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Physical Properties of Low-Molecular Weight Polydimethylsiloxane Fluids

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Physical Properties of Low-Molecular Weight Polydimethylsiloxane Fluids

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Abstract

Physical property measurements including viscosity, density, thermal conductivity, and heat capacity of low-molecular weight polydimethylsiloxane (PDMS) fluids were measured over a wide temperature range (-50 ^{o}C to 150 ^{o}C when possible). Properties of blends of 1 cSt and 20 cSt PDMS fluids were also investigated. Uncertainties in the measurements are cited. These measurements will provide greater fidelity predictions of environmental sensing device behavior in hot and cold environments.

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NOMENCLATURE

CO_2	Carbon dioxide
ESD	Environmental sensing device
LA	Launch accelerometer
NSC	National Security Campus
N_2	Nitrogen
PDMS	Polydimethylsiloxane
VTC	Viscosity temperature coefficient

1. INTRODUCTION

Low-viscosity silicone oils (polydimethylsiloxane, PDMS) are used as damping fluids in a variety of environmental sensing devices (ESDs) such as accelerometers and rolamites. The behavior of these devices is strongly dependent on the material properties of the fluid such as the density and viscosity. Some of these properties change significantly over the temperature range in which ESDs are expected to perform and due to blending of multiple oils. This work aims to characterize the density, viscosity, thermal conductivity, and specific heat of PDMS fluids over a wide temperature range (-40 ^oC to 150 ^oC) and a range of blends. Moreover, uncertainties in property measurements are provided when possible. These measurements will then be used to more accurately model ESD performance in realistic operating temperatures.

PDMS fluids are commercially available liquids that are typically specified by their nominal kinematic viscosity at 25 ^oC. PDMS fluids with viscosities of 20 cSt and below are relevant to ESDs, and in particular blends of 20 cSt and 1 cSt PDMS. The fluids of interest are linear-chain polymers with chemical formulas of $(SiO(CH_3)_2)_x$. The lowest-viscosity silicone oils (1.5 cSt, 1 cSt, and 0.65 cSt) are short-chain polymers, with 0.65 cSt fluid being the shortest chain possible $(O(Si(CH_3)_3)_2$, hexamethyldisiloxane). The lowest-viscosity oils ($\nu < 5$ cSt) show significant evaporation in the temperature range of interest. Evaporation presents a challenge for providing viscosity and density measurements for these fluids over the entire temperature range.

To create a PDMS fluid with the desired viscosity, either the polymer chain length is altered, or silicone oils of different chain lengths are blended. There is no guarantee that all suppliers of these fluids create the same blends. Suppliers of PDMS fluids include Clearco, Xiameter, Gelest, and Dow Corning. It is expected that if there are differences in the products between these sources, the temperature dependence of the density and the viscosity would be sensitive to the differences in producing these products with the same nominal kinematic viscosity at 25 ^oC. Numerous 20 cSt PDMS fluids of varying age and source were obtained to probe the effect of supplier on the physical properties. Through personal conversations [1] with Clearco, it was found that Clearco PDMS is purchased by the supplier either from Momentive or Xiameter. Xiameter is a rebranding of the Dow Corning product.

Currently, models of ESD behavior are based on physical properties found in supplier data sheets. Some of these data are included in the appendix. Not all of these data agree. In addition, some data are difficult to interpret from plots and some data are presented in a confusing manner. Therefore, it was decided that these data should be confirmed by independent measurements at Sandia National Laboratories.

This report summarizes measurements of physical properties of both pure PDMS fluids and also ESD-relevant blends over a wide temperature range (-40 ^oC to 150 ^oC) when possible. Measurements are compared to data sheets provided by suppliers when available in order to identify areas of discrepancy. Because most of these data sheets were obtained online, they are reproduced in the appendix to archive them. Viscosity measurements are also compared to unpublished data obtained using a capillary viscometer by Sabrina Wells [2] over a limited temperature range. Both viscosity and density of samples sourced from various suppliers are also measured over the temperature range in an effort to determine whether these samples behave

differently. Thermal conductivity and heat capacities of fluids and blends are measured. Finally, an effort to characterize the solubility of nitrogen and carbon dioxide gasses in PDMS is also introduced along with preliminary data.

2. DENSITY MEASUREMENTS

2.1. Methods

PDMS fluids of varying room temperature kinematic viscosity were obtained from Clearco (0.65 cSt, 1 cSt, 1.5 cSt, 2 cSt, 5 cSt, 10 cSt, 20 cSt, 50 cSt). Unless noted, measurements were performed on these fluids. Additionally, five other samples of 20 cSt PDMS from various suppliers and of different ages were obtained by Emily Stirrup and Joel Hey. The exact provenances of these stores are unknown beyond the owner of the supply.

Density measurements were obtained using a Mettler Toledo DE40 oscillating U-tube density meter. Assuming a perfectly clean U-tube, this apparatus has a reported accuracy of $1x10^{-4}$ g/cm^3 reported accuracy, but was only able to deliver density measurements between 5 ^{o}C – 50 ^{o}C. This instrument was calibrated at each temperature using dry air and water that was purified through a Milli-Q system.

Density measurements outside of the 5 ^{o}C – 50 ^{o}C temperature range were obtained using a Le Chatelier type pycnometer (Kimble Chase) with a 250 mL capacity in the bulb, 17 mL neck capacity, and 0.1 mL gradations in the neck (see inset in Figure 1). Room temperature PDMS in the flask was weighed using a Mettler Toledo PB4002-s laboratory scale before or after the sample temperature was changed. Thermal control for these measurements was provided by a Tenney Junior TJR environmental chamber with an accuracy of ±0.3 °C. The sample was allowed to come to an equilibrium temperature for at least an hour for each temperature change. To observe the volume of fluid in the pycnometer, the door to the chamber had to be opened quickly; it is estimated that the temperature of the 250 mL flask changes less than a degree during this process. Considering these factors, the uncertainties in the density measurements were estimated to be approximately ±0.002 g/mL.

Any change of the volume of the pycnometer with temperature was checked using a viscosity and density standard (Cannon N14) below 20 ^{o}C. No thermal expansion of the volume was detected. These density measurements for the Canon N14 reference fluid are shown in Figure 1.

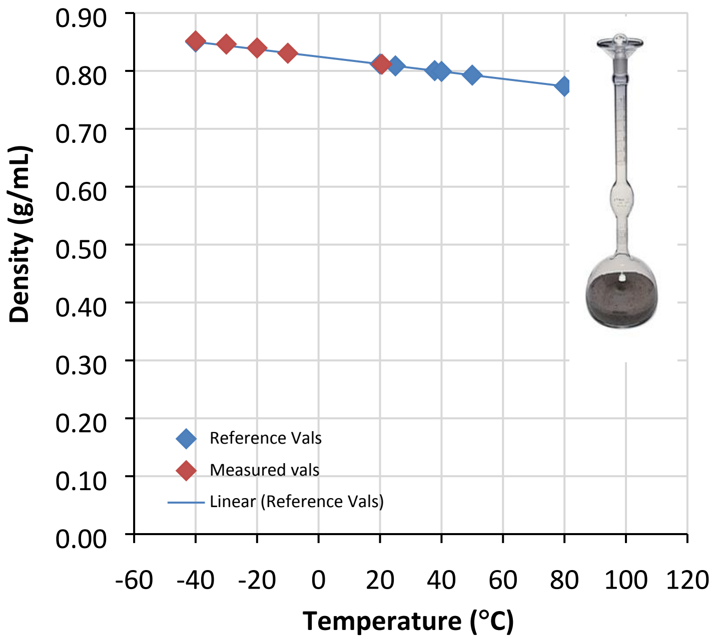


Figure 1. Density measurements of Cannon N14 standard in a large pycnometer using thermal chamber as compared to reference values. No correction to pycnometer volume was applied. Inset: image of a pycnometer.

2.2. Density of Pure Silicone Fluids

The density of Clearco 20 cSt PDMS was measured over a wide temperature range using both the density meter and the pycnometer. These measurements are displayed in Figure 2. Triplicate measurements were taken with the density meter at three separate temperatures. All of these points are plotted in Figure 2, but the points overlap and cannot be distinguished. Single measurements were obtained at each temperature with the pycnometer. The data are effectively linear with temperature over the entire temperature range (-40 ^{o}C to 77 ^{o}C). Good agreement is apparent between the pycnometer and the density meter values. The measured data compares reasonably well with values provided by Clearco in a personal communication for 20 cSt PDMS [1] (Figure A 6).

PDMS (20 cSt) samples were obtained from several different sources. The densities of these samples were measured using the Mettler Toledo density meter and are displayed in Figure 3. Although the exact histories of these samples are unknown, it is probable that these samples are of different ages and suppliers. The density is consistent across all samples.

Density measurements for all low-viscosity PDMS fluids of varying viscosity are displayed in the leftmost inset on Figure 4 as a function of the temperature. Average values collected with the density meter are tabulated in Table 1. At very low temperatures, the Clearco density data deviates from a linear relationship slightly, whereas the measured data for 5, 10, and 20 cSt PDMS follow a linear relationship very well over the entire temperature range. It is speculated that there may be a typo in the 10 cSt, -40 ^{o}C Clearco value.

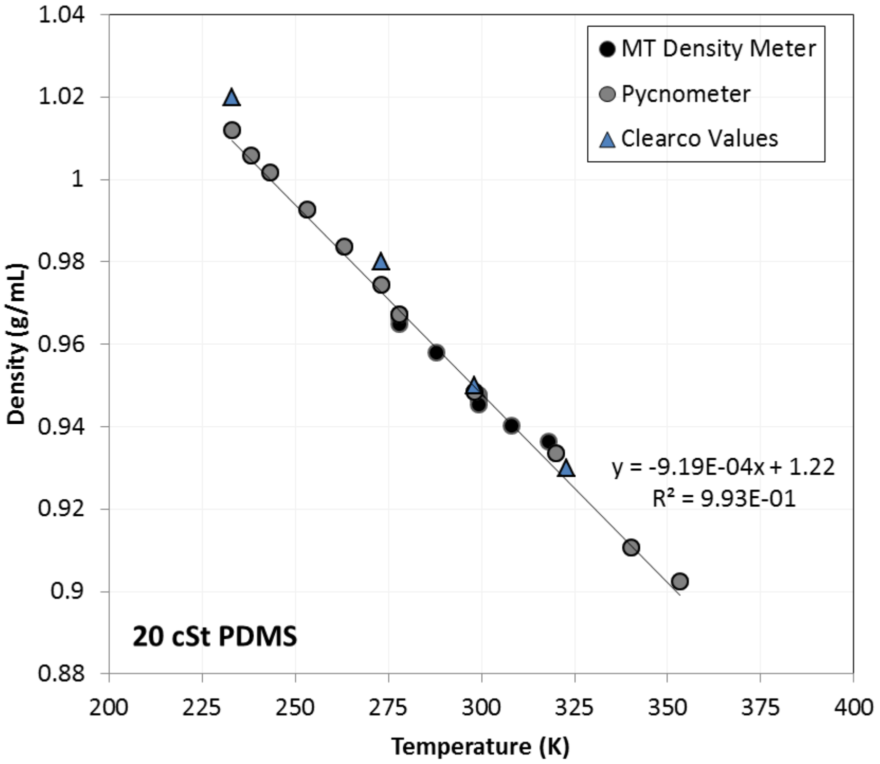


Figure 2. Density measurements of Clearco 20 cSt PDMS using both the pycnometer and the Mettler Toledo density meter as compared to values provided by Clearco (Figure A 6) [3].

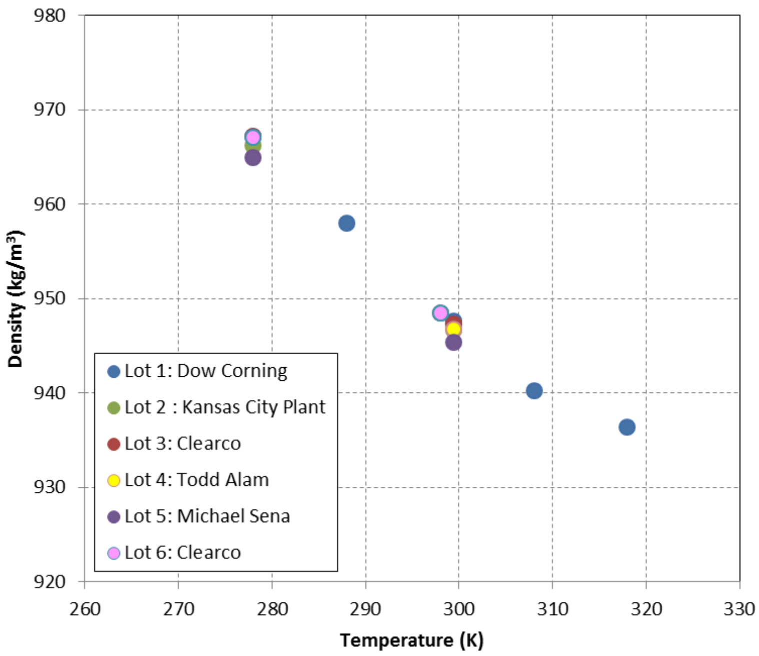


Figure 3. Density measurements of various lots of 20 cSt PDMS. Sources for PDMS are cited.

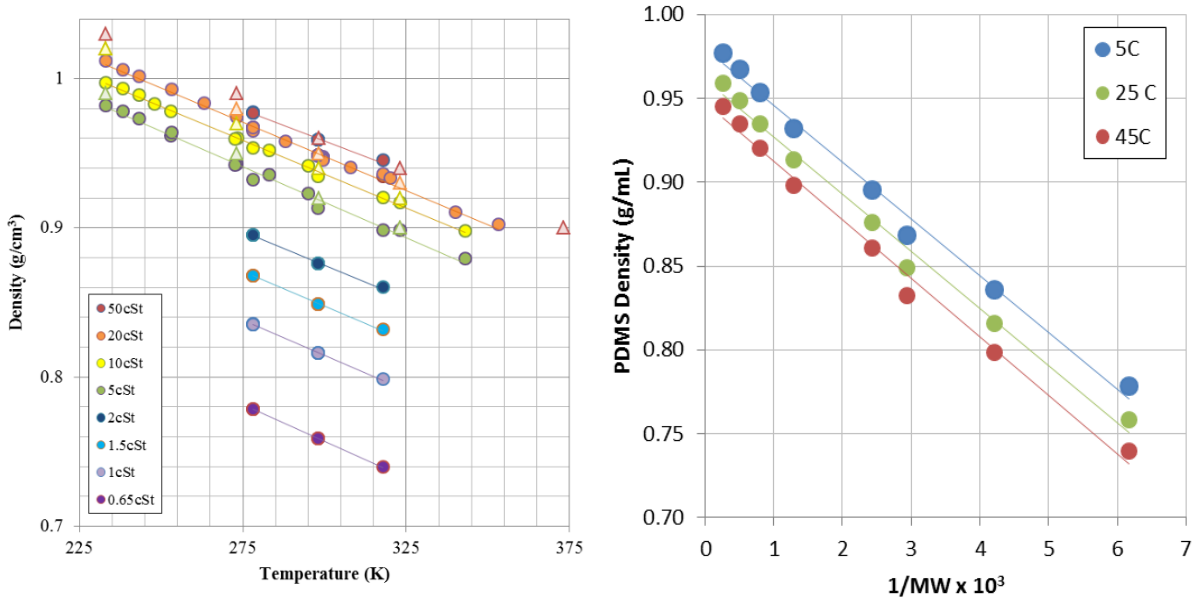


Figure 4. Left: Density measurements of a variety of Clearco PDMS fluids over a range of temperatures. Dots represent values measured in this work. Lines of best fit for each PDMS fluid are plotted based on the measured data. Triangles represent reference data provided by Clearco [1]. Right: PDMS density as a function of PDMS molecular weight given by Clearco.

