



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 2490

### Non-Newtonian Polymer Solution for Rheological Measurements

#### 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. Information values on the dynamic mechanical storage and loss moduli and shift factors are also given. SRM 2490 consists of a polyisobutylene dissolved in 2,6,10,14-tetramethylpentadecane. A unit of SRM 2490 consists of 100 mL of the solution, which contains a mass fraction of 0.114 polyisobutylene and is 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. The uncertainties in Tables 4a thru 4c 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 and NIST Guides [1]. Tables 5a, 5b, and 5c list the information values of the storage modulus  $G'$  and loss modulus  $G''$  as functions of frequency at 0 °C, 25 °C, and 50 °C, respectively. No statements are made regarding the uncertainty associated with the information values.

**Expiration of Certification:** The certification of **SRM 2490** is valid, within the measurement uncertainty specified, until **30 September 2015**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

**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 the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The technical assistance and advice was provided by G.F. Strouse and C.D. Cross of the NIST Process Measurements Division and G.B. McKenna of Texas Tech University.

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

The information values for the oscillatory measurements in this certificate were provided by J. Laeuger of Anton Paar Germany GmbH.

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

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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*Certificate Revision History on Last Page*

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## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE<sup>1</sup>

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

**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 within 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 steady shear testing was carried out using a Rheometric Scientific, Inc. ARES controlled-strain rheometer. 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. All oscillatory data was obtained on a Physica MCR 501 Rheometer from Anton Paar with Peltier temperature control. A CP 50-1 geometry (truncation 51 μm) was used for all measurements. At 0 °C the following procedure was followed for oscillatory measurements: (1) zero gap at 0 °C, (2) fill at 10 °C and set gap to 200 μm, (3) cool to 0 °C followed by 10 min equilibration time, (4) set gap to 51 μm and trim<sup>2</sup>, and (5) and then equilibrate for 10 min before starting the measurement. For all other temperatures: (1) zero gap and fill at the desired temperature, (2) set gap to 200 μm, (3) equilibrate for 10 min, (4) set gap to 51 μm and trim, and (5) then wait 10 min before starting the measurement.

**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

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

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  $\alpha = 6 \times 10^{-4} \text{ cm}^3/(\text{cm}^3 \text{ K})$  is the volumetric coefficient of thermal expansion. The shift factor  $a_T$  was fitted with a function of the WLF type [3], giving

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

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<sup>1</sup>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.

<sup>2</sup>“Trimming” refers to removing excess sample with a small laboratory spatula. This is done carefully to ensure no sample is removed out of the gap. Note also that the lower plate is larger than the upper plate for all oscillatory measurements.

The parameters  $\eta_R$ ,  $\xi_0$ ,  $n$ ,  $C_1$ , and  $C_2$  fitted 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:

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

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 fit to the data. The density was again approximated as a linear function of temperature. 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 <sup>2</sup>	5 Pa·s <sup>2</sup>
$\xi_1$	1.69 s	0.13 s
$\xi_2$	0.247 s	0.026 s
$p$	1.67	0.047

**Models for the Oscillatory Data:** For the storage modulus  $G'(\Omega, T)$  and loss modulus  $G''(\Omega, T)$ , the shift factor  $a_T$  again was fitted with a function of the WLF type [3], with the following parameters:

Table 3. WLF parameters for  $a_T$  for  $G'$  and  $G''$ .

Parameter	Value	Standard Uncertainty
$C_1$	7.61	0.25
$C_2$	160 °C	5 °C

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

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

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 <sup>-1</sup>	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

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. Information Values of the Storage Modulus G', Loss Modulus G'', and Complex Viscosity  $\eta^*$ 

Temperature	Frequency of Oscillation	Information Value of the Storage Modulus G'	Information Value of the Loss Modulus G''	Information Value of the Complex Viscosity $\eta^*$
°C	rad/s	Pa	Pa	Pa·s
0.0	0.00631	0.0528	2.405	382.3
0.0	0.01000	0.1228	3.793	380.4
0.0	0.01585	0.3065	5.973	377.9
0.0	0.02512	0.7543	9.354	373.8
0.0	0.03981	1.653	14.52	367.0
0.0	0.06310	3.534	22.20	356.2
0.0	0.1000	7.287	33.26	340.5
0.0	0.1585	13.97	48.67	319.5
0.0	0.2512	25.21	69.22	293.4
0.0	0.3981	43.30	95.36	263.2
0.0	0.6310	70.23	126.9	230.3
0.0	1.000	108.3	163.2	196.4
0.0	1.585	159.0	203.2	163.4
0.0	2.512	223.4	245.1	132.7
0.0	3.981	302.2	287.3	105.3
0.0	6.310	394.8	328.1	81.87
0.0	10.00	500.1	366.3	62.40
0.0	15.85	616.9	401.1	46.71
0.0	25.12	743.2	432.5	34.41
0.0	39.81	877.7	461.1	24.98
0.0	63.1	1017	487.6	17.93
0.0	100.0	1164	519.5	12.77
0.0	158.5	1313	553.5	9.087
0.0	251.2	1483	603.6	6.498
0.0	398.1	1686	664.1	4.683
0.0	631.0	1921	738.8	3.334

