National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2491

Non-Newtonian Polymer Melt for Rheology

Polydimethylsiloxane

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 2491 consists of polydimethylsiloxane. The supplier identifies the polydimethylsiloxane as having a number average molecular mass of 308,000 g/mol. One unit of SRM 2491 consists of 100 mL of the fluid 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 2491 is valid until **31 December 2012**, 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 and K.M. Flynn 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 Group by J.W.L. Thomas and B.S. MacDonald.

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Technical assistance and advice were provided by G.F. Strouse and D.C. Vaughn of the NIST Process Measurements Division and G.B. McKenna of the Texas Tech University.

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

Source of Material: The polydimethylsiloxane was supplied by Gelest, Inc., Tullytown, PA.[1] The material was also homogenized and packaged by Gelest, Inc., Tullytown, 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 2491 was assessed using the zero-shear-rate viscosity measured at 25 °C using samples from 10 bottles randomly chosen from the 273 bottles available. The characterization of this polymer melt 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, 0.04 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 2 % 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 Carreau model [3,4,5] of the form

$$\eta(\dot{\gamma},T) = \left(\frac{T\rho}{T_R\rho_R}\right)\eta_R a(T)\left[1+(\xi_0 a(T)\dot{\gamma})^2\right]^{(n-1)/2} \tag{1}$$

where ρ is the density at temperature T, η_R is the zero-shear-rate viscosity at the reference temperature T_R = 25 °C, ρ_R is the density at the reference temperature T_R, ξ_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, *α(T)* is the temperature shift factor, and n is the power at which the shear stress increases with shear rate. Based on data from the manufacturer [6], n has been set equal to 0.3. The density was approximated as a linear function of temperature, with $\rho(T)=\rho_R(1-\alpha(T-T_R))$, where α = 9.2 × 10^{-4} $cm^3/(cm^3\ K)$ [6] 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, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

The parameters η_R, ξ_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
η_R	843.9 Pa·s	3.8 Pa·s
ξ_0	0.2037 s	0.0040 s
n	0.3 [6]	----
C_1	5.20	0.48
C_2	232 °C	21 °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:

$$N_1(\dot{\gamma}, T) = \left(\frac{T\rho}{T_R \rho_R}\right) \psi_R (a(T)\dot{\gamma})^2 \left[1 + (\xi_1 a(T)\dot{\gamma})^2\right]^{(m-1)/2} \tag{3}$$

where ρ is the density at temperature T; ψ_R is the zero-shear-rate first normal stress coefficient at the reference temperature T_R = 25 °C; ρ_R is the density at the reference temperature T_R; and ξ_1 and m 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 α = 9.2 × 10^{-4} $cm^3/(cm^3\ K)$ [6]. 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
ψ_R	200.2 Pa·s^2	9.6 Pa·s^2
ξ_1	1.22 s	0.33 s
m	0.474	0.069

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

$$\begin{aligned} \ln\left(\frac{G'(\Omega, T)}{1\ \mathrm{Pa}}\right) &= \ln\left(\frac{T\rho}{T_R \rho_R}\right) + \sum_{k=0}^{3} p_k \left(\ln\left(\frac{a(T)\Omega}{1\ \mathrm{rad/s}}\right)\right)^k \\ \ln\left(\frac{G''(\Omega, T)}{1\ \mathrm{Pa}}\right) &= \ln\left(\frac{T\rho}{T_R \rho_R}\right) + \sum_{k=0}^{3} q_k \left(\ln\left(\frac{a(T)\Omega}{1\ \mathrm{rad/s}}\right)\right)^k \end{aligned} \tag{4}$$

where ρ is the density at temperature T, and ρ_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 α = 9.2 × 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 oscillatory data are given in Table 3.

