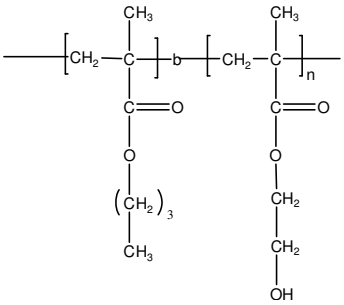
Sample Name:

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

Sample #: P5910-1-nBuMAHEMA

Structure:



Composition:

Mn × 10^3 nBuMA-b-HEMA	PDI
23.0-b-3.5	1.18
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 methnol/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 trimethylsiloxyl 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}H-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 or in DMF by comparing the peak area of the -CH2-CH2 of nBuMA protons at 1.8 and 1.6 ppm with the peak area of ethyl methacrylate at (α-CH3) 0.86-1.2 ppm by substracting the 6 protons ((α-CH3 and terminal CH3 alkyl chain) from n-butyl methacryate Block copolymer PDI is determined by SEC.

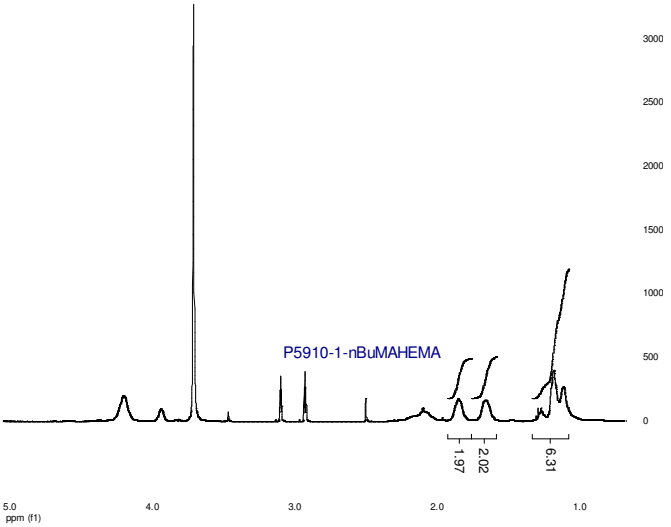
Thermal analysis:

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).

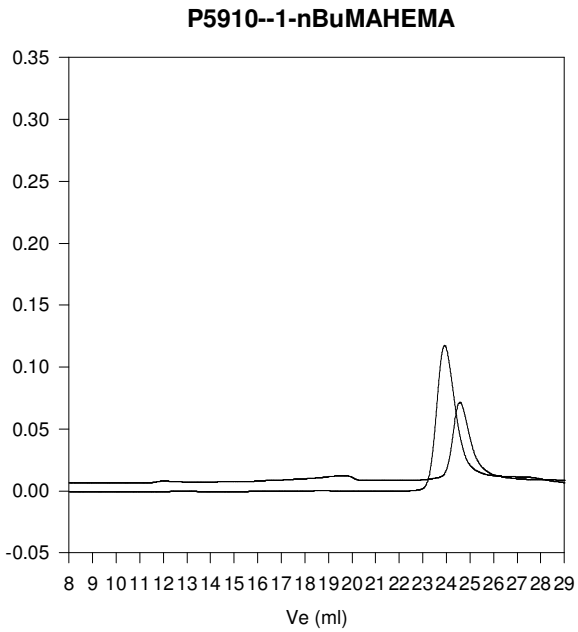
Solubility:

Polymer is soluble in DMF.

^{1}H-NMR Spectrum of the block in DMF:



SEC of the block copolymer:



Size exclusion chromatography of
1. Poly nBuMA: Mn 23000 Mw: 26500 Mw/Mn 1.15
Poly(nBuMA)-b- Poly 2-Hydroxy ethyl methacrylate (Protected with TMS)
Mn 23,000-b-5500 Mw/Mn 1.18
After Deprotection of HEMA TMS : Mn 23000-b-3500 Mw/Mn 1.18

DSC thermogram for the polymer: