



National Institute of Standards & Technology

Certificate

Standard Reference Material[®] 2881

Polystyrene Absolute Molecular Mass Distribution Standard

This Standard Reference Material (SRM) is intended for the calibration and the performance evaluation of instruments used to determine the average molecular mass and molecular mass distribution of synthetic polymers (where mass is taken to be relative to the mass of ^{12}C). These methods include size exclusion chromatography (SEC) [1] and mass spectrometry (MS). [2] The fractional contribution of each oligomer from 1 % to 99 % of the cumulative molecular mass distribution (MMD) was certified and is given in Table 1. The term *absolute* means certification of the molecular mass distribution was performed without reference to another polymer or other polydisperse material.

A unit of SRM 2881 consists of approximately 0.3 g of polystyrene powder. The polystyrene was synthesized by anionic polymerization where initiation was with an *n*-octyl group and termination was with a proton.

Certified Uncertainties: The certified measurement uncertainty is expressed as a combined expanded uncertainty with a coverage factor $k = 2$, calculated in accordance with accepted NIST procedure. [3] The Type A (random) and Type B (systematic) contributions to the expanded uncertainty of the measured molecular mass fractions include uncertainties in sample preparation, instrumentation, and data analysis.

Expiration of Certification: The certification of SRM 2881 is valid, within the measurement uncertainties specified, until **01 January 2019**, provided that the SRM is handled in accordance with the storage instructions given below (see “Instructions for Use”). This certification is nullified if the SRM is modified, contaminated, or stored improperly.

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. Registration will facilitate notification (see attached sheet).

Supplemental Information: Nuclear magnetic resonance spectroscopy (NMR) and matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-TOF MS) were used to analyze the end groups on the polymer. Only the set of end groups specified above were found.

NIST Certification Method: The certified values for the oligomeric fractions in the molecular mass distribution were measured using MALDI-TOF MS. [4]

W.E. Wallace of the NIST Polymers Division provided technical coordination leading to certification of this SRM.

C.M. Guttman, W.E. Wallace, K.M. Flynn, and D.L. VanderHart of the NIST Polymers Division provided technical measurement and data interpretation. A.J. Kearsley of the NIST Mathematical and Computational Sciences Division provided methodology for instrument optimization and for unbiased peak integration methods for the data.

Statistical consultation was provided by J. Lu of the NIST Statistical Engineering Division.

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

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Certificate Issue Date: 12 December 2008

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INSTRUCTIONS FOR USE

Storage: The SRM should be stored in the original bottle with the lid tightly closed under normal laboratory conditions.

Homogeneity and Characterization: SRM homogeneity was tested by SEC analysis as described in reference [4].

Table 1. Number fraction β_i for each oligomer in the molecular mass distribution (MMD) and associated uncertainties for SRM 2881

Repeat Unit Number	Oligomer Mass	Number Fraction of MMD	Uncertainty in Number Fraction	Cumulative Percentage of MMD
i	g/mol	β_i	$k = 2$	%
63	6675.80	0.0020	0.0003	1.0
64	6779.95	0.0025	0.0005	1.2
65	6884.10	0.0033	0.0006	1.6
66	6988.26	0.0041	0.0007	2.0
67	7092.41	0.0053	0.0008	2.5
68	7196.56	0.0067	0.0010	3.2
69	7300.71	0.0084	0.0011	4.0
70	7404.86	0.0105	0.0013	5.1
71	7509.01	0.0129	0.0014	6.4
72	7613.17	0.0157	0.0016	7.9
73	7717.32	0.0186	0.0017	9.8
74	7821.47	0.0217	0.0017	12.0
75	7925.62	0.0254	0.0017	14.5
76	8029.77	0.0287	0.0017	17.4
77	8133.93	0.0321	0.0017	20.6
78	8238.08	0.0353	0.0016	24.1
79	8342.23	0.0383	0.0015	28.0
80	8446.38	0.0410	0.0011	32.1
81	8550.53	0.0431	0.0010	36.4
82	8654.69	0.0449	0.0007	40.8
83	8758.84	0.0456	0.0007	45.4
84	8862.99	0.0459	0.0006	50.0
85	8967.14	0.0457	0.0008	54.6
86	9071.29	0.0450	0.0010	59.1
87	9175.44	0.0437	0.0012	63.4
88	9279.60	0.0416	0.0014	67.6
89	9383.75	0.0397	0.0016	71.6
90	9487.90	0.0368	0.0017	75.2
91	9592.05	0.0339	0.0017	78.6
92	9696.20	0.0310	0.0017	81.7
93	9800.36	0.0279	0.0016	84.5
94	9904.51	0.0249	0.0016	87.0
95	10 008.66	0.0219	0.0015	89.2
96	10 112.81	0.0191	0.0014	91.1
97	10 216.96	0.0164	0.0013	92.7
98	10 321.12	0.0140	0.0011	94.1
99	10 425.27	0.0118	0.0010	95.3
100	10 529.42	0.0098	0.0009	96.3
101	10 633.57	0.0081	0.0007	97.1
102	10 737.72	0.0066	0.0006	97.8
103	10 841.87	0.0053	0.0006	98.3
104	10 946.03	0.0042	0.0005	98.7
105	11 050.18	0.0034	0.0005	99.1

REFERENCES

- [1] ASTM D5296-05; “*Standard Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography*”; Annu. Book ASTM Stand., Vol. 08.03 (2007).
- [2] ASTM D7134-05; “*Standard Test Method for Molecular Mass Averages and Molecular Mass Distribution of Atactic Polystyrene by Matrix Assisted Laser Desorption/Ionization (MALDI) Time of Flight (TOF) Mass Spectrometry (MS)*”; Annu. Book ASTM Stand., Vol. 08.03 (2007).
- [3] 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/guidelines/TN1297/tn1297s.pdf>.
- [4] Guttman, C.M.; Flynn, K.M.; Wallace, W.E.; Kearsley, A.J.; “*Report on the Certification of an Absolute Molecular Mass Distribution Polymer Standard: Standard Reference Material 2881*”; Internal Report 7512, National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (October 2008); available at <http://nvl.nist.gov/pub/nistpubs/ir/2008/ir7512.pdf>.

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