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

Parameter	Value	Standard Uncertainty
p_0	4.2508	2.0 × 10^{-3}
p_1	1.72838	8.7 × 10^{-4}
p_2	-9.658 × 10^{-2}	4.6 × 10^{-4}
p_3	-3.09 × 10^{-3}	1.1 × 10^{-4}
q_0	6.6701	1.8 × 10^{-3}
q_1	0.95974	8.6 × 10^{-4}
q_2	-2.790 × 10^{-2}	4.5 × 10^{-4}
q_3	-7.13 × 10^{-3}	1.1 × 10^{-4}
C_1	6.64	0.21
C_2	299.4 °C	9.2 °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, η	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	1472	31	----	----
0.0	0.001585	1473	31	----	----
0.0	0.002512	1474	30	----	----
0.0	0.003981	1477	30	----	----
0.0	0.006310	1475	30	----	----
0.0	0.01000	1476	30	----	----
0.0	0.01585	1476	30	----	----
0.0	0.02512	1476	30	----	----
0.0	0.03981	1475	30	----	----
0.0	0.06310	1475	30	----	----
0.0	0.1000	1474	30	7.26	5.20
0.0	0.1585	1470	29	19.8	5.8
0.0	0.2512	1465	29	39.9	5.0
0.0	0.3981	1455	29	94.8	5.7
0.0	0.6310	1436	28	201.9	8.7
0.0	1.000	1405	26	423.7	13.9
0.0	1.585	1356	24	872.0	27.9
0.0	2.512	1258	21	1784	55
0.0	3.981	1034	16	3325	99
0.0	6.310	714.0*	12.9*	5546*	169*

* These data are provided as reference values only [2].

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, η	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	841.6	19.1	----	----
25.0	0.001585	842.6	18.3	----	----
25.0	0.002512	844.6	17.6	----	----
25.0	0.003981	843.5	17.6	----	----
25.0	0.006310	844.9	17.1	----	----
25.0	0.01000	844.8	17.0	----	----
25.0	0.01585	844.8	17.0	----	----
25.0	0.02512	844.8	16.9	----	----
25.0	0.03981	844.6	16.9	----	----
25.0	0.06310	844.2	16.9	----	----
25.0	0.1000	844.1	16.9	2.59	1.83
25.0	0.1585	843.2	16.9	5.40	1.91
25.0	0.2512	841.7	16.8	11.9	2.1
25.0	0.3981	839.5	16.8	29.2	2.3
25.0	0.6310	833.8	16.6	68.0	3.4
25.0	1.000	826.1	16.3	154.5	5.8
25.0	1.585	810.0	15.5	337.5	11.4
25.0	2.512	785.8	14.6	712.2	22.3
25.0	3.981	727.7	13.4	1477	45
25.0	6.310	591.1*	13.7*	2756*	81*

* These data are provided as reference values only [2].

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, η	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	541.4	13.4	----	----
50.0	0.001585	541.3	12.0	----	----
50.0	0.002512	541.8	11.3	----	----
50.0	0.003981	542.3	10.9	----	----
50.0	0.006310	542.1	10.8	----	----
50.0	0.01000	543.5	10.8	----	----
50.0	0.01585	542.4	10.7	----	----
50.0	0.02512	542.9	10.6	----	----
50.0	0.03981	542.5	10.6	----	----
50.0	0.06310	542.7	10.6	----	----
50.0	0.1000	542.2	10.6	----	----
50.0	0.1585	541.8	10.6	----	----
50.0	0.2512	541.4	10.6	----	----
50.0	0.3981	540.5	10.5	8.8	1.9
50.0	0.6310	539.0	10.5	23.4	2.1
50.0	1.000	535.6	10.4	58.9	2.9
50.0	1.585	530.6	10.2	137.3	6.4
50.0	2.512	520.0	9.8	308.4	10.4
50.0	3.981	499.8	8.9	662.5	20.5
50.0	6.310	422.1*	6.8*	1390*	52*

* These data are provided as reference values only [2].

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	0.1000	2.95	0.98	148.2	3.9
0.0	0.1585	7.27	1.54	234.0	6.2
0.0	0.2512	17.7	2.4	368.5	9.7
0.0	0.3981	40.9	3.9	577.8	15.3
0.0	0.6310	90.4	6.3	897.8	23.7
0.0	1.000	193.6	10.4	1381	36
0.0	1.585	395.9	17.5	2092	55
0.0	2.512	775.4	29.7	3105	81
0.0	3.981	1449	50	4492	117
0.0	6.310	2579	82	6302	162
0.0	10.00	4359	131	8527	217
0.0	15.85	6978	199	11080	280
0.0	25.12	10590	291	13798	347
0.0	39.81	15240	405	16433	412
0.0	63.10	20891	541	18744	474
0.0	100.0	27364	695	20409	524

