

Sample Name:

Poly(n-butyl methacrylate-b-2-hydroxy ethyl methacrylate)

Sample #: P5931-nBuMAHEMA**Structure:****Composition:**

Mn × 10 ³ nBuMA-b-HEMA	PDI
23.0-b-8.0	1.15
T _g for nBuMA block: 53 °C	T _g for HEMA block: 103 °C

Synthesis Procedure:

Poly(n-butyl methacrylate-b-2-hydroxy ethyl methacrylate) block copolymer is synthesized by living anionic polymerization with sequential addition of n-butyl methacrylate and protecting hydroxyl HEMA (trimethyl siloxy ethyl methacrylate monomer). The obtained polymer was precipitated in methanol/acidic to deprotect the hydroxyl group.

Characterization:

SEC analysis of the obtained block copolymer in THF in presence of triethyl amine as eluent resulting in an ambiguity of the result because some of the trimethylsiloxy ethyl methacrylate units are deprotected to convert hydroxy ethyl methacrylate.

The SEC analysis of the final polymer is carried out after protecting OH groups of hydroxy ethyl methacrylate to acetate group was treated with acetic anhydride in presence of pyridine. The SEC analysis of the obtained polymer gives more reliable results.

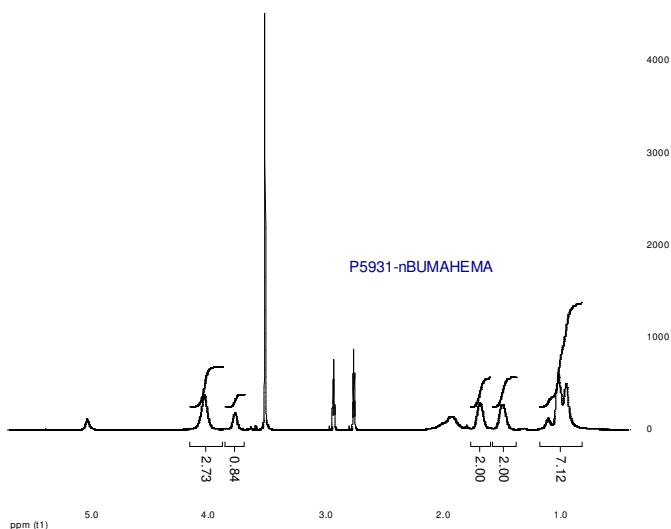
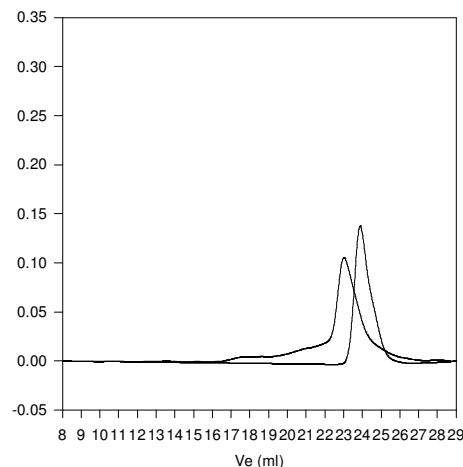
The final block copolymer composition by ¹H-NMR spectroscopy in CdCl₃ also yield the uncertainty of the analysis because of poor solubility of poly HEMA block in CdCl₃. The composition of the obtained polymer therefore, carried out in CdCl₃ after protecting the OH group with acetic anhydride by comparing the peak area CH₂ alkyl ester (4 protons) group at 1.6 and 1.5 ppm and subtracting 6 protons values for nBuMA monomers (3 from α-CH₃ and 3 protons from terminal CH₃ alkyl ester group) between 0.9ppm to 1.2ppm to calculate 3 protons (α-CH₃) from HEMA monomers. Block copolymer PDI is determined by SEC.

Thermal analysis:

Thermal analysis of the samples was carried out on a TA Q100 differential scanning calorimeter at a heating rate of 10°C/min. The midpoint of the slope change of the heat flow plot of the second heating scan was considered as the glass transition temperature (T_g).

Solubility:

Polymer is soluble in DMF.

¹H-NMR Spectrum of the block in DMF:**SEC of the block copolymer:****P5931--nBuMAHEMA**

Size exclusion chromatography of

1. Poly nBuMA: Mn 23000 Mw: 25000 Mw/Mn 1.08

Poly(nBuMA)-b- Poly 2-Hydroxy ethyl methacrylate (Protected with TMS)

Mn 23,000-b-12500 Mw/Mn 1.15

After Deprotection of HEMA TMS : Mn 23000-b-8000 Mw/Mn 1.15

The deprotected product could not be eluted in THF.