



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1924

Poly(ethylene oxide)

This Standard Reference Material (SRM) is intended primarily for use in calibration and performance evaluation of instruments used to determine molecular weight and molecular weight distribution by size exclusion chromatography (SEC) or gel filtration chromatography. SRM 1924 is supplied in the form of a white porous mat easily sampled by cutting with clean scissors and in units of 0.2 g.

This material is certified for weight-average molecular weight (M_w):

$$M_w = 120.9 \times 10^3 \pm 9.0 \times 10^3 \text{ g/mole}^a$$

^aCombined expanded uncertainty, U (see Table 1).

The above uncertainty was calculated by a procedure complying with the description in NIST Technical Note 1297 [1].

Source: This poly(ethylene oxide) was prepared by Polymer Labs, Church Stretton, Shropshire, England.

Expiration of Certification: This certificate will be valid for five years from the date of shipment from NIST, when maintained under recommended storage conditions.

Homogeneity and Material Characterization: The homogeneity of the polymer was tested by SEC analysis of solutions in water at 30 °C. The M_w was determined by Rayleigh light scattering. The M_w/M_n ratio (M_n is the number-average molecular weight) was estimated as 1.04 from the SEC analyses in water. The characterization of this poly(ethylene oxide) by SEC and by light scattering is described in NISTIR 5286 [2].

A high molecular weight peak, distinctly remote from the main peak, occasionally appeared in chromatograms during an aging study of another poly(ethylene oxide) standard by aqueous SEC. The high molecular weight peak can be removed by heating the solution at 50 °C for 1 h. In order to avoid degradation, solutions of the standard should be freshly prepared before each SEC calibration.

Storage: SRM 1924 is provided bottled in an environment of dry argon. The polymer sample should be stored in the original bottle tightly closed and in a desiccator, under normal laboratory conditions.

The technical coordination leading to certification of this material was provided by C.C. Han, with technical measurement and data interpretation provided by C.M. Guttman and J.R. Maurey all of the NIST Polymers Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
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(over)

Table 1. Components of Uncertainty in Weight-Average Molecular Weight of SRM 1924

<u>Source of Uncertainty</u>	<u>Degrees of Freedom</u>	<u>u_i g/mole $\times 10^3$</u>	<u>Type of Uncertainty</u>
Standard deviation of the mean M_w^b	3	0.5	A ^c
Solvent index of refraction		0.06	B ^d
Calibration of differential refractometer	5	1.21	A
Differential refractive index	10	0.48	A
Wavelength of light		<0.04	B
Rayleigh ratio of scattering standard		3.63	B
Light polarizers		0.24	B
Ratio of standard scattering to sample scattering		<0.04	B
Solvent density		0.06	B
Solute weights and solvent weights		0.12	B
Light reflection		<0.04	B
Optical alignment		1.33	B
Refraction correction		0.36	B
Anisotropy of polymer in solution		0.12	B
Truncation of virial expansion		1.69	B
Solute degradation		0.36	B

Combined standard uncertainty: $u_c = 4.48 \times 10^3$ g/mole by root-sum-of-squares of u_i [2].

Combined expanded uncertainty: $U = 8.97 \times 10^3$ g/mole, obtained by multiplying u_c by a coverage factor of $k = 2$ [1].

- b. The standard uncertainty of a mean, u_i , is equated with the standard deviation of the mean when computed from analysis of variance of a statistical population of measurements [1].
- c. Type A uncertainties are evaluated by statistical methods [1,2].
- d. Type B uncertainties are evaluated by other means [1,2]. Degrees of freedom for all Type B uncertainties are infinite [2].

REFERENCES

- [1] Taylor, B.N. and Kuyatt, C.E., Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, NIST Tech. Note 1297, Jan. 1993.
- [2] Guttman, C.M. and Maurey, J.R., Determination of the Weight-Average Molecular Weight of Two Poly(ethylene oxides), SRM 1923 and SRM 1924, NISTIR 5286, 1994.