As the polymer chain length of the PDMS decreases, the density of the fluid also decreases. This can be explained since, per mass of PDMS, shorter chains will have greater conformational degrees of freedom as compared to a mass of longer polymer chains due to the number of bonds between the atoms. Following Lichtenthaler [4], the density was found to be a linear function of the inverse of the fluid molecular weight. This relationship was obeyed for low-molecular weight (low-viscosity) PDMS fluids below a molecular weight of 6000 g/mol (100 cSt), where it is assumed that the maximum packing of bonds per unit volume is attained [4]. On the right side of Figure 4, the density is shown as a function of the inverse of the molecular weight of the fluids.

The volumetric thermal expansion coefficient, α_v, is defined as

$$\alpha_v = \frac{1}{V}\left(\frac{dV}{dT}\right)_P \quad , \qquad (1)$$

where V is the specific volume of the material and the subscript P indicates that the pressure is held constant. The thermal expansion coefficient of PDMS was calculated using the slopes of the curves in Figure 4 (left) as well as the density at 5 °C (Table 1). The calculated thermal expansion coefficient is then compared to the Clearco values in Figure 5. As the chain length of the PDMS decreases, the thermal expansion coefficient increases, consistent with the idea that the shorter chain polymers have greater available conformational degrees of freedom. The thermal expansion coefficients determined form the measurements agree well with Clearco values, despite the density being known at only three temperatures. The exception is at low-viscosity PDMS samples, where

Table 1. Average density values over a range of temperatures measured for low-viscosity PDMS fluids.

viscosity cSt	MW g/mol	Measured Density (g/mL) 5 ^{o}C	 25 ^{o}C	 45 ^{o}C	α_v cm^3/(cm^3 ^{o}C)
0.65	162	0.779	0.759	0.740	0.00216
1	237	0.836	0.816	0.799	0.00167
1.5	340	0.868	0.849	0.832	0.00145
2	410	0.895	0.876	0.861	0.00129
5	770	0.939	0.913	0.898	0.00114
10	1250	0.963	0.934	0.920	0.00105
20	2000	0.967	0.949	0.935	0.00103
50	3780	0.977	0.959	0.945	0.000994

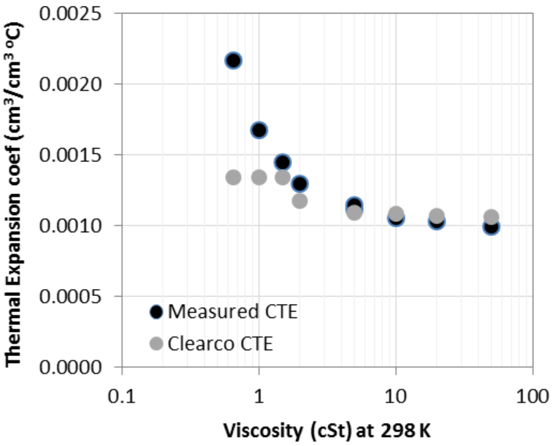


Figure 5. Thermal expansion coefficient as compared to Clearco values.

Clearco cites a constant thermal expansion coefficient with chain length whereas our measurements show the thermal expansion coefficient continues to increase. There is no known physical reason why the thermal expansion coefficient would be constant for low-viscosity PDMS fluids.

2.3. Density and Thermal Expansion Coefficients of Blends of Silicone Fluids

The densities of blends of 20 cSt and 1 cSt PDMS are also of interest. Blends were created on a mass basis, and their densities were measured following the same procedures as the pure fluids. These results are shown in Figure 6 and tabulated in Table 2, where the blend percentage represents the mass fraction of 1 cSt oil. Addition of small amounts of 1 cSt fluid to 20 cSt PDMS decreases the density but does not substantially affect the thermal expansion coefficient.

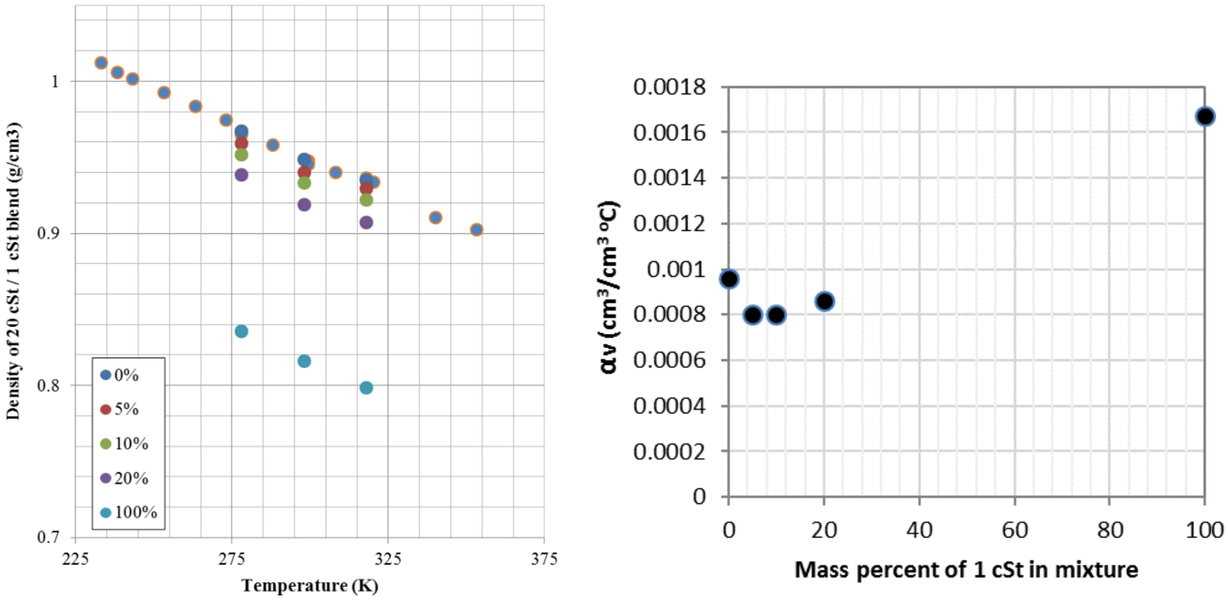


Figure 6. Left: Density of blends of 20 cSt PDMS and 1 cSt PDMS. Legend shows the mass percent of 1 cSt in the mixture. Right: Calculated thermal expansion coefficient of PDMS blends at 5 °C.

Table 2. Average density values over a range of temperatures measured for 1 cSt, 20 cSt blends.

Mass Percent 1cSt	Density (g/cm^3) 5 °C	25 °C	45 °C	α_v cm^3/(cm^3 °C)
0%	0.967	0.949	0.935	0.000961
5%	0.959	0.940	0.929	0.000804
10%	0.952	0.933	0.922	0.000803
20%	0.939	0.919	0.907	0.000864
100%	0.836	0.816	0.799	0.001673

3. VISCOSITY MEASUREMENTS

PDMS fluids are sold and named with respect to their kinematic viscosity at room temperature; however, their viscosity changes dramatically with temperature.

Note that term "viscosity" can refer to two separate parameters. As shown in Eq. 2, the kinematic viscosity (ν), with units of m^2/s, and the dynamic viscosity (μ), with units of Pa·s, are related through the density (ρ). All three of these parameters are temperature-dependent.

$$\nu = \frac{\mu}{\rho} \tag{2}$$

The viscosities of pure silicone fluids and blends were obtained using two separate methods. First, a TA Instruments AR-G2 rheometer was used to obtain viscosity measurements efficiently over a wide temperature range (0 – 150 °C) with varying shear rate. However, the lowest-viscosity fluid measurements contained an unacceptable amount of error using this method due to the limitations of the instrument. Therefore, the viscosities of these fluids were also measured using a capillary viscometer method that did not allow for control over the shear rate and assumed Newtonian behavior of the PDMS liquids. These capillary measurements are more accurate and have less uncertainty than the measurements made with the rotational rheometer.

3.1. Methods

The viscosity measurements obtained on a TA Instruments AR-G2 rheometer used a 1 inch diameter double-gap Couette geometry. The temperature was ramped at a 1 °C/min rate from 0 °C to 150 °C when possible. Note that this method inherently measures the dynamic viscosity of the sample. A shear-rate sweep up to 200 1/s did not detect any non-Newtonian behavior for any of the PDMS liquids, so the shear rate was kept constant at 100 1/s for temperature sweep measurements. The onsets of shear thinning for PDMS fluids of various zero-shear viscosities have been studied by others [5]–[7], and shear thinning for these low-molecular weight PDMS fluids is not expected to be observable within the achievable shear rates of our instruments.

The viscosities of two standards were measured using this procedure and equipment to provide an estimate of the uncertainty in the measurements (Cannon N14, Cannon N44). The data are shown in Figure 7. As the viscosity of the standard decreases below 10 mPa·s, the instrument becomes less able to measure the viscosity accurately, and an alternate method is needed. However, since most relevant ESD fluids have viscosities greater than 10 mPa·s, the AR-G2 rheometer is an attractive choice of equipment for obtaining measurements over a wide range of temperatures. The heating rate had a moderate effect on the data, with slower heating rates having less error. This is due to the large thermal mass of the Couette cell.

Capillary viscometry was also used to obtain viscosity measurements. The unique design of Cannon Ubbelohde viscometers (see Figure 8) with their separate measurement and thermal compensation tubes is such that the calibration constant is independent of temperature. Cannon viscometers were used in this investigation. Both standard three-tube Ubbelohde viscometers and the Cannon brand two-tube variants in which the thermal and measurement tubes are coaxial (the Cannon-Ubbelohde viscometers) were used in these experiments according to ASTM standards [5], [6].

All of the purchased Ubbelohde viscometers have been calibrated against NIST standards at Cannon and have calibration certificates. Independent measurements in our laboratory on the Cannon N14 viscosity standard and for pure water verify these calibrations and reproducible and accurate within ±1% for the temperature range from 5 ^{o}C to 35 ^{o}C. There was no measureable difference in either the accuracy or reproducibility between the Cannon-Ubbelohde viscometers and Ubbelohde viscometers. PDMS fluid measurements in the range of 5 ^{o}C to 35 ^{o}C were performed in a water bath. The temperature was held constant within ±0.1 ^{o}C and the viscosities measured are believed to be accurate to within same ±1% measured in the standards described above.

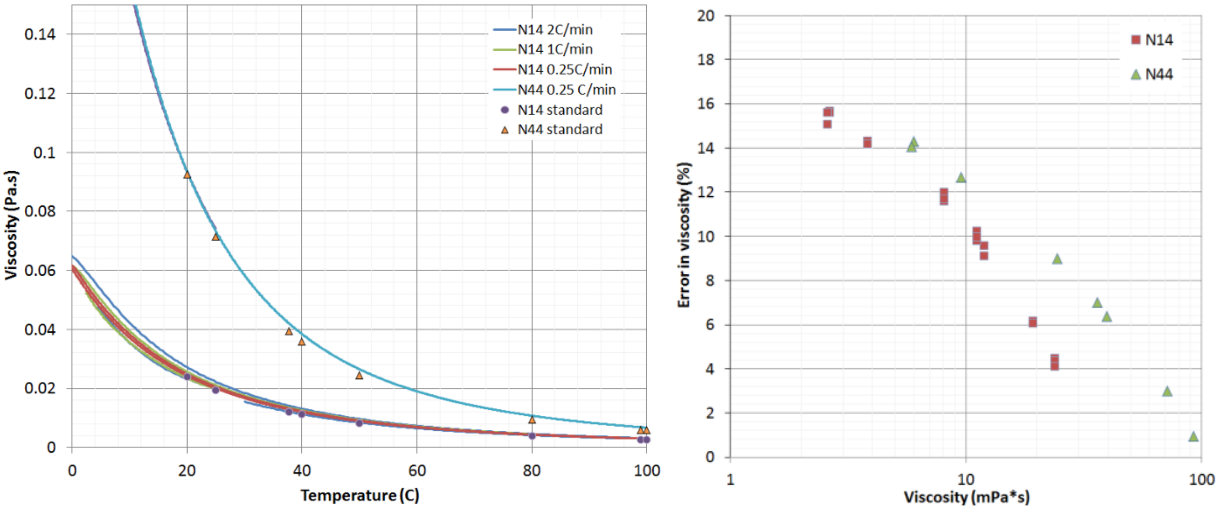


Figure 7. Left: Viscosity measurements of standards Cannon N14 and Cannon N44 using the TA Instruments AR-G2 rheometer at various heating rates. Right: Errors with respect to the cited viscosity values.

(a) **(b)**

Figure 8. Ubbelohde (a) viscometer and Cannon-Ubbelohde (b) viscometer. Ubbelohde viscometers contain independent measurement and temperature-compensation tubes but tend to be somewhat fragile. The Cannon-Ubbelohde viscometer in the center has these tubes running coaxially near the top to make the viscometers more robust in handling. The measurement tube is to the far right in both types of rheometers.

Figure 9. Therm-Craft Lab-Temp chamber with 180 liter liquid nitrogen dewar. Up to three different capillary rheometers were placed in the chamber for measurements at any one time.

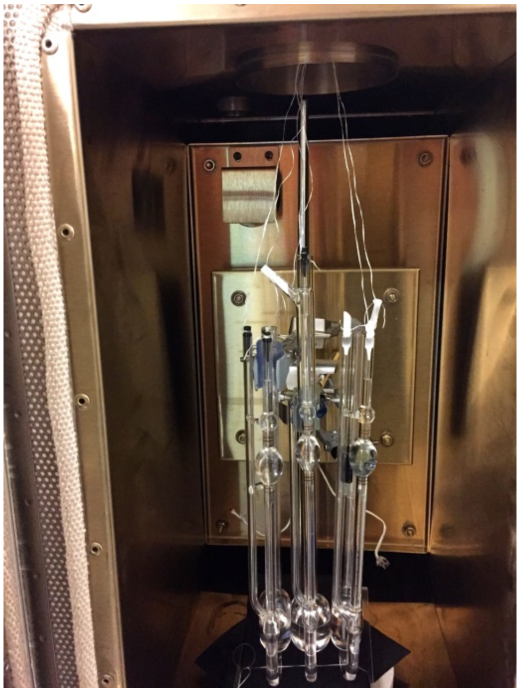
Figure 10. Temperature control unit where the set temperature is shown left and the temperature measured by the thermocouples built into the chamber are displayed right. The chamber temperature can fluctuate as much as ± 2 °C as the evaporated nitrogen is blown into the chamber. The platinum resistance thermometer is immersed in a beaker of PDMS fluid with approximately the same volume as that held in the capillary rheometers.

It was this value that was recorded for the measurement after the 0.5 – 1 hour equilibration time.

