |
| 0.0 | 6.310 | 75.066 | 0.051 | 0.153 | 0.151 | 0.128 | 0.130 |
| 0.0 | 10.00 | 55.594 | 0.037 | 0.097 | 0.111 | 0.083 | 0.084 |
| 0.0 | 15.85 | 40.259 | 0.030 | 0.062 | 0.081 | 0.054 | 0.054 |
| 0.0 | 25.12 | 28.670 | 0.036 | 0.040 | 0.057 | 0.035 | 0.035 |
| 0.0 | 39.81 | 20.230 | 0.055 | 0.026 | 0.041 | 0.023 | 0.023 |
| 0.0 | 63.10 | 13.876 | 0.062 | 0.017 | 0.028 | 0.015 | 0.015 |
| 0.0 | 100.0 | 9.085 | 0.045 | 0.011 | 0.018 | $9.0\times10^{-3}$ | $9.0\times10^{-3}$ |

| Table 3. (continued) Components of Standard Uncertainty in the Viscosity $\eta$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Shear Rate | Measured Viscosity | $u(\eta, h_0)$ | $u(\eta, R)$ | Cone Truncation | Tilt | Evaporation |
| °C | s$^{-1}$ | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s |
| | | | | | | | |
| 0.0 | 0.001000 | 382.900 | 3.104 | 0.161 | 0.091 | 0.020 | 0.042 |
| 0.0 | 0.001585 | 380.049 | 3.097 | 0.160 | 0.091 | 0.020 | 0.045 |
| 0.0 | 0.002512 | 382.381 | 3.085 | 0.161 | 0.091 | 0.020 | 0.048 |
| 0.0 | 0.003981 | 382.901 | 3.067 | 0.161 | 0.090 | 0.019 | 0.052 |
| 0.0 | 0.006310 | 383.973 | 3.042 | 0.161 | 0.090 | 0.019 | 0.055 |
| 0.0 | 0.01000 | 383.078 | 3.006 | 0.161 | 0.089 | 0.019 | 0.058 |
| 0.0 | 0.01585 | 382.897 | 2.956 | 0.161 | 0.088 | 0.019 | 0.060 |
| 0.0 | 0.02512 | 379.825 | 2.885 | 0.160 | 0.086 | 0.018 | 0.063 |
| 0.0 | 0.03981 | 375.059 | 2.785 | 0.158 | 0.083 | 0.017 | 0.064 |
| 0.0 | 0.06310 | 365.510 | 2.649 | 0.154 | 0.080 | 0.016 | 0.065 |
| 0.0 | 0.1000 | 350.006 | 2.468 | 0.147 | 0.076 | 0.015 | 0.066 |
| 0.0 | 0.1585 | 328.432 | 2.241 | 0.138 | 0.070 | 0.013 | 0.065 |
| 0.0 | 0.2512 | 300.793 | 1.962 | 0.126 | 0.062 | 0.011 | 0.062 |
| 0.0 | 0.3981 | 268.517 | 1.648 | 0.113 | 0.053 | $8.0 \times 10^{-3}$ | 0.058 |
| 0.0 | 0.6310 | 232.977 | 1.314 | 0.098 | 0.043 | $6.0 \times 10^{-3}$ | 0.053 |
| 0.0 | 1.000 | 196.534 | 0.995 | 0.083 | 0.034 | $4.0 \times 10^{-3}$ | 0.047 |
| 0.0 | 1.585 | 161.096 | 0.714 | 0.068 | 0.025 | $2.0 \times 10^{-3}$ | 0.039 |
| 0.0 | 2.512 | 128.413 | 0.489 | 0.054 | 0.017 | $1.0 \times 10^{-3}$ | 0.032 |
| 0.0 | 3.981 | 99.449 | 0.323 | 0.042 | 0.012 | $1.0 \times 10^{-3}$ | 0.026 |
| 0.0 | 6.310 | 75.066 | 0.208 | 0.032 | $8.0 \times 10^{-3}$ | $7.0 \times 10^{-4}$ | 0.020 |
| 0.0 | 10.00 | 55.594 | 0.133 | 0.023 | $5.1 \times 10^{-3}$ | $4.0 \times 10^{-4}$ | 0.015 |
| 0.0 | 15.85 | 40.259 | 0.085 | 0.017 | $3.3 \times 10^{-3}$ | $3.0 \times 10^{-4}$ | 0.011 |
| 0.0 | 25.12 | 28.670 | 0.055 | 0.012 | $2.2 \times 10^{-3}$ | $2.0 \times 10^{-4}$ | $8.1 \times 10^{-3}$ |
| 0.0 | 39.81 | 20.230 | 0.036 | $8.0 \times 10^{-3}$ | $1.3 \times 10^{-3}$ | $1.0 \times 10^{-4}$ | $5.9 \times 10^{-3}$ |
| 0.0 | 63.10 | 13.876 | 0.023 | $6.0 \times 10^{-3}$ | $9.0 \times 10^{-4}$ | $1.0 \times 10^{-4}$ | $4.3 \times 10^{-3}$ |
| 0.0 | 100.0 | 9.085 | 0.016 | $4.0 \times 10^{-3}$ | $5.8 \times 10^{-4}$ | $5.0 \times 10^{-5}$ | $3.1 \times 10^{-3}$ |

| Table 3. (continued) Components of Standard Uncertainty in the Viscosity $\eta$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Shear Rate | Measured Viscosity | Standard Uncertainty (Type A) | $u(\eta,T)$ | $u(\eta,M)$ | $u(\eta,\omega)$ | $u(\eta,\beta)$ |
| °C | s$^{-1}$ | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s |
| | | | | | | | |
| 25.0 | 0.001000 | 97.929 | 0.869 | 0.454 | 3.252 | 0.489 | 0.497 |
| 25.0 | 0.001585 | 98.050 | 0.674 | 0.454 | 2.130 | 0.490 | 0.497 |
| 25.0 | 0.002512 | 98.250 | 0.618 | 0.453 | 1.414 | 0.490 | 0.498 |
| 25.0 | 0.003981 | 97.927 | 0.335 | 0.452 | 0.964 | 0.488 | 0.496 |
| 25.0 | 0.006310 | 98.352 | 0.249 | 0.451 | 0.681 | 0.490 | 0.497 |
| 25.0 | 0.01000 | 98.057 | 0.242 | 0.449 | 0.502 | 0.488 | 0.495 |
| 25.0 | 0.01585 | 98.717 | 0.188 | 0.446 | 0.390 | 0.490 | 0.497 |
| 25.0 | 0.02512 | 98.778 | 0.179 | 0.442 | 0.319 | 0.488 | 0.496 |
| 25.0 | 0.03981 | 98.613 | 0.180 | 0.436 | 0.274 | 0.485 | 0.492 |
| 25.0 | 0.06310 | 98.373 | 0.177 | 0.428 | 0.245 | 0.480 | 0.487 |
| 25.0 | 0.1000 | 97.522 | 0.177 | 0.417 | 0.226 | 0.470 | 0.477 |
| 25.0 | 0.1585 | 96.130 | 0.168 | 0.401 | 0.212 | 0.456 | 0.463 |
| 25.0 | 0.2512 | 93.706 | 0.158 | 0.380 | 0.200 | 0.435 | 0.441 |
| 25.0 | 0.3981 | 89.999 | 0.149 | 0.353 | 0.188 | 0.404 | 0.411 |
| 25.0 | 0.6310 | 84.592 | 0.135 | 0.318 | 0.174 | 0.364 | 0.369 |
| 25.0 | 1.000 | 77.608 | 0.116 | 0.277 | 0.158 | 0.315 | 0.320 |
| 25.0 | 1.585 | 69.160 | 0.102 | 0.231 | 0.140 | 0.260 | 0.264 |
| 25.0 | 2.512 | 59.985 | 0.079 | 0.183 | 0.121 | 0.204 | 0.207 |
| 25.0 | 3.981 | 50.557 | 0.062 | 0.137 | 0.102 | 0.153 | 0.155 |
| 25.0 | 6.310 | 41.440 | 0.053 | 0.097 | 0.083 | 0.110 | 0.111 |
| 25.0 | 10.00 | 33.036 | 0.045 | 0.066 | 0.066 | 0.075 | 0.077 |
| 25.0 | 15.85 | 25.605 | 0.041 | 0.043 | 0.051 | 0.050 | 0.051 |
| 25.0 | 25.12 | 19.361 | 0.040 | 0.027 | 0.039 | 0.033 | 0.033 |
| 25.0 | 39.81 | 14.262 | 0.043 | 0.017 | 0.029 | 0.021 | 0.022 |
| 25.0 | 63.10 | 10.220 | 0.058 | 0.011 | 0.020 | 0.014 | 0.014 |
| 25.0 | 100.0 | 7.223 | 0.056 | $7.0\times10^{-3}$ | 0.014 | $9.0\times10^{-3}$ | $9.0\times10^{-3}$ |

| Table 3. (continued) Components of Standard Uncertainty in the Viscosity $\eta$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Shear Rate | Measured Viscosity | $u(\eta, h_0)$ | $u(\eta, R)$ | Cone Truncation | Tilt | Evaporation |
| °C | s⁻¹ | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s |
| | | | | | | | |
| 25.0 | 0.001000 | 97.929 | 0.802 | 0.041 | 0.024 | $5.1\times10^{-3}$ | 0.098 |
| 25.0 | 0.001585 | 98.050 | 0.801 | 0.041 | 0.024 | $5.1\times10^{-3}$ | 0.105 |
| 25.0 | 0.002512 | 98.250 | 0.800 | 0.041 | 0.024 | $5.1\times10^{-3}$ | 0.112 |
| 25.0 | 0.003981 | 97.927 | 0.799 | 0.041 | 0.024 | $5.1\times10^{-3}$ | 0.119 |
| 25.0 | 0.006310 | 98.352 | 0.797 | 0.041 | 0.023 | $5.0\times10^{-3}$ | 0.125 |
| 25.0 | 0.01000 | 98.057 | 0.794 | 0.041 | 0.023 | $5.1\times10^{-3}$ | 0.132 |
| 25.0 | 0.01585 | 98.717 | 0.789 | 0.041 | 0.023 | $5.0\times10^{-3}$ | 0.139 |
| 25.0 | 0.02512 | 98.778 | 0.782 | 0.041 | 0.023 | $5.0\times10^{-3}$ | 0.145 |
| 25.0 | 0.03981 | 98.613 | 0.773 | 0.041 | 0.023 | $4.9\times10^{-3}$ | 0.151 |
| 25.0 | 0.06310 | 98.373 | 0.760 | 0.041 | 0.022 | $4.8\times10^{-3}$ | 0.156 |
| 25.0 | 0.1000 | 97.522 | 0.741 | 0.041 | 0.022 | $4.6\times10^{-3}$ | 0.160 |
| 25.0 | 0.1585 | 96.130 | 0.715 | 0.040 | 0.021 | $4.3\times10^{-3}$ | 0.164 |
| 25.0 | 0.2512 | 93.706 | 0.679 | 0.039 | 0.021 | $4.1\times10^{-3}$ | 0.165 |
| 25.0 | 0.3981 | 89.999 | 0.633 | 0.038 | 0.019 | $3.7\times10^{-3}$ | 0.165 |
| 25.0 | 0.6310 | 84.592 | 0.573 | 0.036 | 0.018 | $3.2\times10^{-3}$ | 0.162 |
| 25.0 | 1.000 | 77.608 | 0.501 | 0.033 | 0.016 | $2.7\times10^{-3}$ | 0.155 |
| 25.0 | 1.585 | 69.160 | 0.419 | 0.029 | 0.014 | $2.1\times10^{-3}$ | 0.145 |
| 25.0 | 2.512 | 59.985 | 0.334 | 0.025 | 0.011 | $1.6\times10^{-3}$ | 0.131 |
| 25.0 | 3.981 | 50.557 | 0.252 | 0.021 | $8.6\times10^{-3}$ | $1.0\times10^{-3}$ | 0.115 |
| 25.0 | 6.310 | 41.440 | 0.180 | 0.017 | $6.4\times10^{-3}$ | $7.0\times10^{-4}$ | 0.098 |
| 25.0 | 10.00 | 33.036 | 0.123 | 0.014 | $4.5\times10^{-3}$ | $4.0\times10^{-4}$ | 0.080 |
| 25.0 | 15.85 | 25.605 | 0.081 | 0.011 | $3.1\times10^{-3}$ | $3.0\times10^{-4}$ | 0.063 |
| 25.0 | 25.12 | 19.361 | 0.052 | $8.0\times10^{-3}$ | $2.0\times10^{-3}$ | $1.0\times10^{-4}$ | 0.049 |
| 25.0 | 39.81 | 14.262 | 0.033 | $6.0\times10^{-3}$ | $1.2\times10^{-3}$ | $1.0\times10^{-4}$ | 0.037 |
| 25.0 | 63.10 | 10.220 | 0.021 | $4.0\times10^{-3}$ | $8.0\times10^{-4}$ | $1.0\times10^{-4}$ | 0.027 |
| 25.0 | 100.0 | 7.223 | 0.014 | $3.0\times10^{-3}$ | $5.3\times10^{-4}$ | $5.0\times10^{-5}$ | 0.020 |

| Temperature | Shear Rate | Measured Viscosity | Standard Uncertainty (Type A) | $u(\eta,T)$ | $u(\eta,M)$ | $u(\eta,\omega)$ | $u(\eta,\beta)$ |
|---|---|---|---|---|---|---|---|
| °C | s$^{-1}$ | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s |
| | | | | | | | |
| 50.0 | 0.001000 | 36.744 | 0.741 | 0.125 | 3.129 | 0.184 | 0.186 |
| 50.0 | 0.001585 | 37.196 | 0.470 | 0.125 | 2.008 | 0.186 | 0.189 |
| 50.0 | 0.002512 | 37.623 | 0.252 | 0.125 | 1.293 | 0.188 | 0.191 |
| 50.0 | 0.003981 | 37.303 | 0.182 | 0.125 | 0.842 | 0.186 | 0.189 |
| 50.0 | 0.006310 | 37.723 | 0.086 | 0.125 | 0.560 | 0.188 | 0.191 |
| 50.0 | 0.01000 | 37.727 | 0.091 | 0.125 | 0.381 | 0.188 | 0.191 |
| 50.0 | 0.01585 | 37.503 | 0.075 | 0.124 | 0.268 | 0.187 | 0.190 |
| 50.0 | 0.02512 | 37.548 | 0.056 | 0.124 | 0.197 | 0.187 | 0.190 |
| 50.0 | 0.03981 | 37.830 | 0.056 | 0.123 | 0.152 | 0.188 | 0.191 |
| 50.0 | 0.06310 | 37.800 | 0.050 | 0.122 | 0.124 | 0.187 | 0.190 |
| 50.0 | 0.1000 | 37.761 | 0.051 | 0.121 | 0.106 | 0.186 | 0.189 |
| 50.0 | 0.1585 | 37.658 | 0.050 | 0.119 | 0.095 | 0.184 | 0.187 |
| 50.0 | 0.2512 | 37.394 | 0.048 | 0.116 | 0.087 | 0.180 | 0.183 |
| 50.0 | 0.3981 | 36.930 | 0.046 | 0.112 | 0.082 | 0.175 | 0.178 |
| 50.0 | 0.6310 | 36.097 | 0.044 | 0.106 | 0.077 | 0.168 | 0.170 |
| 50.0 | 1.000 | 34.770 | 0.040 | 0.099 | 0.073 | 0.157 | 0.159 |
| 50.0 | 1.585 | 32.794 | 0.036 | 0.090 | 0.068 | 0.142 | 0.144 |
| 50.0 | 2.512 | 30.200 | 0.032 | 0.079 | 0.062 | 0.123 | 0.125 |
| 50.0 | 3.981 | 27.049 | 0.024 | 0.066 | 0.055 | 0.102 | 0.104 |
| 50.0 | 6.310 | 23.543 | 0.020 | 0.053 | 0.048 | 0.081 | 0.082 |
| 50.0 | 10.00 | 19.946 | 0.020 | 0.040 | 0.040 | 0.061 | 0.062 |
| 50.0 | 15.85 | 16.408 | 0.016 | 0.029 | 0.033 | 0.044 | 0.045 |
| 50.0 | 25.12 | 13.138 | 0.016 | 0.020 | 0.026 | 0.030 | 0.031 |
| 50.0 | 39.81 | 10.242 | 0.017 | 0.013 | 0.021 | 0.020 | 0.021 |
| 50.0 | 63.10 | 7.751 | 0.018 | $8.2\times10^{-3}$ | 0.016 | 0.013 | 0.014 |
| 50.0 | 100.0 | 5.721 | 0.025 | $5.1\times10^{-3}$ | 0.011 | $9.0\times10^{-3}$ | $9.0\times10^{-3}$ |

Table 3. (continued)  Components of Standard Uncertainty in the Viscosity $\eta$

| Temperature | Shear Rate | Measured Viscosity | $u(\eta, h_0)$ | $u(\eta, R)$ | Cone Truncation | Tilt | Evaporation |
|---|---|---|---|---|---|---|---|
| °C | s⁻¹ | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s | Pa·s |
| | | | | | | | |
| 50.0 | 0.001000 | 36.744 | 0.308 | 0.015 | $9.0\times10^{-3}$ | $2.0\times10^{-3}$ | 0.243 |
| 50.0 | 0.001585 | 37.196 | 0.307 | 0.016 | $9.0\times10^{-3}$ | $1.9\times10^{-3}$ | 0.258 |
| 50.0 | 0.002512 | 37.623 | 0.307 | 0.016 | $9.0\times10^{-3}$ | $2.0\times10^{-3}$ | 0.273 |
| 50.0 | 0.003981 | 37.303 | 0.307 | 0.016 | $9.0\times10^{-3}$ | $2.0\times10^{-3}$ | 0.288 |
| 50.0 | 0.006310 | 37.723 | 0.306 | 0.016 | $9.0\times10^{-3}$ | $2.0\times10^{-3}$ | 0.303 |
| 50.0 | 0.01000 | 37.727 | 0.306 | 0.016 | $9.0\times10^{-3}$ | $2.0\times10^{-3}$ | 0.318 |
| 50.0 | 0.01585 | 37.503 | 0.305 | 0.016 | $9.0\times10^{-3}$ | $1.9\times10^{-3}$ | 0.332 |
| 50.0 | 0.02512 | 37.548 | 0.304 | 0.016 | $8.9\times10^{-3}$ | $1.9\times10^{-3}$ | 0.346 |
| 50.0 | 0.03981 | 37.830 | 0.302 | 0.016 | $8.9\times10^{-3}$ | $1.9\times10^{-3}$ | 0.360 |
| 50.0 | 0.06310 | 37.800 | 0.300 | 0.016 | $8.8\times10^{-3}$ | $1.9\times10^{-3}$ | 0.373 |
| 50.0 | 0.1000 | 37.761 | 0.297 | 0.016 | $8.7\times10^{-3}$ | $1.9\times10^{-3}$ | 0.386 |
| 50.0 | 0.1585 | 37.658 | 0.291 | 0.016 | $8.7\times10^{-3}$ | $1.9\times10^{-3}$ | 0.397 |
| 50.0 | 0.2512 | 37.394 | 0.285 | 0.016 | $8.5\times10^{-3}$ | $1.8\times10^{-3}$ | 0.406 |
| 50.0 | 0.3981 | 36.930 | 0.275 | 0.016 | $8.3\times10^{-3}$ | $1.7\times10^{-3}$ | 0.413 |
| 50.0 | 0.6310 | 36.097 | 0.262 | 0.015 | $7.9\times10^{-3}$ | $1.6\times10^{-3}$ | 0.415 |
| 50.0 | 1.000 | 34.770 | 0.244 | 0.015 | $7.4\times10^{-3}$ | $1.4\times10^{-3}$ | 0.413 |
| 50.0 | 1.585 | 32.794 | 0.222 | 0.014 | $6.9\times10^{-3}$ | $1.3\times10^{-3}$ | 0.405 |
| 50.0 | 2.512 | 30.200 | 0.194 | 0.013 | $6.1\times10^{-3}$ | $1.0\times10^{-3}$ | 0.389 |
| 50.0 | 3.981 | 27.049 | 0.164 | 0.011 | $5.3\times10^{-3}$ | $8.0\times10^{-4}$ | 0.364 |
| 50.0 | 6.310 | 23.543 | 0.131 | 0.010 | $4.4\times10^{-3}$ | $6.0\times10^{-4}$ | 0.332 |
| 50.0 | 10.00 | 19.946 | 0.099 | $8.0\times10^{-3}$ | $3.4\times10^{-3}$ | $4.0\times10^{-4}$ | 0.292 |
| 50.0 | 15.85 | 16.408 | 0.071 | $7.0\times10^{-3}$ | $2.5\times10^{-3}$ | $3.0\times10^{-4}$ | 0.248 |
| 50.0 | 25.12 | 13.138 | 0.049 | $6.0\times10^{-3}$ | $1.8\times10^{-3}$ | $2.0\times10^{-4}$ | 0.204 |
| 50.0 | 39.81 | 10.242 | 0.032 | $4.0\times10^{-3}$ | $1.2\times10^{-3}$ | $1.0\times10^{-4}$ | 0.163 |
| 50.0 | 63.10 | 7.751 | 0.021 | $3.0\times10^{-3}$ | $8.0\times10^{-4}$ | $7.0\times10^{-5}$ | 0.126 |
| 50.0 | 100.0 | 5.721 | 0.013 | $2.0\times10^{-3}$ | $5.1\times10^{-4}$ | $4.0\times10^{-5}$ | 0.095 |

