

Sample Name:

Six-Arm Poly(ethylene oxide) Sulfonic Acid Terminated, Dipentaerythritol Core

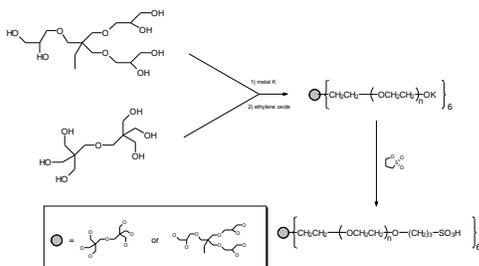
Sample #:

P2833-6EOSO3H

$M_n \times 10^3$ (total)	PDI
10.0	1.08

Synthesis Procedure:

The polymer was prepared by anionic living polymerization of ethylene oxide using dipenta erythritol potassium salt or other hexa functional compound as the initiator. The scheme of the reaction is illustrated below:



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% (Et)₃N 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 NaNO₃ 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.

The sulfonic acid functionality of the polymer was determined by two-phase titration with Hyamine[®] 1622 (Fluka) in water-chloroform using the methylene blue (Fluka) as indicator.

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:

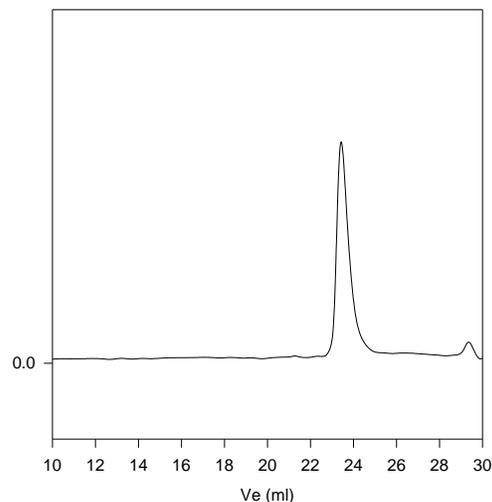
1. Dissolved the polymer in de-ionized distilled water to remove the any insoluble organic catalyst side product.
2. Polymer extracted from water with dichloromethane.
3. Polymer solution in dichloromethane was dried over anhydrous sodium sulfate.
4. Solution filtered and than passed through a column packed with basic Al₂O₃.
5. Solution concentrated on rota-evaporator
6. Solution precipitated in cold diethyl ether.
7. Dried under vacuum for 48h at 38 oC.

Solubility:

Polymer is soluble in toluene, THF, water and CHCl₃. The polymer is insoluble in hexane, ether, cold isopropanol and ethanol.

SEC of the product

P2833-6EO Before End Functionalization



Size Exclusion Chromatogram of Six-Arm Poly(ethylene glycol)

— P2833: $M_n=10000$, $M_w=10800$, $M_w/M_n=1.08$