Table 5b. Information Values of the Storage Modulus  $G'$ , Loss Modulus  $G''$ , and Complex Viscosity  $\eta^*$ 

Temperature	Frequency of Oscillation	Information Value of the Storage Modulus $G'$	Information Value of the Loss Modulus $G''$	Information Value of the Complex Viscosity $\eta^*$
°C	rad/s	Pa	Pa	Pa·s
25.0	0.00631		0.6180	99.84
25.0	0.01000		0.9827	99.78
25.0	0.01585	0.02062	1.562	99.86
25.0	0.02512	0.05525	2.477	99.8
25.0	0.03981	0.1379	3.922	99.63
25.0	0.0631	0.3343	6.189	99.01
25.0	0.1000	0.7819	9.704	97.9
25.0	0.1585	1.738	15.07	96.1
25.0	0.2512	3.711	23.06	93.3
25.0	0.3981	7.565	34.59	89.24
25.0	0.631	14.56	50.63	83.77
25.0	1.000	26.45	71.98	76.94
25.0	1.585	45.34	99.10	68.98
25.0	2.512	73.51	131.9	60.3
25.0	3.981	113.1	169.7	51.38
25.0	6.31	166.0	211.2	42.69
25.0	10.00	232.7	254.8	34.63
25.0	15.85	314.8	298.8	27.47
25.0	25.12	411.0	341.5	21.33
25.0	39.81	518.6	381.2	16.25
25.0	63.1	637.4	421.7	12.15
25.0	100.0	764.7	459.1	8.923
25.0	158.5	897.8	494.8	6.444
25.0	251.2	1026	529.7	4.603
25.0	398.1	1124	565.1	3.295



Table 5c. Information Values of the Storage Modulus G', Loss Modulus G'', and Complex Viscosity  $\eta^*$ 

Temperature	Frequency of Oscillation	Information Value of the Storage Modulus G'	Information Value of the Loss Modulus G''	Information Value of the Complex Viscosity $\eta^*$
°C	rad/s	Pa	Pa	Pa·s
50.0	0.00631		0.2532	40.14
50.0	0.01		0.4018	40.18
50.0	0.01585		0.6379	40.22
50.0	0.02512		1.012	40.26
50.0	0.03981	0.01614	1.606	40.27
50.0	0.0631	0.05306	2.545	40.25
50.0	0.1000	0.1299	4.024	40.16
50.0	0.1585	0.3051	6.340	39.93
50.0	0.2512	0.6963	9.923	39.51
50.0	0.3981	1.639	15.38	38.79
50.0	0.631	3.563	23.50	37.66
50.0	1.000	7.340	35.24	36.05
50.0	1.585	14.31	51.64	33.90
50.0	2.512	26.28	73.63	31.21
50.0	3.981	45.57	101.8	28.08
50.0	6.31	74.59	136.3	24.65
50.0	10.00	115.2	176.5	21.09
50.0	15.85	170.8	221.2	17.60
50.0	25.12	241.6	268.6	14.33
50.0	39.81	325.9	316.7	11.40
50.0	63.10	428.4	363.8	8.893
50.0	100.0	546.9	408.7	6.814
50.0	158.5	673.5	450.9	5.147
50.0	251.2	829.8	490.4	3.847
50.0	398.1	996.4	528.4	2.855

## REFERENCES

- [1] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1<sup>st</sup> ed.; International Organization for Standardization: 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> (accessed Apr 2011).
- [2] Schultheisz, C.R.; 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. Dept. of Commerce, Tech. Admin., NIST (2001).
- [3] Macosko, C.W., *Rheology - Principles, Measurements and Applications*, Wiley-VCH, NY (1994).
- [4] Gordon, G.V.; Shaw, M.T.; *Computer Programs for Rheologists*, Hanser Publishers, Munich (1994).

**Certificate Revision History:** 24 May 2011 (Extension of the certification period and editorial changes); 17 November 2008 (Extension of certification period); 23 June 2006 (This revision reflects a technical change in that all the data in Tables 5a thru 5c become information values only); 18 September 2001 (Original certificate date).

*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*