Table 3. (continued)  Components of Standard Uncertainty in the Viscosity $\eta$

| Table 4.  Components of Standard Uncertainty in the First Normal Stress Difference $N_1$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper-ature | Shear Rate | Measured $N_1$ | Standard Uncertainty (Type A) | $u(N_1,T)$ | $u(N_1,F)$ | $u(N_1,\omega)$ | $u(N_1,\beta)$ |
| °C | s$^{-1}$ | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 0.0 | 0.1585 | 16.30 | 5.41 | 0.24 | 0.85 | 0.12 | 0.12 |
| 0.0 | 0.2512 | 43.78 | 4.69 | 0.42 | 0.90 | 0.30 | 0.30 |
| 0.0 | 0.3981 | 91.85 | 5.40 | 0.68 | 1.00 | 0.57 | 0.58 |
| 0.0 | 0.6310 | 149.06 | 6.54 | 1.06 | 1.11 | 0.85 | 0.86 |
| 0.0 | 1.000 | 257.45 | 6.61 | 1.60 | 1.33 | 1.35 | 1.37 |
| 0.0 | 1.585 | 372.45 | 5.65 | 2.35 | 1.56 | 1.81 | 1.84 |
| 0.0 | 2.512 | 572.64 | 4.98 | 3.37 | 1.96 | 2.60 | 2.64 |
| 0.0 | 3.981 | 845.17 | 4.40 | 4.70 | 2.51 | 3.58 | 3.63 |
| 0.0 | 6.310 | 1218.81 | 6.68 | 6.36 | 3.25 | 4.80 | 4.88 |
| 0.0 | 10.00 | 1717.43 | 5.57 | 8.34 | 4.25 | 6.26 | 6.36 |
| 0.0 | 15.85 | 2362.56 | 5.75 | 10.59 | 5.54 | 7.93 | 8.06 |
| 0.0 | 25.12 | 3195.73 | 7.34 | 13.03 | 7.21 | 9.84 | 9.99 |
| 0.0 | 39.81 | 4251.21 | 11.56 | 15.61 | 9.32 | 11.97 | 12.16 |
| 0.0 | 63.10 | 5518.63 | 19.91 | 18.30 | 11.85 | 14.24 | 14.46 |
| 0.0 | 100.0 | 7125.98 | 26.43 | 21.12 | 15.07 | 16.92 | 17.18 |

| Table 4. (continued)  Components of Standard Uncertainty in the First Normal Stress Difference $N_1$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper-ature | Shear Rate | Measured $N_1$ | $u(N_1,h_0)$ | $u(N_1,R)$ | Trunc-ation | Tilt | Evapor-ation | Inertia |
| °C | s$^{-1}$ | Pa | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | | |
| 0.0 | 0.1585 | 16.30 | 0.26 | $5\times10^{-3}$ | 0.03 | $2\times10^{-3}$ | $5\times10^{-3}$ | $9\times10^{-7}$ |
| 0.0 | 0.2512 | 43.78 | 0.64 | 0.01 | 0.07 | $3\times10^{-3}$ | 0.01 | $2\times10^{-6}$ |
| 0.0 | 0.3981 | 91.85 | 1.22 | 0.03 | 0.16 | $5\times10^{-3}$ | 0.02 | $6\times10^{-6}$ |
| 0.0 | 0.6310 | 149.06 | 1.82 | 0.04 | 0.33 | $8\times10^{-3}$ | 0.03 | $1\times10^{-5}$ |
| 0.0 | 1.000 | 257.45 | 2.90 | 0.07 | 0.64 | 0.01 | 0.05 | $4\times10^{-5}$ |
| 0.0 | 1.585 | 372.45 | 3.90 | 0.10 | 1.14 | 0.02 | 0.09 | $9\times10^{-5}$ |
| 0.0 | 2.512 | 572.64 | 5.59 | 0.16 | 1.92 | 0.02 | 0.14 | $2\times10^{-4}$ |
| 0.0 | 3.981 | 845.17 | 7.70 | 0.24 | 3.12 | 0.03 | 0.22 | $6\times10^{-4}$ |
| 0.0 | 6.310 | 1218.81 | 10.33 | 0.34 | 4.89 | 0.04 | 0.33 | $1\times10^{-3}$ |
| 0.0 | 10.00 | 1717.43 | 13.48 | 0.48 | 7.44 | 0.05 | 0.49 | $4\times10^{-3}$ |
| 0.0 | 15.85 | 2362.56 | 17.07 | 0.66 | 10.99 | 0.06 | 0.69 | $9\times10^{-3}$ |
| 0.0 | 25.12 | 3195.73 | 21.17 | 0.89 | 15.78 | 0.07 | 0.96 | 0.02 |
| 0.0 | 39.81 | 4251.21 | 25.76 | 1.19 | 22.01 | 0.08 | 1.30 | 0.06 |
| 0.0 | 63.10 | 5518.63 | 30.64 | 1.55 | 29.85 | 0.11 | 1.72 | 0.14 |
| 0.0 | 100.0 | 7125.98 | 36.42 | 2.00 | 39.45 | 0.12 | 2.22 | 0.36 |

| Table 4. (continued) Components of Standard Uncertainty in the First Normal Stress Difference $N_1$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper-ature | Shear Rate | Measured $N_1$ | Standard Uncertainty (Type A) | $u(N_1,T)$ | $u(N_1,F)$ | $u(N_1,\omega)$ | $u(N_1,\beta)$ |
| °C | s$^{-1}$ | Pa | Pa | Pa | Pa | Pa | Pa |
| 25.0 | 0.1585 | 2.35 | 0.48 | 0.02 | 0.82 | 0.02 | 0.02 |
| 25.0 | 0.2512 | 5.51 | 0.32 | 0.04 | 0.83 | 0.05 | 0.05 |
| 25.0 | 0.3981 | 12.92 | 0.34 | 0.09 | 0.84 | 0.10 | 0.10 |
| 25.0 | 0.6310 | 26.54 | 0.42 | 0.16 | 0.87 | 0.20 | 0.20 |
| 25.0 | 1.000 | 50.05 | 0.39 | 0.29 | 0.91 | 0.34 | 0.34 |
| 25.0 | 1.585 | 87.62 | 0.45 | 0.47 | 0.99 | 0.54 | 0.55 |
| 25.0 | 2.512 | 148.18 | 0.59 | 0.74 | 1.11 | 0.84 | 0.85 |
| 25.0 | 3.981 | 236.76 | 1.87 | 1.12 | 1.29 | 1.23 | 1.25 |
| 25.0 | 6.310 | 377.26 | 3.92 | 1.64 | 1.57 | 1.83 | 1.86 |
| 25.0 | 10.00 | 585.07 | 4.70 | 2.36 | 1.99 | 2.64 | 2.68 |
| 25.0 | 15.85 | 880.00 | 4.88 | 3.30 | 2.57 | 3.71 | 3.77 |
| 25.0 | 25.12 | 1280.30 | 5.39 | 4.48 | 3.38 | 5.02 | 5.10 |
| 25.0 | 39.81 | 1800.04 | 6.42 | 5.89 | 4.41 | 6.53 | 6.63 |
| 25.0 | 63.10 | 2462.22 | 14.06 | 7.50 | 5.74 | 8.22 | 8.35 |
| 25.0 | 100.0 | 3318.59 | 16.77 | 9.25 | 7.45 | 10.16 | 10.32 |

| Table 4. (continued) Components of Standard Uncertainty in the First Normal Stress Difference $N_1$ | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper-ature | Shear Rate | Measured $N_1$ | $u(N_1,h_0)$ | $u(N_1,R)$ | Trunc-ation | Tilt | Evapor-ation | Inertia |
| °C | s$^{-1}$ | Pa | Pa | Pa | Pa | Pa | Pa | Pa |
| 25.0 | 0.1585 | 2.35 | 0.05 | $7\times10^{-4}$ | $1\times10^{-3}$ | $3\times10^{-4}$ | $5\times10^{-3}$ | $9\times10^{-7}$ |
| 25.0 | 0.2512 | 5.51 | 0.10 | $2\times10^{-3}$ | $3\times10^{-3}$ | $6\times10^{-4}$ | 0.01 | $2\times10^{-6}$ |
| 25.0 | 0.3981 | 12.92 | 0.22 | $4\times10^{-3}$ | 0.01 | $1\times10^{-3}$ | 0.02 | $6\times10^{-6}$ |
| 25.0 | 0.6310 | 26.54 | 0.42 | $7\times10^{-3}$ | 0.03 | $2\times10^{-3}$ | 0.05 | $1\times10^{-5}$ |
| 25.0 | 1.000 | 50.05 | 0.73 | 0.01 | 0.07 | $4\times10^{-3}$ | 0.09 | $4\times10^{-5}$ |
| 25.0 | 1.585 | 87.62 | 1.16 | 0.02 | 0.17 | $5\times10^{-3}$ | 0.18 | $9\times10^{-5}$ |
| 25.0 | 2.512 | 148.18 | 1.80 | 0.04 | 0.35 | $9\times10^{-3}$ | 0.31 | $2\times10^{-4}$ |
| 25.0 | 3.981 | 236.76 | 2.65 | 0.07 | 0.66 | 0.01 | 0.53 | $6\times10^{-4}$ |
| 25.0 | 6.310 | 377.26 | 3.93 | 0.11 | 1.17 | 0.02 | 0.86 | $1\times10^{-3}$ |
| 25.0 | 10.00 | 585.07 | 5.69 | 0.16 | 1.97 | 0.02 | 1.37 | $4\times10^{-3}$ |
| 25.0 | 15.85 | 880.00 | 7.98 | 0.25 | 3.20 | 0.03 | 2.11 | $9\times10^{-3}$ |
| 25.0 | 25.12 | 1280.30 | 10.80 | 0.36 | 5.01 | 0.04 | 3.18 | 0.02 |
| 25.0 | 39.81 | 1800.04 | 14.06 | 0.50 | 7.60 | 0.06 | 4.65 | 0.06 |
| 25.0 | 63.10 | 2462.22 | 17.70 | 0.69 | 11.20 | 0.07 | 6.62 | 0.14 |
| 25.0 | 100.0 | 3318.59 | 21.86 | 0.93 | 16.06 | 0.08 | 9.18 | 0.36 |

| Temper-ature | Shear Rate | Measured $N_1$ | Standard Uncertainty (Type A) | $u(N_1,T)$ | $u(N_1,F)$ | $u(N_1,\omega)$ | $u(N_1,\beta)$ |
|---|---|---|---|---|---|---|---|
| °C | s$^{-1}$ | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 50.0 | 0.3981 | 2.38 | 0.17 | 0.01 | 0.82 | 0.02 | 0.02 |
| 50.0 | 0.6310 | 5.08 | 0.20 | 0.03 | 0.83 | 0.04 | 0.04 |
| 50.0 | 1.000 | 10.73 | 0.26 | 0.06 | 0.84 | 0.09 | 0.09 |
| 50.0 | 1.585 | 21.47 | 0.22 | 0.11 | 0.86 | 0.16 | 0.16 |
| 50.0 | 2.512 | 41.05 | 0.43 | 0.20 | 0.90 | 0.28 | 0.28 |
| 50.0 | 3.981 | 68.85 | 1.15 | 0.32 | 0.95 | 0.43 | 0.43 |
| 50.0 | 6.310 | 114.48 | 2.62 | 0.51 | 1.04 | 0.65 | 0.66 |
| 50.0 | 10.00 | 202.50 | 3.69 | 0.78 | 1.22 | 1.06 | 1.08 |
| 50.0 | 15.85 | 344.80 | 3.97 | 1.16 | 1.50 | 1.68 | 1.71 |
| 50.0 | 25.12 | 557.75 | 4.66 | 1.67 | 1.93 | 2.53 | 2.57 |
| 50.0 | 39.81 | 860.20 | 5.33 | 2.35 | 2.54 | 3.65 | 3.70 |
| 50.0 | 63.10 | 1269.10 | 6.36 | 3.21 | 3.35 | 5.01 | 5.08 |
| 50.0 | 100.0 | 1796.79 | 8.63 | 4.25 | 4.41 | 6.56 | 6.66 |

Table 4. (continued)  Components of Standard Uncertainty in the First Normal Stress Difference $N_1$

| Temper-ature | Shear Rate | Measured $N_1$ | $u(N_1,h_0)$ | $u(N_1,R)$ | Trunc-ation | Tilt | Evapor-ation | Inertia |
|---|---|---|---|---|---|---|---|---|
| °C | s$^{-1}$ | Pa | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | | |
| 50.0 | 0.3981 | 2.38 | 0.05 | $7\times10^{-4}$ | $9\times10^{-4}$ | $3\times10^{-4}$ | 0.03 | $6\times10^{-6}$ |
| 50.0 | 0.6310 | 5.08 | 0.09 | $1\times10^{-3}$ | $3\times10^{-3}$ | $6\times10^{-4}$ | 0.06 | $1\times10^{-5}$ |
| 50.0 | 1.000 | 10.73 | 0.19 | $3\times10^{-3}$ | 0.01 | $1\times10^{-3}$ | 0.13 | $4\times10^{-5}$ |
| 50.0 | 1.585 | 21.47 | 0.34 | $6\times10^{-3}$ | 0.03 | $2\times10^{-3}$ | 0.28 | $9\times10^{-5}$ |
| 50.0 | 2.512 | 41.05 | 0.60 | 0.01 | 0.07 | $3\times10^{-3}$ | 0.56 | $2\times10^{-4}$ |
| 50.0 | 3.981 | 68.85 | 0.92 | 0.02 | 0.16 | $5\times10^{-3}$ | 1.05 | $6\times10^{-4}$ |
| 50.0 | 6.310 | 114.48 | 1.40 | 0.03 | 0.33 | $8\times10^{-3}$ | 1.87 | $1\times10^{-3}$ |
| 50.0 | 10.00 | 202.50 | 2.28 | 0.06 | 0.63 | 0.01 | 3.20 | $4\times10^{-3}$ |
| 50.0 | 15.85 | 344.80 | 3.61 | 0.01 | 1.12 | 0.02 | 5.26 | $9\times10^{-3}$ |
| 50.0 | 25.12 | 557.75 | 5.45 | 0.16 | 1.89 | 0.02 | 8.38 | 0.02 |
| 50.0 | 39.81 | 860.20 | 7.85 | 0.24 | 3.07 | 0.03 | 12.97 | 0.06 |
| 50.0 | 63.10 | 1269.10 | 10.77 | 0.36 | 4.82 | 0.05 | 19.55 | 0.14 |
| 50.0 | 100.0 | 1796.79 | 14.12 | 0.50 | 7.34 | 0.05 | 28.70 | 0.36 |

4.2 Dynamic Testing in Parallel Plates

For the dynamic tests, the uncertainties in the measurements of G' and G" at each temperature and frequency will be estimated. Master curves and the associated shift factors will be calculated using polynomial functions fit to the data in logarithmic space. The curve fitting procedure also provides estimates of the uncertainties in the parameters describing the master curves and shift factors.

The Type A uncertainties associated with variability in the material and random influences on the test conditions were assessed through multiple measurements.

Those sources of uncertainty considered to be Type B are listed below.
1. Temperature
2. Frequency of oscillation
3. Cross-correlation procedure
    a. Transducer reading
    b. Oscillation magnitude
4. Geometry
    a. Gap
    b. Plate diameter
    c. Tilt
    d. Concentricity
5. Solvent evaporation
6. Inertia
7. Edge effects

4.2.1 Uncertainty in the Independent Variables: Temperature and Frequency of Oscillation

The storage modulus G' and loss modulus G" are tabulated as functions of temperature and frequency of oscillation. The standard uncertainty in the temperature is estimated to be 0.1 °C. The standard uncertainty in the frequency of oscillation $\Omega$ is estimated to be $10^{-4} \times \Omega$.

4.2.2 Uncertainties in G' and G"

The Type A uncertainty in each modulus is again calculated from the standard deviation of the repeated experiments divided by the square root of the number of measurements, which gives the uncertainty in the mean of the repeated measurements. These components of uncertainty are listed in Tables 6 and 7.

Type B uncertainties are calculated through the propagation of uncertainties formula in equation (2). These calculations require some analysis of the method by which G' and G" are determined from the geometry, the boundary conditions imposed, and the measured transducer output. The fluid is deformed by oscillating the bottom plate with respect to the upper plate. The strain $\gamma(t)$ in the fluid can be described as

$$\gamma(t) = \frac{r}{h}\phi(t)$$

$$= \frac{r}{h}\phi_0 \sin(\Omega t + \varepsilon) \qquad (32)$$

where $r$ is the radial position, $h$ is the gap between plates, and $\phi(t)$ describes the angular position of the lower plate with respect to the upper plate. The function $\phi(t)$ is assumed to be a sine wave with magnitude of oscillation $\phi_0$, frequency of oscillation $\Omega$, and a possible phase offset of $\varepsilon$ between the master driving signal at $\sin \Omega t$ and the resulting oscillation of the plate. The resulting shear stress $\tau(t)$ in the fluid is assumed as

$$\tau(t) = G'\frac{r}{h}\phi_0 \sin(\Omega t + \varepsilon) + G''\frac{r}{h}\phi_0 \cos(\Omega t + \varepsilon)$$

$$= |G^*|\frac{r}{h}\phi_0 \sin(\Omega t + \delta + \varepsilon) \qquad (33)$$

where G' is the storage modulus, G" is the loss modulus, $|G^*|$ is the magnitude of the complex modulus and $\delta$ is the phase offset between the strain and the stress. These parameters are related, with

$$\begin{aligned} G' &= |G^*|\cos\delta & |G^*|^2 &= (G')^2 + (G'')^2 \\ G'' &= |G^*|\sin\delta & \tan\delta &= \frac{G''}{G'} \end{aligned} \qquad (34)$$

The moment $M$ measured by the transducer is a function of time, and is calculated by integrating over the area of the plate the product of the shear stress and the radial position.

$$M(t) = \int_0^{2\pi}\int_0^R \left[ G'\frac{r^3}{h}\phi_0 \sin(\Omega t + \varepsilon) + G''\frac{r^3}{h}\phi_0 \cos(\Omega t + \varepsilon) \right] dr d\theta$$

$$= \frac{\pi\phi_0 R^4}{2h}\left[G'\sin(\Omega t + \varepsilon) + G''\cos(\Omega t + \varepsilon)\right]$$

$$= \frac{\pi\phi_0 R^4}{2h}|G^*|\sin(\Omega t + \delta + \varepsilon) \qquad (35)$$

$$= M_0 \sin(\Omega t + \delta + \varepsilon)$$

A cross-correlation procedure is used to calculate the storage modulus G' and loss modulus G" from the measured moment $M(t)$ and the measured oscillation $\phi(t)$ [6, 24, 30, 31]. In principle, this procedure correlates the moment $M(t)$ with the strain to calculate the in-phase storage modulus and with a signal $\pi/2$ radians out of phase with the strain to calculate the loss modulus. In practice, the cross-correlation procedure is used to calculate the in-phase and out-of-phase components of both the strain and moment compared to master signals. In this procedure, a measured signal is multiplied together with a master signal (either $\sin \Omega t$ or $\cos \Omega t$) and the result integrated over one or more periods of oscillation to calculate the magnitude and phase angle of the measured signal with respect to the master signal. This procedure also acts a filter to remove unwanted harmonics and noise [6, 24, 30]. The resulting four calculations are

$$\Phi_1 = \frac{\Omega}{k\pi} \int_0^{2k\pi/\Omega} \phi(t)\sin\Omega t\, dt \quad = \quad \phi_0\cos\varepsilon$$

$$\Phi_2 = \frac{\Omega}{k\pi} \int_0^{2k\pi/\Omega} \phi(t)\cos\Omega t\, dt \quad = \quad \phi_0\sin\varepsilon$$

$$\mathbf{M}_1 = \frac{\Omega}{k\pi} \int_0^{2k\pi/\Omega} M(t)\sin\Omega t\, dt \quad = \quad \frac{\pi\phi_0 R^4}{2h}\left[G'\cos\varepsilon - G''\sin\varepsilon\right]$$

$$\mathbf{M}_2 = \frac{\Omega}{k\pi} \int_0^{2k\pi/\Omega} M(t)\cos\Omega t\, dt \quad = \quad \frac{\pi\phi_0 R^4}{2h}\left[G'\sin\varepsilon + G''\cos\varepsilon\right]$$

(36)

where $k$ is the number of cycles over which the integration is performed. The storage and loss moduli are then given by

$$G' = \frac{2h}{\pi R^4}\left[\frac{\mathbf{M}_1\Phi_1 + \mathbf{M}_2\Phi_2}{\Phi_1^2 + \Phi_2^2}\right]$$

$$G'' = \frac{2h}{\pi R^4}\left[\frac{-\mathbf{M}_1\Phi_2 + \mathbf{M}_2\Phi_1}{\Phi_1^2 + \Phi_2^2}\right]$$

(37)

The four quantities $\mathbf{M}_1$, $\mathbf{M}_2$, $\Phi_1$ and $\Phi_2$ are the fundamental outputs from the measurements and the cross-correlation procedure.

The measurements of $G'(\Omega, T)$ and $G''(\Omega, T)$ are shifted to create master curves through time-frequency superposition using a shift factor function $a(T)$, with

$$G'(\Omega,T) = \frac{T\rho}{T_R\rho_R}G'(a(T)\Omega, T_R)$$

$$G''(\Omega,T) = \frac{T\rho}{T_R\rho_R}G''(a(T)\Omega, T_R)$$

(38)

where $T_R = 25\ ^\circ\text{C}$ is the reference temperature, and $a(T)$ has the same WLF functional form used in equation (7), giving

$$a(T) = \exp\left(\frac{-C_1(T - T_R)}{C_2 + T - T_R}\right)$$

(39)

The logarithms of the storage and loss moduli have been fit to polynomial functions of the logarithm of the frequency for calculation of the shift factors [25]. The polynomial functions converge more quickly than an expansion in functions associated with the Rouse modes, and the resulting master curves appear to be satisfactory. The data were fit to functions of the form

$$\ln\left(\frac{G'(\Omega,T)}{1\,\text{Pa}}\right) = \ln\left(\frac{T\rho}{T_R\rho_R}\right) + \sum_{i=0}^{4} p_i\left(\ln\left(\frac{a(T)\Omega}{1\,\text{rad/s}}\right)\right)^i$$

$$\ln\left(\frac{G''(\Omega,T)}{1\,\text{Pa}}\right) = \ln\left(\frac{T\rho}{T_R\rho_R}\right) + \sum_{i=0}^{4} q_i\left(\ln\left(\frac{a(T)\Omega}{1\,\text{rad/s}}\right)\right)^i$$

(40)

Both the storage and loss modulus data were fit simultaneously to the functions in equation (40) to determine the parameters $C_1$ and $C_2$ in the temperature shift factor $a(T)$. These functional representations for the storage and loss moduli are also used to calculate the propagation of the

uncertainties in the temperature and in the frequency of oscillation into the uncertainties in the moduli. The calculated parameters are given in Table 5.

| Table 5. Parameters for G'($\Omega$, $T$), G"($\Omega$, $T$) and $a(T)$ in the models found in equations (39) and (40). | | |
|---|---|---|
| Parameter | Value | Standard Uncertainty |
| $p_0$ | 3.177 | 0.005 |
| $p_1$ | 1.235 | 0.003 |
| $p_2$ | -0.134 | 0.001 |
| $p_3$ | $2.36 \times 10^{-3}$ | $2.7 \times 10^{-4}$ |
| $p_4$ | $5.20 \times 10^{-4}$ | $6.1 \times 10^{-5}$ |
| | | |
| $q_0$ | 4.196 | 0.005 |
| $q_1$ | 0.720 | 0.003 |
| $q_2$ | -0.0719 | 0.0011 |
| $q_3$ | $-3.18 \times 10^{-3}$ | $2.6 \times 10^{-4}$ |
| $q_4$ | $7.06 \times 10^{-4}$ | $6.0 \times 10^{-5}$ |
| | | |
| $C_1$ | 8.85 | 0.30 |
| $C_2$ | 192 °C | 6 °C |

### 4.2.2.1 Uncertainties Associated with Temperature

The components of the standard uncertainty in G' and G" associated with the standard uncertainty in the temperature can be calculated from the functions in equation (40) using the formula for propagation of uncertainty from equation (2).

$$u(G',T) = \frac{\partial G'}{\partial T} u(T)$$

$$= G'(\Omega,T) \left\{ \frac{1}{T} - \frac{\alpha}{1-\alpha(T-T_R)} + \left( \frac{-C_1 C_2}{(C_2+T-T_R)^2} \right) \left[ \sum_{i=1}^{4} ip_i \left( \ln\left( \frac{a(T)\Omega}{1 \text{ rad/s}} \right) \right)^{i-1} \right] \right\} u(T)$$

$$u(G'',T) = \frac{\partial G''}{\partial T} u(T)$$

(41)

$$= G''(\Omega,T) \left\{ \frac{1}{T} - \frac{\alpha}{1-\alpha(T-T_R)} + \left( \frac{-C_1 C_2}{(C_2+T-T_R)^2} \right) \left[ \sum_{i=1}^{4} iq_i \left( \ln\left( \frac{a(T)\Omega}{1 \text{ rad/s}} \right) \right)^{i-1} \right] \right\} u(T)$$

The standard uncertainty in the temperature is $u(T) = 0.1$ °C. The components of the standard uncertainty in G' and G" associated with the standard uncertainty in the temperature are listed in Tables 6 and 7.

