

Polymer Reference Materials-Polyethylene Oxide

Introduction

Reference materials are used for calibration and performance evaluation of instruments used as part of overall quality assurance programs of polymer. These materials support the development of new measurement methods and characterize new materials. The synthesis and characterization of our polymer reference materials are achieved meticulously. Above all, the user can be assured of receiving a well-characterized and quality product.

Chemical structure of the polyethylene oxide:

R-CH2·CH2·O-H

R could be HO—, H_3C –O– CH_2 ·CH–O—, CH_3 ·N— depending on the initiator used for anionic polymerization.

Molecular weight based reference polymers

Polymer Source offers a wide range of reference polymeric materials (RM) for both organic and aqueous phase applications. New reference polymers are added regularly. A wide range of polymers with number average molecular masses (M_n) (oligomers *to 10 million*) have been covered.

Molecular weight values are characterized by various analytical techniques. Interfacing chromatographic methods with other analytical techniques can significantly increase the amount of information available for polymer characterization. The techniques used for characterization are: size exclusion chromatography (SEC), nuclear magnetic resonance (NMR), intrinsic viscosity, thermal analysis, and Matrix Assisted Laser Desorption Ionization-Time of Flight-Mass Spectrometry (MALDI-TOF-MS).

Organic phase soluble	Polystyrene	Polymethyl methacrylate	Polybutadiene	Polyethylene oxide (PEO)
Aqueous phase	Polyethylene	Polyacrylic	Polystyrene	Polystyrene

soluble	oxide (PEO)	acid	sulfonic acid	sodium sulfonate
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The polymer standards are available either *individually* (in the widest range of molecular weights) or *as kits* containing wide range of M_n . Care has been taken to develop standards with the narrowest molecular weight distribution to ensure reliable calibrations of the instruments or for basic research. A *Certificate of analysis* that accompanies each product provides the characterization information indicating the type of end groups and microstructure of the polymer.

Purification of Polymer samples:

Purification of the polyethylene oxide was carried out rigorously to ensure the removal of the catalyst by following steps:

- 1. Dissolved the polymer in de-ionized distilled water to remove the any insoluble organic catalyst side product.
- 2. Polymer extracted from water with dichloromethane.
- 3. Polymer solution in dichloromethane was dried over anhydrous sodium sulfate.
- 4. Solution filtered and then passed through a column packed with basic Al_2O_3 .
- 5. Solution concentrated on rota-evaporator.
- 6. Solution precipitated in cold diethyl ether.
- 7. Dried under vacuum for 48h at 38 °C.

Gel Permeation or Size Exclusion Chromatography

Gel permeation chromatography (GPC) also known as size exclusion chromatography (SEC) is employed to obtain number average molecular masses (M_n) and weight average molecular weight (M_w) . Both these values result to obtain polydispersity index (PDI) (PDI= M_w/M_n). It guides the application level of reference polymers.

SEC analysis was performed on a Varian liquid chromatograph equipped with refractive index detector at 60 °C with DMF as eluent.

Average molecular weight by weight:

$$M_{w} = \frac{\sum w_{i}M_{i}}{\sum w_{i}}$$

Average molecular weight by number:

$$M_n = \frac{\sum n_i M_i}{\sum n_i}$$

where: w_i is the weight in fraction i; n_i is the molecular number in fraction i. M_i is the molecular weight of fraction i.

$$M_i = \frac{W_i}{n_i}$$

Due to some polymer chains distribution in polymer sample, weight average molecular weights (Mw) is always greater than number average molecular weights (Mn). The index of Mw/Mn determine the molecular distribution (polydispersity: PDI) is introduced.

$$PI = \frac{M_w}{M_n}$$

If PDI=1, all the chain lengths are same. Usually, the sample prepared by living process is of narrow distribution character, the PI should be less than 1.15.

