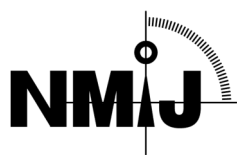


National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5004-a

No. +++



Polystyrene 1000

This certified reference material (CRM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in the calibration of instruments, validation of measurements as well as performance evaluation of instruments used to determine polymer average molecular masses and molecular mass distribution.

Certified Values

(1) The certified values for the mass fraction and the mole fraction for each polystyrene component of different degree of polymerization are given in the table below. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95%.

Degree of polymerization i	Molecular weight M_i	Certified value, Mass fraction w_i	Expanded uncertainty $U(w_i) (k = 2)$	Certified value, Mole fraction x_i	Expanded uncertainty $U(x_i) (k = 2)$
2	266.42	0.0104	0.0012	0.0340	0.0039
3	370.57	0.0306	0.0012	0.0716	0.0026
4	474.72	0.0399	0.0014	0.0730	0.0025
5	578.87	0.0629	0.0022	0.0943	0.0031
6	683.02	0.0866	0.0027	0.1101	0.0033
7	787.17	0.1055	0.0032	0.1164	0.0033
8	891.32	0.1158	0.0034	0.1128	0.0031
9	995.46	0.1151	0.0034	0.1004	0.0028
10	1099.61	0.1056	0.0031	0.0834	0.0024
11	1203.76	0.0898	0.0027	0.0648	0.0019
12	1307.91	0.0718	0.0021	0.0476	0.0014
13	1412.06	0.0541	0.0016	0.0332	0.00103
14	1516.21	0.0391	0.0012	0.0224	0.00071
15	1620.36	0.0268	0.0008	0.0144	0.00047
16	1724.51	0.0178	0.0006	0.0090	0.00029
17	1828.66	0.0114	0.0004	0.0054	0.00018
18	1932.81	0.0070	0.0002	0.0032	0.00010
19	2036.96	0.0042	0.0001	0.0018	0.00006
20	2141.10	0.0025	0.0001	0.0010	0.00003
21	2245.25	0.0015	0.00005	0.00056	0.00002
22	2349.40	0.00081	0.00003	0.00030	0.00001
23	2453.55	0.00043	0.00002	0.00015	0.00001
24	2557.70	0.00022	0.00001	0.000074	0.000003
25	2661.85	0.00013	0.00001	0.000042	0.000004
26	2766.00	0.00008	0.00001	0.000024	0.000003

(2) The certified values of the polystyrene component in this CRM for the mass average molecular mass, M_w , the mole average molecular mass, M_n , the peak average molecular mass, M_p , and the polydispersity index, $P(=M_w/M_n)$ are given in the table below. The certified values of M_w and M_n were calculated from the mass fractions and mole fractions, respectively, of each polystyrene component. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95%.

	Certified value	Expanded uncertainty ($k = 2$)
M_w	1012.9	8.6
M_n	868.3	7.3
M_p	1021.4	6.1
$P(=M_w/M_n)$	1.167	0.019

Analysis

The degree of polymerization of each component was identified by the matrix-assisted laser desorption ionization time of flight mass spectrometry (MALDI-TOFMS). The molecular weight of each component was calculated using "Atomic Weights of the Elements 2001" published by IUPAC. The values of the mass and mole fractions, the mass average molecular mass, the mole average molecular mass, and the polydispersity index were calculated from peak area fractions of the supercritical fluid chromatography (SFC) chromatograms. The SFC chromatograms were obtained by an ultraviolet absorption detector. Polymerization degree dependence of relative ultraviolet absorbance coefficient, with degree of polymerization ∞ of 1, was determined by using uniform polystyrene as a reference. The degree of polymerization of the uniform polystyrene oligomers was identified by the MALDI-TOFMS and the molecular weights were calculated using "Atomic Weights of the Elements 2001" published by IUPAC. Peak average molecular mass is defined as the molecular mass corresponding to the peak elution time in the size-exclusion chromatography (SEC) chromatograms, and it was determined by the calibration curve obtained by using the above-mentioned uniform polystyrenes as a reference. The theoretical plate number for the two SEC columns (4.8 mm I.D. \times 250 mm) was found to be 32,100 for polystyrene with a degree of polymerization $i = 1$. The calibration function was linear.

Expiration of Certification

This certificate is valid until March 31, 2024, provided that the material remains unopened and is stored in accordance with the instructions given in this certificate.

Sample Form

This CRM is in the form of a colorless and highly-viscous liquid at room temperature. This CRM of ca. 0.5 g in net volume is kept in an amber glass bottle.

Homogeneity

The homogeneity of this CRM was determined by the SFC analysis for five bottles out of 151 bottles. The analysis of the variance has proven the homogeneity of this CRM and no differences among bottles.

Instructions for Storage

This CRM should be stored at a temperature between 5 °C and 35 °C, and shielded from light. The bottle should be stored with the lid tightly closed once it is opened.

Instructions for Use

This CRM is for laboratory use only. This CRM should be used promptly once the bottle is opened.

Precautions for Handling

Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

This CRM was synthesized by anionic polymerization in Tosoh Corp., Japan.

Technical Information

The apparent values of M_w , M_n , and $P(=M_w/M_n)$ determined by the SEC are 1009.2 ± 6.2 , 868.4 ± 6.1 , and 1.162 ± 0.0024 , respectively. The numbers following the symbol \pm correspond to 95% confidence intervals. In the SEC measurements, ten uniform polystyrenes from $i = 1$ to 19 were used to obtain the calibration curve. The polymerization degree dependence of the refractive index increment was taken into account when the concentration was calculated at each elution time. Owing to chromatographic band broadening, the apparent values given above essentially do not agree with the certified values. The apparent values of M_{wuc} , M_{nuc} , and $P_{uc}(=M_{wuc}/M_{nuc})$ without the correction of degree of polymerization for the refractive index increment were determined to be 1025.2 ± 3.0 , 889.5 ± 3.0 , and 1.153 ± 0.001 , respectively. The numbers following \pm are the standard deviations in the repeated SEC measurements.

NMIJ Analysts

The technical and production managers for this CRM are KINUGASA S. and the analysts are KINUGASA S. and KISHINE K.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this CRM can be obtained from the contact details given below.

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

April 1, 2020

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:

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National Metrology Institute of Japan,

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Phone: +81-29-861-4059; Fax: +81-29-861-4009, <https://unit.aist.go.jp/nmij/english/refmate/>

Revision history

July 22, 2013: The expiration date was extended from March 31, 2015 to March 31, 2024.

April 1, 2015: "Metrology Management Center" was renamed to "Center for Quality Management of Metrology."