### 4.2.2.2 Uncertainties Associated with Frequency of Oscillation

The components of the standard uncertainty in G' and G" associated with the standard uncertainty in the frequency of oscillation $\Omega$ are also calculated from the functions above fit to the data.

$$u(G',\Omega) = \frac{\partial G'}{\partial \Omega} u(\Omega)$$

$$= \frac{G'(\Omega,T)}{\Omega} \left[ \sum_{i=1}^{4} ip_i \left( \ln\left( \frac{a(T)\Omega}{1 \text{ rad/s}} \right) \right)^{i-1} \right] u(\Omega)$$

$$u(G'',\Omega) = \frac{\partial G''}{\partial \Omega} u(\Omega)$$

(42)

$$= \frac{G''(\Omega,T)}{\Omega} \left[ \sum_{i=1}^{4} iq_i \left( \ln\left( \frac{a(T)\Omega}{1 \text{ rad/s}} \right) \right)^{i-1} \right] u(\Omega)$$

The standard uncertainty in the frequency of oscillation is $u(\Omega) = 10^{-4} \times \Omega$. The components of the standard uncertainty in G' and G" associated with the standard uncertainty in the frequency of oscillation are listed in Tables 6 and 7.

### 4.2.2.3 Uncertainties Associated with Cross-correlation of the Transducer Signal and Oscillation Magnitude

The storage modulus G' and loss modulus G" are calculated from cross-correlating the moment and the magnitude of oscillation with master signals of $\sin \Omega t$ and $\cos \Omega t$, as described in connection with equations (36) and (37) above. The cross-correlation procedure does filter out noise and higher harmonics [6, 24, 30]. It seems reasonable to assume that the unwanted signals are no more likely to be correlated with $\sin \Omega t$ than with $\cos \Omega t$, in which case the standard uncertainty in $M_1$ should be equal to the standard uncertainty in $M_2$: $u(M_1) = u(M_2)$. Similarly, the standard uncertainties in $\Phi_1$ and $\Phi_2$ should be equal: $u(\Phi_1) = u(\Phi_2)$. For this special case, where $u(M_1) = u(M_2)$ and $u(\Phi_1) = u(\Phi_2)$, the propagation of the uncertainties into G' and G" have simple forms. Define $u(M) \equiv u(M_1) = u(M_2)$ and $u(\Phi) \equiv u(\Phi_1) = u(\Phi_2)$. The component of standard uncertainty in G' arising from the standard uncertainties in $M_1$ and $M_2$ is

$$
u^2(G',M) \equiv u^2(G',M_1,M_2) = \left(\frac{\partial G'}{\partial M_1}\right)^2 u^2(M_1) + \left(\frac{\partial G'}{\partial M_2}\right)^2 u^2(M_2)
$$
$$
= \left(\frac{2h}{\pi R^4 \phi_0}\right)^2 u^2(M)
$$

(43)

The component of standard uncertainty in G" arising from the standard uncertainties in $M_1$ and $M_2$ is the same as that found above for G' (assuming $u(M_1) = u(M_2)$ and $u(\Phi_1) = u(\Phi_2)$):

$$
u^2(G'',M) \equiv u^2(G'',M_1,M_2) = \left(\frac{2h}{\pi R^4 \phi_0}\right)^2 u^2(M)
$$

(44)

Similarly, the components of standard uncertainty in G' and G" arising from the standard uncertainties in $\Phi_1$ and $\Phi_2$ are

$$
u^2(G',\Phi) \equiv u^2(G',\Phi_1,\Phi_2) = \left(\frac{|G^*|}{\phi_0}\right)^2 u^2(\Phi)
$$
$$
u^2(G'',\Phi) \equiv u^2(G'',\Phi_1,\Phi_2) = \left(\frac{|G^*|}{\phi_0}\right)^2 u^2(\Phi)
$$

(45)

As mentioned above, the cross-correlation procedure does filter out some extraneous signals. The instrument manufacturer indicates that the constant term in the transducer standard uncertainty is decreased by approximately one order of magnitude, so the standard uncertainty in M is taken as $u(M) = 10^{-8}$ N·m + $(0.002)M_0$, where $M_0$ is the magnitude of the sinusoidally varying moment, as defined by equation (35). The instrument manufacturer also indicates that the standard uncertainty in the magnitude of oscillation is $u(\Phi) = (0.0025)\phi_0$. The components of the standard uncertainty in G' and G" associated with the cross correlation procedure are listed in Tables 6 and 7.

### 4.2.2.4 Uncertainties Associated with Geometry

#### 4.2.2.4.1 Gap

The components of the standard uncertainties in G' and G" associated with the standard uncertainty in the gap $h$ are given by

$$u(G',h) = \frac{G'}{h_0}u(h)$$

$$u(G'',h) = \frac{G''}{h_0}u(h)$$

(46)

where $h_0$ is the specified gap. Uncertainty in the gap arises from the uncertainty in the position at zero gap and at the specified setting, uncertainty in the flatness of the upper and lower plates, uncertainty in the thermal expansion of the fixtures, compliance of the transducer and thermal expansion of the transducer [16, 20-22]. A deviation from flatness of the parallel plates could result in a single point of contact at the edge, so the standard uncertainty associated with the position at zero gap was assigned a standard uncertainty of 2.5 μm. The standard uncertainty in the thermal expansion of the fixtures is 0.1 μm/°C, and the gap was zeroed at 25 °C, so the maximum temperature variation from the known gap dimension is 25 °C. Thus, a value of 2.5 μm was assigned as a conservative estimate of the standard uncertainty in the gap resulting from uncertainty in the thermal expansion of the fixtures. Each of the other five influences was assigned a standard uncertainty of 1 μm. These components were added in quadrature to calculate a combined standard uncertainty in the gap of $u(h) = 4.2$ μm. The minimum gap used in the tests was 1.071 mm, so a conservative estimate of $u(h)/h_0$ is $(0.0042/1.071) = 0.004$. The components of the standard uncertainty in G' and G" associated with the standard uncertainty in the gap are listed in Tables 6 and 7.

#### 4.2.2.4.2 Plate Diameter

The components of the standard uncertainties in G' and G" associated with the standard uncertainty in the plate diameter are given by

$$u(G',R) = 4\frac{G'}{R_0}u(R)$$

$$u(G'',R) = 4\frac{G''}{R_0}u(R)$$

(47)

where $R_0 = 25$ mm is the specified plate radius. The standard uncertainty in the radius of the upper or lower plate is estimated to be 0.0025 mm. The combined standard uncertainty calculated by adding these two components in quadrature is $u(R) = 0.0035$ mm. The components of the standard uncertainty in G' and G" associated with the standard uncertainty in the plate diameter are listed in Tables 6 and 7.

### 4.2.2.4.3 Tilt

An angle of tilt between the axis of the upper plate and the axis of the lower plate introduces a bias in the measurement of G' and G". This angle is expressed as a standard uncertainty estimated to be $2 \times 10^{-4}$ rad, and the effects of such a tilt were determined by a two dimensional numerical solution of the integral in equation (35). For a gap of 1 mm, the tilt introduces a component of standard uncertainty in G' of (0.005)G' and a similar component of standard uncertainty in G" of (0.005)G". The components of the standard uncertainty in G' and G" associated with a tilt are listed in Tables 6 and 7.

### 4.2.2.4.4 Concentricity

An offset between the axis of the upper plate and the axis of the lower plate also introduces a bias in the measurement of G' and G". This offset is expressed as a standard uncertainty estimated to be 25 μm. As with the case of measurements in steady shear, the uncertainties in G' and G" associated with this amount of offset are negligible.

### 4.2.2.5 Solvent Evaporation

Solvent evaporation should affect the measurements of G' and G" to an extent that is similar to the effects of solvent evaporation on the steady shear viscosity measurements. An estimate of the effect of evaporation on G' and G" has been made using the measured rates of change in the viscosity at 25 °C and 50 °C coupled with an Arrhenius temperature dependence. The components of standard uncertainty in G' and G" associated with solvent evaporation are listed in Tables 6 and 7.

### 4.2.2.6 Inertia

Inertial effects have been neglected in the equations describing the dynamics of the fluid and the instrument. These influences are not expected to be large, particularly in a strain-controlled rheometer, but the uncertainties associated with inertial effects have not been quantified, so no component of uncertainty has been assigned to the data.

### 4.2.2.7 Edge Effects

In the equations describing the response of the fluid, the geometry is assumed to be a perfect cylinder. The exact shape of the fluid edge will be affected by thermal expansion, surface tension and fluid migration, and the shape of the edge will affect the measurements of G' and G". Again, these effects are not expected to be large, but they have not been quantified, so no component of uncertainty has been assigned to the data.

| Table 6. Components of Standard Uncertainty in the Storage Modulus G' | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | Standard Uncertainty (Type A) | $u(G',T)$ | $u(G',\Omega)$ | $u(G',M)$ | $u(G',\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 0.0 | 0.02512 | 0.605 | $7.7\times10^{-3}$ | $6.3\times10^{-3}$ | $1.1\times10^{-4}$ | 0.018 | 0.020 |
| 0.0 | 0.03981 | 1.385 | $9.4\times10^{-3}$ | 0.014 | $2.3\times10^{-4}$ | 0.027 | 0.031 |
| 0.0 | 0.06310 | 2.973 | $8.3\times10^{-3}$ | 0.027 | $4.6\times10^{-4}$ | 0.040 | 0.048 |
| 0.0 | 0.1000 | 6.067 | 0.012 | 0.052 | $8.8\times10^{-4}$ | 0.060 | 0.072 |
| 0.0 | 0.1585 | 11.699 | 0.028 | 0.092 | $1.6\times10^{-3}$ | 0.088 | 0.107 |
| 0.0 | 0.2512 | 21.207 | 0.048 | 0.152 | $2.6\times10^{-3}$ | 0.126 | 0.155 |
| 0.0 | 0.3981 | 36.214 | 0.066 | 0.236 | $4.1\times10^{-3}$ | 0.177 | 0.219 |
| 0.0 | 0.6310 | 58.511 | 0.123 | 0.343 | $5.9\times10^{-3}$ | 0.244 | 0.302 |
| 0.0 | 1.000 | 89.636 | 0.196 | 0.470 | $8.2\times10^{-3}$ | 0.327 | 0.406 |
| 0.0 | 1.585 | 130.948 | 0.263 | 0.606 | 0.011 | 0.428 | 0.533 |
| 0.0 | 2.512 | 182.959 | 0.400 | 0.740 | 0.013 | 0.547 | 0.681 |
| 0.0 | 3.981 | 246.085 | 0.565 | 0.858 | 0.015 | 0.684 | 0.852 |
| 0.0 | 6.310 | 319.791 | 0.804 | 0.953 | 0.017 | 0.838 | 1.044 |
| 0.0 | 10.00 | 403.264 | 1.035 | 1.022 | 0.019 | 1.007 | 1.256 |
| 0.0 | 15.85 | 495.279 | 1.284 | 1.075 | 0.020 | 1.190 | 1.485 |
| 0.0 | 25.12 | 593.784 | 1.610 | 1.129 | 0.022 | 1.385 | 1.728 |
| 0.0 | 39.81 | 697.210 | 1.966 | 1.210 | 0.023 | 1.589 | 1.984 |
| 0.0 | 63.10 | 804.155 | 2.290 | 1.355 | 0.026 | 1.802 | 2.250 |
| 0.0 | 100.0 | 912.794 | 2.672 | 1.615 | 0.031 | 2.022 | 2.525 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | $u(G',h)$ | $u(G',R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 0.0 | 0.02512 | 0.605 | $2.4\times10^{-3}$ | $3.4\times10^{-4}$ | $3.0\times10^{-3}$ | $2.4\times10^{-5}$ |
| 0.0 | 0.03981 | 1.385 | $5.5\times10^{-3}$ | $7.8\times10^{-4}$ | $6.9\times10^{-3}$ | $9.7\times10^{-5}$ |
| 0.0 | 0.06310 | 2.973 | 0.012 | $1.7\times10^{-3}$ | 0.015 | $2.6\times10^{-4}$ |
| 0.0 | 0.1000 | 6.067 | 0.024 | $3.4\times10^{-3}$ | 0.030 | $6.0\times10^{-4}$ |
| 0.0 | 0.1585 | 11.699 | 0.047 | $6.6\times10^{-3}$ | 0.058 | $1.2\times10^{-3}$ |
| 0.0 | 0.2512 | 21.207 | 0.085 | 0.012 | 0.106 | $2.3\times10^{-3}$ |
| 0.0 | 0.3981 | 36.214 | 0.145 | 0.020 | 0.181 | $4.0\times10^{-3}$ |
| 0.0 | 0.6310 | 58.511 | 0.234 | 0.033 | 0.293 | $6.6\times10^{-3}$ |
| 0.0 | 1.000 | 89.636 | 0.359 | 0.050 | 0.448 | 0.010 |
| 0.0 | 1.585 | 130.948 | 0.524 | 0.073 | 0.655 | 0.015 |
| 0.0 | 2.512 | 182.959 | 0.732 | 0.102 | 0.915 | 0.021 |
| 0.0 | 3.981 | 246.085 | 0.984 | 0.138 | 1.230 | 0.029 |
| 0.0 | 6.310 | 319.791 | 1.279 | 0.179 | 1.599 | 0.037 |
| 0.0 | 10.00 | 403.264 | 1.613 | 0.226 | 2.016 | 0.047 |
| 0.0 | 15.85 | 495.279 | 1.981 | 0.277 | 2.476 | 0.058 |
| 0.0 | 25.12 | 593.784 | 2.375 | 0.333 | 2.969 | 0.070 |
| 0.0 | 39.81 | 697.210 | 2.789 | 0.390 | 3.486 | 0.082 |
| 0.0 | 63.10 | 804.155 | 3.217 | 0.450 | 4.021 | 0.095 |
| 0.0 | 100.0 | 912.794 | 3.651 | 0.511 | 4.564 | 0.109 |

| Table 6. (continued)  Components of Standard Uncertainty in the Storage Modulus G' | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | Standard Uncertainty (Type A) | $u(G',T)$ | $u(G',\Omega)$ | $u(G',M)$ | $u(G',\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 10.0 | 0.03981 | 0.486 | $6.1\times10^{-3}$ | $4.7\times10^{-3}$ | $9.0\times10^{-5}$ | 0.017 | 0.018 |
| 10.0 | 0.06310 | 1.099 | $9.8\times10^{-3}$ | 0.010 | $2.0\times10^{-4}$ | 0.025 | 0.029 |
| 10.0 | 0.1000 | 2.438 | 0.012 | 0.021 | $4.0\times10^{-4}$ | 0.037 | 0.044 |
| 10.0 | 0.1585 | 5.076 | 0.014 | 0.041 | $7.8\times10^{-4}$ | 0.056 | 0.067 |
| 10.0 | 0.2512 | 10.023 | 0.026 | 0.073 | $1.4\times10^{-3}$ | 0.082 | 0.100 |
| 10.0 | 0.3981 | 18.531 | 0.041 | 0.123 | $2.4\times10^{-3}$ | 0.119 | 0.146 |
| 10.0 | 0.6310 | 32.329 | 0.077 | 0.194 | $3.8\times10^{-3}$ | 0.169 | 0.209 |
| 10.0 | 1.000 | 53.217 | 0.138 | 0.287 | $5.6\times10^{-3}$ | 0.234 | 0.290 |
| 10.0 | 1.585 | 83.154 | 0.199 | 0.399 | $7.8\times10^{-3}$ | 0.317 | 0.394 |
| 10.0 | 2.512 | 123.313 | 0.303 | 0.523 | 0.010 | 0.418 | 0.520 |
| 10.0 | 3.981 | 174.767 | 0.393 | 0.646 | 0.013 | 0.538 | 0.670 |
| 10.0 | 6.310 | 238.200 | 0.537 | 0.757 | 0.015 | 0.678 | 0.846 |
| 10.0 | 10.00 | 313.204 | 0.738 | 0.848 | 0.017 | 0.837 | 1.044 |
| 10.0 | 15.85 | 398.816 | 1.002 | 0.916 | 0.019 | 1.013 | 1.263 |
| 10.0 | 25.12 | 493.868 | 1.272 | 0.965 | 0.020 | 1.204 | 1.502 |
| 10.0 | 39.81 | 596.352 | 1.585 | 1.010 | 0.022 | 1.408 | 1.757 |
| 10.0 | 63.10 | 704.450 | 1.921 | 1.073 | 0.024 | 1.623 | 2.026 |
| 10.0 | 100.0 | 816.167 | 2.268 | 1.186 | 0.026 | 1.847 | 2.306 |

| Table 6. (continued)  Components of Standard Uncertainty in the Storage Modulus G' | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | $u(G',h)$ | $u(G',R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 10.0 | 0.03981 | 0.486 | $1.9\times10^{-3}$ | $2.7\times10^{-4}$ | $2.4\times10^{-3}$ | $1.1\times10^{-4}$ |
| 10.0 | 0.06310 | 1.099 | $4.4\times10^{-3}$ | $6.2\times10^{-4}$ | $5.5\times10^{-3}$ | $3.1\times10^{-4}$ |
| 10.0 | 0.1000 | 2.438 | $9.8\times10^{-3}$ | $1.4\times10^{-3}$ | 0.012 | $7.5\times10^{-4}$ |
| 10.0 | 0.1585 | 5.076 | 0.020 | $2.8\times10^{-3}$ | 0.025 | $1.7\times10^{-3}$ |
| 10.0 | 0.2512 | 10.023 | 0.040 | $5.6\times10^{-3}$ | 0.050 | $3.4\times10^{-3}$ |
| 10.0 | 0.3981 | 18.531 | 0.074 | 0.010 | 0.093 | $6.4\times10^{-3}$ |
| 10.0 | 0.6310 | 32.329 | 0.129 | 0.018 | 0.162 | 0.011 |
| 10.0 | 1.000 | 53.217 | 0.213 | 0.030 | 0.266 | 0.019 |
| 10.0 | 1.585 | 83.154 | 0.333 | 0.047 | 0.416 | 0.029 |
| 10.0 | 2.512 | 123.313 | 0.493 | 0.069 | 0.617 | 0.044 |
| 10.0 | 3.981 | 174.767 | 0.699 | 0.098 | 0.874 | 0.062 |
| 10.0 | 6.310 | 238.200 | 0.953 | 0.133 | 1.191 | 0.085 |
| 10.0 | 10.00 | 313.204 | 1.253 | 0.175 | 1.566 | 0.112 |
| 10.0 | 15.85 | 398.816 | 1.595 | 0.223 | 1.994 | 0.143 |
| 10.0 ´ | 25.12 | 493.868 | 1.975 | 0.277 | 2.469 | 0.178 |
| 10.0 | 39.81 | 596.352 | 2.385 | 0.334 | 2.982 | 0.216 |
| 10.0 | 63.10 | 704.450 | 2.818 | 0.394 | 3.522 | 0.255 |
| 10.0 | 100.0 | 816.167 | 3.265 | 0.457 | 4.081 | 0.296 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | Standard Uncertainty (Type A) | $u(G',T)$ | $u(G',\Omega)$ | $u(G',M)$ | $u(G',\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 20.0 | 0.03981 | 0.182 | $5.8\times10^{-3}$ | $1.7\times10^{-3}$ | $3.6\times10^{-5}$ | 0.011 | 0.012 |
| 20.0 | 0.06310 | 0.430 | $8.6\times10^{-3}$ | $4.0\times10^{-3}$ | $8.4\times10^{-5}$ | 0.017 | 0.018 |
| 20.0 | 0.1000 | 1.023 | $8.8\times10^{-3}$ | $8.7\times10^{-3}$ | $1.9\times10^{-4}$ | 0.025 | 0.028 |
| 20.0 | 0.1585 | 2.275 | 0.012 | 0.018 | $3.8\times10^{-4}$ | 0.037 | 0.044 |
| 20.0 | 0.2512 | 4.809 | 0.017 | 0.035 | $7.4\times10^{-4}$ | 0.056 | 0.067 |
| 20.0 | 0.3981 | 9.571 | 0.031 | 0.063 | $1.4\times10^{-3}$ | 0.082 | 0.100 |
| 20.0 | 0.6310 | 17.918 | 0.050 | 0.107 | $2.3\times10^{-3}$ | 0.120 | 0.147 |
| 20.0 | 1.000 | 31.620 | 0.084 | 0.170 | $3.7\times10^{-3}$ | 0.171 | 0.211 |
| 20.0 | 1.585 | 52.610 | 0.129 | 0.253 | $5.5\times10^{-3}$ | 0.238 | 0.295 |
| 20.0 | 2.512 | 82.735 | 0.188 | 0.354 | $7.8\times10^{-3}$ | 0.323 | 0.401 |
| 20.0 | 3.981 | 123.780 | 0.274 | 0.466 | 0.010 | 0.428 | 0.532 |
| 20.0 | 6.310 | 176.784 | 0.384 | 0.580 | 0.013 | 0.553 | 0.689 |
| 20.0 | 10.00 | 242.540 | 0.490 | 0.683 | 0.015 | 0.700 | 0.873 |
| 20.0 | 15.85 | 320.640 | 0.637 | 0.768 | 0.018 | 0.867 | 1.081 |
| 20.0 | 25.12 | 410.368 | 0.834 | 0.831 | 0.019 | 1.052 | 1.313 |
| 20.0 | 39.81 | 510.091 | 1.069 | 0.876 | 0.021 | 1.254 | 1.565 |
| 20.0 | 63.10 | 617.780 | 1.312 | 0.915 | 0.022 | 1.470 | 1.836 |
| 20.0 | 100.0 | 731.238 | 1.575 | 0.968 | 0.024 | 1.698 | 2.120 |

