

Sample Name:

Amino end functionalized Poly(ethylene oxide -b- methacrylic acid)

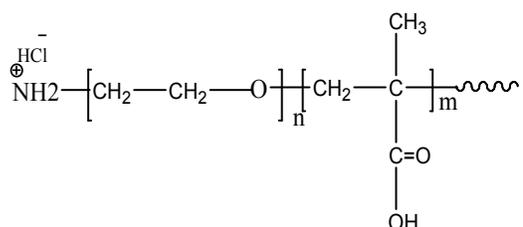
Sample #: P4739A- NH2EGMAA

Composition:

| | |
|---------------------------------------|-----|
| Mn x 10 ³ NH2PEG-b-PMAA | PDI |
| 11.5-b-2.2 | 1.2 |

Synthesis Procedure:

NH2 end functionalized Poly(ethylene oxide -b- methacrylic acid) is prepared by living anionic polymerization of ethylene oxide and tert. Butyl methacrylate followed by hydrolysis of tert.butyl ester to methacrylic acid form.



Characterization:

An aliquot of the anionic poly(ethylene oxide) block was terminated before addition of tert.butyl methacrylate and analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI). The polymer obtained at each step and the final block copolymer composition was calculated from ¹H-NMR spectroscopy by comparing the peak area of the ethylene oxide protons at about 3.6 ppm with the one proton at about 1.2ppm.

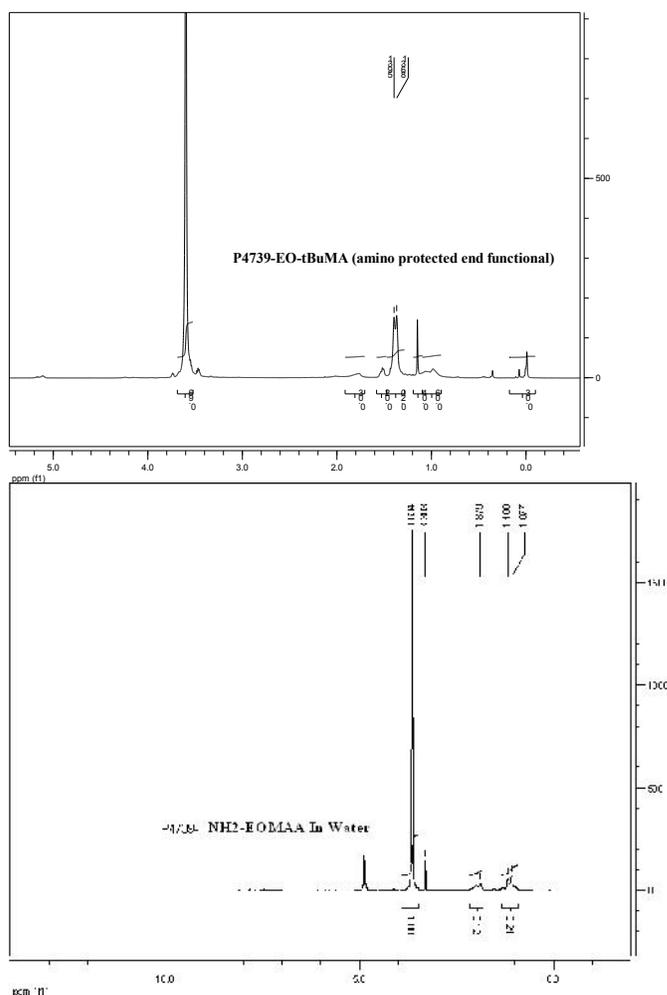
FTIR:

The degree of hydrolysis of tert.butyl ester to methacrylic acid was determined from the FTIR by disappearance of characteristic absorbance of tert.butyl ester at 1265 cm⁻¹ and broadening of ester C=O absorbance.

