

National Institute of Advanced Industrial Science and Technology

## National Metrology Institute of Japan



## Reference Material Certificate

NMIJ CRM 5011-a

No. +++



Poly(ethylene glycol) (23mer)

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 the calibration of instruments, the validation of measurements, and the evaluation of analytical performance used to determine the molecular weight of polymers.

**Certified Value**

The certified value of the mass fraction of the specified degree of polymerization of 23 against the total amount of poly(ethylene glycol) is given in the table below. The uncertainty of the certified value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor ( $k$ ) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Substance	Certified value Mass fraction (kg/kg)	Expanded uncertainty Mass fraction (kg/kg)
Poly(ethylene glycol) 23mer	0.998	0.005

**Analysis**

The mass fraction of the 23mer was determined from the relative intensities of the peaks observed in the chromatograms for different degrees of polymerization that were obtained with a supercritical fluid chromatograph equipped with an evaporative light scattering detector (SFC-ELSD) of which the sensitivity had been evaluated as a function of the degree of polymerization. The contributions from components of degrees of polymerization other than 23 whose peak intensities were less than the limit of detection, were included in the uncertainty of the mass fraction evaluated based on the limit of detection.

**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 42, (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 was carried out by a JCSS-calibrated balance.

**Expiration of Certification**

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

**Description of the Material**

This CRM consists of poly(ethylene glycol) 23mer as the major content and is in the form of a colorless solid at room temperature. A unit of the CRM consists of approximately 50  $\mu\text{g}$  in a glass bottle kept in an environment of dry argon. The cap of the bottle is

made of polypropylene and polyethylene.

### Homogeneity

The homogeneity of this CRM was evaluated by SFC-ELSD analysis for 5 bottles picked up randomly from 107 bottles, and counted in the uncertainty analysis. This CRM is homogeneous within the range of the uncertainty of the certified value.

### Instructions for Storage

This CRM should be stored below 25 °C in the original bottle tightly closed and shielded from light. It is recommended to be stored below 5 °C if storage for a period longer than 1 month is needed.

### Instructions for Use

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

### Precautions for Handling

Keep away from fire, heat and sparks and ventilate the air. Wear suitable protective clothing and gloves. This CRM must be stored and disposed of in accordance with relevant laws. Refer to the safety data sheet (SDS) on this CRM before use.

### Preparation

The source material of this CRM is the same as NMIJ CRM 5006-a Poly(ethylene glycol) 1000. The source material of NMIJ CRM 5006-a was manufactured by Wako Pure Chemical Industries, Ltd., Osaka, Japan. The 23mer was prepared by SFC from the source material.

### Technical Information

- 1) The average molecular weight of this CRM is 1031.2, which is calculated based on “ATOMIC WEIGHT OF THE ELEMENTS 2011” published by IUPAC, and the standard uncertainty of the average molecular weight is 1.2. The most abundant molecular weight of 23mer is 1030.61, which is calculated from the isotopic ratios in “ATOMIC WEIGHT OF THE ELEMENTS 2011”.
- 2) This CRM contains a small amount of 22mer as an impurity.
- 3) The relationship between molecular weight  $M$  and diffusion coefficient  $D_0$  for uniform poly(ethylene glycol) oligomers was measured in dilute solutions of deuterium oxide at 30 °C by pulsed-field gradient nuclear magnetic resonance as shown in Equation (1) [Ref.: Kayori Shimada, et al., *J. Chem. Phys.*, **122**, 244914 (2005)].

$$D_0 = (6.28 \times 10^{-9} \pm 0.38 \times 10^{-9}) M^{-0.43 \pm 0.01} \text{ (m}^2 \text{ s}^{-1}\text{)} \quad (1)$$

By applying this equation and the viscosity of deuterium oxide at 30 °C,  $\eta = 0.973 \times 10^{-3} \text{ Pa s}$  [Ref.: N. Matsunaga and A. Nagashima, *J. Phys. Chem. Ref. Data* **12**, 933 (1983)], particle size  $d$  (twice of hydrodynamic radius) was calculated from the Stokes-Einstein relationship shown in Equation (2). Here,  $k_B$  is the Boltzmann constant and  $T$  is the absolute temperature. The results are summarized in Table A.

$$d = \frac{k_B T}{3\pi\eta D_0} \quad (2)$$

Table A. Stokes-Einstein particle size of uniform poly(ethylene glycol) 23mer in deuterium oxide at 30 °C

Molecular weight $M$	Diffusion coefficient $D_0 / \text{m}^2 \text{ s}^{-1}$	Stokes-Einstein particle size $d / \text{nm}$
1031.2	$3.18 \times 10^{-10}$	1.4

### NMIJ Analysts

The technical manager for this CRM is SAKURAI H., the production manager is TAKAHASHI K., and the analyst is

Date of Shipment: Xxxxx xx, 20xx

5011a00-140326-230320

TAKAHASHI K.

### Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customers. 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,  
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."  
November 18, 2019: The expanded uncertainty in "Certified Value" was changed. The description in "Expiration of Certification" was changed to "one year from the date of shipment."  
March 20, 2023: The expanded uncertainty in "Certified Value" was changed.