Low-temperature measurements of PDMS fluids provided additional and unexpected challenges. Because nuances in the technique were found to be necessary for generating acceptable measurements, they are described in detail here. As shown in Figure 9, measurements below 0 ^{o}C were performed in a Therm-Craft Lab-Temp chamber cooled with liquid nitrogen. The chamber is capable of holding temperatures of 50 ^{o}C to -100 ^{o}C within ±1 ^{o}C. Actual chamber temperatures were measured with a NIST traceable platinum thermometer that is accurate within ±0.08 ^{o}C. The temperature probe was immersed in a separate reservoir of the same volume of the test PDMS fluid. Note that the Lab-Temp controller unit was able to maintain a constant temperature to within ±2 ^{o}C.

Using a suction bulb, the fluid in the measurement tube was drawn up to a level above the bulb above the upper measurement mark. After release of the suction bulb, the level of the fluid fell. The time for the meniscus to fall between the two measurement marks was recorded with a stopwatch. The kinematic viscosity was determined by multiplying this time by the calibration constant for that rheometer.

The initial technique used to make low-temperature measurements with the capillary rheometers was to use a suction bulb to draw the PDMS fluid above the bulb above the upper measurement line. Before the liquid could fall into the second bulb, a variable-diameter stopper with a string attached was inserted into the top of the measurement tube. The string was labeled and passed through the upper port in the chamber. After thermal equilibration, the stopper was pulled and the time for the meniscus to fall between the two marks was observed and recorded. A rack of three viscometers being prepared for insertion into the cryogenic chamber is shown in Figure 11.



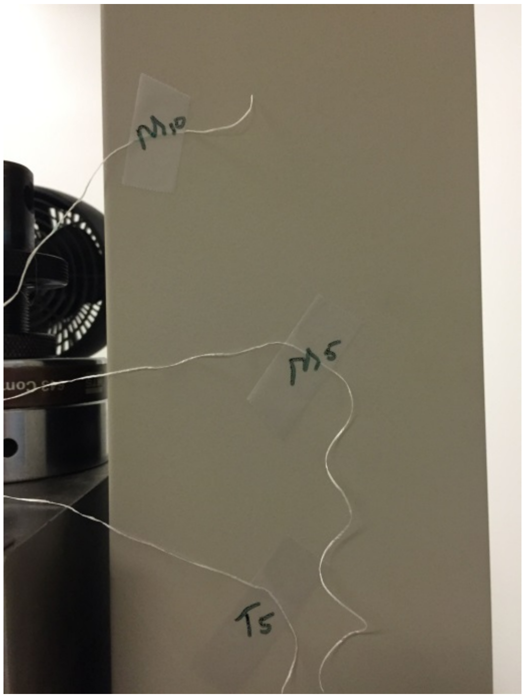


Figure 11. Rack of three capillary rheometers in cryogenic chamber. Stoppers for the different tubes were passed through the top of the chamber and identified for testing.

This technique worked well for temperatures of -10 °C and above. Excellent agreement with water bath measurements made using low-viscosity silicone fluid for a heat transfer medium was observed at -5 °C. Below -10 °C, low apparent values of the viscosity were observed. Careful observation of the rheometers during thermal equilibration revealed that a bubble of gas was being drawn from the thermal tube into the measurement tube. This was due to the contribution of the significant thermal expansion of the PDMS fluids and the large temperature drops of the capillary rheometers in the cryogenic chamber. The onset of this phenomenon occurred at a higher temperature for the higher-viscosity and hence higher-density PDMS fluids. It was determined that inserting a stopper into both the thermal and measurement tubes prevented bubble formation in the temperature range of above -25 °C. The procedure was to pull the measurement-tube stopper first and then the thermal-tube stopper and then make the measurement.

Unfortunately, even with both tubes stoppered, as the viscometers were cooled to less than -30 °C bubbles formed. The technique to allow measurements down to -40 °C was to wait during the cool-down process until a bubble appeared. Then the front door of the chamber was opened, and the suction bulb was used to draw more PDMS fluid into the measurement arm and move the bubble to the free surface at the top of the arm. Frost was introduced into the chamber as moist room air condensed on the window and the viscometers. Allowing the samples to remain in the dry nitrogen stream for 30 minutes resulted in clear surfaces as the frost sublimed. Usually this "burping" of the measurement arm needed to occur once or twice to allow the capillary rheometers to reach -40 °C.

During our post-experiments review, a method to prevent these problems was conceived. In future experiments, we recommend that either special viscometers be ordered from Cannon or a glass shop be employed to add extenders to both the temperature and measurement tubes of the rheometers. These longer tubes would pass through specially designed insulation at the top of the chamber. Hence, the fluid could be cooled to very low temperatures and then be raised into the measurement tubes. Thermal effects could be modeled to provide greater understanding of the heat transfer through the extended tubes.

3.2 Viscosity of Pure Silicone Fluids

The viscosities of pure silicone fluids were measured using both the double-walled Couette geometry on the AR-G2 rheometer and also the Cannon Ubbelohde viscometers. Good agreement is seen between the two data sets (Figure 12). Raw data are tabulated in Appendix B.

As expected, the viscosity follows Arrhenius-type behavior, with the log of the viscosity dependent on 1/T. When plotted on these axes, the viscosity follows a linear relationship. To describe this relationship, each PDMS fluid viscosity was fit to an equation of the form [10], [11]:

$$log_{10}\mu = \frac{A}{T} + B \tag{3}$$

where μ is the viscosity in Pa·s and T is the temperature in Kelvin. In order to not bias the line of best fit towards the rheometer data, which contains many more points than the capillary viscometry data, the rheometer data were reduced to selected points every 10 ^{o}C for the fit. Both sets of data were then fit together. The resulting fitting parameter A and viscosity measured at 25 ^{o}C are displayed in Table 3 for each PDMS fluid and are plotted in Figure 13. Error bars for the A fitting parameter are drawn to represent the standard error in the linear regression of the fit. As the chain length of the PDMS decreases, the temperature dependence of the viscosity decreases, as seen by a decreasing fitting parameter A. Parameter A stays relatively constant for PDMS fluids with room temperature kinematic viscosities greater than 20 cSt, however.

The Clearco viscosity temperature coefficient (VTC), which is used to determine viscosity at a temperature, is defined as:

$$VTC = 1 - \frac{\nu_{100}}{\nu_{35}} \tag{4}$$

where ν_{100} is the kinematic viscosity at 100 ^{o}C, and ν_{35} is the kinematic viscosity at 35 ^{o}C (Figure A2).

To compare viscosity/temperature relationships reported by Clearco[1] with the measurements, a dynamic viscosity at 25 ^{o}C was calculated using the specific gravity and kinematic viscosity measurements reported in Figure A2. The A parameter was calculated from the Clearco viscosity temperature coefficient (VTC, defined in Equation 1) using the following relationship:

$$A = \frac{-\,log_{10}\left(\frac{\rho_{373}}{\rho_{308}}(1 - VTC)\right)}{\frac{1}{308} - \frac{1}{373}} \tag{5}$$

Here, ρ_{373} is the density of the PDMS at 373 K (100 ^{o}C). The Clearco-published density value was used for this calculation and also to convert between kinematic and dynamic viscosity.

---

[1] Clearco also reports a “computational expression” for the viscosity of PDMS (see Figure A3 in the appendix). Using this expression, it would be assumed that the temperature dependence of the viscosity would be the same for all PDMS liquids, but for low-viscosity PDMS fluids (< 20 cSt) this is not true. For the purposes of this report, this computational expression is assumed to apply only to high viscosity PDMS liquids, and the viscosity temperature coefficient (which does depend on PDMS chain length) is referenced instead.

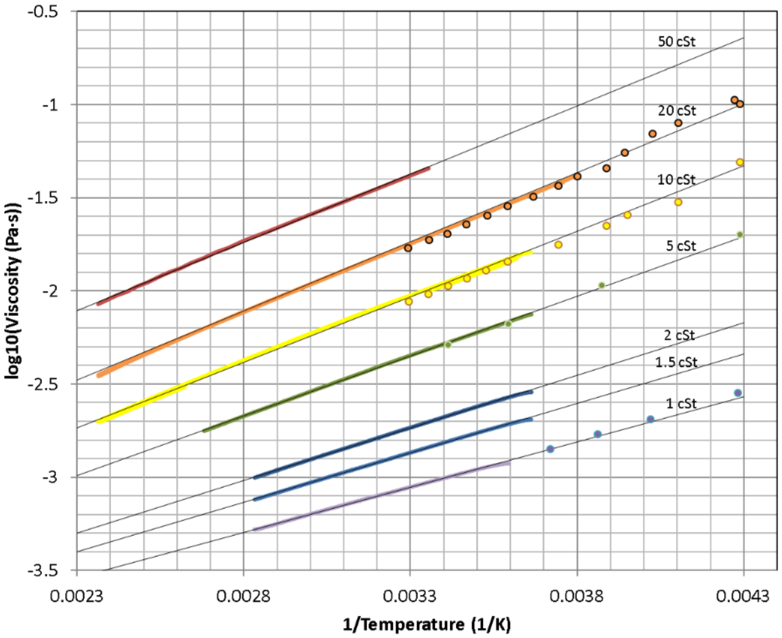


Figure 12. Viscosity of pure PDMS fluids over a wide temperature range (-40 to 150 °C). High-temperature data were obtained using the AR-G2 rheometer, whereas discrete points at low temperatures were obtained using the capillary viscometer method. Lines of best fit for the combined data sets are shown.

Table 3. Viscosity fitting parameters

Fluid ID (cSt)	A (log(Pa·s)K) measured	Clearco VTC	A (log(Pa·s K) Clearco	Measured viscosity 25 °C (mPa·s)	Clearco viscosity 25 °C (mPa·s)
50	732 ± 5	0.59	740	45±2	48
20	752 ± 6	0.59	740	19.7±0.9	19
10	687 ± 6	0.56	686	10.0±0.05	9.4
5	647 ± 5	0.54	652	4.8±0.2	4.6
2	565 ± 5	0.48	558	2.0±0.09	1.7
1.5	531 ± 5	0.46	529	1.45±0.07	1.3
1	499 ± 5	0.37	410	0.94±0.04	0.82

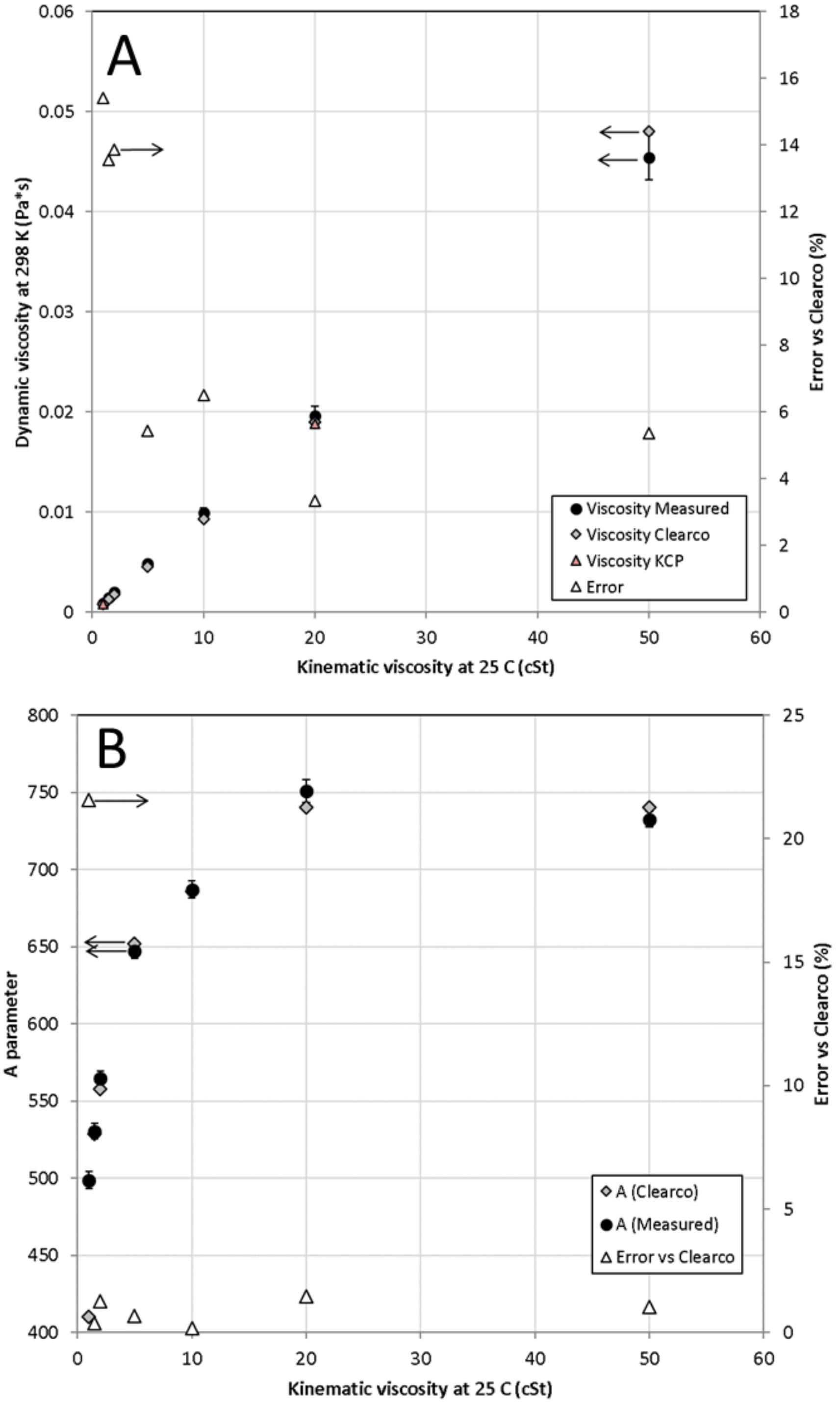


Figure 13 a,b: Fitting parameters for PDMS fluid viscosity data.

As expected from the rheology of low-viscosity calibration fluids, low-viscosity measurements (< 0.01 Pa·s) contain as much as 15% error as compared to the Clearco-reported values. As the viscosity increases, more stress is applied to the rheometer fixture, and the error decreases to about 6%. Error bars are drawn on Figure 13a to represent the estimated error in the viscosity measurements found using the calibration fluids. These uncertainties are also cited in Table 3. The temperature dependence of the viscosity compares very well to the Clearco dependence. Note that the Clearco VTC is the slope between viscosity values taken at only two temperatures.

Room temperature viscosity values are also plotted along with measurements obtained at the National Security Center by Sabrina Wells using a capillary viscometer [10], [11]. Again, the Clearco-cited density values at 25 ^{o}C were used to convert kinematic to dynamic viscosity. Low-viscosity measurements obtained by the capillary viscometer and Clearco were more comparable, but the capillary viscometer method was able to measure over only a small temperature range near room temperature (15, 25, 35 ^{o}C) with the available equipment.

3.3 Viscosity of Silicone Fluid Blends

The viscosity of blends of 1 cSt and 20 cSt PDMS fluids are measured with respect to temperature. In all instances in this report, the concentration of 1 cSt fluid is reported on a mass basis. Again, a double-walled Couette cylinder geometry is used to measure the viscosity with the AR-G2 rheometer at high temperatures. Although a cover is used to limit evaporation of 1 cSt PDMS from this geometry, it was expected that some concentration change would result during the heating ramp. Therefore, the temperature is first decreased, then increased to the maximum value, and finally returned to room temperature to determine whether the viscosity changes during the experiment. In some instances, a 5% increase in viscosity is measured due to the temperature ramp. Also, blends were measured using capillary viscometry using the methods described previously. It is estimated that evaporation is minimal from the capillary viscometers since the glassware does not allow much surface area for evaporation and the measurements were all taken at depressed temperatures.

The viscosity data are linear when plotted according to Equation 4, as shown in Figure 14. Fitting parameters are listed in Table 4, combining both data sets as described above. Uncertainties in the A parameter are cited from the standard error of the fit, whereas uncertainties in the room temperature viscosity values are estimated from the inaccuracy of measuring the standards, as before. The A parameter seems to be a linear function of the blend composition, as shown in Figure 15. More data should be obtained to make a more definite conclusion.

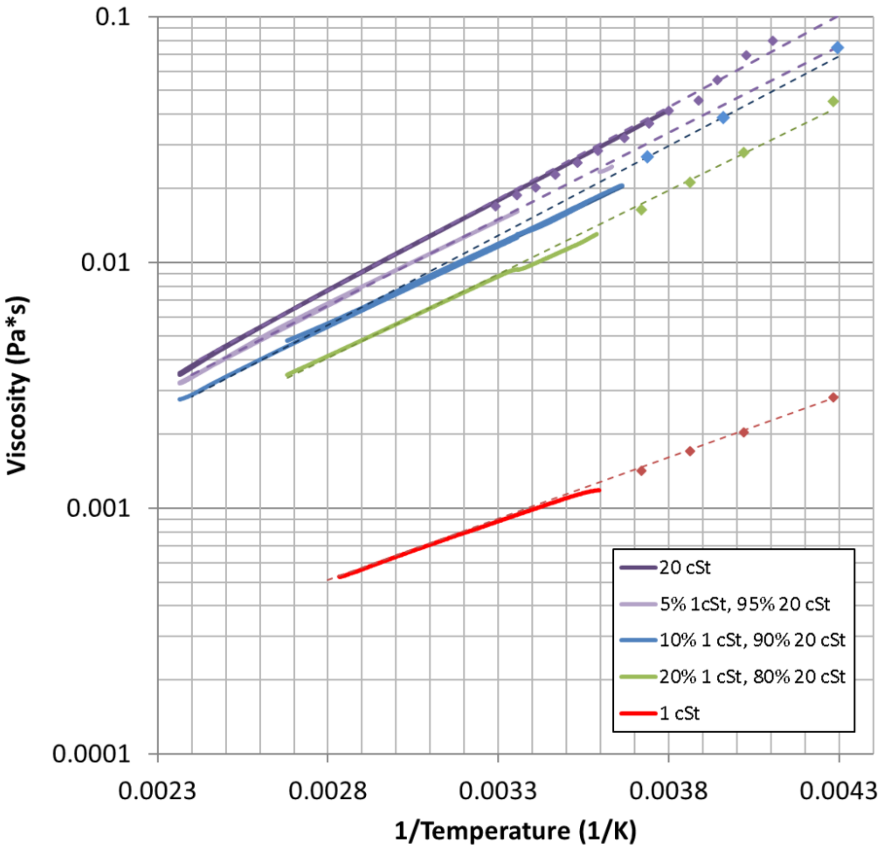


Figure 14. Viscosity of 20 cSt/1 cSt PDMS fluid blends over a wide temperature range (-40 to 150 °C). High-temperature data, denoted with a thick line, were obtained using the AR-G2 rheometer, whereas discrete points at low temperatures were obtained using the capillary viscometer method. Lines of best fit (dotted) for the combined data sets are shown.

Table 4. Viscosity fitting parameters for PDMS blends of 1 cSt and 20 cSt liquids (mass percent cited).

Fluid ID	*A* (log(Pa·s)K) measured	Measured viscosity 25 °C (mPa·s)	Blend prediction 25 °C (mPa·s)
5% 1 cSt, 95% 20 cSt	704±7	(18±1)	16
10% 1 cSt, 90% 20 cSt	727±8	(12±0.5)	14
20% 1 cSt, 80% 20 cSt	682±4	(9.4±0.5)	10

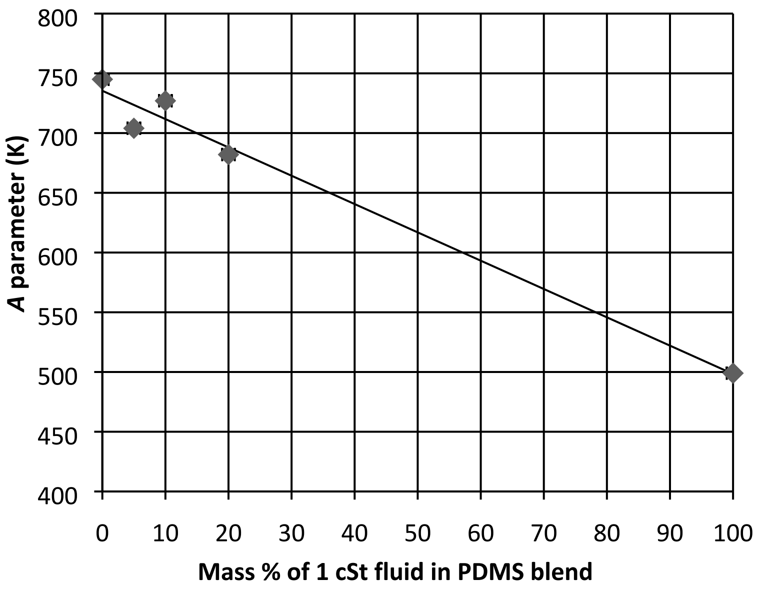


Figure 15. The *A* parameter of the viscosity fit is a linear function of the amount of 1 cSt fluid in the 1 cSt/20 cSt blend.

The room temperature data are compared to the measurements obtained using a capillary viscometer in Figure 16. Also shown is a simple mixing rule prediction for a PDMS blend viscosity (μ_{1+2}) of a blend containing φ fraction of fluid 1 cited by Gelest [12]:

$$\varphi\, log_{10}(\mu_1) + (1 - \varphi) log_{10}(\mu_2) = log_{10}(\mu_{1+2}) \tag{6}$$

Gelest cautions that this mixing rule is valid only for mixtures of PDMS fluids that have viscosities within one order of magnitude. Although blends of 20 cSt and 1 cSt fluids are outside of its cited applicable range, Figure 16 shows that this rule may give an acceptable starting point for the creation of new, targeted viscosity PDMS blends. It seems as though the mixing rule overpredicts the viscosity of 20 cSt/1 cSt PDMS blends by about 7%. Other mixing rules have been attempted to predict the viscosity of oils with varying success [13], [14].

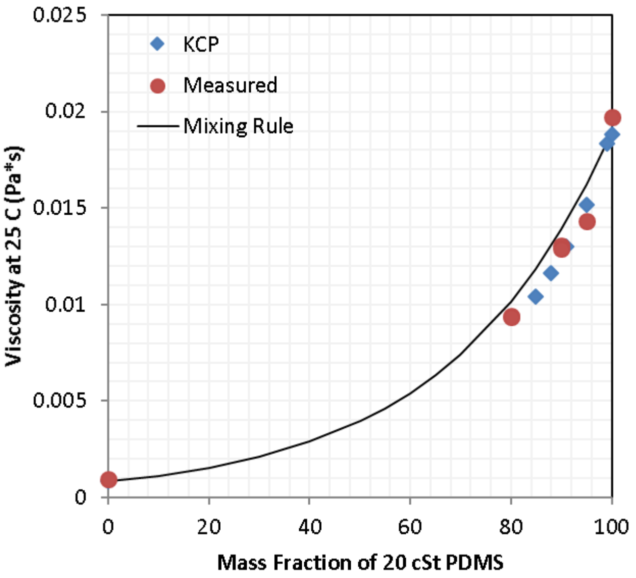


Figure 16. Room temperature viscosity of PDMS fluids measured in this report, by S. Wells [2] and predicted using a simple mixing rule [12].

4. THERMAL PROPERTY MEASUREMENTS

In order to estimate heat transfer through environmental sensing devices, both the thermal conductivity and the specific heat of the PDMS liquids are of interest. However, thermal properties of low-viscosity liquids are difficult to measure. For example, when a temperature gradient is imposed across the sample, as is common in many thermal conductivity measurement techniques, a density gradient is unavoidable in the liquid. This density gradient can drive convection of the liquid during the measurement. This density gradient can lead to instabilities which are undesirable.

In this report, two separate apparatuses designed to measure the thermal conductivity of fluids were compared for their effectiveness in measuring the thermal properties of the target PDMS liquids. The specific heat of PDMS liquids was also measured.

4.1. Methods

Two separate apparatuses designed to measure the thermal conductivity of the fluids were used. The first is a Thermal Conductivity Instruments thermal conductivity analyzer (TCi C-Therm). The measurement device imparts a transient heat flux to the sample using a planar source consisting of a 1.5 cm diameter wire coil, as shown in Figure 17. The sample is contained within a thin well that sits on top of the coil. The decay in the electrical resistance of the wire is then related to the thermal properties of the fluid using a thermal model for heat conduction from a planar source. The apparatus is contained within a Tenney Jr. thermal chamber, which has the ability to control the sample temperature between -73 ^{o}C to 200 ^{o}C. The temperature set point was held constant until the measured sample temperature stabilized at the chamber temperature set point, and then the thermal conductivity was measured at least 10 times at a rate of 1 measurement/minute. These values were then averaged for each data point. This method has a ±5% uncertainty in measuring the thermal conductivity as specified by the manufacturer. This uncertainty was verified using deionized water as a standard, as shown in Figure 17 [15].

The second apparatus was a ThermTest Transient Hot Wire (THW) Liquid Thermal Conductivity Meter. The sample chamber of this apparatus is a closed vessel, allowing the sample to be sealed from evaporation, in contrast to the TCi measurement. A known heat flux is imparted to the fluid using a vertical wire that runs through the middle of the sample chamber. Again, the electrical resistance of the wire is used to determine the temperature of the wire. A simple model for the heat conduction from a cylinder is then used to obtain the thermal conductivity of the surrounding fluid. Tests with deionized water as a standard showed the uncertainty of these measurements to also be within ±5% (Figure 17).

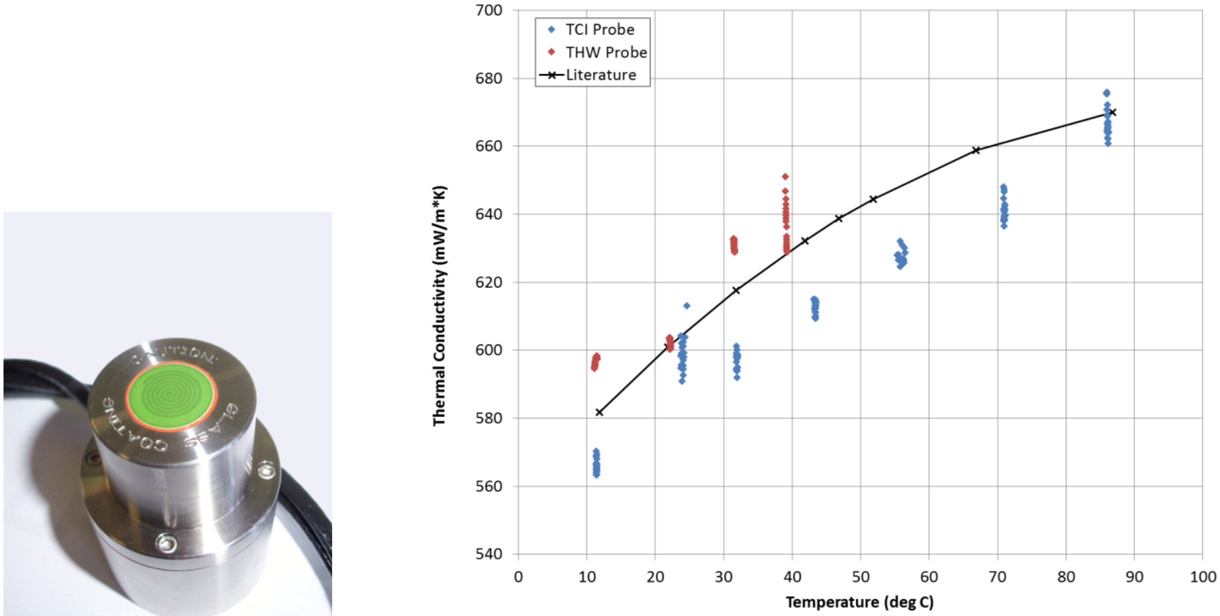


Figure 17. Left: TCi thermal conductivity analyzer showing coil. Right: Thermal conductivity of water measured with both the TCi and THW probes. The literature value is within the stated ±5% uncertainty for both apparatuses.

Figure 18. ThermTest transient hot wire apparatus showing sample holder on the right.

The specific heats of the PDMS liquids were measured using a TA Instruments Q200 differential scanning calorimeter (DSC), which measures the amount of heat required to increase the temperature of a sample as compared to a reference standard. Non-evaporating samples were placed in open aluminum DSC pans. After a 30 minute equilibration period, they were exposed to a thermal ramp rate of 20 ^{o}C/min from -40 ^{o}C to 150 ^{o}C under a flow of dry nitrogen. This cycle was repeated at least five times. A baseline obtained using a sapphire standard was subtracted from the data. Evaporating samples (2 cSt, 5 cSt) were placed in hermetically sealed pans and exposed to a more limited temperature range (-60 ^{o}C to 40 ^{o}C). In all cases, measurements for a baseline, a sapphire reference sample, and the PDMS sample were all collected using the same DSC pan. Then the baseline was subtracted from both the sapphire and the sample. The heat capacity of the sample (*Cp)* was then calculated as

$$Cp_{sample} = \frac{Cp_{sapphire,\,known} \cdot q_{sample}}{q_{sapphire}} \tag{7}$$

Here q is the measured heat flow in W/g for either the sapphire or the sample and $Cp_{sapphire,\,known}$ is given by Reference [16].

4.2. Results

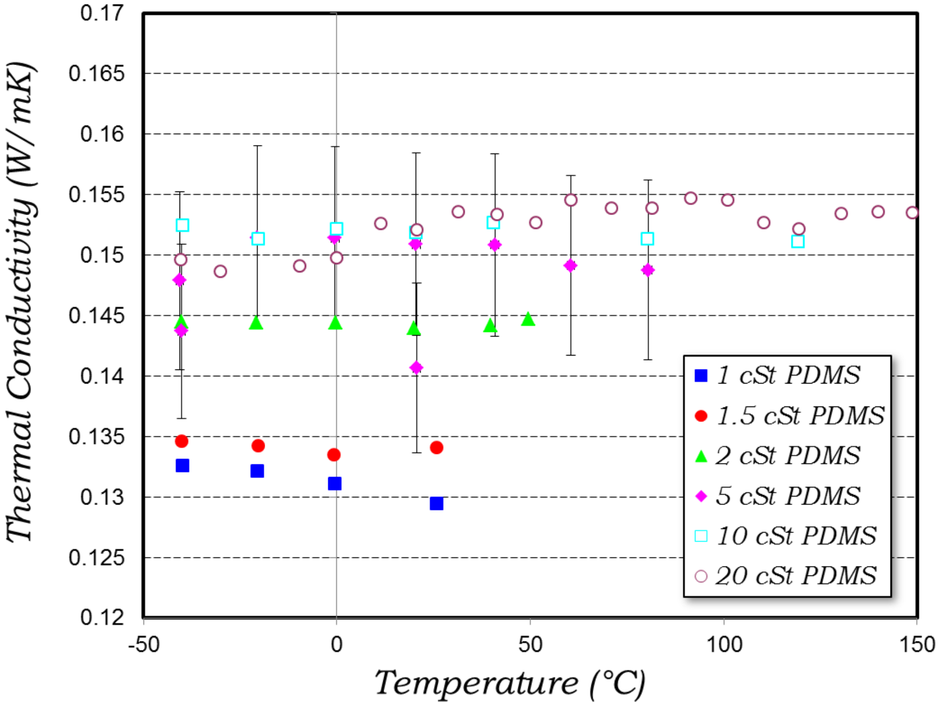


Figure 19. Thermal conductivity of PDMS fluids measured using the TCi apparatus with respect to temperature. Error bars are included for 10 and 5 cSt PDMS only so as to not clutter the graph.