| Table 6. (continued)  Components of Standard Uncertainty in the Storage Modulus G' | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | $u(G',h)$ | $u(G',R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 20.0 | 0.03981 | 0.182 | $7.3\times10^{-4}$ | $1.0\times10^{-4}$ | $9.1\times10^{-4}$ | $1.1\times10^{-4}$ |
| 20.0 | 0.06310 | 0.430 | $1.7\times10^{-3}$ | $2.4\times10^{-4}$ | $2.2\times10^{-3}$ | $3.2\times10^{-4}$ |
| 20.0 | 0.1000 | 1.023 | $4.1\times10^{-3}$ | $5.7\times10^{-4}$ | $5.1\times10^{-3}$ | $8.2\times10^{-4}$ |
| 20.0 | 0.1585 | 2.275 | $9.1\times10^{-3}$ | $1.3\times10^{-3}$ | 0.011 | $1.9\times10^{-3}$ |
| 20.0 | 0.2512 | 4.809 | 0.019 | $2.7\times10^{-3}$ | 0.024 | $4.2\times10^{-3}$ |
| 20.0 | 0.3981 | 9.571 | 0.038 | $5.4\times10^{-3}$ | 0.048 | $8.5\times10^{-3}$ |
| 20.0 | 0.6310 | 17.918 | 0.072 | 0.010 | 0.090 | 0.016 |
| 20.0 | 1.000 | 31.620 | 0.126 | 0.018 | 0.158 | 0.029 |
| 20.0 | 1.585 | 52.610 | 0.210 | 0.029 | 0.263 | 0.048 |
| 20.0 | 2.512 | 82.735 | 0.331 | 0.046 | 0.414 | 0.076 |
| 20.0 | 3.981 | 123.780 | 0.495 | 0.069 | 0.619 | 0.114 |
| 20.0 | 6.310 | 176.784 | 0.707 | 0.099 | 0.884 | 0.163 |
| 20.0 | 10.00 | 242.540 | 0.970 | 0.136 | 1.213 | 0.224 |
| 20.0 | 15.85 | 320.640 | 1.283 | 0.180 | 1.603 | 0.298 |
| 20.0 | 25.12 | 410.368 | 1.641 | 0.230 | 2.052 | 0.382 |
| 20.0 | 39.81 | 510.091 | 2.040 | 0.286 | 2.550 | 0.476 |
| 20.0 | 63.10 | 617.780 | 2.471 | 0.346 | 3.089 | 0.578 |
| 20.0 | 100.0 | 731.238 | 2.925 | 0.409 | 3.656 | 0.685 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | Standard Uncertainty (Type A) | $u(G',T)$ | $u(G',\Omega)$ | $u(G',M)$ | $u(G',\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 30.0 | 0.03981 | 0.077 | $2.9\times10^{-3}$ | $6.3\times10^{-4}$ | $1.5\times10^{-5}$ | $8.1\times10^{-3}$ | $7.6\times10^{-3}$ |
| 30.0 | 0.06310 | 0.178 | $7.1\times10^{-3}$ | $1.6\times10^{-3}$ | $3.7\times10^{-5}$ | 0.012 | 0.012 |
| 30.0 | 0.1000 | 0.440 | $7.5\times10^{-3}$ | $3.7\times10^{-3}$ | $8.6\times10^{-5}$ | 0.017 | 0.019 |
| 30.0 | 0.1585 | 1.039 | $8.1\times10^{-3}$ | $8.0\times10^{-3}$ | $1.9\times10^{-4}$ | 0.025 | 0.029 |
| 30.0 | 0.2512 | 2.323 | 0.012 | 0.017 | $3.9\times10^{-4}$ | 0.038 | 0.045 |
| 30.0 | 0.3981 | 4.899 | 0.021 | 0.032 | $7.6\times10^{-4}$ | 0.057 | 0.069 |
| 30.0 | 0.6310 | 9.776 | 0.037 | 0.058 | $1.4\times10^{-3}$ | 0.085 | 0.103 |
| 30.0 | 1.000 | 18.376 | 0.058 | 0.099 | $2.4\times10^{-3}$ | 0.124 | 0.152 |
| 30.0 | 1.585 | 32.459 | 0.094 | 0.157 | $3.8\times10^{-3}$ | 0.176 | 0.218 |
| 30.0 | 2.512 | 54.041 | 0.139 | 0.234 | $5.7\times10^{-3}$ | 0.246 | 0.305 |
| 30.0 | 3.981 | 85.240 | 0.186 | 0.327 | $8.0\times10^{-3}$ | 0.334 | 0.415 |
| 30.0 | 6.310 | 127.700 | 0.262 | 0.430 | 0.011 | 0.443 | 0.552 |
| 30.0 | 10.00 | 182.644 | 0.340 | 0.534 | 0.013 | 0.574 | 0.715 |
| 30.0 | 15.85 | 250.728 | 0.433 | 0.628 | 0.016 | 0.726 | 0.905 |
| 30.0 | 25.12 | 331.736 | 0.548 | 0.706 | 0.018 | 0.900 | 1.122 |
| 30.0 | 39.81 | 424.451 | 0.663 | 0.763 | 0.020 | 1.092 | 1.363 |
| 30.0 | 63.10 | 527.345 | 0.798 | 0.803 | 0.022 | 1.302 | 1.626 |
| 30.0 | 100.0 | 638.186 | 0.947 | 0.838 | 0.023 | 1.527 | 1.906 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | $u(G',h)$ | $u(G',R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 30.0 | 0.03981 | 0.077 | $3.1\times10^{-4}$ | $4.3\times10^{-5}$ | $3.8\times10^{-4}$ | $1.2\times10^{-4}$ |
| 30.0 | 0.06310 | 0.178 | $7.1\times10^{-4}$ | $1.0\times10^{-4}$ | $8.9\times10^{-4}$ | $3.2\times10^{-4}$ |
| 30.0 | 0.1000 | 0.440 | $1.8\times10^{-3}$ | $2.5\times10^{-4}$ | $2.2\times10^{-3}$ | $8.5\times10^{-4}$ |
| 30.0 | 0.1585 | 1.039 | $4.2\times10^{-3}$ | $5.8\times10^{-4}$ | $5.2\times10^{-3}$ | $2.1\times10^{-3}$ |
| 30.0 | 0.2512 | 2.323 | $9.3\times10^{-3}$ | $1.3\times10^{-3}$ | 0.012 | $4.8\times10^{-3}$ |
| 30.0 | 0.3981 | 4.899 | 0.020 | $2.7\times10^{-3}$ | 0.024 | 0.010 |
| 30.0 | 0.6310 | 9.776 | 0.039 | $5.5\times10^{-3}$ | 0.049 | 0.021 |
| 30.0 | 1.000 | 18.376 | 0.074 | 0.010 | 0.092 | 0.040 |
| 30.0 | 1.585 | 32.459 | 0.130 | 0.018 | 0.162 | 0.071 |
| 30.0 | 2.512 | 54.041 | 0.216 | 0.030 | 0.270 | 0.118 |
| 30.0 | 3.981 | 85.240 | 0.341 | 0.048 | 0.426 | 0.186 |
| 30.0 | 6.310 | 127.700 | 0.511 | 0.072 | 0.639 | 0.280 |
| 30.0 | 10.00 | 182.644 | 0.731 | 0.102 | 0.913 | 0.401 |
| 30.0 | 15.85 | 250.728 | 1.003 | 0.140 | 1.254 | 0.553 |
| 30.0 | 25.12 | 331.736 | 1.327 | 0.186 | 1.659 | 0.733 |
| 30.0 | 39.81 | 424.451 | 1.698 | 0.238 | 2.122 | 0.940 |
| 30.0 | 63.10 | 527.345 | 2.109 | 0.295 | 2.637 | 1.171 |
| 30.0 | 100.0 | 638.186 | 2.553 | 0.357 | 3.191 | 1.419 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | Standard Uncertainty (Type A) | $u(G',T)$ | $u(G',\Omega)$ | $u(G',M)$ | $u(G',\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 40.0 | 0.03981 | 0.030 | $4.9\times10^{-3}$ | $2.4\times10^{-4}$ | $6.3\times10^{-6}$ | $6.1\times10^{-3}$ | $5.1\times10^{-3}$ |
| 40.0 | 0.06310 | 0.062 | $4.7\times10^{-3}$ | $6.4\times10^{-4}$ | $1.7\times10^{-5}$ | $8.5\times10^{-3}$ | $8.1\times10^{-3}$ |
| 40.0 | 0.1000 | 0.200 | $6.4\times10^{-3}$ | $1.6\times10^{-3}$ | $4.1\times10^{-5}$ | 0.012 | 0.013 |
| 40.0 | 0.1585 | 0.478 | $3.1\times10^{-3}$ | $3.7\times10^{-3}$ | $9.6\times10^{-5}$ | 0.018 | 0.020 |
| 40.0 | 0.2512 | 1.142 | $8.8\times10^{-3}$ | $8.0\times10^{-3}$ | $2.1\times10^{-4}$ | 0.027 | 0.031 |
| 40.0 | 0.3981 | 2.562 | 0.015 | 0.016 | $4.3\times10^{-4}$ | 0.041 | 0.048 |
| 40.0 | 0.6310 | 5.391 | 0.020 | 0.032 | $8.3\times10^{-4}$ | 0.061 | 0.074 |
| 40.0 | 1.000 | 10.695 | 0.035 | 0.057 | $1.5\times10^{-3}$ | 0.090 | 0.110 |
| 40.0 | 1.585 | 19.966 | 0.054 | 0.096 | $2.6\times10^{-3}$ | 0.131 | 0.161 |
| 40.0 | 2.512 | 35.035 | 0.084 | 0.152 | $4.1\times10^{-3}$ | 0.187 | 0.231 |
| 40.0 | 3.981 | 58.033 | 0.121 | 0.224 | $6.0\times10^{-3}$ | 0.260 | 0.322 |
| 40.0 | 6.310 | 91.010 | 0.165 | 0.311 | $8.4\times10^{-3}$ | 0.353 | 0.438 |
| 40.0 | 10.00 | 135.717 | 0.218 | 0.407 | 0.011 | 0.467 | 0.581 |
| 40.0 | 15.85 | 193.163 | 0.289 | 0.503 | 0.014 | 0.602 | 0.750 |
| 40.0 | 25.12 | 264.093 | 0.369 | 0.588 | 0.017 | 0.761 | 0.948 |
| 40.0 | 39.81 | 347.741 | 0.469 | 0.657 | 0.019 | 0.940 | 1.172 |
| 40.0 | 63.10 | 443.102 | 0.576 | 0.707 | 0.021 | 1.139 | 1.421 |
| 40.0 | 100.0 | 548.169 | 0.708 | 0.742 | 0.022 | 1.355 | 1.691 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | $u(G',h)$ | $u(G',R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 40.0 | 0.03981 | 0.030 | $1.2\times10^{-4}$ | $1.7\times10^{-5}$ | $1.5\times10^{-4}$ | $1.1\times10^{-4}$ |
| 40.0 | 0.06310 | 0.062 | $2.5\times10^{-4}$ | $3.4\times10^{-5}$ | $3.1\times10^{-4}$ | $2.5\times10^{-4}$ |
| 40.0 | 0.1000 | 0.200 | $8.0\times10^{-4}$ | $1.1\times10^{-4}$ | $1.0\times10^{-3}$ | $8.7\times10^{-4}$ |
| 40.0 | 0.1585 | 0.478 | $1.9\times10^{-3}$ | $2.7\times10^{-4}$ | $2.4\times10^{-3}$ | $2.2\times10^{-3}$ |
| 40.0 | 0.2512 | 1.142 | $4.6\times10^{-3}$ | $6.4\times10^{-4}$ | $5.7\times10^{-3}$ | $5.3\times10^{-3}$ |
| 40.0 | 0.3981 | 2.562 | 0.010 | $1.4\times10^{-3}$ | 0.013 | 0.012 |
| 40.0 | 0.6310 | 5.391 | 0.022 | $3.0\times10^{-3}$ | 0.027 | 0.026 |
| 40.0 | 1.000 | 10.695 | 0.043 | $6.0\times10^{-3}$ | 0.053 | 0.052 |
| 40.0 | 1.585 | 19.966 | 0.080 | 0.011 | 0.100 | 0.098 |
| 40.0 | 2.512 | 35.035 | 0.140 | 0.020 | 0.175 | 0.172 |
| 40.0 | 3.981 | 58.033 | 0.232 | 0.032 | 0.290 | 0.285 |
| 40.0 | 6.310 | 91.010 | 0.364 | 0.051 | 0.455 | 0.449 |
| 40.0 | 10.00 | 135.717 | 0.543 | 0.076 | 0.679 | 0.671 |
| 40.0 | 15.85 | 193.163 | 0.773 | 0.108 | 0.966 | 0.957 |
| 40.0 | 25.12 | 264.093 | 1.056 | 0.148 | 1.320 | 1.312 |
| 40.0 | 39.81 | 347.741 | 1.391 | 0.195 | 1.739 | 1.732 |
| 40.0 | 63.10 | 443.102 | 1.772 | 0.248 | 2.216 | 2.211 |
| 40.0 | 100.0 | 548.169 | 2.193 | 0.307 | 2.741 | 2.740 |

| Table 6. (continued)  Components of Standard Uncertainty in the Storage Modulus G' | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | Standard Uncertainty (Type A) | $u(G',T)$ | $u(G',\Omega)$ | $u(G',\mathbf{M})$ | $u(G',\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 50.0 | 0.03981 | 0.013 | $5.6\times10^{-3}$ | $9.9\times10^{-5}$ | $2.8\times10^{-6}$ | $5.0\times10^{-3}$ | $3.6\times10^{-3}$ |
| 50.0 | 0.06310 | 0.034 | $5.9\times10^{-3}$ | $2.7\times10^{-4}$ | $7.7\times10^{-6}$ | $6.7\times10^{-3}$ | $5.8\times10^{-3}$ |
| 50.0 | 0.1000 | 0.088 | $4.7\times10^{-3}$ | $7.0\times10^{-4}$ | $2.0\times10^{-5}$ | $9.3\times10^{-3}$ | $9.1\times10^{-3}$ |
| 50.0 | 0.1585 | 0.241 | $4.1\times10^{-3}$ | $1.7\times10^{-3}$ | $4.9\times10^{-5}$ | 0.013 | 0.014 |
| 50.0 | 0.2512 | 0.592 | $5.2\times10^{-3}$ | $3.9\times10^{-3}$ | $1.1\times10^{-4}$ | 0.020 | 0.022 |
| 50.0 | 0.3981 | 1.375 | 0.011 | $8.5\times10^{-3}$ | $2.5\times10^{-4}$ | 0.030 | 0.035 |
| 50.0 | 0.6310 | 3.058 | 0.013 | 0.017 | $5.0\times10^{-4}$ | 0.045 | 0.054 |
| 50.0 | 1.000 | 6.385 | 0.027 | 0.033 | $9.6\times10^{-4}$ | 0.068 | 0.082 |
| 50.0 | 1.585 | 12.509 | 0.041 | 0.059 | $1.7\times10^{-3}$ | 0.100 | 0.122 |
| 50.0 | 2.512 | 23.028 | 0.067 | 0.097 | $2.9\times10^{-3}$ | 0.145 | 0.179 |
| 50.0 | 3.981 | 39.969 | 0.105 | 0.152 | $4.5\times10^{-3}$ | 0.205 | 0.254 |
| 50.0 | 6.310 | 65.434 | 0.148 | 0.222 | $6.6\times10^{-3}$ | 0.284 | 0.353 |
| 50.0 | 10.00 | 101.548 | 0.207 | 0.305 | $9.1\times10^{-3}$ | 0.384 | 0.477 |
| 50.0 | 15.85 | 149.841 | 0.280 | 0.394 | 0.012 | 0.505 | 0.629 |
| 50.0 | 25.12 | 211.567 | 0.363 | 0.482 | 0.015 | 0.649 | 0.809 |
| 50.0 | 39.81 | 286.640 | 0.458 | 0.558 | 0.017 | 0.816 | 1.018 |
| 50.0 | 63.10 | 374.506 | 0.576 | 0.618 | 0.020 | 1.004 | 1.253 |
| 50.0 | 100.0 | 473.567 | 0.703 | 0.661 | 0.022 | 1.212 | 1.512 |

| Table 6. (continued) Components of Standard Uncertainty in the Storage Modulus G' | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Storage Modulus G' | $u(G',h)$ | $u(G',R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 50.0 | 0.03981 | 0.013 | $5.1\times10^{-5}$ | $7.1\times10^{-6}$ | $6.3\times10^{-5}$ | $9.7\times10^{-5}$ |
| 50.0 | 0.06310 | 0.034 | $1.4\times10^{-4}$ | $1.9\times10^{-5}$ | $1.7\times10^{-4}$ | $3.0\times10^{-4}$ |
| 50.0 | 0.1000 | 0.088 | $3.5\times10^{-4}$ | $4.9\times10^{-5}$ | $4.4\times10^{-4}$ | $8.3\times10^{-4}$ |
| 50.0 | 0.1585 | 0.241 | $9.7\times10^{-4}$ | $1.4\times10^{-4}$ | $1.2\times10^{-3}$ | $2.4\times10^{-3}$ |
| 50.0 | 0.2512 | 0.592 | $2.4\times10^{-3}$ | $3.3\times10^{-4}$ | $3.0\times10^{-3}$ | $6.0\times10^{-3}$ |
| 50.0 | 0.3981 | 1.375 | $5.5\times10^{-3}$ | $7.7\times10^{-4}$ | $6.9\times10^{-3}$ | 0.014 |
| 50.0 | 0.6310 | 3.058 | 0.012 | $1.7\times10^{-3}$ | 0.015 | 0.032 |
| 50.0 | 1.000 | 6.385 | 0.026 | $3.6\times10^{-3}$ | 0.032 | 0.067 |
| 50.0 | 1.585 | 12.509 | 0.050 | $7.0\times10^{-3}$ | 0.063 | 0.132 |
| 50.0 | 2.512 | 23.028 | 0.092 | 0.013 | 0.115 | 0.243 |
| 50.0 | 3.981 | 39.969 | 0.160 | 0.022 | 0.200 | 0.424 |
| 50.0 | 6.310 | 65.434 | 0.262 | 0.037 | 0.327 | 0.695 |
| 50.0 | 10.00 | 101.548 | 0.406 | 0.057 | 0.508 | 1.081 |
| 50.0 | 15.85 | 149.841 | 0.599 | 0.084 | 0.749 | 1.600 |
| 50.0 ́ | 25.12 | 211.567 | 0.846 | 0.118 | 1.058 | 2.264 |
| 50.0 | 39.81 | 286.640 | 1.147 | 0.161 | 1.433 | 3.075 |
| 50.0 | 63.10 | 374.506 | 1.498 | 0.210 | 1.873 | 4.025 |
| 50.0 | 100.0 | 473.567 | 1.894 | 0.265 | 2.368 | 5.098 |

| Table 7. Components of Standard Uncertainty in the Loss Modulus G" | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | Standard Uncertainty (Type A) | $u(G",T)$ | $u(G",\Omega)$ | $u(G",M)$ | $u(G",\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 0.0 | 0.02512 | 7.976 | 0.019 | 0.045 | $7.8\times10^{-4}$ | 0.018 | 0.020 |
| 0.0 | 0.03981 | 12.346 | 0.031 | 0.068 | $1.2\times10^{-3}$ | 0.027 | 0.031 |
| 0.0 | 0.06310 | 18.852 | 0.047 | 0.099 | $1.7\times10^{-3}$ | 0.040 | 0.048 |
| 0.0 | 0.1000 | 28.201 | 0.067 | 0.139 | $2.4\times10^{-3}$ | 0.060 | 0.072 |
| 0.0 | 0.1585 | 41.113 | 0.098 | 0.188 | $3.3\times10^{-3}$ | 0.088 | 0.107 |
| 0.0 | 0.2512 | 58.262 | 0.143 | 0.245 | $4.3\times10^{-3}$ | 0.126 | 0.155 |
| 0.0 | 0.3981 | 79.846 | 0.202 | 0.303 | $5.4\times10^{-3}$ | 0.177 | 0.219 |
| 0.0 | 0.6310 | 105.855 | 0.300 | 0.358 | $6.4\times10^{-3}$ | 0.244 | 0.302 |
| 0.0 | 1.000 | 135.566 | 0.360 | 0.402 | $7.3\times10^{-3}$ | 0.327 | 0.406 |
| 0.0 | 1.585 | 168.058 | 0.481 | 0.427 | $7.9\times10^{-3}$ | 0.428 | 0.533 |
| 0.0 | 2.512 | 201.883 | 0.592 | 0.429 | $8.1\times10^{-3}$ | 0.547 | 0.681 |
| 0.0 | 3.981 | 235.898 | 0.724 | 0.406 | $7.9\times10^{-3}$ | 0.684 | 0.852 |
| 0.0 | 6.310 | 268.794 | 0.871 | 0.363 | $7.4\times10^{-3}$ | 0.838 | 1.044 |
| 0.0 | 10.00 | 299.722 | 0.994 | 0.305 | $6.6\times10^{-3}$ | 1.007 | 1.256 |
| 0.0 | 15.85 | 328.134 | 1.122 | 0.244 | $5.8\times10^{-3}$ | 1.190 | 1.485 |
| 0.0 | 25.12 | 354.144 | 1.244 | 0.191 | $5.0\times10^{-3}$ | 1.385 | 1.728 |
| 0.0 | 39.81 | 378.885 | 1.361 | 0.157 | $4.6\times10^{-3}$ | 1.589 | 1.984 |
| 0.0 | 63.10 | 404.134 | 1.499 | 0.152 | $4.6\times10^{-3}$ | 1.802 | 2.250 |
| 0.0 | 100.0 | 432.131 | 1.685 | 0.189 | $5.3\times10^{-3}$ | 2.022 | 2.525 |