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	0.1000	1.81	0.79	118.5	3.2
10.0	0.1585	4.46	1.24	187.5	5.0
10.0	0.2512	11.1	2.0	295.9	7.9
10.0	0.3981	26.4	3.1	465.3	12.4
10.0	0.6310	59.8	5.0	727.0	19.3
10.0	1.000	130.6	8.1	1126	30
10.0	1.585	275.2	13.6	1721	46
10.0	2.512	553.7	22.9	2584	68
10.0	3.981	1061	38	3795	99
10.0	6.310	1946	65	5421	141
10.0	10.00	3388	105	7492	193
10.0	15.85	5594	164	9958	254
10.0	25.12	8752	247	12698	322
10.0	39.81	12976	353	15494	392
10.0	63.10	18287	483	18081	457
10.0	100.0	24559	632	20125	515

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	0.1000	1.12	0.66	96.4	2.6
20.0	0.1585	2.88	1.01	152.3	4.0
20.0	0.2512	7.11	1.58	240.8	6.3
20.0	0.3981	17.2	2.5	379.3	10.0
20.0	0.6310	40.1	4.0	594.4	15.7
20.0	1.000	89.7	6.5	925.8	24.4
20.0	1.585	193.5	10.7	1426	38
20.0	2.512	398.0	18.1	2164	57
20.0	3.981	784.0	30.3	3215	84
20.0	6.310	1474	51	4665	121
20.0	10.00	2636	84	6560	169
20.0	15.85	4477	135	8901	227
20.0	25.12	7208	207	11592	294
20.0	39.81	10986	304	14463	365
20.0	63.10	15898	427	17258	436
20.0	100.0	21873	571	19603	499

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	0.1000	0.704	0.548	79.3	2.1
30.0	0.1585	1.81	0.85	125.6	3.4
30.0	0.2512	4.57	1.32	198.6	5.3
30.0	0.3981	11.4	2.1	313.7	8.3
30.0	0.6310	27.4	3.3	492.8	13.0
30.0	1.000	63.0	5.3	770.1	20.4
30.0	1.585	138.1	8.6	1193	31
30.0	2.512	290.0	14.4	1821	49
30.0	3.981	584.5	24.2	2738	72
30.0	6.310	1125	41	4022	105
30.0	10.00	2066	68	5746	149
30.0	15.85	3598	111	7935	204
30.0	25.12	5947	174	10537	268
30.0	39.81	9300	262	13418	340
30.0	63.10	13793	375	16331	414
30.0	100.0	19436	512	18939	481

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	0.1000	0.451	0.466	66.1	1.8
40.0	0.1585	1.24	0.71	104.5	2.8
40.0	0.2512	3.13	1.11	165.5	4.4
40.0	0.3981	7.77	1.72	261.6	7.0
40.0	0.6310	19.0	2.7	411.7	11.0
40.0	1.000	44.7	4.4	645.4	17.2
40.0	1.585	100.2	7.1	1004	27
40.0	2.512	213.9	11.7	1544.71	41
40.0	3.981	439.4	19.5	2339.77	62
40.0	6.310	863.6	33.3	3472.58	92
40.0	10.00	1621	56	5026.86	132
40.0	15.85	2891	92	7051.73	183
40.0	25.12	4896	148	9531.25	245
40.0	39.81	7846	226	12365.63	316
40.0	63.10	11916	330	15341.24	389
40.0	100.0	17189	462	18149.89	461

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	0.1000	0.301	0.401	55.4	1.5
50.0	0.1585	0.878	0.607	87.9	2.4
50.0	0.2512	2.10	0.93	138.9	3.7
50.0	0.3981	5.52	1.45	219.7	5.9
50.0	0.6310	13.5	2.3	346.3	9.2
50.0	1.000	32.2	3.6	544.4	14.5
50.0	1.585	72.7	5.9	850.1	22.4
50.0	2.512	159.1	9.6	1314	35
50.0	3.981	332.5	16.0	2005	53
50.0	6.310	665.4	27.2	3002	80
50.0	10.00	1274	46	4397	116
50.0	15.85	2323	77	6251	163
50.0	25.12	4025	125	8587	223
50.0	39.81	6602	195	11331	291
50.0	63.10	10260	290	14321	365
50.0	100.0	15120	413	17249	439

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.; 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/.

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[6] *Gelest Silicone Fluids: Stable, Inert Media*, Gelest, Inc., Tullytown, PA (1997); available at http://www.gelest.com/Library/SiFluids.pdf.

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