Both apparatuses were able to measure the thermal conductivity of PDMS acceptably below the temperature at which they normally would evaporate. Although the sample chamber of the thermal hot wire probe is sealed and therefore in theory this probe can measure the thermal conductivity of pressurized liquid PDMS, high-temperature data for low-viscosity PDMS liquids were unacceptably noisy and are not included in this report.

Thermal conductivity results for 1 – 20 cSt PDMS fluids are displayed in Figure 19 with 5% error bars plotted for the 20 cSt measurements for reference. Within the expected error, the thermal conductivity of each of the liquids is not a function of temperature within the range of temperatures studied. Therefore, an average thermal conductivity was calculated for each liquid as listed in Table 5 and plotted in Figure 20.

The measured thermal conductivity value for 20 cSt PDMS corresponds closely with values published in the Polymer Data Handbook [17] and also those reported by Clearco (Figure A2). As the room temperature viscosity of PDMS is decreased below 5 cSt, the thermal conductivity of the fluid is also decreased. This is because heat is efficiently transported down the length of a polymer backbone by way of vibrations and chain motion. Clearco values for the heat capacity of low-viscosity fluids are lower than those that were measured; without more information about the provenance of these values, we cannot speculate on the discrepancy.

Table 5. Measured thermal conductivities of PDMS liquids.

PDMS Viscosity (25°C, cSt)	Thermal Conductivity (W/m·K)	Temperature Range (°C)
10 wt% 1cSt in 20cSt	0.147 ± 5%	-40°C – 0°C
1	0.131 ± 5%	-40°C – 25°C
1.5	0.134 ± 5%	-40°C – 25°C
2	0.144 ± 5%	-40°C – 50°C
5	0.148 ± 5%	-40°C – 80°C
10	0.152 ± 5%	-40°C – 120°C
20	0.153 ± 5%	-40°C – 150°C

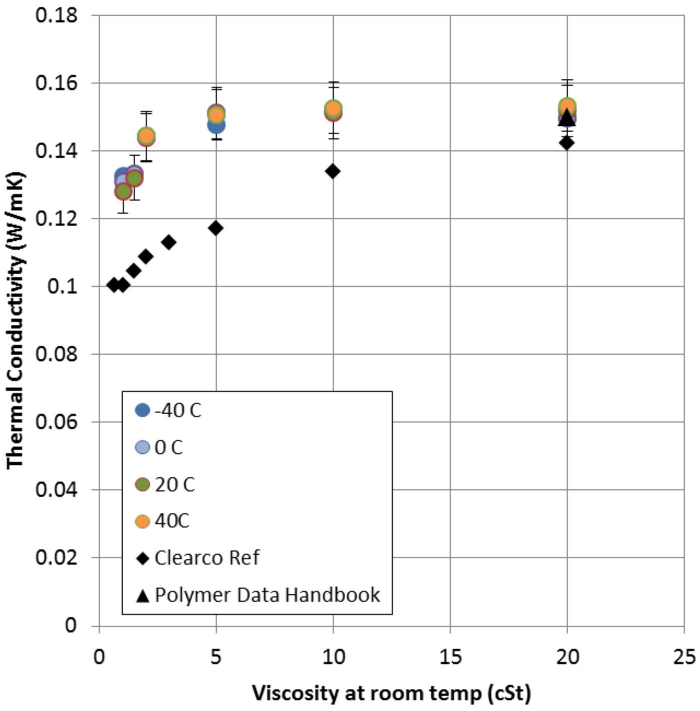


Figure 20. Thermal conductivity of PDMS as a function of room temperature viscosity.

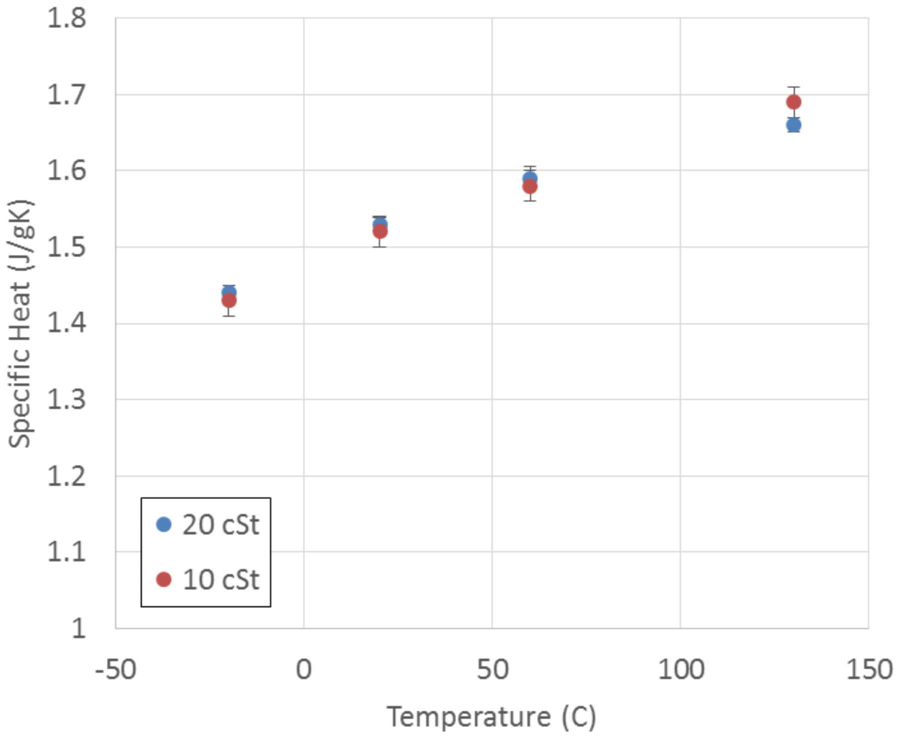


Figure 21. Specific heat of PDMS fluids as a function of temperature.

Table 6. Measured specific heats of PDMS liquids.

	Specific Heat (J/g·K)											
Viscosity	**-20 °C**			**20 °C**			**60 °C**			**130 °C**		
2	1.65	±	0.05	1.71	±	0.05	NA			NA		
5	1.69	±	0.05	1.75	±	0.05	NA			NA		
10	1.43	±	0.02	1.52	±	0.02	1.58	±	0.02	1.69	±	0.02
20	1.44	±	0.01	1.53	±	0.01	1.59	±	0.02	1.66	±	0.01
10% 1 cSt/90% 20 cSt	1.56	±	0.05	1.63	±	0.05	NA			NA		

The heat capacity of PDMS fluids is a mildly increasing function of temperature, as shown in Figure 21. This behavior is characteristic of many polymers; as the temperature increases, more modes of motion are available for the polymer chains, and the heat capacity increases accordingly. Even at low temperatures, the PDMS chains are comparatively flexible, so the change of heat capacity with temperature is less than is reported for other polymers [23]. Recommended heat capacity values and their corresponding uncertainties are reported in Table 6.

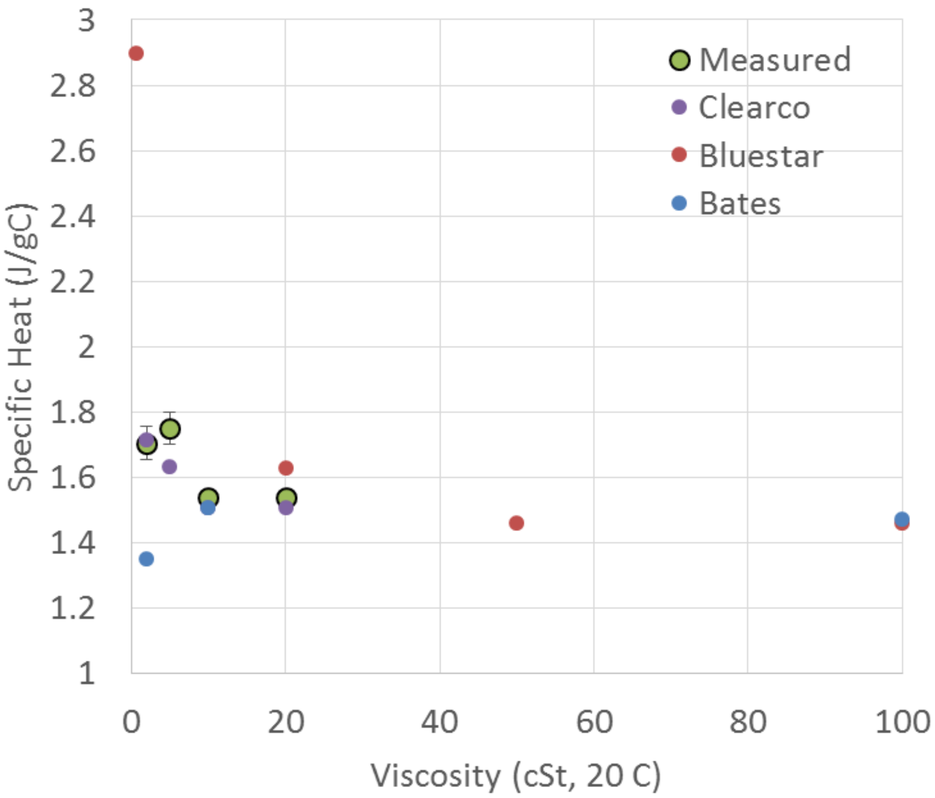


Figure 22. Heat capacity of PDMS fluids at 20 ^oC as a function of room temperature viscosity.

The measured heat capacities of PDMS fluids are shown in Figure 22 along with published values including Clearco [3] and Bluestar Silicones [18] data sheets and values from the peer reviewed literature [19]. Low-molecular weight (low-viscosity, low-density) PDMS fluids have slightly higher specific heat values due to the increased chain mobility of these fluids. As the molecular weight of PDMS increases, the specific heat becomes independent of molecular weight. The measured data correspond well with the published values with the exception of one point at low viscosity published by Bates [19]. It may be that this value was skewed due to evaporation of the liquid during the experiment.

5. SOLUBILITY OF GASES IN PDMS

The solubility of gases in PDMS fluids is a parameter of interest since many ESDs contain bubbles to account for thermal expansion of the liquid inside the device. The solubility of air in PDMS fluids as a function of temperature is one parameter needed to determine the pressure of the bubble throughout temperature excursions.

Preliminary work has been accomplished to create a simple device to measure the solubility of gases in PDMS. The apparatus is shown below in Figure 23. Here, a sample vessel is filled partially with degassed fluid that has been maintained within an evacuated desiccator vessel for weeks. A vacuum is pulled on the sample, and the pressure is monitored over days until it becomes stable at the vapor pressure of the PDMS fluid. Then gas (nitrogen or carbon dioxide) is introduced to the sample chamber to pressurize the system to a predetermined value. As the gas dissolves into the sample fluid, the pressure and temperature of the vessel are monitored over time using an Omega calibrated thermocouple placed on the outside of the sample vessel (Figure 23) and an Omega pressure transducer.

The apparatus has been tested to be free of leaks by observing no measurable pressure drop during a two week hold of a 20 psia initial gas pressure. Leaks were prevented by welding the tubing and sample chamber together. The volume contained between the valves was determined to be 31.85 mL by flowing a known amount of nitrogen into the apparatus, first with the outlet valve closed and second with the inlet valve closed so that the dead volume of the tubing could be accounted for.

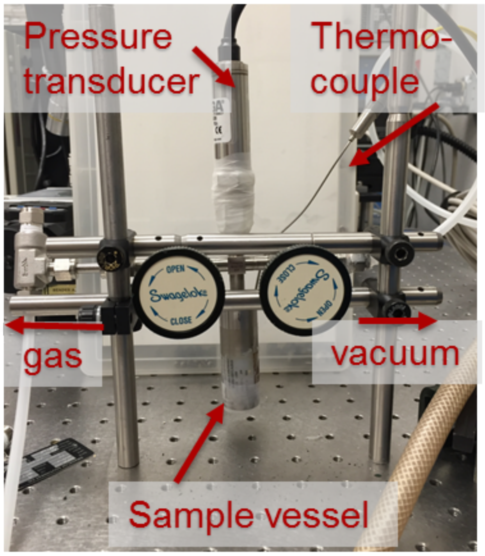


Figure 23. Apparatus for measuring the solubility of gases in PDMS.

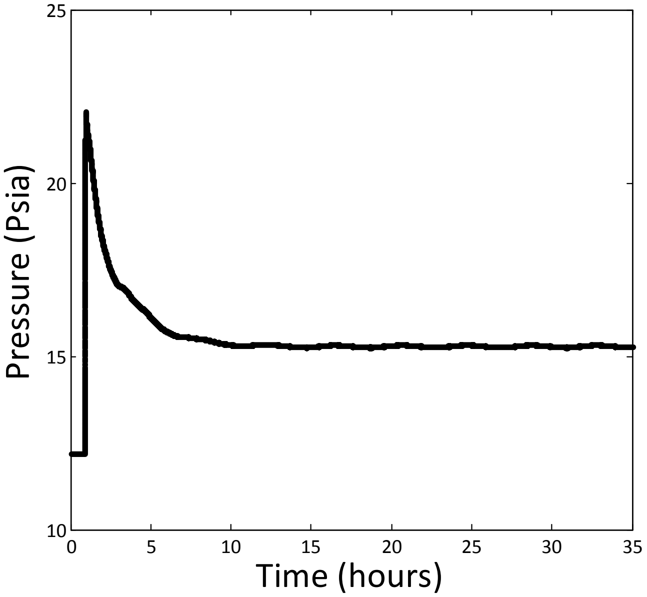


Figure 24. Example pressure decrease (CO_2 dissolves into 20 cSt PDMS).

Table 7. Measured solubilities of gases into PDMS fluids at room temperature (22 °C) as compared to literature values (Lit val 1: [20]; Lit val 2: [21]).

Gas	Initial P	Final P	Solubility	Lit val 1	Lit val 2
	psi	psi	mL/g	mL/g	mL/g
N_2	27.31	25.42	0.18±0.05	0.166	0.15
N_2	27.14	25.25	0.18±0.05	0.166	0.15
CO_2	28.65	19.22	1.207±0.003	1.497	2.2
CO_2	22.07	15.29	1.09±0.003	1.497	2.2

An example of the raw experimental pressure data is shown in Figure 24 for CO_2 introduced to degassed 20 cSt PDMS at room temperature (22 °C). This sample was pressurized with CO_2 to about 23 psia. As the gas dissolved into the PDMS, the headspace pressure drops in the sample chamber. Small fluctuations of pressure in time are correlated with fluctuations in the room temperature as the HVAC system cycles.

