

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 5006-a

No. +++



Poly(ethylene glycol) 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, 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 7 to 40 are given in the table below. The mass and number fractions were calculated from the compositions of degrees of polymerization from 7 to 40 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) 1000 | | | |
|---------------------------------|----------------------------------|--------------------------------|--------------------------|---|---|
| Degree of Polymerization i | Relative Molecular Mass M_i | Certified Values | | | |
| | | Mass Fraction w_i (kg/kg) | Number Fraction x_i | Expanded Uncertainty of Mass Fraction $U(w_i)$ (kg/kg) | Expanded Uncertainty of Number Fraction $U(x_i)$ |
| 7 | 326.38 | 0.0004 | 0.0013 | 0.0003 | 0.0008 |
| 8 | 370.44 | 0.0005 | 0.0013 | 0.0003 | 0.0008 |
| 9 | 414.49 | 0.0005 | 0.0013 | 0.0003 | 0.0007 |
| 10 | 458.54 | 0.0007 | 0.0016 | 0.0003 | 0.0007 |
| 11 | 502.60 | 0.0011 | 0.0022 | 0.0004 | 0.0007 |
| 12 | 546.65 | 0.0019 | 0.0037 | 0.0003 | 0.0006 |
| 13 | 590.70 | 0.0037 | 0.0066 | 0.0003 | 0.0005 |
| 14 | 634.75 | 0.0069 | 0.0114 | 0.0003 | 0.0005 |
| 15 | 678.81 | 0.0121 | 0.0186 | 0.0003 | 0.0005 |
| 16 | 722.86 | 0.0196 | 0.0282 | 0.0008 | 0.0012 |
| 17 | 766.91 | 0.0289 | 0.0393 | 0.0009 | 0.0014 |
| 18 | 810.97 | 0.0389 | 0.0499 | 0.0014 | 0.0018 |
| 19 | 855.02 | 0.0498 | 0.0606 | 0.0024 | 0.0029 |
| 20 | 899.07 | 0.0600 | 0.0694 | 0.0027 | 0.0031 |
| 21 | 943.12 | 0.0688 | 0.0760 | 0.0034 | 0.0036 |
| 22 | 987.18 | 0.0763 | 0.0805 | 0.0039 | 0.0039 |
| 23 | 1031.23 | 0.0800 | 0.0807 | 0.0048 | 0.0046 |
| 24 | 1075.28 | 0.0809 | 0.0783 | 0.0051 | 0.0047 |

| | | | | | |
|----|---------|--------|--------|--------|--------|
| 25 | 1119.33 | 0.0792 | 0.0737 | 0.0054 | 0.0048 |
| 26 | 1163.39 | 0.0752 | 0.0673 | 0.0057 | 0.0049 |
| 27 | 1207.44 | 0.0676 | 0.0583 | 0.0057 | 0.0047 |
| 28 | 1251.49 | 0.0592 | 0.0492 | 0.0057 | 0.0046 |
| 29 | 1295.55 | 0.0494 | 0.0397 | 0.0051 | 0.0040 |
| 30 | 1339.60 | 0.0397 | 0.0309 | 0.0045 | 0.0034 |
| 31 | 1383.65 | 0.0308 | 0.0232 | 0.0038 | 0.0028 |
| 32 | 1427.70 | 0.0228 | 0.0166 | 0.0030 | 0.0022 |
| 33 | 1471.76 | 0.0165 | 0.0117 | 0.0030 | 0.0021 |
| 34 | 1515.81 | 0.0110 | 0.0075 | 0.0017 | 0.0012 |
| 35 | 1559.86 | 0.0071 | 0.0047 | 0.0013 | 0.0008 |
| 36 | 1603.92 | 0.0044 | 0.0029 | 0.0012 | 0.0008 |
| 37 | 1647.97 | 0.0027 | 0.0017 | 0.0010 | 0.0006 |
| 38 | 1692.02 | 0.0015 | 0.0009 | 0.0004 | 0.0002 |
| 39 | 1736.07 | 0.0011 | 0.0006 | 0.0005 | 0.0003 |
| 40 | 1780.13 | 0.0006 | 0.0004 | 0.0006 | 0.0003 |

(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 7 to 40. The uncertainties 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 %.

| Average Molecular Mass | Certified Value | Expanded Uncertainty |
|--|-----------------|----------------------|
| Mass-average Molecular Mass M_w | 1084.7 | 20.8 |
| Number-average Molecular Mass M_n | 1040.6 | 17.6 |

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:

$$x_i = \frac{w_i / M_i}{\sum_j (w_j / M_j)}$$

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:

$$M_n = \sum_i x_i M_i$$

$$M_w = \sum_i w_i M_i$$

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 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 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 wax like material 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. 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.

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) 1000 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,
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."

Sample