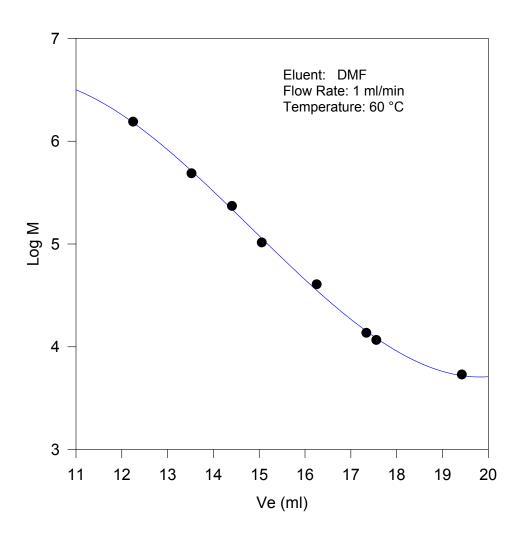
Mp is the molecular weight at peak maximum.

It is worth to note that all the results listed here might have about $\pm 5\%$ deviation.

Typical SEC of PEO Reference Materials

Part No.	Lot No.	M _n	M _w	M _p	M _w /M _n
PEO 5K	P9236	5,100	5,200	5,100	1.02
PEO 11K	P3755	11,500	12,600	13,200	1.09
PEO 13K	P3771	13,200	14,700	15,500	1.12
PEO 35K	P2359A	34,500	39,000	40,400	1.14
PEO 100K	P1893	107,000	122,000	125,000	1.14
PEO 180K	P1904	171,000	202,000	219,000	1.18
PEO 420K	P5617	415,000	493,000	505,500	1.18

Polyethylene oxide standard samples



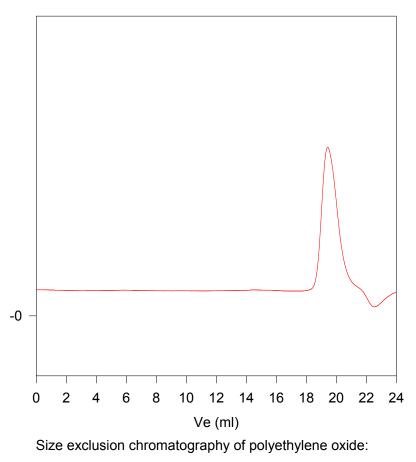
Calibration Curve-PEO in DMF

Characterization report for the each sample analysis:

PEO: 5K lot P9236

Part No.	Lot No.	$\mathbf{M}_{\mathbf{n}}$	$\mathbf{M}_{\mathbf{w}}$	\mathbf{M}_{p}	M_w/M_n
PEO 5K	P9236-S	5,100	5,200	5,100	1.02



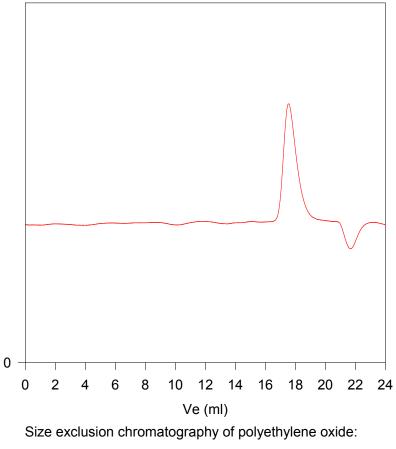


M_n=5,100 M_w=5,200, PDI=1.02

PEO: 11K lot P3755

	Lot No.	$\mathbf{M}_{\mathbf{n}}$	\mathbf{M}_{w}	\mathbf{M}_{p}	M_w/M_n
PEO 11K	P3755	11,500	12,600	13,200	1.09

P3755-EO

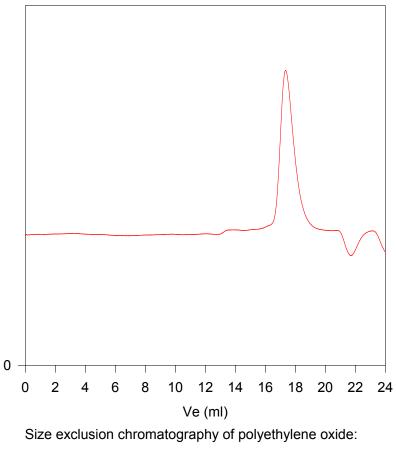


M_n=11,500 M_w=12,600, PDI=1.09

PEO: 13K lot P3771

Part No.	Lot No.	\mathbf{M}_{n}	$\mathbf{M}_{\mathbf{w}}$	\mathbf{M}_{p}	M_w/M_n
PEO 13K	P3771	13,200	14,700	15,500	1.12