Measured solubility values for both nitrogen and CO_2 in 20 cSt PDMS are given in Table 7. Triplicate measurements were done for each. Solubility values are compared to literature values cited by references [20] and [21] for crosslinked PDMS membranes. The measured values are within the same range as the literature values. Although currently there is no temperature control installed on this experimental setup, future plans are to use ovens or freezers to obtain data away from room temperature. These data will be compared to the published data of Shah *et al.* [22].

In order to gather solubility data quickly without a complicated experimental setup, design tradeoffs were made to the experimental apparatus that limit its utility for simultaneously determining the diffusion coefficient of gas through the liquids. Although the relatively large-diameter sample chamber allows for a greater gas/liquid interface for mass transfer, the large-diameter chamber also does not prevent convection of the fluid. Comparing the expected diffusion time of the gas into the PDMS using published diffusion coefficients [17], [23], [24] with the experimentally observed equilibration time, it is clear that convection in the apparatus is likely. Convection would occur if, for example, gas-saturated PDMS had a higher density than degassed PDMS, which would drive a Rayleigh-Taylor instability type flow. Convection could be slowed by investigating higher-viscosity PDMS fluids or by decreasing the radius of the sample chamber dramatically. Another disadvantage of the current setup is that the change of density of PDMS with gas concentration is unaccounted for. This density change is suspected to be a factor in the case of CO_2 dissolving into the fluid and could be measured directly using pycnometry of PDMS containing different concentrations of gas.

6. CONCLUSIONS

Low-molecular weight polydimethylsiloxanes are widely used in industry as lubricants and defoamers, within the nuclear weapons community as inert fluids, and within academia as standard non-aqueous test liquids that can be obtained for a wide range of viscosities. Yet, despite their wide use, it seems as though published property measurements on these fluids are mostly found in technical data sheets without citations for how the data were collected. Here, a variety of fluid properties were characterized from -40 ^{o}C to 150 ^{o}C in order to inform models of component performance.

When combined, property measurements of PDMS fluids demonstrate the uniqueness of these polymers. Nearly all of the measured physical properties (density, thermal expansion coefficient, thermal conductivity, specific heat) are independent of the polymer chain length above a certain threshold. As the polymer chain grows, there is less influence of the end groups, and the entire chain on average behaves homogeneously. A notable exception to this rule is viscosity, which is affected by the ability of entangled polymer chains to move past one another; this property is influenced by molecular weight greatly. Many properties are also temperature-independent ($d\rho/dT_p$, thermal conductivity). Although viscosity, density, and specific heat are functions of temperature, their dependence is much less than that of a polymer with a carbon backbone, for example. In fact, PDMS fluids remain a liquid at much lower temperatures than hydrocarbons and do not experience glass transitions until well below -100 ^{o}C [25]. This behavior is due to the relative flexibility of the PDMS chain, even at low temperatures, due to small methyl side groups and the relatively long Si-O bond [25]. That is, it does not require much thermal energy for the PDMS polymer chain to be in motion or for its atoms to vibrate. Physical properties are monotonic with temperature due to the lack of complicated side chain moieties, branching, or crosslinking whose motion could be activated at intermediate temperatures. These physical properties of PDMS make it a good polymeric fluid choice for weapons systems, which are expected to perform in many different environmental conditions.

7. REFERENCES

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APPENDIX A

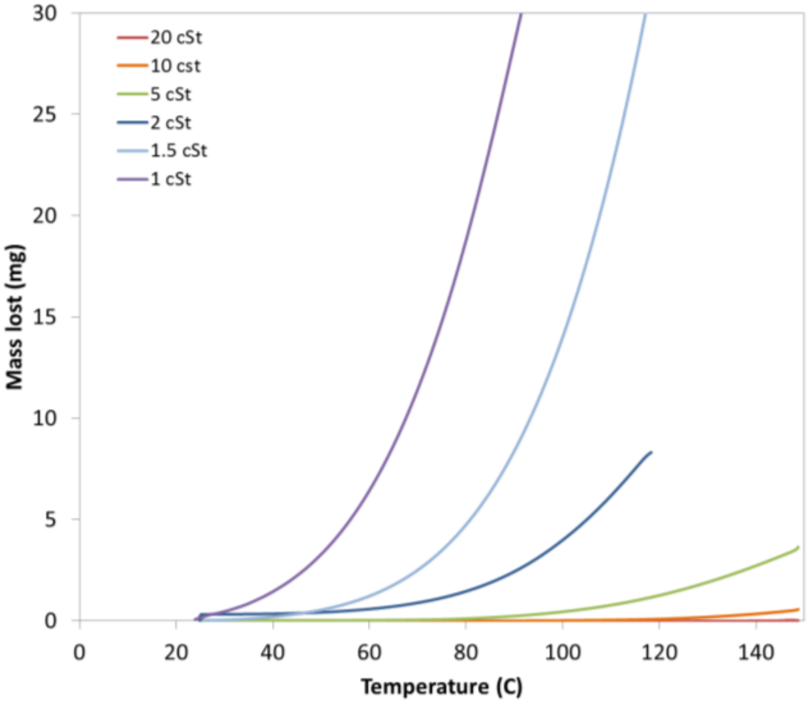
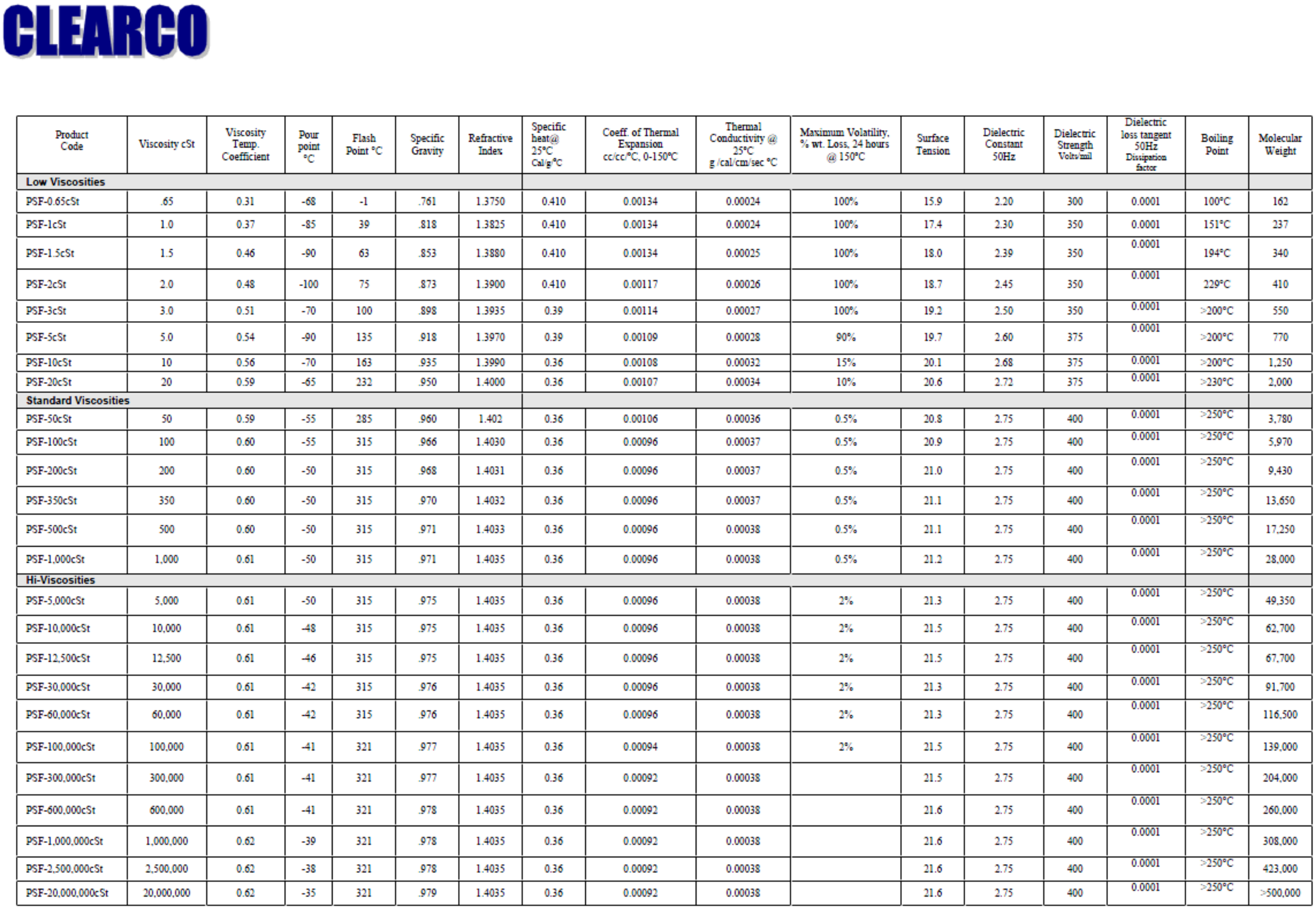


Figure A 1. Mass lost from PDMS samples of varying viscosity from a thermogravimetric analysis pan under flowing nitrogen at 20 ^{o}C/min ramp rate. PDMS fluids with viscosities less than 5 cSt lost significant mass in the temperature range.



CLEARCO

Product Code	Viscosity cSt	Viscosity Temp. Coefficient	Pour point °C	Flash Point °C	Specific Gravity	Refractive Index	Specific heat@ 25°C Cal/g/°C	Coeff. of Thermal Expansion cc/cc/°C, 0-150°C	Thermal Conductivity @ 25°C g /cal/cm/sec °C	Maximum Volatility, % wt. Loss, 24 hours @ 150°C	Surface Tension	Dielectric Constant 50Hz	Dielectric Strength Volts/mil	Dielectric loss tangent 50Hz Dissipation factor	Boiling Point	Molecular Weight
Low Viscosities																
PSF-0.65cSt	.65	0.31	-68	-1	.761	1.3750	0.410	0.00134	0.00024	100%	15.9	2.20	300	0.0001	100°C	162
PSF-1cSt	1.0	0.37	-85	39	.818	1.3825	0.410	0.00134	0.00024	100%	17.4	2.30	350	0.0001	151°C	237
PSF-1.5cSt	1.5	0.46	-90	63	.853	1.3880	0.410	0.00134	0.00025	100%	18.0	2.39	350	0.0001	194°C	340
PSF-2cSt	2.0	0.48	-100	75	.873	1.3900	0.410	0.00117	0.00026	100%	18.7	2.45	350	0.0001	229°C	410
PSF-3cSt	3.0	0.51	-70	100	.898	1.3935	0.39	0.00114	0.00027	100%	19.2	2.50	350	0.0001	>200°C	550
PSF-5cSt	5.0	0.54	-90	135	.918	1.3970	0.39	0.00109	0.00028	90%	19.7	2.60	375	0.0001	>200°C	770
PSF-10cSt	10	0.56	-70	163	.935	1.3990	0.36	0.00108	0.00032	15%	20.1	2.68	375	0.0001	>200°C	1,250
PSF-20cSt	20	0.59	-65	232	.950	1.4000	0.36	0.00107	0.00034	10%	20.6	2.72	375	0.0001	>230°C	2,000
Standard Viscosities																
PSF-50cSt	50	0.59	-55	285	.960	1.402	0.36	0.00106	0.00036	0.5%	20.8	2.75	400	0.0001	>250°C	3,780
PSF-100cSt	100	0.60	-55	315	.966	1.4030	0.36	0.00096	0.00037	0.5%	20.9	2.75	400	0.0001	>250°C	5,970
PSF-200cSt	200	0.60	-50	315	.968	1.4031	0.36	0.00096	0.00037	0.5%	21.0	2.75	400	0.0001	>250°C	9,430
PSF-350cSt	350	0.60	-50	315	.970	1.4032	0.36	0.00096	0.00037	0.5%	21.1	2.75	400	0.0001	>250°C	13,650
PSF-500cSt	500	0.60	-50	315	.971	1.4033	0.36	0.00096	0.00038	0.5%	21.1	2.75	400	0.0001	>250°C	17,250
PSF-1,000cSt	1,000	0.61	-50	315	.971	1.4035	0.36	0.00096	0.00038	0.5%	21.2	2.75	400	0.0001	>250°C	28,000
Hi-Viscosities																
PSF-5,000cSt	5,000	0.61	-50	315	.975	1.4035	0.36	0.00096	0.00038	2%	21.3	2.75	400	0.0001	>250°C	49,350
PSF-10,000cSt	10,000	0.61	-48	315	.975	1.4035	0.36	0.00096	0.00038	2%	21.5	2.75	400	0.0001	>250°C	62,700
PSF-12,500cSt	12,500	0.61	-46	315	.975	1.4035	0.36	0.00096	0.00038	2%	21.5	2.75	400	0.0001	>250°C	67,700
PSF-30,000cSt	30,000	0.61	-42	315	.976	1.4035	0.36	0.00096	0.00038	2%	21.3	2.75	400	0.0001	>250°C	91,700
PSF-60,000cSt	60,000	0.61	-42	315	.976	1.4035	0.36	0.00096	0.00038	2%	21.3	2.75	400	0.0001	>250°C	116,500
PSF-100,000cSt	100,000	0.61	-41	321	.977	1.4035	0.36	0.00094	0.00038	2%	21.5	2.75	400	0.0001	>250°C	139,000
PSF-300,000cSt	300,000	0.61	-41	321	.977	1.4035	0.36	0.00092	0.00038		21.5	2.75	400	0.0001	>250°C	204,000
PSF-600,000cSt	600,000	0.61	-41	321	.978	1.4035	0.36	0.00092	0.00038		21.6	2.75	400	0.0001	>250°C	260,000
PSF-1,000,000cSt	1,000,000	0.62	-39	321	.978	1.4035	0.36	0.00092	0.00038		21.6	2.75	400	0.0001	>250°C	308,000
PSF-2,500,000cSt	2,500,000	0.62	-38	321	.978	1.4035	0.36	0.00092	0.00038		21.6	2.75	400	0.0001	>250°C	423,000
PSF-20,000,000cSt	20,000,000	0.62	-35	321	.979	1.4035	0.36	0.00092	0.00038		21.6	2.75	400	0.0001	>250°C	>500,000

Figure A 2. Clearco data sheet for PDMS fluids [3].