| Table 7. (continued)  Components of Standard Uncertainty in the Loss Modulus G" | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | $u(G",h)$ | $u(G",R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 0.0 | 0.02512 | 7.976 | 0.032 | $4.5 \times 10^{-3}$ | 0.040 | $3.2 \times 10^{-4}$ |
| 0.0 | 0.03981 | 12.346 | 0.049 | $6.9 \times 10^{-3}$ | 0.062 | $8.6 \times 10^{-4}$ |
| 0.0 | 0.06310 | 18.852 | 0.075 | 0.011 | 0.094 | $1.6 \times 10^{-3}$ |
| 0.0 | 0.1000 | 28.201 | 0.113 | 0.016 | 0.141 | $2.8 \times 10^{-3}$ |
| 0.0 | 0.1585 | 41.113 | 0.164 | 0.023 | 0.206 | $4.3 \times 10^{-3}$ |
| 0.0 | 0.2512 | 58.262 | 0.233 | 0.033 | 0.291 | $6.4 \times 10^{-3}$ |
| 0.0 | 0.3981 | 79.846 | 0.319 | 0.045 | 0.399 | $8.9 \times 10^{-3}$ |
| 0.0 | 0.6310 | 105.855 | 0.423 | 0.059 | 0.529 | 0.012 |
| 0.0 | 1.000 | 135.566 | 0.542 | 0.076 | 0.678 | 0.016 |
| 0.0 | 1.585 | 168.058 | 0.672 | 0.094 | 0.840 | 0.019 |
| 0.0 | 2.512 | 201.883 | 0.808 | 0.113 | 1.009 | 0.023 |
| 0.0 | 3.981 | 235.898 | 0.944 | 0.132 | 1.179 | 0.027 |
| 0.0 | 6.310 | 268.794 | 1.075 | 0.151 | 1.344 | 0.031 |
| 0.0 | 10.00 | 299.722 | 1.199 | 0.168 | 1.499 | 0.035 |
| 0.0 ´ | 15.85 | 328.134 | 1.313 | 0.184 | 1.641 | 0.039 |
| 0.0 | 25.12 | 354.144 | 1.417 | 0.198 | 1.771 | 0.042 |
| 0.0 | 39.81 | 378.885 | 1.516 | 0.212 | 1.894 | 0.045 |
| 0.0 | 63.10 | 404.134 | 1.617 | 0.226 | 2.021 | 0.048 |
| 0.0 | 100.0 | 432.131 | 1.729 | 0.242 | 2.161 | 0.051 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | Standard Uncertainty (Type A) | $u(G",T)$ | $u(G",\Omega)$ | $u(G",M)$ | $u(G",\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 10.0 | 0.03981 | 7.295 | 0.018 | 0.037 | $7.2\times10^{-4}$ | 0.017 | 0.018 |
| 10.0 | 0.06310 | 11.352 | 0.031 | 0.056 | $1.1\times10^{-3}$ | 0.025 | 0.029 |
| 10.0 | 0.1000 | 17.419 | 0.044 | 0.082 | $1.6\times10^{-3}$ | 0.037 | 0.044 |
| 10.0 | 0.1585 | 26.287 | 0.066 | 0.117 | $2.3\times10^{-3}$ | 0.056 | 0.067 |
| 10.0 | 0.2512 | 38.734 | 0.098 | 0.160 | $3.2\times10^{-3}$ | 0.082 | 0.100 |
| 10.0 | 0.3981 | 55.454 | 0.138 | 0.210 | $4.2\times10^{-3}$ | 0.119 | 0.146 |
| 10.0 | 0.6310 | 76.894 | 0.197 | 0.264 | $5.3\times10^{-3}$ | 0.169 | 0.209 |
| 10.0 | 1.000 | 103.161 | 0.278 | 0.315 | $6.4\times10^{-3}$ | 0.234 | 0.290 |
| 10.0 | 1.585 | 133.741 | 0.357 | 0.357 | $7.3\times10^{-3}$ | 0.317 | 0.394 |
| 10.0 | 2.512 | 167.622 | 0.468 | 0.384 | $8.0\times10^{-3}$ | 0.418 | 0.520 |
| 10.0 | 3.981 | 203.424 | 0.568 | 0.391 | $8.3\times10^{-3}$ | 0.538 | 0.670 |
| 10.0 | 6.310 | 240.105 | 0.688 | 0.375 | $8.3\times10^{-3}$ | 0.678 | 0.846 |
| 10.0 | 10.00 | 276.093 | 0.826 | 0.338 | $7.8\times10^{-3}$ | 0.837 | 1.044 |
| 10.0 | 15.85 | 310.338 | 0.970 | 0.287 | $7.0\times10^{-3}$ | 1.013 | 1.263 |
| 10.0 | 25.12 | 342.151 | 1.099 | 0.231 | $6.1\times10^{-3}$ | 1.204 | 1.502 |
| 10.0 | 39.81 | 371.952 | 1.224 | 0.178 | $5.3\times10^{-3}$ | 1.408 | 1.757 |
| 10.0 | 63.10 | 400.674 | 1.369 | 0.141 | $4.8\times10^{-3}$ | 1.623 | 2.026 |
| 10.0 | 100.0 | 429.896 | 1.547 | 0.129 | $4.7\times10^{-3}$ | 1.847 | 2.306 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | $u(G",h)$ | $u(G",R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 10.0 | 0.03981 | 7.295 | 0.029 | $4.1 \times 10^{-3}$ | 0.036 | $1.7 \times 10^{-3}$ |
| 10.0 | 0.06310 | 11.352 | 0.045 | $6.4 \times 10^{-3}$ | 0.057 | $3.2 \times 10^{-3}$ |
| 10.0 | 0.1000 | 17.419 | 0.070 | $9.8 \times 10^{-3}$ | 0.087 | $5.3 \times 10^{-3}$ |
| 10.0 | 0.1585 | 26.287 | 0.105 | 0.015 | 0.131 | $8.6 \times 10^{-3}$ |
| 10.0 | 0.2512 | 38.734 | 0.155 | 0.022 | 0.194 | 0.013 |
| 10.0 | 0.3981 | 55.454 | 0.222 | 0.031 | 0.277 | 0.019 |
| 10.0 | 0.6310 | 76.894 | 0.308 | 0.043 | 0.384 | 0.027 |
| 10.0 | 1.000 | 103.161 | 0.413 | 0.058 | 0.516 | 0.036 |
| 10.0 | 1.585 | 133.741 | 0.535 | 0.075 | 0.669 | 0.047 |
| 10.0 | 2.512 | 167.622 | 0.670 | 0.094 | 0.838 | 0.060 |
| 10.0 | 3.981 | 203.424 | 0.814 | 0.114 | 1.017 | 0.072 |
| 10.0 | 6.310 | 240.105 | 0.960 | 0.134 | 1.201 | 0.086 |
| 10.0 | 10.00 | 276.093 | 1.104 | 0.155 | 1.380 | 0.099 |
| 10.0 | 15.85 | 310.338 | 1.241 | 0.174 | 1.552 | 0.112 |
| 10.0 | 25.12 | 342.151 | 1.369 | 0.192 | 1.711 | 0.123 |
| 10.0 | 39.81 | 371.952 | 1.488 | 0.208 | 1.860 | 0.134 |
| 10.0 | 63.10 | 400.674 | 1.603 | 0.224 | 2.003 | 0.145 |
| 10.0 | 100.0 | 429.896 | 1.720 | 0.241 | 2.149 | 0.156 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | Standard Uncertainty (Type A) | $u(G",T)$ | $u(G",\Omega)$ | $u(G",M)$ | $u(G",\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 20.0 | 0.03981 | 4.605 | 0.011 | 0.021 | $4.6\times10^{-4}$ | 0.011 | 0.012 |
| 20.0 | 0.06310 | 7.259 | 0.017 | 0.032 | $7.1\times10^{-4}$ | 0.017 | 0.018 |
| 20.0 | 0.1000 | 11.277 | 0.029 | 0.049 | $1.1\times10^{-3}$ | 0.025 | 0.028 |
| 20.0 | 0.1585 | 17.376 | 0.041 | 0.073 | $1.6\times10^{-3}$ | 0.037 | 0.044 |
| 20.0 | 0.2512 | 26.304 | 0.063 | 0.103 | $2.3\times10^{-3}$ | 0.056 | 0.067 |
| 20.0 | 0.3981 | 38.913 | 0.086 | 0.142 | $3.2\times10^{-3}$ | 0.082 | 0.100 |
| 20.0 | 0.6310 | 56.012 | 0.133 | 0.187 | $4.2\times10^{-3}$ | 0.120 | 0.147 |
| 20.0 | 1.000 | 78.152 | 0.173 | 0.236 | $5.3\times10^{-3}$ | 0.171 | 0.211 |
| 20.0 | 1.585 | 105.458 | 0.238 | 0.283 | $6.4\times10^{-3}$ | 0.238 | 0.295 |
| 20.0 | 2.512 | 137.487 | 0.307 | 0.323 | $7.4\times10^{-3}$ | 0.323 | 0.401 |
| 20.0 | 3.981 | 173.320 | 0.390 | 0.349 | $8.2\times10^{-3}$ | 0.428 | 0.532 |
| 20.0 | 6.310 | 211.561 | 0.482 | 0.357 | $8.6\times10^{-3}$ | 0.553 | 0.689 |
| 20.0 | 10.00 | 250.993 | 0.577 | 0.344 | $8.5\times10^{-3}$ | 0.700 | 0.873 |
| 20.0 | 15.85 | 290.060 | 0.682 | 0.311 | $8.1\times10^{-3}$ | 0.867 | 1.081 |
| 20.0 | 25.12 | 327.457 | 0.792 | 0.265 | $7.3\times10^{-3}$ | 1.052 | 1.313 |
| 20.0 | 39.81 | 362.955 | 0.909 | 0.212 | $6.4\times10^{-3}$ | 1.254 | 1.565 |
| 20.0 | 63.10 | 396.760 | 1.036 | 0.163 | $5.6\times10^{-3}$ | 1.470 | 1.836 |
| 20.0 | 100.0 | 429.671 | 1.199 | 0.126 | $5.0\times10^{-3}$ | 1.698 | 2.120 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | $u(\mathrm{G}",h)$ | $u(\mathrm{G}",R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 20.0 | 0.03981 | 4.605 | 0.018 | $2.6\times10^{-3}$ | 0.023 | $2.9\times10^{-3}$ |
| 20.0 | 0.06310 | 7.259 | 0.029 | $4.1\times10^{-3}$ | 0.036 | $5.3\times10^{-3}$ |
| 20.0 | 0.1000 | 11.277 | 0.045 | $6.3\times10^{-3}$ | 0.056 | $9.1\times10^{-3}$ |
| 20.0 | 0.1585 | 17.376 | 0.070 | $9.7\times10^{-3}$ | 0.087 | 0.015 |
| 20.0 | 0.2512 | 26.304 | 0.105 | 0.015 | 0.132 | 0.023 |
| 20.0 | 0.3981 | 38.913 | 0.156 | 0.022 | 0.195 | 0.035 |
| 20.0 | 0.6310 | 56.012 | 0.224 | 0.031 | 0.280 | 0.051 |
| 20.0 | 1.000 | 78.152 | 0.313 | 0.044 | 0.391 | 0.071 |
| 20.0 | 1.585 | 105.458 | 0.422 | 0.059 | 0.527 | 0.096 |
| 20.0 | 2.512 | 137.487 | 0.550 | 0.077 | 0.687 | 0.126 |
| 20.0 | 3.981 | 173.320 | 0.693 | 0.097 | 0.867 | 0.160 |
| 20.0 | 6.310 | 211.561 | 0.846 | 0.118 | 1.058 | 0.195 |
| 20.0 | 10.00 | 250.993 | 1.004 | 0.141 | 1.255 | 0.232 |
| 20.0 | 15.85 | 290.060 | 1.160 | 0.162 | 1.450 | 0.269 |
| 20.0 ` | 25.12 | 327.457 | 1.310 | 0.183 | 1.637 | 0.305 |
| 20.0 | 39.81 | 362.955 | 1.452 | 0.203 | 1.815 | 0.339 |
| 20.0 | 63.10 | 396.760 | 1.587 | 0.222 | 1.984 | 0.371 |
| 20.0 | 100.0 | 429.671 | 1.719 | 0.241 | 2.148 | 0.403 |

| Table 7. (continued)  Components of Standard Uncertainty in the Loss Modulus G" | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | Standard Uncertainty (Type A) | $u(G",T)$ | $u(G",\Omega)$ | $u(G",M)$ | $u(G",\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 30.0 | 0.03981 | 3.028 | $9.3\times10^{-3}$ | 0.012 | $3.0\times10^{-4}$ | $8.1\times10^{-3}$ | $7.6\times10^{-3}$ |
| 30.0 | 0.06310 | 4.773 | 0.011 | 0.019 | $4.7\times10^{-4}$ | 0.012 | 0.012 |
| 30.0 | 0.1000 | 7.480 | 0.016 | 0.030 | $7.3\times10^{-4}$ | 0.017 | 0.019 |
| 30.0 | 0.1585 | 11.650 | 0.023 | 0.045 | $1.1\times10^{-3}$ | 0.025 | 0.029 |
| 30.0 | 0.2512 | 17.936 | 0.032 | 0.067 | $1.6\times10^{-3}$ | 0.038 | 0.045 |
| 30.0 | 0.3981 | 27.172 | 0.055 | 0.095 | $2.3\times10^{-3}$ | 0.057 | 0.069 |
| 30.0 | 0.6310 | 40.190 | 0.077 | 0.131 | $3.2\times10^{-3}$ | 0.085 | 0.103 |
| 30.0 | 1.000 | 57.922 | 0.099 | 0.172 | $4.3\times10^{-3}$ | 0.124 | 0.152 |
| 30.0 | 1.585 | 80.873 | 0.133 | 0.217 | $5.5\times10^{-3}$ | 0.176 | 0.218 |
| 30.0 | 2.512 | 109.231 | 0.163 | 0.261 | $6.6\times10^{-3}$ | 0.246 | 0.305 |
| 30.0 | 3.981 | 142.586 | 0.209 | 0.297 | $7.7\times10^{-3}$ | 0.334 | 0.415 |
| 30.0 | 6.310 | 180.008 | 0.249 | 0.321 | $8.4\times10^{-3}$ | 0.443 | 0.552 |
| 30.0 | 10.00 | 219.989 | 0.298 | 0.327 | $8.8\times10^{-3}$ | 0.574 | 0.715 |
| 30.0 | 15.85 | 261.329 | 0.357 | 0.314 | $8.8\times10^{-3}$ | 0.726 | 0.905 |
| 30.0 | 25.12 | 302.326 | 0.424 | 0.284 | $8.3\times10^{-3}$ | 0.900 | 1.122 |
| 30.0 | 39.81 | 342.115 | 0.489 | 0.240 | $7.5\times10^{-3}$ | 1.092 | 1.363 |
| 30.0 | 63.10 | 380.371 | 0.571 | 0.191 | $6.6\times10^{-3}$ | 1.302 | 1.626 |
| 30.0 | 100.0 | 417.237 | 0.675 | 0.144 | $5.7\times10^{-3}$ | 1.527 | 1.906 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | $u(\text{G}",h)$ | $u(\text{G}",R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 30.0 | 0.03981 | 3.028 | 0.012 | $1.7\times10^{-3}$ | 0.015 | $4.7\times10^{-3}$ |
| 30.0 | 0.06310 | 4.773 | 0.019 | $2.7\times10^{-3}$ | 0.024 | $8.5\times10^{-3}$ |
| 30.0 | 0.1000 | 7.480 | 0.030 | $4.2\times10^{-3}$ | 0.037 | 0.014 |
| 30.0 | 0.1585 | 11.650 | 0.047 | $6.5\times10^{-3}$ | 0.058 | 0.024 |
| 30.0 | 0.2512 | 17.936 | 0.072 | 0.010 | 0.090 | 0.037 |
| 30.0 | 0.3981 | 27.172 | 0.109 | 0.015 | 0.136 | 0.058 |
| 30.0 | 0.6310 | 40.190 | 0.161 | 0.023 | 0.201 | 0.086 |
| 30.0 | 1.000 | 57.922 | 0.232 | 0.032 | 0.290 | 0.125 |
| 30.0 | 1.585 | 80.873 | 0.323 | 0.045 | 0.404 | 0.176 |
| 30.0 | 2.512 | 109.231 | 0.437 | 0.061 | 0.546 | 0.238 |
| 30.0 | 3.981 | 142.586 | 0.570 | 0.080 | 0.713 | 0.312 |
| 30.0 | 6.310 | 180.008 | 0.720 | 0.101 | 0.900 | 0.395 |
| 30.0 | 10.00 | 219.989 | 0.880 | 0.123 | 1.100 | 0.484 |
| 30.0 | 15.85 | 261.329 | 1.045 | 0.146 | 1.307 | 0.576 |
| 30.0 | 25.12 | 302.326 | 1.209 | 0.169 | 1.512 | 0.668 |
| 30.0 | 39.81 | 342.115 | 1.368 | 0.192 | 1.711 | 0.758 |
| 30.0 | 63.10 | 380.371 | 1.521 | 0.213 | 1.902 | 0.845 |
| 30.0 | 100.0 | 417.237 | 1.669 | 0.234 | 2.086 | 0.928 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | Standard Uncertainty (Type A) | $u(G",T)$ | $u(G",\Omega)$ | $u(G",M)$ | $u(G",\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 40.0 | 0.03981 | 2.047 | $6.7\times10^{-3}$ | $7.4\times10^{-3}$ | $2.0\times10^{-4}$ | $6.1\times10^{-3}$ | $5.1\times10^{-3}$ |
| 40.0 | 0.06310 | 3.236 | 0.011 | 0.012 | $3.2\times10^{-4}$ | $8.5\times10^{-3}$ | $8.1\times10^{-3}$ |
| 40.0 | 0.1000 | 5.101 | $8.0\times10^{-3}$ | 0.019 | $5.0\times10^{-4}$ | 0.012 | 0.013 |
| 40.0 | 0.1585 | 7.998 | 0.011 | 0.029 | $7.8\times10^{-4}$ | 0.018 | 0.020 |
| 40.0 | 0.2512 | 12.447 | 0.021 | 0.044 | $1.2\times10^{-3}$ | 0.027 | 0.031 |
| 40.0 | 0.3981 | 19.162 | 0.030 | 0.064 | $1.7\times10^{-3}$ | 0.041 | 0.048 |
| 40.0 | 0.6310 | 28.944 | 0.041 | 0.091 | $2.5\times10^{-3}$ | 0.061 | 0.074 |
| 40.0 | 1.000 | 42.757 | 0.061 | 0.125 | $3.4\times10^{-3}$ | 0.090 | 0.110 |
| 40.0 | 1.585 | 61.428 | 0.083 | 0.163 | $4.5\times10^{-3}$ | 0.131 | 0.161 |
| 40.0 | 2.512 | 85.515 | 0.108 | 0.205 | $5.7\times10^{-3}$ | 0.187 | 0.231 |
| 40.0 | 3.981 | 115.104 | 0.138 | 0.244 | $6.9\times10^{-3}$ | 0.260 | 0.322 |
| 40.0 | 6.310 | 149.769 | 0.183 | 0.277 | $7.9\times10^{-3}$ | 0.353 | 0.438 |
| 40.0 | 10.00 | 188.496 | 0.234 | 0.297 | $8.7\times10^{-3}$ | 0.467 | 0.581 |
| 40.0 | 15.85 | 229.749 | 0.290 | 0.301 | $9.1\times10^{-3}$ | 0.602 | 0.750 |
| 40.0 | 25.12 | 272.239 | 0.361 | 0.286 | $9.0\times10^{-3}$ | 0.761 | 0.948 |
| 40.0 | 39.81 | 314.635 | 0.442 | 0.256 | $8.5\times10^{-3}$ | 0.940 | 1.172 |
| 40.0 | 63.10 | 356.105 | 0.532 | 0.214 | $7.7\times10^{-3}$ | 1.139 | 1.421 |
| 40.0 | 100.0 | 396.285 | 0.670 | 0.167 | $6.7\times10^{-3}$ | 1.355 | 1.691 |

| Temperature | Frequency | Loss Modulus G" | $u(G",h)$ | $u(G",R)$ | Tilt | Evaporation |
|---|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 40.0 | 0.03981 | 2.047 | $8.2\times10^{-3}$ | $1.2\times10^{-3}$ | 0.010 | $7.2\times10^{-3}$ |
| 40.0 | 0.06310 | 3.236 | 0.013 | $1.8\times10^{-3}$ | 0.016 | 0.013 |
| 40.0 | 0.1000 | 5.101 | 0.020 | $2.9\times10^{-3}$ | 0.026 | 0.022 |
| 40.0 | 0.1585 | 7.998 | 0.032 | $4.5\times10^{-3}$ | 0.040 | 0.036 |
| 40.0 | 0.2512 | 12.447 | 0.050 | $7.0\times10^{-3}$ | 0.062 | 0.058 |
| 40.0 | 0.3981 | 19.162 | 0.077 | 0.011 | 0.096 | 0.091 |
| 40.0 | 0.6310 | 28.944 | 0.116 | 0.016 | 0.145 | 0.140 |
| 40.0 | 1.000 | 42.757 | 0.171 | 0.024 | 0.214 | 0.208 |
| 40.0 | 1.585 | 61.428 | 0.246 | 0.034 | 0.307 | 0.300 |
| 40.0 | 2.512 | 85.515 | 0.342 | 0.048 | 0.428 | 0.419 |
| 40.0 | 3.981 | 115.104 | 0.460 | 0.064 | 0.576 | 0.566 |
| 40.0 | 6.310 | 149.769 | 0.599 | 0.084 | 0.749 | 0.738 |
| 40.0 | 10.00 | 188.496 | 0.754 | 0.106 | 0.942 | 0.932 |
| 40.0 | 15.85 | 229.749 | 0.919 | 0.129 | 1.149 | 1.139 |
| 40.0 | 25.12 | 272.239 | 1.089 | 0.152 | 1.361 | 1.352 |
| 40.0 | 39.81 | 314.635 | 1.259 | 0.176 | 1.573 | 1.567 |
| 40.0 | 63.10 | 356.105 | 1.424 | 0.199 | 1.781 | 1.777 |
| 40.0 | 100.0 | 396.285 | 1.585 | 0.222 | 1.981 | 1.981 |

Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G"

| Table 7. (continued)  Components of Standard Uncertainty in the Loss Modulus G" | | | | | | | |
|:---:|:---:|:---:|:---:|:---:|:---:|:---:|:---:|
| Temperature | Frequency | Loss Modulus G" | Standard Uncertainty (Type A) | $u(G",T)$ | $u(G",\Omega)$ | $u(G",M)$ | $u(G",\Phi)$ |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 50.0 | 0.03981 | 1.457 | $4.8\times10^{-3}$ | $4.6\times10^{-3}$ | $1.4\times10^{-4}$ | $5.0\times10^{-3}$ | $3.6\times10^{-3}$ |
| 50.0 | 0.06310 | 2.316 | $7.9\times10^{-3}$ | $7.5\times10^{-3}$ | $2.2\times10^{-4}$ | $6.7\times10^{-3}$ | $5.8\times10^{-3}$ |
| 50.0 | 0.1000 | 3.630 | $8.7\times10^{-3}$ | 0.012 | $3.6\times10^{-4}$ | $9.3\times10^{-3}$ | $9.1\times10^{-3}$ |
| 50.0 | 0.1585 | 5.726 | 0.014 | 0.019 | $5.6\times10^{-4}$ | 0.013 | 0.014 |
| 50.0 | 0.2512 | 8.980 | 0.018 | 0.029 | $8.6\times10^{-4}$ | 0.020 | 0.022 |
| 50.0 | 0.3981 | 13.949 | 0.028 | 0.044 | $1.3\times10^{-3}$ | 0.030 | 0.035 |
| 50.0 | 0.6310 | 21.392 | 0.040 | 0.064 | $1.9\times10^{-3}$ | 0.045 | 0.054 |
| 50.0 | 1.000 | 32.210 | 0.057 | 0.090 | $2.7\times10^{-3}$ | 0.068 | 0.082 |
| 50.0 | 1.585 | 47.339 | 0.081 | 0.122 | $3.7\times10^{-3}$ | 0.100 | 0.122 |
| 50.0 | 2.512 | 67.610 | 0.108 | 0.159 | $4.8\times10^{-3}$ | 0.145 | 0.179 |
| 50.0 | 3.981 | 93.473 | 0.140 | 0.197 | $6.1\times10^{-3}$ | 0.205 | 0.254 |
| 50.0 | 6.310 | 125.025 | 0.177 | 0.233 | $7.3\times10^{-3}$ | 0.284 | 0.353 |
| 50.0 | 10.00 | 161.601 | 0.223 | 0.261 | $8.3\times10^{-3}$ | 0.384 | 0.477 |
| 50.0 | 15.85 | 201.983 | 0.270 | 0.276 | $9.0\times10^{-3}$ | 0.505 | 0.629 |
| 50.0 | 25.12 | 244.960 | 0.326 | 0.276 | $9.3\times10^{-3}$ | 0.649 | 0.809 |
| 50.0 | 39.81 | 289.041 | 0.389 | 0.260 | $9.1\times10^{-3}$ | 0.816 | 1.018 |
| 50.0 | 63.10 | 333.071 | 0.465 | 0.228 | $8.6\times10^{-3}$ | 1.004 | 1.253 |
| 50.0 | 100.0 | 376.368 | 0.572 | 0.187 | $7.7\times10^{-3}$ | 1.212 | 1.512 |

| Table 7. (continued) Components of Standard Uncertainty in the Loss Modulus G" | | | | | | |
|---|---|---|---|---|---|---|
| Temperature | Frequency | Loss Modulus G" | $u(G",h)$ | $u(G",R)$ | Tilt | Evaporation |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa |
| | | | | | | |
| 50.0 | 0.03981 | 1.457 | $5.8\times10^{-3}$ | $8.2\times10^{-4}$ | $7.3\times10^{-3}$ | 0.011 |
| 50.0 | 0.06310 | 2.316 | $9.3\times10^{-3}$ | $1.3\times10^{-3}$ | 0.012 | 0.020 |
| 50.0 | 0.1000 | 3.630 | 0.015 | $2.0\times10^{-3}$ | 0.018 | 0.034 |
| 50.0 | 0.1585 | 5.726 | 0.023 | $3.2\times10^{-3}$ | 0.029 | 0.056 |
| 50.0 | 0.2512 | 8.980 | 0.036 | $5.0\times10^{-3}$ | 0.045 | 0.091 |
| 50.0 | 0.3981 | 13.949 | 0.056 | $7.8\times10^{-3}$ | 0.070 | 0.143 |
| 50.0 | 0.6310 | 21.392 | 0.086 | 0.012 | 0.107 | 0.223 |
| 50.0 | 1.000 | 32.210 | 0.129 | 0.018 | 0.161 | 0.338 |
| 50.0 | 1.585 | 47.339 | 0.189 | 0.027 | 0.237 | 0.499 |
| 50.0 | 2.512 | 67.610 | 0.270 | 0.038 | 0.338 | 0.715 |
| 50.0 | 3.981 | 93.473 | 0.374 | 0.052 | 0.467 | 0.991 |
| 50.0 | 6.310 | 125.025 | 0.500 | 0.070 | 0.625 | 1.328 |
| 50.0 | 10.00 | 161.601 | 0.646 | 0.090 | 0.808 | 1.721 |
| 50.0 | 15.85 | 201.983 | 0.808 | 0.113 | 1.010 | 2.157 |
| 50.0 ´ | 25.12 | 244.960 | 0.980 | 0.137 | 1.225 | 2.621 |
| 50.0 | 39.81 | 289.041 | 1.156 | 0.162 | 1.445 | 3.101 |
| 50.0 | 63.10 | 333.071 | 1.332 | 0.187 | 1.665 | 3.580 |
| 50.0 | 100.0 | 376.368 | 1.505 | 0.211 | 1.882 | 4.052 |

## 5. Conclusion

The measured viscosity and first normal stress difference data are given in Table 8, along with the combined standard uncertainties and the models fit to the data using equations (6) through (8).

The measured storage modulus G' and loss modulus G" are given in Table 9, along with the combined standard uncertainties and models fit to the data using equations (39) and (40).

| Table 8. Viscosity and First Normal Stress Difference ($N_1$) with Combined Standard Uncertainties and Models Fit to the Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temperature | Shear Rate | Viscosity | Combined Standard Uncertainty | Model Fit to Viscosity | $N_1$ | Combined Standard Uncertainty | Model Fit to $N_1$ |
| °C | s⁻¹ | Pa·s | Pa·s | Pa·s | Pa | Pa | Pa |
| | | | | | | | |
| 0.0 | 0.001000 | 382.90 | 5.97 | 393.97 | | | |
| 0.0 | 0.001585 | 380.05 | 5.27 | 393.30 | | | |
| 0.0 | 0.002512 | 382.38 | 4.93 | 392.33 | | | |
| 0.0 | 0.003981 | 382.90 | 4.74 | 390.93 | | | |
| 0.0 | 0.006310 | 383.97 | 4.62 | 388.91 | | | |
| 0.0 | 0.01000 | 383.08 | 4.52 | 386.04 | | | |
| 0.0 | 0.01585 | 382.90 | 4.42 | 381.95 | | | |
| 0.0 | 0.02512 | 379.83 | 4.31 | 376.18 | | | |
| 0.0 | 0.03981 | 375.06 | 4.15 | 368.12 | | | |
| 0.0 | 0.06310 | 365.51 | 3.95 | 357.03 | | | |
| 0.0 | 0.1000 | 350.01 | 3.66 | 342.11 | | | |
| 0.0 | 0.1585 | 328.43 | 3.30 | 322.58 | 16.30 | 5.49 | 24.88 |
| 0.0 | 0.2512 | 300.79 | 2.87 | 297.94 | 43.78 | 4.84 | 46.98 |
| 0.0 | 0.3981 | 268.52 | 2.39 | 268.25 | 91.85 | 5.64 | 83.99 |
| 0.0 | 0.6310 | 232.98 | 1.90 | 234.42 | 149.06 | 6.93 | 143.09 |
| 0.0 | 1.000 | 196.53 | 1.44 | 198.21 | 257.45 | 7.41 | 234.03 |
| 0.0 | 1.585 | 161.10 | 1.04 | 161.96 | 372.45 | 7.41 | 369.92 |
| 0.0 | 2.512 | 128.41 | 0.73 | 128.04 | 572.64 | 8.43 | 567.68 |
| 0.0 | 3.981 | 99.45 | 0.49 | 98.23 | 845.17 | 10.41 | 847.95 |
| 0.0 | 6.310 | 75.07 | 0.33 | 73.46 | 1218.81 | 14.41 | 1234.06 |
| 0.0 | 10.00 | 55.59 | 0.22 | 53.81 | 1717.43 | 17.68 | 1750.11 |
| 0.0 | 15.85 | 40.26 | 0.1● | 38.78 | 2362.56 | 22.19 | 2418.51 |
| 0.0 | 25.12 | 28.67 | 0.10 | 27.60 | 3195.73 | 27.63 | 3258.06 |
| 0.0 | 39.81 | 20.23 | 0.08 | 19.48 | 4251.21 | 34.35 | 4283.30 |
| 0.0 | 63.10 | 13.88 | 0.08 | 13.65 | 5518.63 | 43.41 | 5505.89 |
| 0.0 | 100.0 | 9.08 | 0.06 | 9.53 | 7125.98 | 53.74 | 6937.36 |

| Temperature | Shear Rate | Viscosity | Combined Standard Uncertainty | Model Fit to Viscosity | $N_1$ | Combined Standard Uncertainty | Model Fit to $N_1$ |
|---|---|---|---|---|---|---|---|
| °C | $s^{-1}$ | Pa·s | Pa·s | Pa·s | Pa | Pa | Pa |
| | | | | | | | |
| 25.0 | 0.001000 | 97.93 | 3.52 | 100.09 | | | |
| 25.0 | 0.001585 | 98.05 | 2.46 | 100.03 | | | |
| 25.0 | 0.002512 | 98.25 | 1.86 | 99.96 | | | |
| 25.0 | 0.003981 | 97.93 | 1.45 | 99.84 | | | |
| 25.0 | 0.006310 | 98.35 | 1.26 | 99.68 | | | |
| 25.0 | 0.01000 | 98.06 | 1.17 | 99.45 | | | |
| 25.0 | 0.01585 | 98.72 | 1.12 | 99.11 | | | |
| 25.0 | 0.02512 | 98.78 | 1.09 | 98.63 | | | |
| 25.0 | 0.03981 | 98.61 | 1.07 | 97.93 | | | |
| 25.0 | 0.06310 | 98.37 | 1.05 | 96.95 | | | |
| 25.0 | 0.1000 | 97.52 | 1.02 | 95.55 | | | |
| 25.0 | 0.1585 | 96.13 | 0.99 | 93.60 | 2.35 | 0.95 | 2.55 |
| 25.0 | 0.2512 | 93.71 | 0.94 | 90.91 | 5.51 | 0.89 | 5.69 |
| 25.0 | 0.3981 | 90.00 | 0.88 | 87.28 | 12.92 | 0.94 | 12.10 |
| 25.0 | 0.6310 | 84.59 | 0.80 | 82.50 | 26.54 | 1.05 | 24.39 |
| 25.0 | 1.000 | 77.61 | 0.70 | 76.44 | 50.05 | 1.24 | 46.41 |
| 25.0 | 1.585 | 69.16 | 0.58 | 69.09 | 87.62 | 1.64 | 83.57 |
| 25.0 | 2.512 | 59.98 | 0.47 | 60.64 | 148.18 | 2.31 | 143.27 |
| 25.0 | 3.981 | 50.56 | 0.36 | 51.51 | 236.76 | 3.72 | 235.55 |
| 25.0 | 6.310 | 41.44 | 0.27 | 42.29 | 377.26 | 6.07 | 373.98 |
| 25.0 | 10.00 | 33.04 | 0.19 | 33.58 | 585.07 | 8.10 | 576.17 |
| 25.0 | 15.85 | 25.61 | 0.14 | 25.87 | 880.00 | 10.41 | 863.78 |
| 25.0 | 25.12 | 19.36 | 0.10 | 19.41 | 1280.30 | 13.63 | 1261.63 |
| 25.0 | 39.81 | 14.26 | 0.08 | 14.25 | 1800.04 | 17.78 | 1795.66 |
| 25.0 | 63.10 | 10.22 | 0.07 | 10.29 | 2462.22 | 25.58 | 2490.38 |
| 25.0 | 100.0 | 7.22 | 0.06 | 7.34 | 3318.59 | 31.68 | 3366.61 |

Table 8. (continued)  Viscosity and First Normal Stress Difference ($N_1$) with Combined Standard Uncertainties and Models Fit to the Data

| Temperature | Shear Rate | Viscosity | Combined Standard Uncertainty | Model Fit to Viscosity | $N_1$ | Combined Standard Uncertainty | Model Fit to $N_1$ |
|---|---|---|---|---|---|---|---|
| °C | $s^{-1}$ | Pa·s | Pa·s | Pa·s | Pa | Pa | Pa |
| | | | | | | | |
| 50.0 | 0.001000 | 36.74 | 3.25 | 38.07 | | | |
| 50.0 | 0.001585 | 37.20 | 2.11 | 38.06 | | | |
| 50.0 | 0.002512 | 37.62 | 1.40 | 38.05 | | | |
| 50.0 | 0.003981 | 37.30 | 0.98 | 38.03 | | | |
| 50.0 | 0.006310 | 37.72 | 0.74 | 38.00 | | | |
| 50.0 | 0.01000 | 37.73 | 0.63 | 37.97 | | | |
| 50.0 | 0.01585 | 37.50 | 0.57 | 37.91 | | | |
| 50.0 | 0.02512 | 37.55 | 0.55 | 37.83 | | | |
| 50.0 | 0.03981 | 37.83 | 0.54 | 37.71 | | | |
| 50.0 | 0.06310 | 37.80 | 0.54 | 37.54 | | | |
| 50.0 | 0.1000 | 37.76 | 0.55 | 37.30 | | | |
| 50.0 | 0.1585 | 37.66 | 0.55 | 36.96 | | | |
| 50.0 | 0.2512 | 37.39 | 0.55 | 36.47 | | | |
| 50.0 | 0.3981 | 36.93 | 0.55 | 35.78 | 2.38 | 0.84 | 2.23 |
| 50.0 | 0.6310 | 36.10 | 0.54 | 34.84 | 5.08 | 0.86 | 5.02 |
| 50.0 | 1.000 | 34.77 | 0.52 | 33.55 | 10.73 | 0.91 | 10.81 |
| 50.0 | 1.585 | 32.79 | 0.50 | 31.84 | 21.47 | 1.00 | 22.09 |
| 50.0 | 2.512 | 30.20 | 0.46 | 29.66 | 41.05 | 1.33 | 42.62 |
| 50.0 | 3.981 | 27.05 | 0.42 | 26.98 | 68.85 | 2.14 | 77.74 |
| 50.0 | 6.310 | 23.54 | 0.37 | 23.86 | 114.48 | 3.82 | 134.77 |
| 50.0 | 10.00 | 19.95 | 0.32 | 20.43 | 202.50 | 5.71 | 223.69 |
| 50.0 | 15.85 | 16.41 | 0.27 | 16.92 | 344.80 | 7.85 | 357.98 |
| 50.0 | 25.12 | 13.14 | 0.22 | 13.54 | 557.75 | 11.34 | 555.37 |
| 50.0 | 39.81 | 10.24 | 0.17 | 10.50 | 860.20 | 16.37 | 837.99 |
| 50.0 | 63.10 | 7.75 | 0.13 | 7.93 | 1269.10 | 23.53 | 1231.67 |
| 50.0 | 100.0 | 5.72 | 0.10 | 5.85 | 1796.79 | 33.56 | 1764.04 |

Table 8. (continued)  Viscosity and First Normal Stress Difference ($N_1$) with Combined Standard Uncertainties and Models Fit to the Data

| Table 9. Storage Modulus G' and Loss Modulus G" with the Combined Standard Uncertainties and Models Fit to the Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper- ature | Frequency | Storage Modulus G' | Combined Standard Uncertainty in G' | Model Fit to G' | Loss Modulus G" | Combined Standard Uncertainty in G" | Model Fit to G" |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 0.0 | 0.02512 | 0.605 | 0.029 | 0.565 | 7.976 | 0.076 | 8.064 |
| 0.0 | 0.03981 | 1.385 | 0.045 | 1.305 | 12.346 | 0.116 | 12.519 |
| 0.0 | 0.06310 | 2.973 | 0.071 | 2.845 | 18.852 | 0.175 | 19.124 |
| 0.0 | 0.1000 | 6.067 | 0.114 | 5.849 | 28.201 | 0.256 | 28.609 |
| 0.0 | 0.1585 | 11.699 | 0.184 | 11.344 | 41.113 | 0.366 | 41.753 |
| 0.0 | 0.2512 | 21.207 | 0.289 | 20.762 | 58.262 | 0.510 | 59.264 |
| 0.0 | 0.3981 | 36.214 | 0.440 | 35.897 | 79.846 | 0.690 | 81.618 |
| 0.0 | 0.6310 | 58.511 | 0.652 | 58.727 | 105.855 | 0.912 | 108.890 |
| 0.0 | 1.000 | 89.636 | 0.929 | 91.103 | 135.566 | 1.150 | 140.620 |
| 0.0 | 1.585 | 130.948 | 1.270 | 134.379 | 168.058 | 1.431 | 175.769 |
| 0.0 | 2.512 | 182.959 | 1.690 | 189.081 | 201.883 | 1.727 | 212.800 |
| 0.0 | 3.981 | 246.085 | 2.180 | 254.775 | 235.898 | 2.045 | 249.910 |
| 0.0 | 6.310 | 319.791 | 2.752 | 330.190 | 268.794 | 2.381 | 285.327 |
| 0.0 | 10.00 | 403.264 | 3.381 | 413.644 | 299.722 | 2.718 | 317.654 |
| 0.0 | 15.85 | 495.279 | 4.070 | 503.665 | 328.134 | 3.065 | 346.137 |
| 0.0 | 25.12 | 593.784 | 4.832 | 599.721 | 354.144 | 3.417 | 370.841 |
| 0.0 | 39.81 | 697.210 | 5.646 | 702.968 | 378.885 | 3.778 | 392.707 |
| 0.0 | 63.10 | 804.155 | 6.490 | 817.012 | 404.134 | 4.163 | 413.539 |
| 0.0 | 100.0 | 912.794 | 7.392 | 948.855 | 432.131 | 4.589 | 436.001 |

| Table 9. (continued) Storage Modulus G' and Loss Modulus G" with the Combined Standard Uncertainties and Models Fit to the Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper-ature | Frequency | Storage Modulus G' | Combined Standard Uncertainty in G' | Model Fit to G' | Loss Modulus G" | Combined Standard Uncertainty in G" | Model Fit to G" |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 10.0 | 0.03981 | 0.486 | 0.026 | 0.469 | 7.295 | 0.067 | 7.438 |
| 10.0 | 0.06310 | 1.099 | 0.041 | 1.099 | 11.352 | 0.104 | 11.585 |
| 10.0 | 0.1000 | 2.438 | 0.064 | 2.430 | 17.419 | 0.157 | 17.776 |
| 10.0 | 0.1585 | 5.076 | 0.102 | 5.070 | 26.287 | 0.233 | 26.741 |
| 10.0 | 0.2512 | 10.023 | 0.164 | 9.976 | 38.734 | 0.338 | 39.279 |
| 10.0 | 0.3981 | 18.531 | 0.258 | 18.520 | 55.454 | 0.476 | 56.151 |
| 10.0 | 0.6310 | 32.329 | 0.399 | 32.471 | 76.894 | 0.652 | 77.925 |
| 10.0 | 1.000 | 53.217 | 0.598 | 53.844 | 103.161 | 0.870 | 104.795 |
| 10.0 | 1.585 | 83.154 | 0.861 | 84.616 | 133.741 | 1.119 | 136.432 |
| 10.0 | 2.512 | 123.313 | 1.200 | 126.341 | 167.622 | 1.406 | 171.910 |
| 10.0 | 3.981 | 174.767 | 1.605 | 179.799 | 203.424 | 1.712 | 209.757 |
| 10.0 | 6.310 | 238.200 | 2.095 | 244.784 | 240.105 | 2.044 | 248.155 |
| 10.0 | 10.00 | 313.204 | 2.668 | 320.173 | 276.093 | 2.397 | 285.239 |
| 10.0 | 15.85 | 398.816 | 3.325 | 404.283 | 310.338 | 2.763 | 319.442 |
| 10.0 | 25.12 | 493.868 | 4.045 | 495.473 | 342.151 | 3.133 | 349.801 |
| 10.0 | 39.81 | 596.352 | 4.831 | 592.887 | 371.952 | 3.512 | 376.167 |
| 10.0 | 63.10 | 704.450 | 5.670 | 697.213 | 400.674 | 3.910 | 399.285 |
| 10.0 | 100.0 | 816.167 | 6.549 | 811.461 | 429.896 | 4.336 | 420.795 |

| Table 9. (continued) Storage Modulus G' and Loss Modulus G" with the Combined Standard Uncertainties and Models Fit to the Data | | | | | | | |
|---|---|---|---|---|---|---|---|
| Temper-ature | Frequency | Storage Modulus G' | Combined Standard Uncertainty in G' | Model Fit to G' | Loss Modulus G" | Combined Standard Uncertainty in G" | Model Fit to G" |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 20.0 | 0.03981 | 0.182 | 0.017 | 0.175 | 4.605 | 0.041 | 4.622 |
| 20.0 | 0.06310 | 0.430 | 0.027 | 0.437 | 7.259 | 0.064 | 7.272 |
| 20.0 | 0.1000 | 1.023 | 0.040 | 1.029 | 11.277 | 0.100 | 11.341 |
| 20.0 | 0.1585 | 2.275 | 0.063 | 2.291 | 17.376 | 0.151 | 17.437 |
| 20.0 | 0.2512 | 4.809 | 0.100 | 4.812 | 26.304 | 0.227 | 26.296 |
| 20.0 | 0.3981 | 9.571 | 0.160 | 9.530 | 38.913 | 0.329 | 38.738 |
| 20.0 | 0.6310 | 17.918 | 0.252 | 17.811 | 56.012 | 0.470 | 55.557 |
| 20.0 | 1.000 | 31.620 | 0.389 | 31.430 | 78.152 | 0.646 | 77.370 |
| 20.0 | 1.585 | 52.610 | 0.584 | 52.447 | 105.458 | 0.865 | 104.431 |
| 20.0 | 2.512 | 82.735 | 0.845 | 82.918 | 137.487 | 1.123 | 136.466 |
| 20.0 | 3.981 | 123.780 | 1.185 | 124.513 | 173.320 | 1.417 | 172.594 |
| 20.0 | 6.310 | 176.784 | 1.607 | 178.141 | 211.561 | 1.740 | 211.357 |
| 20.0 | 10.00 | 242.540 | 2.107 | 243.709 | 250.993 | 2.088 | 250.910 |
| 20.0 | 15.85 | 320.640 | 2.693 | 320.155 | 290.060 | 2.456 | 289.320 |
| 20.0 | 25.12 | 410.368 | 3.364 | 405.783 | 327.457 | 2.837 | 324.922 |
| 20.0 | 39.81 | 510.091 | 4.112 | 498.867 | 362.955 | 3.233 | 356.644 |
| 20.0 | 63.10 | 617.780 | 4.919 | 598.391 | 396.760 | 3.643 | 384.228 |
| 20.0 | 100.0 | 731.238 | 5.776 | 704.841 | 429.671 | 4.077 | 408.333 |

