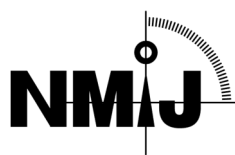


National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5001-a

No. +++



Polystyrene 2400

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, and 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 $U(x_i) (k = 2)$
9	995.46	0.0014	0.0003	0.0033	0.0007
10	1099.61	0.0025	0.0007	0.0053	0.0015
11	1203.76	0.0045	0.0002	0.0086	0.0003
12	1307.91	0.0065	0.0008	0.0114	0.0014
13	1412.06	0.0109	0.0009	0.0178	0.0014
14	1516.21	0.0169	0.0012	0.0258	0.0017
15	1620.36	0.0245	0.0011	0.0349	0.0015
16	1724.51	0.0340	0.0012	0.0455	0.0016
17	1828.66	0.0440	0.0013	0.0556	0.0017
18	1932.81	0.0549	0.0017	0.0656	0.0020
19	2036.96	0.0646	0.0020	0.0731	0.0021
20	2141.10	0.0732	0.0022	0.0789	0.0023
21	2245.25	0.0780	0.0022	0.0802	0.0022
22	2349.40	0.0802	0.0023	0.0787	0.0022
23	2453.55	0.0789	0.0025	0.0742	0.0023
24	2557.70	0.0756	0.0022	0.0682	0.0019
25	2661.85	0.0694	0.0025	0.0602	0.0021
26	2766.00	0.0610	0.0020	0.0509	0.0016
27	2870.15	0.0518	0.0016	0.0417	0.0013
28	2974.30	0.0428	0.0014	0.0332	0.0010
29	3078.45	0.0337	0.0010	0.0253	0.0008
30	3182.60	0.0266	0.0008	0.0193	0.0006
31	3286.74	0.0201	0.0009	0.0141	0.0007
32	3390.89	0.0148	0.0010	0.0101	0.0007
33	3495.04	0.0103	0.0013	0.0068	0.0009
34	3599.19	0.0068	0.0004	0.0044	0.0003

35	3703.34	0.0045	0.0009	0.0028	0.0005
36	3807.49	0.0025	0.0002	0.0015	0.0001
37	3911.64	0.0017	0.0003	0.0010	0.0002
38	4015.79	0.0010	0.0003	0.0006	0.0002
39	4119.94	0.0011	0.0003	0.0006	0.0002
40	4224.09	0.00081	0.00030	0.0004	0.0002

(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.

	Certified value	Expanded uncertainty ( $k = 2$ )
$M_w$	2423	20
$M_n$	2307	18
$M_p$	2469	17
$P(=M_w/M_n)$	1.050	0.016

### Analysis

The degree of polymerization of each component was identified by 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 supercritical fluid chromatography (SFC) chromatograms with the correction of ultraviolet absorbance coefficient as a function of degree of polymerization, which is based on the extrapolation assigned the coefficient of infinite degree of polymerization to 1 by using uniform polystyrene oligomers. The degree of polymerization of the uniform polystyrene oligomers were identified by 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 size-exclusion chromatography (SEC) chromatograms, where calibration between polymer molecular mass and elution time was constructed using uniform polystyrenes. The theoretical plate number for the two SEC columns (4.8 mm I.D. × 250 mm) was found to be 33,400 for polystyrene with a degree of polymerization of  $i = 1$ . The calibration function was a linear one.

### Expiration of Certification

This certificate is valid until July 27, 2023, 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 fine white powder. This CRM of ca. 0.2 g in net volume is kept in a glass bottle.

### Homogeneity

A unit of CRM 5001-a consists of approximately 0.2 g of polystyrene in each bottle. The homogeneity of this CRM was evaluated by the SEC analysis for six bottles out of 150 bottles. Analysis of variance applied to the matching factors of the SEC chromatograms proved the homogeneity of this CRM.

### Instructions for Storage

This CRM is chemically stable. However, it 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 after opening.

**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 by Tosoh Corp., Japan.

**Technical Information**

The apparent  $M_w$ ,  $M_n$ , and  $P(=M_w/M_n)$  values determined by SEC were  $2415 \pm 32$ ,  $2292 \pm 29$ , and  $1.0536 \pm 0.0014$ , respectively. The numbers following the symbol  $\pm$  correspond to 95% confidence intervals. In SEC measurements, fourteen uniform polystyrenes from  $i = 1$  to 41 were used to construct the calibration. The degree of polymerization dependence of the refractive index increment was taken into account when we calculated the concentration at each elution time. Owing to chromatographic band broadening, the apparent values given above essentially do not coincide with the certified values. The apparent values of  $M_{w,uc}$ ,  $M_{n,uc}$ , and  $P_{uc}(=M_{w,uc}/M_{n,uc})$  without the correction of degree of polymerization for the refractive index increment were determined to be  $2419 \pm 11$ ,  $2297 \pm 10$ , and  $1.0532 \pm 0.0008$ , 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., SATO K., 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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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 July 27, 2013 to July 27, 2023.

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