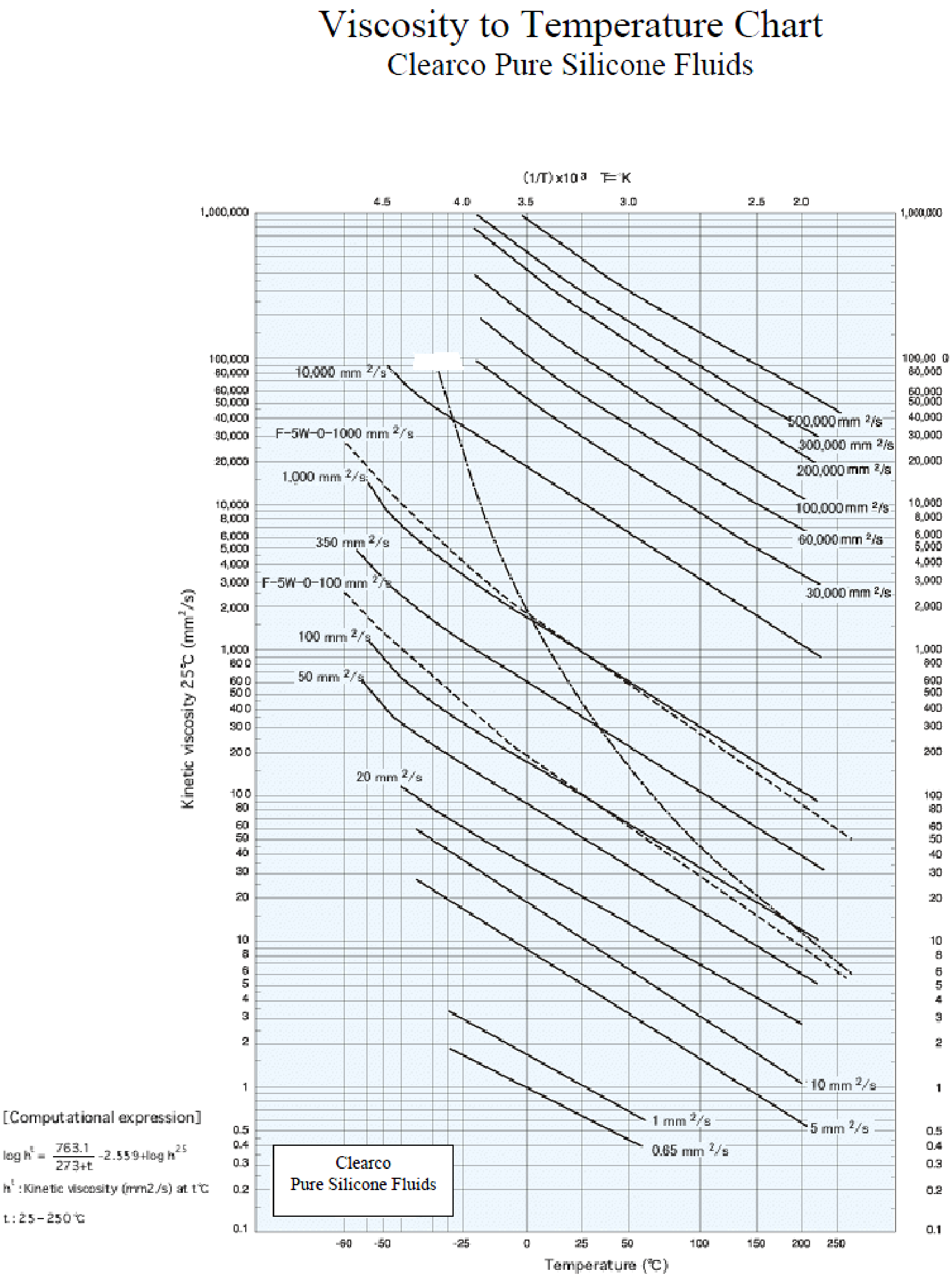


Figure A 3. Viscosity temperature chart for PDMS fluids published by Clearco [3].

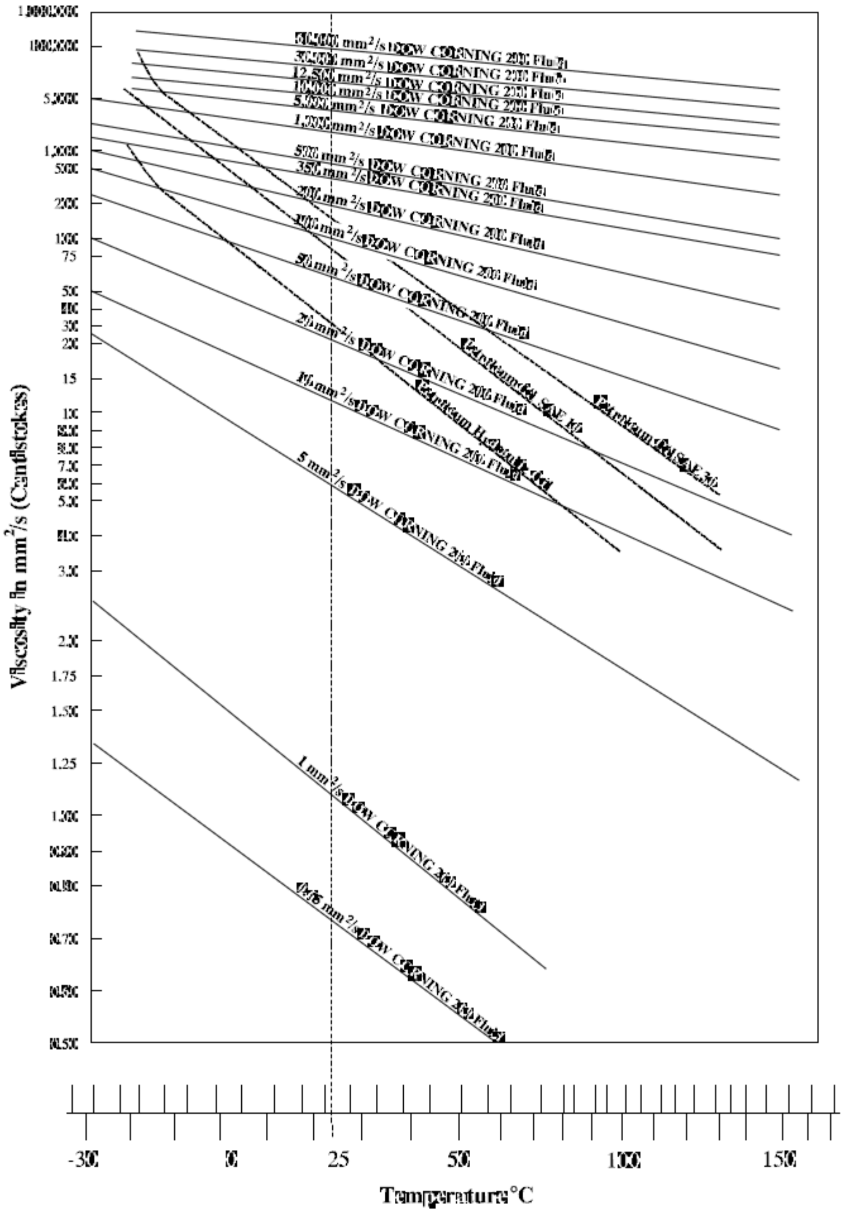


Figure A 4. Viscosity temperature chart for PDMS fluids published by Dow Corning [26].

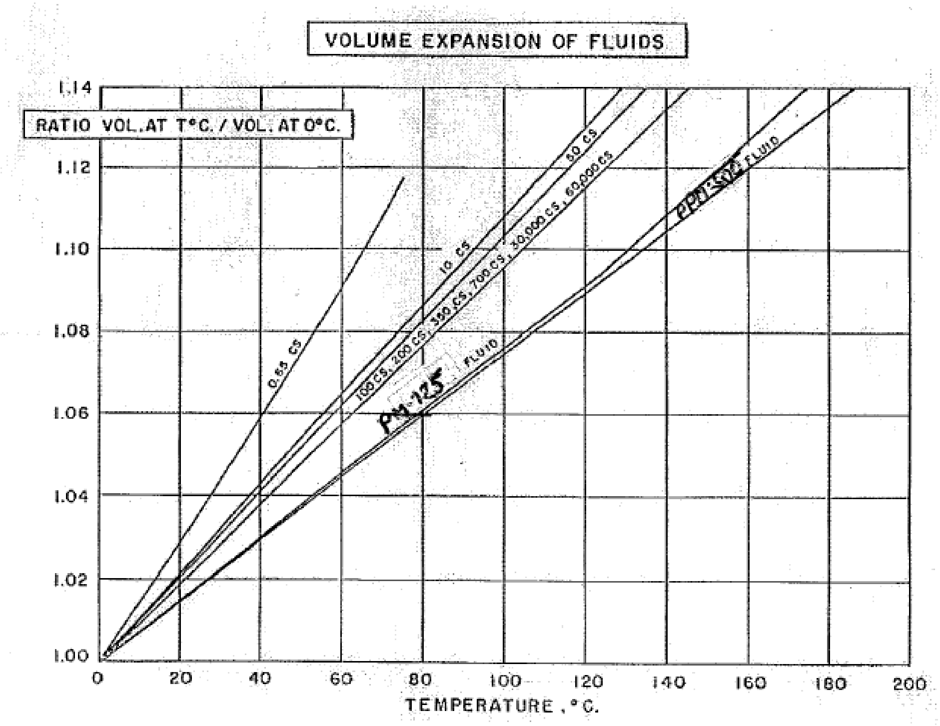
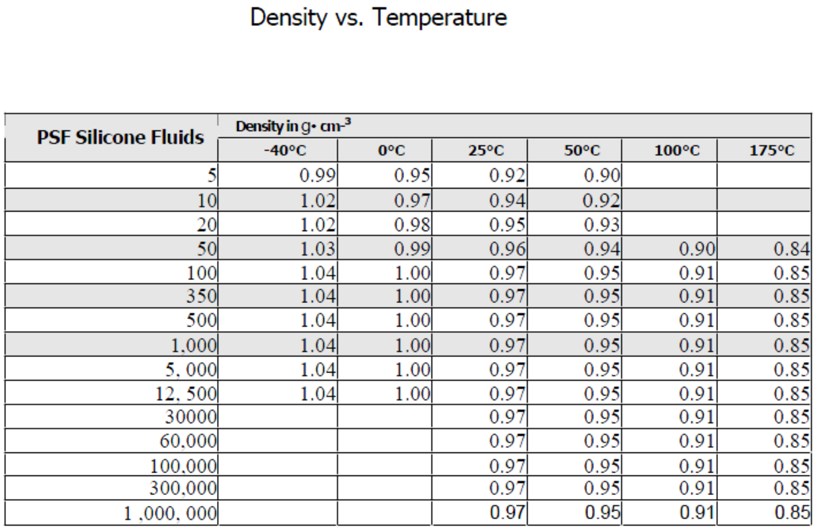


Figure A 5. Thermal expansion of PDMS fluids published by Clearco [3].



Density vs. Temperature

PSF Silicone Fluids	Density in g• cm-3 -40°C	0°C	25°C	50°C	100°C	175°C
5	0.99	0.95	0.92	0.90		
10	1.02	0.97	0.94	0.92		
20	1.02	0.98	0.95	0.93		
50	1.03	0.99	0.96	0.94	0.90	0.84
100	1.04	1.00	0.97	0.95	0.91	0.85
350	1.04	1.00	0.97	0.95	0.91	0.85
500	1.04	1.00	0.97	0.95	0.91	0.85
1,000	1.04	1.00	0.97	0.95	0.91	0.85
5, 000	1.04	1.00	0.97	0.95	0.91	0.85
12, 500	1.04	1.00	0.97	0.95	0.91	0.85
30000			0.97	0.95	0.91	0.85
60,000			0.97	0.95	0.91	0.85
100,000			0.97	0.95	0.91	0.85
300,000			0.97	0.95	0.91	0.85
1 ,000, 000			0.97	0.95	0.91	0.85

Figure A 6. Density data provided by Clearco [1].

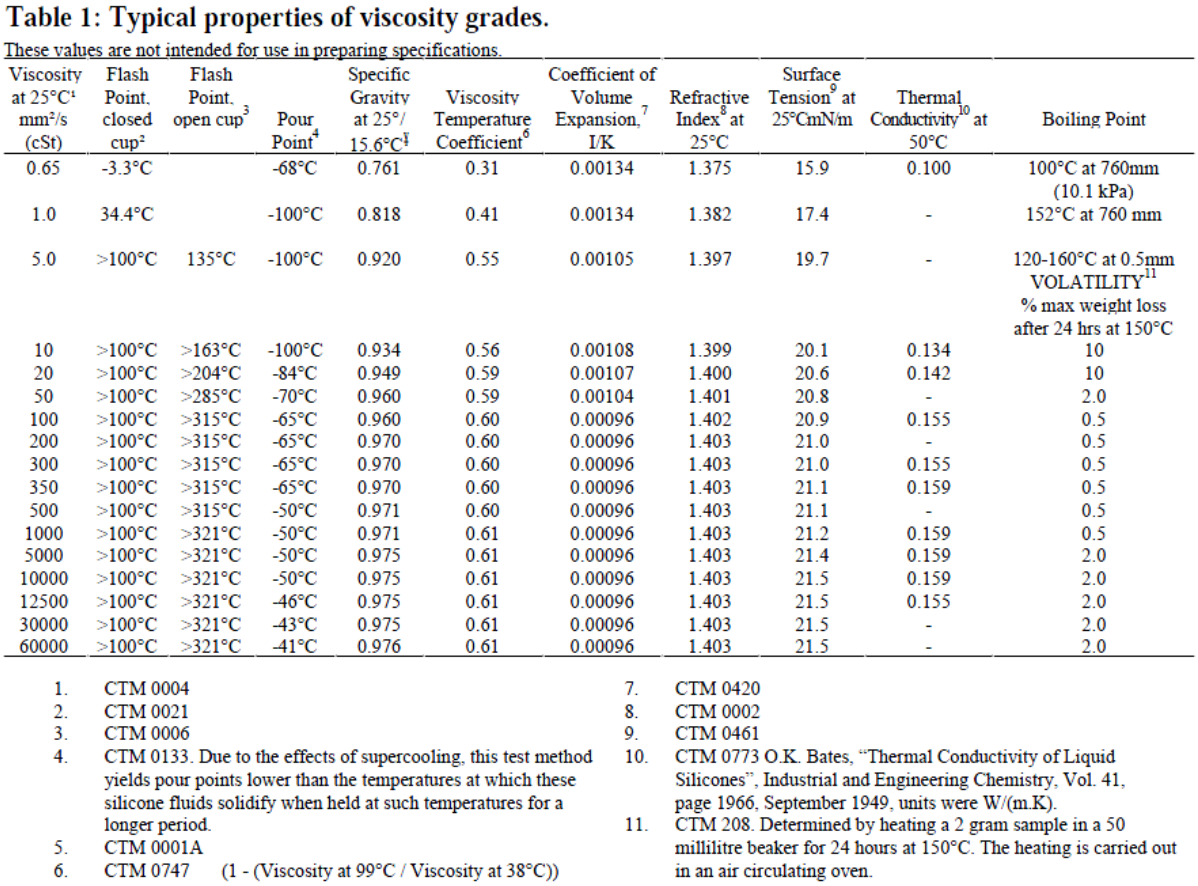


Table 1: Typical properties of viscosity grades.

These values are not intended for use in preparing specifications.