70

| Temper-ature | Frequency | Storage Modulus G' | Combined Standard Uncertainty in G' | Model Fit to G' | Loss Modulus G" | Combined Standard Uncertainty in G" | Model Fit to G" |
|---|---|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 30.0 | 0.03981 | 0.077 | 0.011 | 0.068 | 3.028 | 0.028 | 3.011 |
| 30.0 | 0.06310 | 0.178 | 0.018 | 0.180 | 4.773 | 0.042 | 4.748 |
| 30.0 | 0.1000 | 0.440 | 0.027 | 0.448 | 7.480 | 0.066 | 7.471 |
| 30.0 | 0.1585 | 1.039 | 0.041 | 1.057 | 11.650 | 0.101 | 11.652 |
| 30.0 | 0.2512 | 2.323 | 0.065 | 2.353 | 17.936 | 0.154 | 17.914 |
| 30.0 | 0.3981 | 4.899 | 0.103 | 4.942 | 27.172 | 0.232 | 27.017 |
| 30.0 | 0.6310 | 9.776 | 0.164 | 9.789 | 40.190 | 0.339 | 39.801 |
| 30.0 | 1.000 | 18.376 | 0.259 | 18.295 | 57.922 | 0.482 | 57.084 |
| 30.0 | 1.585 | 32.459 | 0.401 | 32.287 | 80.873 | 0.667 | 79.500 |
| 30.0 | 2.512 | 54.041 | 0.601 | 53.879 | 109.231 | 0.893 | 107.309 |
| 30.0 | 3.981 | 85.240 | 0.872 | 85.188 | 142.586 | 1.163 | 140.234 |
| 30.0 | 6.310 | 127.700 | 1.228 | 127.930 | 180.008 | 1.470 | 177.366 |
| 30.0 | 10.00 | 182.644 | 1.667 | 183.040 | 219.989 | 1.808 | 217.209 |
| 30.0 | 15.85 | 250.728 | 2.198 | 250.425 | 261.329 | 2.174 | 257.867 |
| 30.0 | 25.12 | 331.736 | 2.820 | 328.992 | 302.326 | 2.559 | 297.352 |
| 30.0 | 39.81 | 424.451 | 3.521 | 417.002 | 342.115 | 2.959 | 333.953 |
| 30.0 | 63.10 | 527.345 | 4.299 | 512.676 | 380.371 | 3.375 | 366.566 |
| 30.0 | 100.0 | 638.186 | 5.139 | 614.971 | 417.237 | 3.807 | 394.925 |

Table 9. (continued) Storage Modulus G' and Loss Modulus G" with the Combined Standard Uncertainties and Models Fit to the Data

| Temper-ature | Frequency | Storage Modulus G' | Combined Standard Uncertainty in G' | Model Fit to G' | Loss Modulus G" | Combined Standard Uncertainty in G" | Model Fit to G" |
|---|---|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
|  |  |  |  |  |  |  |  |
| 40.0 | 0.03981 | 0.030 | 0.0094 | 0.028 | 2.047 | 0.020 | 2.056 |
| 40.0 | 0.06310 | 0.062 | 0.013 | 0.077 | 3.236 | 0.032 | 3.229 |
| 40.0 | 0.1000 | 0.200 | 0.019 | 0.202 | 5.101 | 0.048 | 5.092 |
| 40.0 | 0.1585 | 0.478 | 0.028 | 0.501 | 7.998 | 0.075 | 8.007 |
| 40.0 | 0.2512 | 1.142 | 0.044 | 1.174 | 12.447 | 0.118 | 12.474 |
| 40.0 | 0.3981 | 2.562 | 0.070 | 2.600 | 19.162 | 0.180 | 19.147 |
| 40.0 | 0.6310 | 5.391 | 0.111 | 5.429 | 28.944 | 0.271 | 28.815 |
| 40.0 | 1.000 | 10.695 | 0.179 | 10.695 | 42.757 | 0.398 | 42.348 |
| 40.0 | 1.585 | 19.966 | 0.285 | 19.879 | 61.428 | 0.568 | 60.574 |
| 40.0 | 2.512 | 35.035 | 0.445 | 34.894 | 85.515 | 0.787 | 84.114 |
| 40.0 | 3.981 | 58.033 | 0.676 | 57.927 | 115.104 | 1.057 | 113.191 |
| 40.0 | 6.310 | 91.010 | 0.992 | 91.127 | 149.769 | 1.378 | 147.460 |
| 40.0 | 10.00 | 135.717 | 1.407 | 136.200 | 188.496 | 1.742 | 185.926 |
| 40.0 | 15.85 | 193.163 | 1.929 | 194.010 | 229.749 | 2.140 | 227.005 |
| 40.0 | 25.12 | 264.093 | 2.562 | 264.356 | 272.239 | 2.565 | 268.722 |
| 40.0 | 39.81 | 347.741 | 3.302 | 346.035 | 314.635 | 3.011 | 309.050 |
| 40.0 | 63.10 | 443.102 | 4.141 | 437.224 | 356.105 | 3.470 | 346.277 |
| 40.0 | 100.0 | 548.169 | 5.067 | 536.136 | 396.285 | 3.948 | 379.343 |

Table 9. (continued)  Storage Modulus G' and Loss Modulus G" with the Combined Standard Uncertainties and Models Fit to the Data

| Temper-ature | Frequency | Storage Modulus G' | Combined Standard Uncertainty in G' | Model Fit to G' | Loss Modulus G" | Combined Standard Uncertainty in G" | Model Fit to G" |
|---|---|---|---|---|---|---|---|
| | | | Table 9. (continued)  Storage Modulus G' and Loss Modulus G"<br>with the Combined Standard Uncertainties and Models Fit to the Data | | | | |
| °C | rad/s | Pa | Pa | Pa | Pa | Pa | Pa |
| | | | | | | | |
| 50.0 | 0.03981 | 0.013 | 0.0083 | 0.012 | 1.457 | 0.017 | 1.468 |
| 50.0 | 0.06310 | 0.034 | 0.011 | 0.035 | 2.316 | 0.029 | 2.284 |
| 50.0 | 0.1000 | 0.088 | 0.014 | 0.094 | 3.630 | 0.046 | 3.592 |
| 50.0 | 0.1585 | 0.241 | 0.020 | 0.245 | 5.726 | 0.074 | 5.665 |
| 50.0 | 0.2512 | 0.592 | 0.032 | 0.601 | 8.980 | 0.117 | 8.897 |
| 50.0 | 0.3981 | 1.375 | 0.051 | 1.394 | 13.949 | 0.183 | 13.830 |
| 50.0 | 0.6310 | 3.058 | 0.083 | 3.055 | 21.392 | 0.281 | 21.160 |
| 50.0 | 1.000 | 6.385 | 0.139 | 6.313 | 32.210 | 0.424 | 31.720 |
| 50.0 | 1.585 | 12.509 | 0.232 | 12.308 | 47.339 | 0.623 | 46.402 |
| 50.0 | 2.512 | 23.028 | 0.385 | 22.641 | 67.610 | 0.888 | 66.032 |
| 50.0 | 3.981 | 39.969 | 0.622 | 39.343 | 93.473 | 1.228 | 91.190 |
| 50.0 | 6.310 | 65.434 | 0.968 | 64.677 | 125.025 | 1.643 | 122.010 |
| 50.0 | 10.00 | 101.548 | 1.451 | 100.798 | 161.601 | 2.129 | 158.023 |
| 50.0 | 15.85 | 149.841 | 2.091 | 149.329 | 201.983 | 2.671 | 198.091 |
| 50.0 | 25.12 | 211.567 | 2.901 | 210.969 | 244.960 | 3.257 | 240.496 |
| 50.0 | 39.81 | 286.640 | 3.882 | 285.316 | 289.041 | 3.871 | 283.179 |
| 50.0 | 63.10 | 374.506 | 5.029 | 370.982 | 333.071 | 4.499 | 324.088 |
| 50.0 | 100.0 | 473.567 | 6.320 | 466.051 | 376.368 | 5.137 | 361.564 |

## 6. References

1. K. Walters, *Rheometry*, Chapman and Hall, London, 1975.
2. W.R. Schowalter, *Mechanics of Non-Newtonian Fluids*, Pergamon Press, Oxford, 1978.
3. R.I. Tanner, *Engineering Rheology*, Clarendon Press, Oxford, 1985.
4. R.B. Bird, R.C. Armstrong and O. Hassager, *Dynamics of Polymeric Liquids, Vol. 1, Fluid Mechanics*, 2nd Edition, John Wiley and Sons, New York, 1987.
5. R.B. Bird, C.F. Curtiss, R.C. Armstrong and O. Hassager, *Dynamics of Polymeric Liquids, Volume 2, Kinetic Theory*, 2nd Edition, John Wiley and Sons, New York, 1987.
6. C.W. Macosko, *Rheology - Principles, Measurements and Applications*, Wiley-VCH, New York, 1994.
7. L.J. Zapas, and J.C. Phillips, *Certificate for Standard Reference Material 1490, Polyisobutylene Solution in Cetane (Viscosity and First Normal Stress Difference)*, National Bureau of Standards, Office of Standard Reference Materials, 1977.
8. *CRC Handbook of Chemistry and Physics*, 81$^{st}$ Edition, CRC Press, Boca Raton, 2000.
9. L.J. Zapas and J.C. Phillips, "Simple Shearing Flows in Polyisobutylene Solutions," *Journal of Research of the National Bureau of Standards - A. Physics and Chemistry*, Volume 75A, pp. 33-40, 1971.
10. L.J. Zapas and J.C. Phillips, "Nonlinear Behavior of Polyisobutylene Solutions as a Function of Concentration," *Journal of Rheology*, Volume 25, pp. 405-420, 1981.
11. K. Khalil, A. Tougui and D. Sigli, "Relation between some rheological properties of polyisobutylene solutions and their mode of preparation," *Journal of Non-Newtonian Fluid Mechanics*, Volume 52, pp. 375-386, 1994.
12. C.R. Schultheisz and G.B. McKenna, "A Nonlinear Fluid Standard Reference Material: Progress Report," *Proceedings of SPE ANTEC*, Volume 1, Processing, pp. 1125-1129, 1999.
13. C.R. Schultheisz and G.B. McKenna, "Standard Reference Materials: Non-Newtonian Fluids for Rheological Measurements," *Proceedings of SPE ANTEC*, Volume 1, Processing, pp. 1042-1046, 2000.
14. *Merck Index*, Twelfth Edition, Merck and Company, Rahway, New Jersey, 1996.
15. *Webster's Third New International Dictionary of the English Language*, Merriam-Webster, Inc., Springfield, Massachusetts, 1981.
16. Advanced Rheometric Expansion System ARES Instrument Manual, Rheometric Scientific, 1997.
17. J.V. Nicholas and D.R. White, *Traceable Temperatures*, John Wiley and Sons, Chichester, 1994.
18. B.N. Taylor, and C.E. Kuyatt, "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, 1994 Edition, U.S. Government Printing Office, Washington, D.C., 1994.
19. M.E. Mackay and G.K. Dick, "A comparison of the dimensions of a truncated cone measured with various techniques: The cone measurement project," *Journal of Rheology*, Volume 39, pp. 673-677, 1995.
20. R. Garritano, Rheometric Scientific, Incorporated, private communication.
21. J.M. Niemiec, J.-J. Pesce, G.B. McKenna, S. Skocypec and R.F. Garritano, "Anomalies in the Normal Force Measurement when using a Force Rebalance Transducer," *Journal of Rheology*, Volume 40, pp. 323-334, 1996.

22. Schultheisz, C.R. and McKenna, G.B., "Thermal Expansion and Its Effect on the Normal Force for a Modified Force Rebalance Transducer," *Proceedings of SPE ANTEC*, Volume 1, Processing, pp. 1142-1145, May 3-6, 1999.

23. H. Markovitz, L.J. Elyash, F.J. Padden, Jr. and T.W. DeWitt, "A Cone-and-Plate Viscometer," *Journal of Colloid Science*, Volume 10, p. 165-173, 1955.

24. W.H. Press, S.A. Teukolsky, W.T. Vetterling and B.P. Flannery, *Numerical Recipes*, Second Edition, Cambridge University Press, Cambridge, 1992.

25. G.V. Gordon and M.T. Shaw, *Computer Programs for Rheologists*, Hanser Publishers, Munich, 1994.

26. D.W. Van Krevelen, *Properties of Polymers*, Third Edition, Elsevier, Amsterdam, 1990.

27. W.J. Lyman, W.F. Reehl and D.H. Rosenblatt, *Handbook of Chemical Property Estimation Methods*, American Chemical Society, Washington, D.C., 1990.

28. B.D. Marsh and J.R.A. Pearson, "The Measurement of Normal-Stress Differences Using a Cone-and-Plate Total Thrust Apparatus," *Rheologica Acta*, Volume 7, pp. 326-331, 1968.

29. C.L. Yaws, *Handbook of Thermal Conductivity*, Gulf Publishing Company, Houston, 1995.

30. G. Winther, D.M. Parsons and J.L. Schrag, "A High-Speed, High-Precision Data Acquisition and Processing System for Experiments Producing Steady State Periodic Signals," *Journal of Polymer Science: Part B: Polymer Physics*, Volume 32, pp. 659-670, 1994.

31. *RMS-800/RDS-II Instrument Manual*, Rheometric Scientific, 1990.

# National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 2490

### Non-Newtonian Polymer Solution for Rheology

#### Polyisobutylene Dissolved in 2,6,10,14-Tetramethylpentadecane

This Standard Reference Material (SRM) is intended primarily for use in calibration and performance evaluation of instruments used to determine the viscosity and first normal stress difference in steady shear, or to determine the dynamic mechanical storage and loss moduli and shift factors through time-temperature superposition. SRM 2490 consists of a polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane (common name pristane). The solution contains a mass fraction of 0.114 polyisobutylene. The mass average relative molecular mass of the polyisobutylene is reported as 1 000 000 by the supplier. One unit of SRM 2490 consists of 100 mL of the solution packaged in an amber glass bottle.

**Certified Values and Uncertainties:** The certified values of the viscosity and first normal stress difference as functions of shear rate are given in Tables 4a, 4b, and 4c at temperatures of 0 °C, 25 °C, and 50 °C, respectively. Tables 4a through 4c also list the expanded combined uncertainties in the certified values of the viscosity and first normal stress difference. Tables 5a, 5b, 5c, 5d, 5e, and 5f list the certified values of the storage modulus G' and loss modulus G" as functions of frequency at 0 °C, 10 °C, 20 °C, 30 °C, 40 °C, and 50 °C, respectively. Tables 5a through 5f also list the expanded combined uncertainties in the certified values of the storage modulus G' and loss modulus G". The uncertainties in Tables 4a through 4c and 5a through 5f were calculated as $U = ku_c$, where $k = 2$ is the coverage factor for a 95 % level of confidence and $u_c$ is the combined standard uncertainty calculated according to the ISO Guide [1].

**Expiration of Certification:** The certification of SRM 2490 is valid until **31 December 2008**, within the measurement uncertainties specified, provided that the SRM is handled in accordance with the storage instructions given in this certificate. This certification is nullified if the SRM is modified or contaminated.

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Technical coordination leading to the certification of this SRM was provided by B.M. Fanconi of the NIST Polymers Division.

The certification of this SRM was performed by C.R. Schultheisz of the NIST Polymers Division.

The support aspects in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.W.L. Thomas.

Eric J. Amis, Chief
Polymers Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 September 2001

John Rumble, Jr., Acting Chief
Standard Reference Materials Program

Statistical analysis and measurement advice were provided by S.D. Leigh of the NIST Statistical Engineering Division.

Technical assistance and advice were provided by Gregory Strouse and Dawn Vaughn of the NIST Process Measurements Division and Gregory B. McKenna of the Texas Tech University.

**Source of Material:** The polyisobutylene and 2,6,10,14-tetramethylpentadecane were obtained from Aldrich Chemical Company, Milwaukee, Wisconsin.[1] The solution was mixed and packaged by the Cannon Instrument Company, State College, PA.[1]

**Storage and Handling:** The SRM should be stored in the original bottle with the lid tightly closed under normal laboratory conditions. Before taking a sample, the bottle should be turned end-over-end at a rate of approximately 1 revolution per 10 minutes for 30 minutes. This procedure is intended to ensure that the material in each bottle is homogeneous, in case there is any settling caused by gravity.

**Homogeneity and Characterization:** The homogeneity of SRM 2490 was tested by measuring the zero-shear-rate viscosity at 25 °C from 10 bottles randomly chosen from the 438 bottles available. Three samples from each bottle were tested in random order. The characterization of this polymer solution is described in reference [2].

**Measurement Technique:** All rheological testing was carried out using a Rheometric Scientific, Inc., ARES controlled-strain rheometer.[1] Transducer calibration was accomplished, in accordance with the manufacturer's instructions, by hanging a known mass from a fixture mounted to the transducer to apply a known torque or normal force. Phase angle calibration was accomplished, also in accordance with the manufacturer's instructions, by applying an oscillatory strain to an elastic steel test coupon. Temperature calibration in the rheometer was accomplished through comparison with a NIST-calibrated thermistor. The viscosity and first normal stress difference were measured in steady shear using 50 mm diameter, 0.02 rad cone-and-plate fixtures. The storage modulus and loss modulus were measured in 50 mm diameter parallel-plate fixtures with an applied strain magnitude of 20 % at a nominal gap of 1 mm.

**Models for the Data:** The steady shear data (viscosity and first normal stress difference) and the oscillatory data (storage modulus and loss modulus) were fitted to empirical functions to describe master curves and calculate shift factors for time-temperature superposition. These models can be used to estimate the rheological behavior of the material in the temperature range 0 °C to 50 °C.

**Models for the Steady Shear Data:** The viscosity $\eta(\dot{\gamma}, T)$ as a function of the shear rate $\dot{\gamma}$, and the temperature $T$ was fitted to a Cross model [3,4] of the form

$$\eta(\dot{\gamma}, T) = \left( \frac{T\rho}{T_R \rho_R} \right) \left( \frac{\eta_R a(T)}{1 + (\xi_0 a(T) \dot{\gamma})^{1-n}} \right) \tag{1}$$

where $\rho$ is the density at temperature $T$, $\eta_R$ is the zero-shear-rate viscosity at the reference temperature $T_R = 25$ °C, $\rho_R$ is the density at the reference temperature $T_R$, $\xi_0$ is a parameter that governs the transition from the Newtonian regime at low shear rates to the power law regime at high shear rates, and $n$ is the power at which the shear stress increases with shear rate. The density was approximated as a linear function of temperature, with $\rho(T) = \rho_R(1 - \alpha(T - T_R))$, where $\alpha = 6 \times 10^{-4}$ cm$^3$/(cm$^3$ K) is the volumetric coefficient of thermal expansion. The shift factor $a(T)$ was fitted with a function of the WLF type [3],

$$a(T) = \exp\left( \frac{-C_1(T - T_R)}{C_2 + T - T_R} \right) \tag{2}$$

---

[1]Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

The parameters $\eta_R$, $\xi_0$, $n$, $C_1$, and $C_2$ estimated from the fit to the viscosity data are given in Table 1.

Table 1. Parameters for $\eta(\dot{\gamma},T)$ and $a(T)$

| Parameter | Value | Standard Uncertainty |
|-----------|-------|---------------------|
| $\eta_R$ | 100.2 Pa·s | 0.6 Pa·s |
| $\xi_0$ | 0.234 s | 0.004 s |
| $n$ | 0.195 | 0.004 |
| | | |
| $C_1$ | 7.23 | 0.24 |
| $C_2$ | 150 °C | 5 °C |

The first normal stress difference $N_1(\dot{\gamma},T)$ was fitted to a similar empirical model using the same temperature shift factor $a(T)$ calculated from the viscosity data:

$$N_1(\dot{\gamma},T) = \left(\frac{T\rho}{T_R\rho_R}\right)\left(\frac{\psi_R(a(T)\dot{\gamma})^2}{1+\xi_1 a(T)\dot{\gamma}+(\xi_2 a(T)\dot{\gamma})^p}\right) \tag{3}$$

where $\rho$ is the density at temperature $T$; $\psi_R$ is the zero-shear-rate first normal stress coefficient at the reference temperature $T_R = 25$ °C; $\rho_R$ is the density at the reference temperature $T_R$; and $\xi_1$, $\xi_2$, and $p$ are parameters estimated from the fit to the data. The density was approximated as a linear function of temperature, with $\rho(T) = \rho_R(1-\alpha(T-T_R))$, where $\alpha = 6 \times 10^{-4}$ cm$^3$/(cm$^3$ K). Values for the parameters describing $N_1(\dot{\gamma},T)$ are given in Table 2.

Table 2. Parameters for $N_1(\dot{\gamma},T)$

| Parameter | Value | Standard Uncertainty |
|-----------|-------|---------------------|
| $\psi_R$ | 129 Pa·s$^2$ | 5 Pa·s$^2$ |
| $\xi_1$ | 1.69 s | 0.13 s |
| $\xi_2$ | 0.247 s | 0.026 s |
| $p$ | 1.67 | 0.047 |

**Models for the Oscillatory Data:** The storage modulus G'($\Omega$,$T$) and loss modulus G"($\Omega$,$T$) as functions of the frequency of oscillation $\Omega$ and temperature $T$ were modeled using polynomial functions [4]. The data were fitted to functions of the form

$$\ln\left(\frac{\text{G'}(\Omega,T)}{1\text{ Pa}}\right) = \ln\left(\frac{T\rho}{T_R\rho_R}\right) + \sum_{k=0}^{4} p_k\left(\ln\left(\frac{a(T)\Omega}{1\text{ rad/s}}\right)\right)^k$$

$$\ln\left(\frac{\text{G''}(\Omega,T)}{1\text{ Pa}}\right) = \ln\left(\frac{T\rho}{T_R\rho_R}\right) + \sum_{k=0}^{4} q_k\left(\ln\left(\frac{a(T)\Omega}{1\text{ rad/s}}\right)\right)^k \tag{4}$$

where $\rho$ is the density at temperature $T$, and $\rho_R$ is the density at the reference temperature $T_R = 25$ °C. The density was again approximated as a linear function of temperature, with $\rho(T) = \rho_R(1 - \alpha(T - T_R))$, where $\alpha = 6 \times 10^{-4}$ cm$^3$/(cm$^3$ K). The shift factor $a(T)$ again was fitted with a function of the WLF type [3],

$$a(T) = \exp\left(\frac{-C_1(T - T_R)}{C_2 + T - T_R}\right) \tag{5}$$

The parameters estimated from the to the oscillatory data are given in Table 3.

