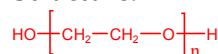


Sample Name:

Poly(ethylene glycol) Oligomers-tetramer

Sample #: P5488-EG2OH

Structure:

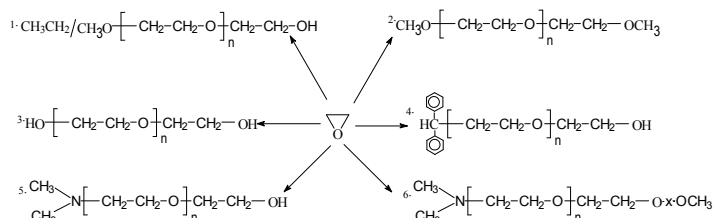


Composition:

Mn x 10 ³	PDI
0.20	1.20

Synthesis Procedure:

Poly (ethylene glycol) is obtained by living anionic polymerization and the reaction. Scheme of the polymerization is illustrated below:



Initiator System	Resulting Polymer
1) $\text{CH}_3\text{OCH}_2\text{CH}(\text{CH}_3)\text{OK}$	polyethylene glycol methyl ether
2) $\text{CH}_3\text{OCH}_2\text{CH}(\text{CH}_3)\text{OK}$	α , ω -term. methyl ether polyethylene glycol
3) $\text{KOCH}_2\text{CH}_2\text{OK}$	polyethylene glycol
4) $\text{CH}(\text{C}_6\text{H}_5)_2\text{CK}$	polyethylene glycol diphenyl ether
5) $(\text{CH}_3)_2\text{N}-\text{CH}_2\text{CH}_2\text{OK}$	methyl amino terminated PEG
6) $(\text{CH}_3)_2\text{N}-\text{CH}_2\text{CH}_2\text{OK}$	α -methyl amino ω -methyl ether term. PEG

Characterization:

By Size exclusion chromatography (SEC): Varian liquid chromatograph equipped with UV and refractive detector. SEC columns from Supelco were used with THF containing 2 vol% $(\text{Et})_3\text{N}$ as the eluent. The molecular weights were determined using light scattering detector and viscosity detector. The molecular weights and the polydispersity indice were calculated.

An aqueous GPC column from Supelco(G5000 PWXL) was also used with 0.5 M acetic acid and 0.8 M NaNO_3 as the eluent. It was kept at a constant temperature of 50°C. The flow rate was 1.0 ml/min. The column was calibrated with monodisperse poly(ethylene oxide) standards. The molecular weights and the polydispersity index of polyethylene oxide were calculated by using a Visual Basic GPC software.

Purification of the obtained polymer:

Purification of the obtained polymer was carried out rigorously as follows to ensure the removal of the catalyst side product:

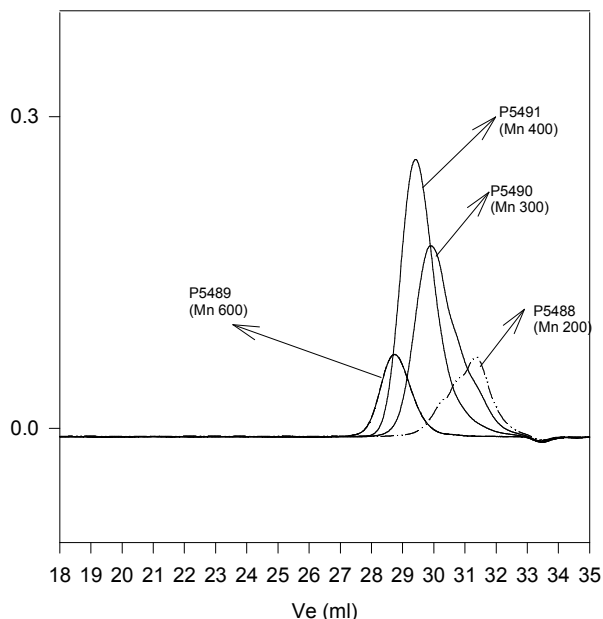
1. Dissolved the polymer in de-ionized distilled water to remove the any insoluble organic catalyst side product.
2. Polymer extracted from water with dichloromethane.
3. Polymer solution in dichloromethane was dried over anhydrous sodium sulfate.
4. Solution filtered and than passed through a column 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 oC.

Solubility:

Poly(ethyl glycol) is soluble in toluene, THF, water and CHCl_3 . The polymer is insoluble in hexane, ether, cold isopropanol and ethanol.

SEC of Sample of the polymer

SEC Profile for PEG Oligomers



Size exclusion chromatography of poly(ethylene glycol):

Lot# P 5488-EG2OH Mn=200, Mw=240, Mw/Mn =1.20

Lot# P 5490-EG2OH Mn 300 Mw: 360 Mw/Mn = 1.20

Lot# P 5491-EG2OH Mn 400 Mw: 480 Mw/Mn = 1.2

Lot# P 5489-EG2OH Mn 600 Mw: 690 Mw/Mn 1.15