Viscosity at 25°C[1] mm²/s (cSt)	Flash Point, closed cup[2]	Flash Point, open cup[3]	Pour Point[4]	Specific Gravity at 25°/ 15.6°C[5]	Viscosity Temperature Coefficient[6]	Coefficient of Volume Expansion,[7] I/K	Refractive Index[8] at 25°C	Surface Tension[9] at 25°CmN/m	Thermal Conductivity[10] at 50°C	Boiling Point
0.65	-3.3°C		-68°C	0.761	0.31	0.00134	1.375	15.9	0.100	100°C at 760mm (10.1 kPa)
1.0	34.4°C		-100°C	0.818	0.41	0.00134	1.382	17.4	-	152°C at 760 mm
5.0	>100°C	135°C	-100°C	0.920	0.55	0.00105	1.397	19.7	-	120-160°C at 0.5mm
										VOLATILITY[11] % max weight loss after 24 hrs at 150°C
10	>100°C	>163°C	-100°C	0.934	0.56	0.00108	1.399	20.1	0.134	10
20	>100°C	>204°C	-84°C	0.949	0.59	0.00107	1.400	20.6	0.142	10
50	>100°C	>285°C	-70°C	0.960	0.59	0.00104	1.401	20.8	-	2.0
100	>100°C	>315°C	-65°C	0.960	0.60	0.00096	1.402	20.9	0.155	0.5
200	>100°C	>315°C	-65°C	0.970	0.60	0.00096	1.403	21.0	-	0.5
300	>100°C	>315°C	-65°C	0.970	0.60	0.00096	1.403	21.0	0.155	0.5
350	>100°C	>315°C	-65°C	0.970	0.60	0.00096	1.403	21.1	0.159	0.5
500	>100°C	>315°C	-50°C	0.971	0.60	0.00096	1.403	21.1	-	0.5
1000	>100°C	>321°C	-50°C	0.971	0.61	0.00096	1.403	21.2	0.159	0.5
5000	>100°C	>321°C	-50°C	0.975	0.61	0.00096	1.403	21.4	0.159	2.0
10000	>100°C	>321°C	-50°C	0.975	0.61	0.00096	1.403	21.5	0.159	2.0
12500	>100°C	>321°C	-46°C	0.975	0.61	0.00096	1.403	21.5	0.155	2.0
30000	>100°C	>321°C	-43°C	0.975	0.61	0.00096	1.403	21.5	-	2.0
60000	>100°C	>321°C	-41°C	0.976	0.61	0.00096	1.403	21.5	-	2.0

1. CTM 0004
2. CTM 0021
3. CTM 0006
4. CTM 0133. Due to the effects of supercooling, this test method yields pour points lower than the temperatures at which these silicone fluids solidify when held at such temperatures for a longer period.
5. CTM 0001A
6. CTM 0747 (1 - (Viscosity at 99°C / Viscosity at 38°C))
7. CTM 0420
8. CTM 0002
9. CTM 0461
10. CTM 0773 O.K. Bates, "Thermal Conductivity of Liquid Silicones", Industrial and Engineering Chemistry, Vol. 41, page 1966, September 1949, units were W/(m.K).
11. CTM 208. Determined by heating a 2 gram sample in a 50 millilitre beaker for 24 hours at 150°C. The heating is carried out in an air circulating oven.

Figure A 7. Various data provided by Dow Corning [26].

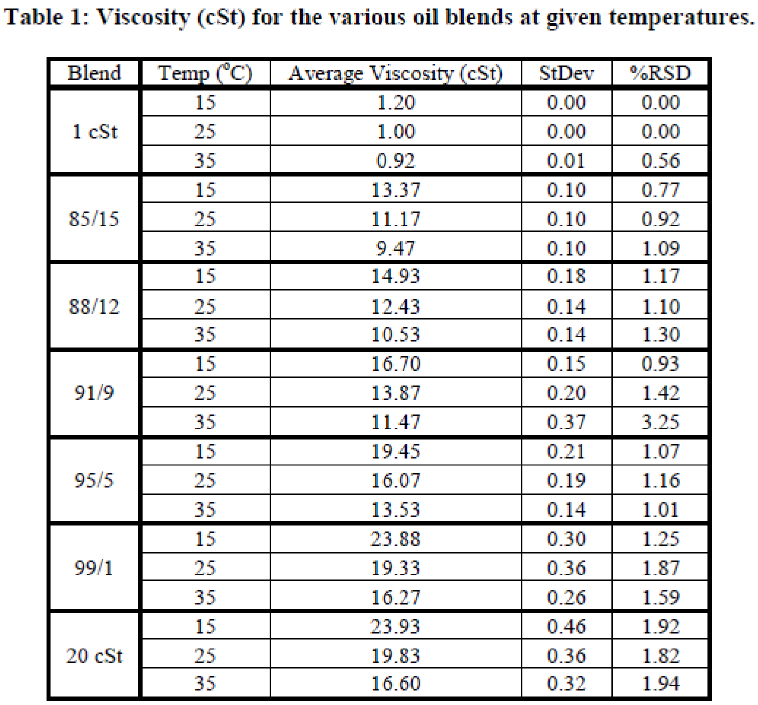


Table 1: Viscosity (cSt) for the various oil blends at given temperatures.

Blend	Temp (°C)	Average Viscosity (cSt)	StDev	%RSD
1 cSt	15	1.20	0.00	0.00
	25	1.00	0.00	0.00
	35	0.92	0.01	0.56
85/15	15	13.37	0.10	0.77
	25	11.17	0.10	0.92
	35	9.47	0.10	1.09
88/12	15	14.93	0.18	1.17
	25	12.43	0.14	1.10
	35	10.53	0.14	1.30
91/9	15	16.70	0.15	0.93
	25	13.87	0.20	1.42
	35	11.47	0.37	3.25
95/5	15	19.45	0.21	1.07
	25	16.07	0.19	1.16
	35	13.53	0.14	1.01
99/1	15	23.88	0.30	1.25
	25	19.33	0.36	1.87
	35	16.27	0.26	1.59
20 cSt	15	23.93	0.46	1.92
	25	19.83	0.36	1.82
	35	16.60	0.32	1.94

Figure A 8. Viscosity data obtained by Sabrina Wells, Honeywell National Security Campus [2].

APPENDIX B

Table B1. Measured viscosity of pure PDMS liquids.

1.5 cSt PDMS				2 cSt PDMS				5 cSt PDMS			
T [C]	mu [Pa*s]	log10(mu)	Method	T [C]	mu [Pa*s]	log10(mu)	Method	T [C]	mu [Pa*s]	log10(mu)	Method
0.1	0.00204	-2.69	rheometer	0	0.00286	-2.54	viscometer	5.326	0.00668	-2.18	viscometer
5	0.00187	-2.73	rheometer	5	0.00261	-2.58	viscometer	19.96	0.00512	-2.29	viscometer
10	0.00172	-2.76	rheometer	10	0.00240	-2.62	viscometer	-14.84	0.01070	-1.97	viscometer
15	0.00161	-2.79	rheometer	15	0.00222	-2.65	viscometer	-39.84	0.02002	-1.70	viscometer
20	0.00150	-2.82	rheometer	20	0.00206	-2.69	viscometer	0.0	0.00749	-2.13	rheometer
25	0.00142	-2.85	rheometer	25	0.00195	-2.71	viscometer	10.0	0.00627	-2.20	rheometer
30	0.00136	-2.87	rheometer	30	0.00185	-2.73	viscometer	20.0	0.00529	-2.28	rheometer
35	0.00127	-2.90	rheometer	35	0.00173	-2.76	viscometer	30.0	0.00451	-2.35	rheometer
40	0.00119	-2.92	rheometer	40	0.00161	-2.79	viscometer	40.0	0.00386	-2.41	rheometer
45	0.00112	-2.95	rheometer	45	0.00150	-2.82	viscometer	50.0	0.00333	-2.48	rheometer
50	0.00106	-2.97	rheometer	50	0.00142	-2.85	viscometer	60.0	0.00289	-2.54	rheometer
55.0	0.00100	-3.00	rheometer	55.0	0.00133	-2.88	rheometer	70.0	0.00254	-2.60	rheometer
60.0	0.00094	-3.03	rheometer	60.0	0.00160	-2.80	rheometer	80.0	0.00224	-2.65	rheometer
65.0	0.00089	-3.05	rheometer	65.0	0.00118	-2.93	rheometer	90.0	0.00199	-2.70	rheometer
70.0	0.00084	-3.07	rheometer	70.0	0.00112	-2.95	rheometer	100.0	0.00177	-2.75	rheometer
75.0	0.00080	-3.10	rheometer	75.0	0.00105	-2.98	rheometer				
80.0	0.00075	-3.12	rheometer	80.0	0.00099	-3.00	rheometer				

20 cSt PDMS				50 cSt PDMS			
T [C]	mu [Pa*s]	log10(mu)	Method	T [C]	mu [Pa*s]	log10(mu)	Method
5.5	0.02842	-1.55	viscometer	30	0.04157	-1.38	rheometer
10.2	0.02544	-1.59	viscometer	35	0.03812	-1.42	rheometer
15.4	0.02282	-1.64	viscometer	40	0.03498	-1.46	rheometer
20.3	0.02017	-1.70	viscometer	45	0.03232	-1.49	rheometer
25.1	0.01870	-1.73	viscometer	50.0	0.02984	-1.53	rheometer
30.8	0.01698	-1.77	viscometer	55.0	0.02771	-1.56	rheometer
-15.8	0.04555	-1.34	viscometer	60.0	0.02579	-1.59	rheometer
-39.8	0.10121	-0.99	viscometer	65.0	0.02400	-1.62	rheometer
-5.8	0.03679	-1.43	viscometer	70.0	0.02234	-1.65	rheometer
-9.8	0.04135	-1.38	viscometer	75.0	0.02083	-1.68	rheometer
-19.4	0.05527	-1.26	viscometer	80.0	0.01956	-1.71	rheometer
-29.3	0.08007	-1.10	viscometer	85.0	0.01833	-1.74	rheometer
-0.5	0.03202	-1.49	viscometer	90.0	0.01703	-1.77	rheometer
-24.7	0.06950	-1.16	viscometer	95.0	0.01611	-1.79	rheometer
-39.0	0.10563	-0.98	viscometer	100.0	0.01501	-1.82	rheometer
-5.0	0.03700	-1.43	rheometer	105	0.01419	-1.85	rheometer
5.0	0.02951	-1.53	rheometer	110	0.01324	-1.88	rheometer
15.0	0.02451	-1.61	rheometer	115	0.0126	-1.90	rheometer
25.0	0.01970	-1.71	rheometer	120	0.01186	-1.93	rheometer
35.0	0.01642	-1.78	rheometer	125	0.01112	-1.95	rheometer
45.0	0.01386	-1.86	rheometer	130	0.01058	-1.98	rheometer
55.0	0.01181	-1.93	rheometer	135	0.009917	-2.00	rheometer
65.0	0.01008	-2.00	rheometer	140	0.009487	-2.02	rheometer
75.0	0.00873	-2.06	rheometer	145	0.008969	-2.05	rheometer
85.0	0.00765	-2.12	rheometer	150	0.008496	-2.07	rheometer
95.0	0.00667	-2.18	rheometer				
105.0	0.00590	-2.23	rheometer				
115.0	0.00525	-2.28	rheometer				
125.0	0.00476	-2.32	rheometer				
135.0	0.00419	-2.38	rheometer				
145.0	0.00367	-2.43	rheometer				
150.0	0.00355	-2.45	rheometer				

Table B2. Measured viscosity of blends of PDMS liquids.

1 cSt PDMS				5% 1 cSt/95 % 20 cSt			
T [C]	**mu [Pa*s]**	**log10(mu)**	**Method**	**T [C]**	**mu [Pa*s]**	**log10(mu)**	**Method**
-4.16	0.00142	-2.85	viscometer	0.2	0.02589	-1.59	rheometer
-14.06	0.00170	-2.77	viscometer	2.2	0.02495	-1.60	rheometer
-24.26	0.00204	-2.69	viscometer	5.0	0.02371	-1.63	rheometer
-39.46	0.00283	-2.55	viscometer	25.0	0.016042	-1.79	rheometer
5.0	0.00118	-2.93	rheometer	30.0	0.014653	-1.83	rheometer
15.0	0.00107	-2.97	rheometer	35.0	0.013425	-1.87	rheometer
25.0	0.00094	-3.03	rheometer	40.0	0.01242	-1.91	rheometer
35.0	0.00083	-3.08	rheometer	45.0	0.011363	-1.94	rheometer
45.0	0.00075	-3.13	rheometer	50.0	0.01051	-1.98	rheometer
55.0	0.00067	-3.17	rheometer	55.0	0.00967	-2.01	rheometer
65.0	0.00060	-3.22	rheometer	60.0	0.008941	-2.05	rheometer
75.0	0.00055	-3.26	rheometer	65.0	0.008284	-2.08	rheometer
80.0	0.00052	-3.28	rheometer	70.0	0.007707	-2.11	rheometer
				75.0	0.007159	-2.15	rheometer
				80.0	0.006672	-2.18	rheometer

10% 1 cSt/90 % 20 cSt				20% 1 cSt/80 % 20 cSt			
T [C]	**mu [Pa*s]**	**log10(mu)**	**Method**	**T [C]**	**mu [Pa*s]**	**log10(mu)**	**Method**
-40.16	0.07511	-1.12	viscometer	-4.16	0.01637	-1.79	viscometer
-20.46	0.03876	-1.41	viscometer	-14.06	0.02110	-1.68	viscometer
-5.36	0.02699	-1.57	viscometer	-24.26	0.02809	-1.55	viscometer
2.4	0.01977	-1.70	rheometer	-39.46	0.04514	-1.35	viscometer
10.0	0.01669	-1.78	rheometer	10.0	0.01188	-1.35	rheometer
15.0	0.01506	-1.82	rheometer	15.0	0.01089	-1.35	rheometer
25.0	0.01292	-1.89	rheometer	20.0	0.01003	-1.35	rheometer
35.0	0.011	-1.96	rheometer	25.0	0.00936	-1.35	rheometer
40.0	0.01017	-1.99	rheometer	30.0	0.00877	-1.35	rheometer
45.0	0.00946	-2.02	rheometer	35.0	0.00812	-1.35	rheometer
50.0	0.00879	-2.06	rheometer	40.0	0.00751	-1.35	rheometer
55.0	0.00818	-2.09	rheometer	45.0	0.00697	-1.35	rheometer
60.0	0.00763	-2.12	rheometer	50.0	0.00646	-1.35	rheometer
65.0	0.00713	-2.15	rheometer	55.0	0.00601	-1.35	rheometer
70.0	0.00669	-2.17	rheometer	60.0	0.00562	-1.35	rheometer
75.0	0.00628	-2.20	rheometer	65.0	0.00525	-1.35	rheometer
80.0	0.00592	-2.23	rheometer	70.0	0.00493	-1.35	rheometer
				75.0	0.00463	-1.35	rheometer
				80.0	0.00436	-1.35	rheometer

Distribution

1	MS 0333	A. Morris-Eckart	2616
1	MS 0333	G. L. Benavides	2616
1	MS 0333	M. G. Goff	2616
1	MS 0333	M. D. Williams	2616
1	MS 0346	M. B. Nemer	1512
1	MS 0346	C. C. Roberts	1512
1	MS 0346	L. M. Phinney	1514
1	MS 0349	C. W. Vanecek	2613
1	MS 0350	C. M. Siefert	2615
1	MS 0350	R. S. Colbert	2613
1	MS 0350	D. Groysman	2613
1	MS 0350	D. E. Petersen	2613
1	MS 0350	R. R. Parker	2615
1	MS 0350	M. P. Sena	2615
1	MS 0825	J. L. Payne	1513
1	MS 0825	E. K. Stirrup	1513
1	MS 0828	B. Hassan	1510
1	MS 0828	T. L. Durbin	1512
1	MS 0828	J. R. Clausen	1516
1	MS 0840	T. J. O'Hern	1512
1	MS 0840	J. R. Torczynski	1513
1	MS 0871	R. M. Garcia	2726
1	MS 1064	M. J. Parrish	2615
1	MS 1064	W. Y.Waller	2615
1	MS 1064	J. T. McLaughlin	2615
1	MS 1064	W. C. Fazio	2615
1	MS 1064	P. T. Martin	2615
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