## National Institute of Advanced Industrial Science and Technology

# National Metrology Institute of Japan



**Reference Material Report** 

NMIJ RM 5012-a No. +++



Polystyrene (for Light Scattering)

This reference material (RM) is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This RM is intended for use in the validation of measurements and the evaluation of analytical performance used to determine the molar mass of polymers.

## **Indicative Value**

The indicative value of the mass-average molar mass of this RM is given in the table below. The uncertainty of the indicative value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 3.18 for the degree of freedom of 3, which gives a level of confidence of approximately 95 %.

	Material	Indicative value,	Expanded uncertainty,			
	IVIALEIIAI	Mass-average molar mass* (g/mol)	Mass-average molar mass* (g/mol)			
	Polystyrene	6.18 x 10⁵	0.86 x 10⁵			

\* "Mass-average molar mass" means the same physical quantity used to be called "weight-average molecular weight".

## Analysis

The indicative value of the mass-average molar mass of this RM was measured by static light scattering method. The Rayleigh ratio  $R_{VV}$  (25 °C, toluene, scattering angle at 90°, vertical-vertical polarized laser light with the wavelength of 532 nm) for measurement of the reference value was determined as  $R_{VV} = 2.34 \times 10^{-5} (\text{cm}^{-1})$  by using NMIJ CRM 5008-a Polystyrene (Polydisperse), NMIJ RM 5009-a Polystyrene 8500, and NIST SRM 705a Polystyrene. A Japan Calibration Service System (JCSS)-calibrated balance was used for weighing of solutes and solvents to prepare solutions for light scattering measurements.

## **Expiration of Report**

This report is valid for one year from the date of shipment, provided that the RM is stored in accordance with the instructions given in this report.

## Sample Form

This RM is in the form of a white solid at room temperature. This RM of ca. 0.5 g in net volume is kept in a glass bottle.

## Homogeneity

The homogeneity of this RM was evaluated by static light scattering method for 5 bottles picked up randomly from 100 bottles. The homogeneity is reflected in the uncertainty of the indicative value.

#### **Instructions for Storage**

This RM should be stored at a temperature between 5 °C and 35 °C, and shielded from light.

#### Instructions for Use

This RM is for laboratory use only. The RM should be used promptly as possible once the bottle is opened. The minimum amount for the use of this RM is 10 mg.

#### **Precautions for Handling**

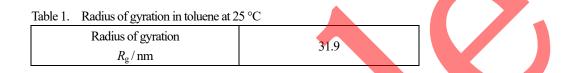
Keep away from fire, heat and sparks and ventilate the air. Wear suitable protective clothing and gloves. Refer to the safety data sheet (SDS) on this RM before use.

#### Preparation

The RM was synthesized by living anionic polymerization with sec-butyl lithium as an initiator, produced by Dr. Atsushi Takano, Associate Professor, Department of Applied Chemistry, Nagoya University (as of 2014).

#### **Technical Information**

The radius of gyration  $R_g$  in toluene at 25 °C measured by static light scattering in 2014 is shown in Table 1. The radius of gyration  $R_g$  was given by the analysis of a Berry Plot for the scattering angle and concentration dependences of scattered light with incident laser-wavelength of 532 nm.



From Stokes-Einstein relationship, the particle sizes (twice of hydrodynamic radii) were determined by dynamic light seattering measurement at 25 °C in toluene with concentration and angular dependences. The results are summarized in Table 2 for the laser-wavelength of 633 nm, and in Table 3 for the laser-wavelength of 532 nm.

Table 2. Particle size (twice of hydrodynamic radius) in toluene at 25 °C with concentration and angular dependences (laser-wavelength of 633 nm)