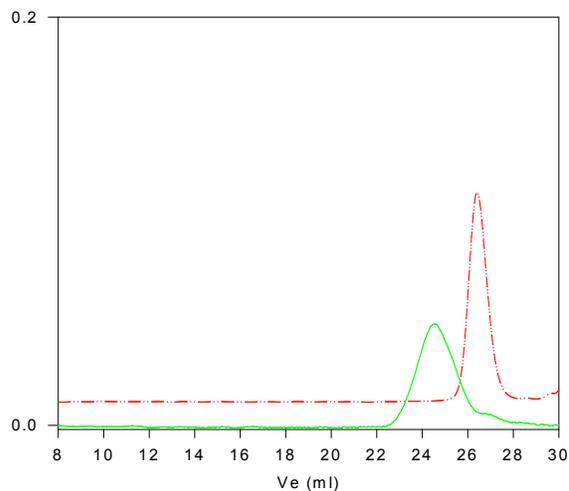
Solubility:

NH2 end functionalized Poly(ethylene oxide -b- methacrylic acid) is soluble in methanol, ethanol and in water cloudy solution to a clear solution – depending on the temperature of the solution, due to less hydrophilic characteristics of methacrylic acid block. It is also soluble in THF.

¹H-NMR Spectrum of the polymer run in CD3OD:



P4739A-NH2EOtBUMA
Precursor for P4739A-NH2EOMAA



Size exclusion chromatography:

- Poly(tert.butylmethacrylate), M_n=3500, M_w=3900, PI=1.10
- Block Copolymer PtBuMA(3500)-b-PEO(11500), PI=1.20
Composition from ¹H NMR
after hydrolysis of tert.butyl ester:
Mn 2200-11500

Thermal analysis of the P4739A- NH2EGMAA

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).

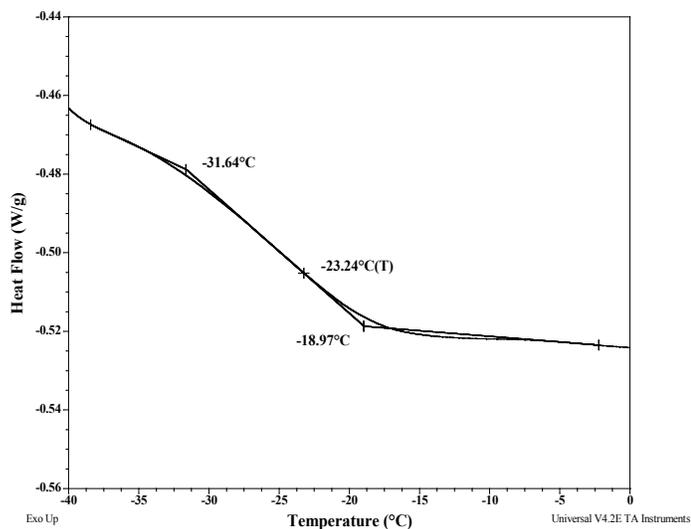
Melting and crystallization curve for the sample

The melting temperature (T_m) was taken as the maximum of the endothermic peak where as the crystallization temperature (T_c) was considered as the minimum of the exothermic peak.

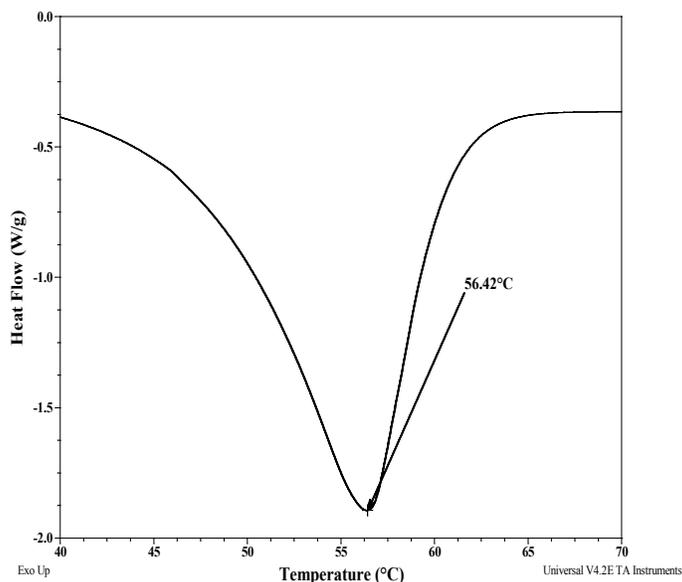
Thermal analysis results at a glance

| Sample | T_m (°C) | T_c (°C) | T_g (°C) |
|----------|------------|------------|------------|
| EO Block | 56 | 04 | -23 |
| AA block | - | - | Not found |

Thermogram for the EO block:



Melting curve for the polymer:



Crystallization curve for the polymer:

