Date of Shipment: Xxxxxx XX, 20XX

#### National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



# Reference Material Certificate NMIJ CRM 5005-a No. +++



### Poly(ethylene glycol) 400

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, the validation of measurements, and the evaluation of analytical performance used to determine the average molecular mass and molecular mass distribution of polymers.

#### **Certified Values**

(1) The certified values for the mass and number fractions of poly(ethylene glycol) with degrees of polymerization from 5 to 18 are given in the table below. The mass and number fractions were calculated from the compositions of degrees of polymerization from 5 to 18 as the mathematical summation equals to 1, and other compositions are not certified as zero contents. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which give a level of confidence of approximately 95 %.

Material		Poly(ethylene glycol) 400			
		Certified Values			
Degree of	Relative			Expanded	Expanded
Polymeri	Molecular	Mass Fraction	Number Fraction	Uncertainty of	Uncertainty of
zation	Mass	w <sub>i</sub> (kg/kg)	$X_i$	Mass Fraction	Number Fraction
i	Mi			<i>U(w<sub>i</sub>)</i> (kg/kg)	U(x <sub>i</sub> )
5	238.28	0.02453	0.04219	0.00224	0.00373
6	282.33	0.06024	0.08745	0.00389	0.00525
7	326.38	0.11677	0.14664	0.00477	0.00539
8	370.44	0.15986	0.17688	0.00383	0.00410
9	414.49	0.17595	0.17400	0.00269	0.00312
10	458.54	0.16577	0.14818	0.00406	0.00360
11	502.60	0.12944	0.10557	0.00538	0.00413
12	546.65	0.08822	0.06615	0.00551	0.00394
13	590.70	0.04842	0.03360	0.00410	0.00278
14	634.75	0.02080	0.01343	0.00232	0.00149
15	678.81	0.00693	0.00418	0.00102	0.00062
16	722.86	0.00212	0.00120	0.00065	0.00037
17	766.91	0.00073	0.00039	0.00040	0.00021
18	810.97	0.00023	0.00012	0.00033	0.00017

(2) The certified values for the mass-average molecular mass and the number-average molecular mass are given in the table below, which were calculated from the values of the mass and number fractions with degrees of polymerization from 5 to 18. The uncertainties of the certified values are the half-width of the expanded uncertainty intervals calculated using a coverage

factor (k) of 2, which give a level of confidence of approximately 95 %.

	Certified Value	Expanded Uncertainty
Mass-average Molecular Mass  M <sub>w</sub>	431.2	5.7
Number-average Molecular Mass <i>M</i> <sub>n</sub>	409.9	4.4

#### **Analysis**

The mass fraction of this CRM was measured by supercritical fluid chromatography (SFC) with an evaporative light scattering detector (ELSD) calibrated by using uniform poly(ethylene glycol) oligomers. The number fraction  $x_i$  was calculated from the mass fraction  $w_i$  by the following equation:

$$x_i = \frac{w_i / M_i}{\sum_i \left( w_j / M_j \right)}$$

The mass- and number-average molecular masses,  $M_w$  and  $M_n$ , of this CRM were calculated from the mass fraction  $w_i$  and the number fraction  $x_i$  by the following equations:

$$M_{\rm n} = \sum_{i} x_i M_i$$

$$M_{\rm w} = \sum_{i} w_{i} M_{i}$$

#### **Metrological Traceability**

The relative sensitivities of the SFC-ELSD to poly(ethylene glycol) as a function of the degree of polymerization were needed to determine the mass fraction of the poly(ethylene glycol) of the specified degree of polymerization. The relative sensitivities were evaluated by comparing the relative peak intensities measured by the SFC-ELSD for an equimass solution that consisted of poly(ethylene glycol)s of different degrees of polymerization. The equimass solution was prepared by (1) making solutions of poly(ethylene glycol) of a certain degree of polymerization that ranged from 6 to 21, (2) measuring the mass concentration of each solution by total organic carbon (TOC) measurement of which the linearity was verified independently, and (3) mixing the solutions to contain equimass poly(ethylene glycol)s of different degrees of polymerization. Weighting were carried out by a JCSS-calibrated balance. The molecular mass of each component was calculated using "ATOMIC WEIGHT OF THE ELEMENTS 2001" published by IUPAC.

#### **Expiration of Certification**

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

#### Sample Form

This CRM is in the form of a colorless liquid at room temperature. This CRM of ca. 1 g in net volume is kept in a polypropylene bottle with dry argon gas.

#### Homogeneity

The homogeneity of this CRM was evaluated by the SFC-ELSD analysis for 7 bottles picked up from 300 bottles. The analysis of variance applied to the SFC chromatograms proved the homogeneity of this CRM.

#### **Instructions for Storage**

This CRM should be stored at a temperature of 25 °C or below in a clean place and shielded from light. However, in case of long-term storage of 1 month and more, the CRM should be stored in a clean place at a temperature of 5 °C or below.

#### **Instructions for Use**

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

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#### **Precautions for Handling**

Keep away from fire, heat and sparks. Use under open air. Wear suitable protective clothing and gloves. Avoid any contamination. Store and dispose of the CRM in accordance with relevant laws. Refer to the safety data sheet (SDS) on this CRM before use.

#### **Preparation**

This poly(ethylene glycol) 400 was prepared by Wako Pure Chemical Industries, Ltd., Osaka, Japan.

#### **NMIJ Analysts**

The technical manager is KINUGASA S. The production manager is SHIMADA K. The analyst is SHIMADA 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
National Institute of Advanced Industrial Science and Technology,
National Metrology Institute of Japan,
Center for Quality Management of Metrology, Reference Materials Office,

t Quality Washingtonion of Wiederlegy, Reference Waterland Only

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."

July 15, 2015: The description in "Expiration of Certification" was changed to "one year from the date of shipment."

June 9, 2020: The description in "Instructions for Storage" was revised."