	,				Scattering a	ngle (°)				
Concentration (g/mL)	90	100	110	120	130	140	150	160	170	173
0.00010	45.6	45.2	44.9	44.6	44.3	44.1	43.9	43.7	43.7	43.6
0.00015	45.4	45.1	44.8	44.5	44.2	43.9	43.8	43.6	43.5	43.5
0.00020	45.2	44.9	44.6	44.3	44.0	43.8	43.6	43.5	43.4	43.4
0.00025	45.1	44.8	44.4	44.2	43.9	43.7	43.5	43.4	43.3	43.3
0.00030	44.9	44.6	44.3	44.0	43.8	43.6	43.4	43.2	43.2	43.2
0.00035	44.7	44.4	44.1	43.9	43.6	43.4	43.3	43.1	43.0	43.0
0.00040	44 <mark>.</mark> 6	44.3	44.0	43.7	43.5	43.3	43.1	43.0	42.9	42.9
0.00045	44.4	44.1	43.8	43.6	43.4	43.2	43.0	42.9	42.8	42.8
0.00050	44.2	44.0	43.7	43.4	43.2	43.0	42.9	42.8	42.7	42.7
0.00055	44.1	43.8	43.5	43.3	43.1	42.9	42.7	42.6	42.6	42.6
0.00060	43.9	43.6	43.4	43.2	42.9	42.8	42.6	42.5	42.4	42.4
0.00065	43.7	43.5	43.2	43.0	42.8	42.6	42.5	42.4	42.3	42.3
0.00070	43.6	43.3	43.1	42.9	42.7	42.5	42.4	42.3	42.2	42.2
0.00075	43.4	43.2	42.9	42.7	42.5	42.4	42.2	42.1	42.1	42.1
0.00080	43.2	43.0	42.8	42.6	42.4	42.2	42.1	42.0	42.0	41.9
0.00085	43.1	42.8	42.6	42.4	42.3	42.1	42.0	41.9	41.8	41.8
0.00090	42.9	42.7	42.5	42.3	42.1	42.0	41.9	41.8	41.7	41.7
0.00095	42.7	42.5	42.3	42.1	42.0	41.8	41.7	41.6	41.6	41.6
0.00100	42.6	42.4	42.2	42.0	41.8	41.7	41.6	41.5	41.5	41.5

Table 3. Particle size (twice of hydrodynamic radius) in toluene at 25 °C with concentration and angular dependences (laser-wavelength of 532 nm)

	Scattering angle (°)									
Concentration (g/mL)	90	100	110	120	130	140	150	160	170	173
0.00010	44.8	44.3	43.8	43.4	43.0	42.7	42.4	42.2	42.1	42.0
0.00015	44.6	44.1	43.7	43.3	42.9	42.6	42.3	42.1	42.0	42.0
0.00020	44.5	44.0	43.6	43.2	42.8	42.5	42.2	42.0	41.9	41.9
0.00025	44.3	43.9	43.4	43.0	42.7	42.4	42.1	41.9	41.8	41.8
0.00030	44.2	43.7	43.3	42.9	42.6	42.3	42.0	41.8	41.7	41.7
0.00035	44.0	43.6	43.2	42.8	42.5	42.2	41.9	41.8	41.6	41.6
0.00040	43.9	43.5	43.1	42.7	42.4	42.1	41.8	41.7	41.6	41.5
0.00045	43.7	43.3	42.9	42.6	42.3	42.0	41.7	41.6	41.5	41.5
0.00050	43.6	43.2	42.8	42.5	42.2	41.9	41.7	41. <mark>5</mark>	41.4	<b>4</b> 1.4
0.00055	43.4	43.1	42.7	42.4	42.0	41.8	41.6	41 <mark>.</mark> 4	41,3	41.3
0.00060	43.3	42.9	42.6	42.2	41.9	41.7	41.5	41.3	41.2	41. <mark>2</mark>
0.00065	43.1	42.8	42.4	42.1	41.8	41.6	41.4	41.2	41.1	41.1
0.00070	43.0	42.6	42.3	42.0	41.7	41.5	41.3	41.1	41.1	41.0
0.00075	42.8	42.5	42.2	41.9	41.6	41.4	41.2	41.1	41.0	41.0
0.00080	42.7	42.4	42.1	41.8	41.5	41.3	41.1	41.0	40.9	40.9
0.00085	42.5	42.2	41.9	41.7	41.4	41.2	41.0	40.9	40.8	40.8
0.00090	42.4	42.1	41.8	41.5	41.3	41.1	40.9	40.8	40.7	40.7
0.00095	42.2	42.0	41.7	41.4	41.2	41.0	40.8	40.7	40.6	40.6
0.00100	42.1	41.8	41.6	41.3	41.1	40.9	40.7	40.6	40.6	40.5

#### NMIJ Analysts

The technical manager is SAKURAI H. The production manager is TAKAHASHI K. The analyst is TAKAHASHI K.

#### Information

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

## **Reproduction of Report**

In reproducing this report, 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 RM, please contact: National Institute of Advanced Industrial Science and Technology, National Metrology Institute of Japan, Center for Quality Management of Metrology, Reference Materials Office, 1-1-1 Umezono, Tsukuba, Ibaraki 305-8563, Japan Phone: +81-29-861-4059; Fax: +81-29-861-4009, https://unit.aist.go.jp/nmij/english/refmate/

### Revision history

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