

Sample Name:

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

Sample #: P5957--nBuMAHEMA

Structure:**Composition:**

$M_n \times 10^3$	PDI
nBuMA-b-HEMA	
18.0-b-7.0	1.3
T_g for nBuMA block: 53 °C	T_g for HEMA block: Not detected

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.

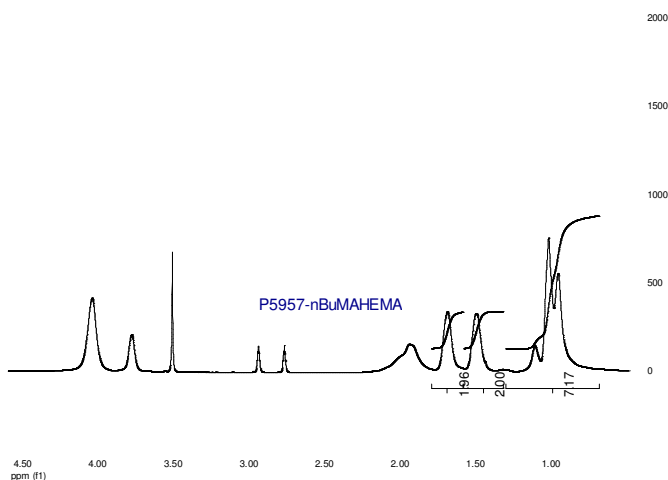
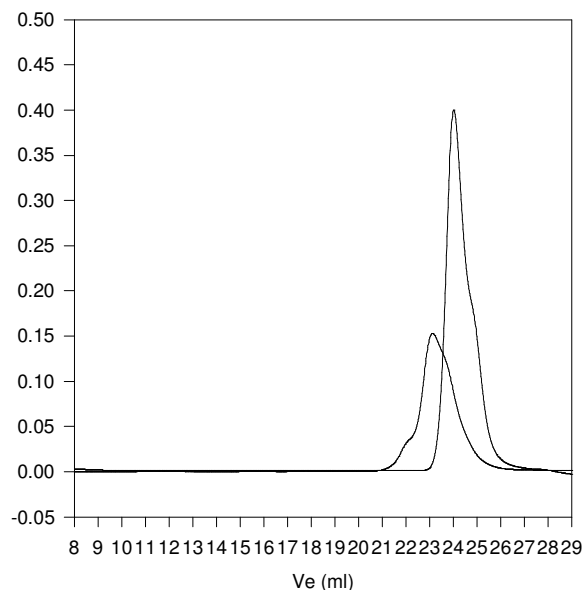
The final block copolymer composition by $^1\text{H-NMR}$ spectroscopy in CdCl_3 also yield the uncertainty of the analysis because of poor solubility of poly HEMA block in CdCl_3 . The composition of the obtained polymer therefore, carried out in CdCl_3 after protecting the OH group with acetic anhydride or in DMF by comparing the peak area of the $-\text{CH}_2-\text{CH}_2$ of nBuMA protons at 1.8 and 1.6 ppm with the peak area of ethyl methacrylate at ($\alpha\text{-CH}_3$) 0.86-1.2 ppm by subtracting the 6 protons ($\alpha\text{-CH}_3$ and terminal CH_3 alkyl chain) from n-butyl methacrylate. 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 $10^\circ\text{C}/\text{min}$. 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.

 $^1\text{H-NMR}$ Spectrum of the block in DMF:**SEC of the block copolymer:****P5957--nBuMAHEMA**

Size exclusion chromatography of

1. Poly nBuMA: M_n 18000 M_w : 20500 M_w/M_n 1.15

Poly(nBuMA)-b- Poly 2-Hydroxy ethyl methacrylate (Protected with TMS)
 M_n 18,000-b-11000 M_w/M_n 1.3

After Deprotection of HEMA TMS : M_n 18000-b-7000 M_w/M_n 1.3

In THF after deprotection , it can not be eluted