P3771-EO

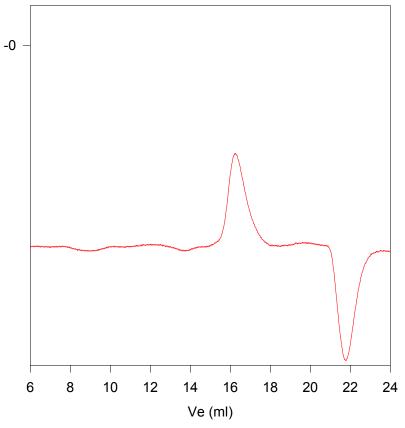


M_n=13,200 M_w=14,700, PDI=1.12

PEO: 35K lot P2359A

Part No.	Lot No.	\mathbf{M}_{n}	$\mathbf{M}_{\mathbf{w}}$	\mathbf{M}_{p}	M_w/M_n
PEO 35k	P2359A	34,500	39,000	40,400	1.14



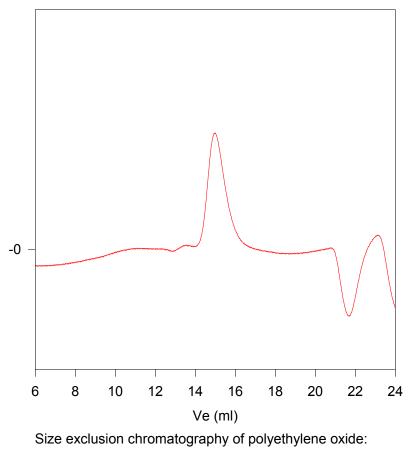


Size exclusion chromatography of polyethylene oxide: M_n =34,500 M_w =39,000, PDI=1.14

PEO: 100K lot P1893

Part No.	Lot No.	\mathbf{M}_{n}	$\mathbf{M}_{\mathbf{w}}$	\mathbf{M}_{p}	M_w/M_n
PEO 100k	P1893	105,000	121,000	125,000	1.15

P1893-EO

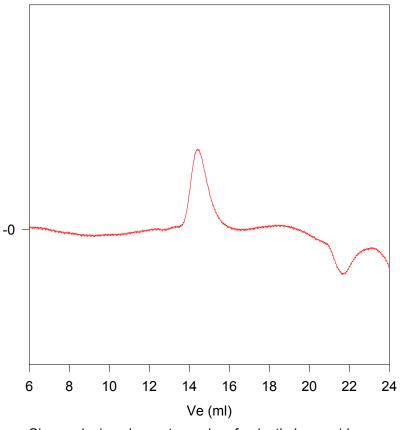


M_n=107,000 M_w=122,000, PDI=1.14

PEO: 180K lot P1904

Part No.	Lot No.	M _n	$\mathbf{M}_{\mathbf{w}}$	M _p	M_w/M_n
PEO 180k	P1904	171,000	202,000	219,000	1.18

P1904-EO

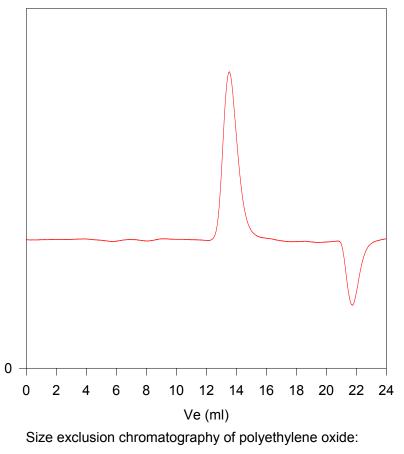


Size exclusion chromatography of polyethylene oxide: M_n =171,000 M_w =202,000, PDI=1.18

PEO: 420K lot P5617

Part No.	Lot No.	M _n	$\mathbf{M}_{\mathbf{w}}$	M _p	M_w/M_n
PEO 420K	P5617	415,000	493.000	505,000	1.18

P5617-EO



M_n=415,000 M_w=493,000, PDI=1.18