Table 3. Parameters for G'($\Omega$, $T$), G"($\Omega$, $T$) and $a(T)$

| Parameter | Value | Standard Uncertainty |
|-----------|-------|----------------------|
| $p_0$ | 3.177 | 0.005 |
| $p_1$ | 1.235 | 0.003 |
| $p_2$ | $-0.134$ | 0.001 |
| $p_3$ | $2.36 \times 10^{-3}$ | $2.7 \times 10^{-4}$ |
| $p_4$ | $5.20 \times 10^{-4}$ | $6.1 \times 10^{-5}$ |
| | | |
| $q_0$ | 4.196 | 0.005 |
| $q_1$ | 0.720 | 0.003 |
| $q_2$ | $-0.0719$ | 0.0011 |
| $q_3$ | $-3.18 \times 10^{-3}$ | $2.6 \times 10^{-4}$ |
| $q_4$ | $7.06 \times 10^{-4}$ | $6.0 \times 10^{-5}$ |
| | | |
| $C_1$ | 8.85 | 0.30 |
| $C_2$ | 192 °C | 6 °C |

Table 4a. Certified Values of Viscosity and First Normal Stress Difference
with Expanded Combined Uncertainties at 0 °C

| Temperature | Shear Rate | Certified Value of the Viscosity, $\eta$ | Uncertainty in the Viscosity | Certified Value of the First Normal Stress Difference, $N_1$ | Uncertainty in $N_1$ |
|---|---|---|---|---|---|
| °C | $s^{-1}$ | Pa·s | Pa·s | Pa | Pa |
| | | | | | |
| 0.0 | 0.001000 | 383 | 12 | | |
| 0.0 | 0.001585 | 380 | 11 | | |
| 0.0 | 0.002512 | 382.4 | 9.9 | | |
| 0.0 | 0.003981 | 382.9 | 9.5 | | |
| 0.0 | 0.006310 | 384.0 | 9.2 | | |
| 0.0 | 0.01000 | 383.1 | 9.0 | | |
| 0.0 | 0.01585 | 382.9 | 8.8 | | |
| 0.0 | 0.02512 | 379.8 | 8.6 | | |
| 0.0 | 0.03981 | 375.1 | 8.3 | | |
| 0.0 | 0.06310 | 365.5 | 7.9 | | |
| 0.0 | 0.1000 | 350.0 | 7.3 | | |
| 0.0 | 0.1585 | 328.4 | 6.6 | 16 | 11 |
| 0.0 | 0.2512 | 300.8 | 5.7 | 44 | 10 |
| 0.0 | 0.3981 | 268.5 | 4.8 | 92 | 11 |
| 0.0 | 0.6310 | 233.0 | 3.8 | 149 | 14 |
| 0.0 | 1.000 | 196.5 | 2.9 | 257 | 15 |
| 0.0 | 1.585 | 161.1 | 2.1 | 372 | 15 |
| 0.0 | 2.512 | 128.4 | 1.5 | 573 | 17 |
| 0.0 | 3.981 | 99.45 | 0.98 | 845 | 21 |
| 0.0 | 6.310 | 75.07 | 0.66 | 1219 | 29 |
| 0.0 | 10.00 | 55.59 | 0.44 | 1717 | 35 |
| 0.0 | 15.85 | 40.26 | 0.29 | 2363 | 44 |
| 0.0 | 25.12 | 28.67 | 0.20 | 3196 | 55 |
| 0.0 | 39.81 | 20.23 | 0.17 | 4251 | 69 |
| 0.0 | 63.10 | 13.88 | 0.15 | 5519 | 87 |
| 0.0 | 100.0 | 9.08 | 0.12 | 7126 | 107 |

Table 4b. Certified Values of Viscosity and First Normal Stress Difference
with Expanded Combined Uncertainties at 25 °C

| Temperature | Shear Rate | Certified Value of the Viscosity, $\eta$ | Uncertainty in the Viscosity | Certified Value of the First Normal Stress Difference, $N_1$ | Uncertainty in $N_1$ |
|---|---|---|---|---|---|
| °C | $s^{-1}$ | Pa·s | Pa·s | Pa | Pa |
| | | | | | |
| 25.0 | 0.001000 | 97.9 | 7.0 | | |
| 25.0 | 0.001585 | 98.1 | 4.9 | | |
| 25.0 | 0.002512 | 98.3 | 3.7 | | |
| 25.0 | 0.003981 | 97.9 | 2.9 | | |
| 25.0 | 0.006310 | 98.4 | 2.5 | | |
| 25.0 | 0.01000 | 98.1 | 2.3 | | |
| 25.0 | 0.01585 | 98.7 | 2.2 | | |
| 25.0 | 0.02512 | 98.8 | 2.2 | | |
| 25.0 | 0.03981 | 98.6 | 2.1 | | |
| 25.0 | 0.06310 | 98.4 | 2.1 | | |
| 25.0 | 0.1000 | 97.5 | 2.0 | | |
| 25.0 | 0.1585 | 96.1 | 2.0 | 2.4 | 1.9 |
| 25.0 | 0.2512 | 93.7 | 1.9 | 5.5 | 1.8 |
| 25.0 | 0.3981 | 90.0 | 1.8 | 12.9 | 1.9 |
| 25.0 | 0.6310 | 84.6 | 1.6 | 26.5 | 2.1 |
| 25.0 | 1.000 | 77.6 | 1.4 | 50.1 | 2.5 |
| 25.0 | 1.585 | 69.2 | 1.2 | 87.6 | 3.3 |
| 25.0 | 2.512 | 59.98 | 0.94 | 148.2 | 4.6 |
| 25.0 | 3.981 | 50.56 | 0.72 | 236.8 | 7.4 |
| 25.0 | 6.310 | 41.44 | 0.54 | 377 | 12 |
| 25.0 | 10.00 | 33.04 | 0.39 | 585 | 16 |
| 25.0 | 15.85 | 25.60 | 0.28 | 880 | 21 |
| 25.0 | 25.12 | 19.36 | 0.20 | 1280 | 27 |
| 25.0 | 39.81 | 14.26 | 0.15 | 1800 | 36 |
| 25.0 | 63.10 | 10.22 | 0.14 | 2462 | 51 |
| 25.0 | 100.0 | 7.22 | 0.13 | 3319 | 63 |

Table 4c. Certified Values of Viscosity and First Normal Stress Difference
with Expanded Combined Uncertainties at 50 °C

| Temperature | Shear Rate | Certified Value of the Viscosity, $\eta$ | Uncertainty in the Viscosity | Certified Value of the First Normal Stress Difference, $N_1$ | Uncertainty in $N_1$ |
|---|---|---|---|---|---|
| °C | $s^{-1}$ | Pa·s | Pa·s | Pa | Pa |
| | | | | | |
| 50.0 | 0.001000 | 36.7 | 6.5 | | |
| 50.0 | 0.001585 | 37.2 | 4.2 | | |
| 50.0 | 0.002512 | 37.6 | 2.8 | | |
| 50.0 | 0.003981 | 37.3 | 2.0 | | |
| 50.0 | 0.006310 | 37.7 | 1.5 | | |
| 50.0 | 0.01000 | 37.7 | 1.3 | | |
| 50.0 | 0.01585 | 37.5 | 1.1 | | |
| 50.0 | 0.02512 | 37.5 | 1.1 | | |
| 50.0 | 0.03981 | 37.8 | 1.1 | | |
| 50.0 | 0.06310 | 37.8 | 1.1 | | |
| 50.0 | 0.1000 | 37.8 | 1.1 | | |
| 50.0 | 0.1585 | 37.7 | 1.1 | | |
| 50.0 | 0.2512 | 37.4 | 1.1 | | |
| 50.0 | 0.3981 | 36.9 | 1.1 | 2.4 | 1.7 |
| 50.0 | 0.6310 | 36.1 | 1.1 | 5.1 | 1.7 |
| 50.0 | 1.000 | 34.8 | 1.0 | 10.7 | 1.8 |
| 50.0 | 1.585 | 32.79 | 0.99 | 21.5 | 2.0 |
| 50.0 | 2.512 | 30.20 | 0.93 | 41.0 | 2.7 |
| 50.0 | 3.981 | 27.05 | 0.84 | 68.8 | 4.3 |
| 50.0 | 6.310 | 23.54 | 0.74 | 114.5 | 7.6 |
| 50.0 | 10.00 | 19.95 | 0.64 | 203 | 11 |
| 50.0 | 15.85 | 16.41 | 0.53 | 345 | 16 |
| 50.0 | 25.12 | 13.14 | 0.43 | 558 | 23 |
| 50.0 | 39.81 | 10.24 | 0.34 | 860 | 33 |
| 50.0 | 63.10 | 7.75 | 0.26 | 1269 | 47 |
| 50.0 | 100.0 | 5.72 | 0.20 | 1797 | 67 |

Table 5a. Certified Values of the Storage Modulus G' and the Loss Modulus G"
with Expanded Combined Uncertainties at 0 °C

| Temperature | Frequency of Oscillation | Certified Value of the Storage Modulus G' | Uncertainty in G' | Certified Value of the Loss Modulus G" | Uncertainty in G" |
|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa |
| | | | | | |
| 0.0 | 0.02512 | 0.605 | 0.058 | 7.98 | 0.15 |
| 0.0 | 0.03981 | 1.385 | 0.090 | 12.35 | 0.23 |
| 0.0 | 0.06310 | 2.97 | 0.14 | 18.85 | 0.35 |
| 0.0 | 0.1000 | 6.07 | 0.23 | 28.20 | 0.51 |
| 0.0 | 0.1585 | 11.70 | 0.37 | 41.11 | 0.73 |
| 0.0 | 0.2512 | 21.21 | 0.58 | 58.3 | 1.0 |
| 0.0 | 0.3981 | 36.21 | 0.88 | 79.8 | 1.4 |
| 0.0 | 0.6310 | 58.5 | 1.3 | 105.9 | 1.8 |
| 0.0 | 1.000 | 89.6 | 1.9 | 135.6 | 2.3 |
| 0.0 | 1.585 | 130.9 | 2.5 | 168.1 | 2.9 |
| 0.0 | 2.512 | 183.0 | 3.4 | 201.9 | 3.5 |
| 0.0 | 3.981 | 246.1 | 4.4 | 235.9 | 4.1 |
| 0.0 | 6.310 | 319.8 | 5.5 | 268.8 | 4.8 |
| 0.0 | 10.00 | 403.3 | 6.8 | 299.7 | 5.4 |
| 0.0 | 15.85 | 495.3 | 8.1 | 328.1 | 6.1 |
| 0.0 | 25.12 | 593.8 | 9.7 | 354.1 | 6.8 |
| 0.0 | 39.81 | 697 | 11 | 378.9 | 7.6 |
| 0.0 | 63.10 | 804 | 13 | 404.1 | 8.3 |
| 0.0 | 100.0 | 913 | 15 | 432.1 | 9.2 |

Table 5b. Certified Values of the Storage Modulus G' and the Loss Modulus G"
with Expanded Combined Uncertainties at 10 °C

| Temperature | Frequency of Oscillation | Certified Value of the Storage Modulus G' | Uncertainty in G' | Certified Value of the Loss Modulus G" | Uncertainty in G" |
|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa |
| | | | | | |
| 10.0 | 0.03981 | 0.486 | 0.052 | 7.29 | 0.13 |
| 10.0 | 0.06310 | 1.099 | 0.082 | 11.35 | 0.21 |
| 10.0 | 0.1000 | 2.44 | 0.13 | 17.42 | 0.31 |
| 10.0 | 0.1585 | 5.08 | 0.20 | 26.29 | 0.47 |
| 10.0 | 0.2512 | 10.02 | 0.33 | 38.73 | 0.68 |
| 10.0 | 0.3981 | 18.53 | 0.52 | 55.45 | 0.95 |
| 10.0 | 0.6310 | 32.33 | 0.80 | 76.9 | 1.3 |
| 10.0 | 1.000 | 53.2 | 1.2 | 103.2 | 1.7 |
| 10.0 | 1.585 | 83.2 | 1.7 | 133.7 | 2.2 |
| 10.0 | 2.512 | 123.3 | 2.4 | 167.6 | 2.8 |
| 10.0 | 3.981 | 174.8 | 3.2 | 203.4 | 3.4 |
| 10.0 | 6.310 | 238.2 | 4.2 | 240.1 | 4.1 |
| 10.0 | 10.00 | 313.2 | 5.3 | 276.1 | 4.8 |
| 10.0 | 15.85 | 398.8 | 6.7 | 310.3 | 5.5 |
| 10.0 | 25.12 | 493.9 | 8.1 | 342.2 | 6.3 |
| 10.0 | 39.81 | 596.4 | 9.7 | 372.0 | 7.0 |
| 10.0 | 63.10 | 704 | 11 | 400.7 | 7.8 |
| 10.0 | 100.0 | 816 | 13 | 429.9 | 8.7 |

Table 5c. Certified Values of the Storage Modulus G' and the Loss Modulus G"
with Expanded Combined Uncertainties at 20 °C

| Temperature | Frequency of Oscillation | Certified Value of the Storage Modulus G' | Uncertainty in G' | Certified Value of the Loss Modulus G" | Uncertainty in G" |
|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa |
| | | | | | |
| 20.0 | 0.03981 | 0.182 | 0.034 | 4.605 | 0.083 |
| 20.0 | 0.06310 | 0.430 | 0.053 | 7.26 | 0.13 |
| 20.0 | 0.1000 | 1.023 | 0.080 | 11.28 | 0.20 |
| 20.0 | 0.1585 | 2.27 | 0.13 | 17.38 | 0.30 |
| 20.0 | 0.2512 | 4.81 | 0.20 | 26.30 | 0.45 |
| 20.0 | 0.3981 | 9.57 | 0.32 | 38.91 | 0.66 |
| 20.0 | 0.6310 | 17.92 | 0.50 | 56.01 | 0.94 |
| 20.0 | 1.000 | 31.62 | 0.78 | 78.2 | 1.3 |
| 20.0 | 1.585 | 52.6 | 1.2 | 105.5 | 1.7 |
| 20.0 | 2.512 | 82.7 | 1.7 | 137.5 | 2.2 |
| 20.0 | 3.981 | 123.8 | 2.4 | 173.3 | 2.8 |
| 20.0 | 6.310 | 176.8 | 3.2 | 211.6 | 3.5 |
| 20.0 | 10.00 | 242.5 | 4.2 | 251.0 | 4.2 |
| 20.0 | 15.85 | 320.6 | 5.4 | 290.1 | 4.9 |
| 20.0 | 25.12 | 410.4 | 6.7 | 327.5 | 5.7 |
| 20.0 | 39.81 | 510.1 | 8.2 | 363.0 | 6.5 |
| 20.0 | 63.10 | 617.8 | 9.8 | 396.8 | 7.3 |
| 20.0 | 100.0 | 731 | 12 | 429.7 | 8.2 |

Table 5d.  Certified Values of the Storage Modulus G' and the Loss Modulus G"
with Expanded Combined Uncertainties at 30 °C

| Temperature | Frequency of Oscillation | Certified Value of the Storage Modulus G' | Uncertainty in G' | Certified Value of the Loss Modulus G" | Uncertainty in G" |
|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa |
| | | | | | |
| 30.0 | 0.03981 | 0.077 | 0.023 | 3.028 | 0.055 |
| 30.0 | 0.06310 | 0.178 | 0.036 | 4.773 | 0.084 |
| 30.0 | 0.1000 | 0.440 | 0.054 | 7.48 | 0.13 |
| 30.0 | 0.1585 | 1.039 | 0.082 | 11.65 | 0.20 |
| 30.0 | 0.2512 | 2.32 | 0.13 | 17.94 | 0.31 |
| 30.0 | 0.3981 | 4.90 | 0.21 | 27.17 | 0.46 |
| 30.0 | 0.6310 | 9.78 | 0.33 | 40.19 | 0.68 |
| 30.0 | 1.000 | 18.38 | 0.52 | 57.92 | 0.96 |
| 30.0 | 1.585 | 32.46 | 0.80 | 80.9 | 1.3 |
| 30.0 | 2.512 | 54.0 | 1.2 | 109.2 | 1.8 |
| 30.0 | 3.981 | 85.2 | 1.7 | 142.6 | 2.3 |
| 30.0 | 6.310 | 127.7 | 2.5 | 180.0 | 2.9 |
| 30.0 | 10.00 | 182.6 | 3.3 | 220.0 | 3.6 |
| 30.0 | 15.85 | 250.7 | 4.4 | 261.3 | 4.3 |
| 30.0 | 25.12 | 331.7 | 5.6 | 302.3 | 5.1 |
| 30.0 | 39.81 | 424.5 | 7.0 | 342.1 | 5.9 |
| 30.0 | 63.10 | 527.3 | 8.6 | 380.4 | 6.8 |
| 30.0 | 100.0 | 638 | 10 | 417.2 | 7.6 |

Table 5e. Certified Values of the Storage Modulus G' and the Loss Modulus G"
with Expanded Combined Uncertainties at 40 °C

| Temperature | Frequency of Oscillation | Certified Value of the Storage Modulus G' | Uncertainty in G' | Certified Value of the Loss Modulus G" | Uncertainty in G" |
|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa |
| | | | | | |
| 40.0 | 0.03981 | 0.030 | 0.019 | 2.047 | 0.039 |
| 40.0 | 0.06310 | 0.062 | 0.025 | 3.236 | 0.063 |
| 40.0 | 0.1000 | 0.200 | 0.038 | 5.101 | 0.096 |
| 40.0 | 0.1585 | 0.478 | 0.055 | 8.00 | 0.15 |
| 40.0 | 0.2512 | 1.142 | 0.088 | 12.45 | 0.23 |
| 40.0 | 0.3981 | 2.56 | 0.14 | 19.16 | 0.36 |
| 40.0 | 0.6310 | 5.39 | 0.22 | 28.94 | 0.54 |
| 40.0 | 1.000 | 10.69 | 0.36 | 42.76 | 0.80 |
| 40.0 | 1.585 | 19.97 | 0.57 | 61.4 | 1.1 |
| 40.0 | 2.512 | 35.03 | 0.89 | 85.5 | 1.6 |
| 40.0 | 3.981 | 58.0 | 1.4 | 115.1 | 2.1 |
| 40.0 | 6.310 | 91.0 | 2.0 | 149.8 | 2.8 |
| 40.0 | 10.00 | 135.7 | 2.8 | 188.5 | 3.5 |
| 40.0 | 15.85 | 193.2 | 3.9 | 229.7 | 4.3 |
| 40.0 | 25.12 | 264.1 | 5.1 | 272.2 | 5.1 |
| 40.0 | 39.81 | 347.7 | 6.6 | 314.6 | 6.0 |
| 40.0 | 63.10 | 443.1 | 8.3 | 356.1 | 6.9 |
| 40.0 | 100.0 | 548 | 10 | 396.3 | 7.9 |

Table 5f. Certified Values of the Storage Modulus G' and the Loss Modulus G"
with Expanded Combined Uncertainties at 50 °C

| Temperature | Frequency of Oscillation | Certified Value of the Storage Modulus G' | Uncertainty in G' | Certified Value of the Loss Modulus G" | Uncertainty in G" |
|---|---|---|---|---|---|
| °C | rad/s | Pa | Pa | Pa | Pa |
| | | | | | |
| 50.0 | 0.03981 | 0.013 | 0.017 | 1.457 | 0.034 |
| 50.0 | 0.06310 | 0.034 | 0.021 | 2.316 | 0.058 |
| 50.0 | 0.1000 | 0.088 | 0.028 | 3.63 | 0.092 |
| 50.0 | 0.1585 | 0.241 | 0.041 | 5.73 | 0.15 |
| 50.0 | 0.2512 | 0.592 | 0.063 | 8.98 | 0.23 |
| 50.0 | 0.3981 | 1.37 | 0.10 | 13.95 | 0.37 |
| 50.0 | 0.6310 | 3.06 | 0.17 | 21.39 | 0.56 |
| 50.0 | 1.000 | 6.39 | 0.28 | 32.21 | 0.85 |
| 50.0 | 1.585 | 12.51 | 0.46 | 47.3 | 1.2 |
| 50.0 | 2.512 | 23.03 | 0.77 | 67.6 | 1.8 |
| 50.0 | 3.981 | 40.0 | 1.2 | 93.5 | 2.5 |
| 50.0 | 6.310 | 65.4 | 1.9 | 125.0 | 3.3 |
| 50.0 | 10.00 | 101.5 | 2.9 | 161.6 | 4.3 |
| 50.0 | 15.85 | 149.8 | 4.2 | 202.0 | 5.3 |
| 50.0 | 25.12 | 211.6 | 5.8 | 245.0 | 6.5 |
| 50.0 | 39.81 | 286.6 | 7.8 | 289.0 | 7.7 |
| 50.0 | 63.10 | 375 | 10 | 333.1 | 9.0 |
| 50.0 | 100.0 | 474 | 13 | 376 | 10 |

## REFERENCES

[1]  *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994); available at http://physics.nist.gov/Pubs/.

[2]  Schultheisz, C.R. and Leigh, S.J., "Certification of the Rheological Behavior of SRM 2490, Polyisobutylene Dissolved in 2,6,10,14-Tetramethylpentadecane," NIST Special Publication 260-143, U.S. Department of Commerce, Technology Administration, National Institute of Standards and Technology, (2001).

[3]  Macosko, C.W., *Rheology - Principles, Measurements and Applications*, Wiley-VCH, New York, (1994).

[4]  Gordon, G.V. and Shaw, M.T., *Computer Programs for Rheologists*, Hanser Publishers, Munich, (1994).

*Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.*

# *NIST* *Technical Publications*

## *Periodical*

**Journal of Research of the National Institute of Standards and Technology**—Reports NIST research and development in those disciplines of the physical and engineering sciences in which the Institute is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Institute's technical and scientific programs. Issued six times a year.

## *Nonperiodicals*

**Monographs**—Major contributions to the technical literature on various subjects related to the Institute's scientific and technical activities.

**Handbooks**—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

**Special Publications**—Include proceedings of conferences sponsored by NIST, NIST annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

**National Standard Reference Data Series**—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a worldwide program coordinated by NIST under the authority of the National Standard Data Act (Public Law 90-396). NOTE: The Journal of Physical and Chemical Reference Data (JPCRD) is published bimonthly for NIST by the American Institute of Physics (AIP). Subscription orders and renewals are available from AIP, P.O. Box 503284, St. Louis, MO 63150-3284.

**Building Science Series**—Disseminates technical information developed at the Institute on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

**Technical Notes**—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NIST under the sponsorship of other government agencies.

**Voluntary Product Standards**—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The standards establish nationally recognized requirements for products, and provide all concerned interests with a basis for common understanding of the characteristics of the products. NIST administers this program in support of the efforts of private-sector standardizing organizations.

*Order the* **following** *NIST publications—FIPS and NISTIRs—from the National Technical Information Service, Springfield, VA 22161.*

**Federal Information Processing Standards Publications (FIPS PUB)**—Publications in this series collectively constitute the Federal Information Processing Standards Register. The Register serves as the official source of information in the Federal Government regarding standards issued by NIST pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

**NIST Interagency or Internal Reports (NISTIR)**—The series includes interim or final reports on work performed by NIST for outside sponsors (both government and nongovernment). In general, initial distribution is handled by the sponsor; public distribution is handled by sales through the National Technical Information Service, Springfield, VA 22161, in hard copy, electronic media, or microfiche form. NISTIR's may also report results of NIST projects of transitory or limited interest, including those that will be published subsequently in more comprehensive form.