

Sample Name: Biotin end functionalized

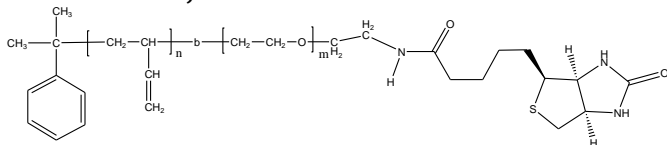
Poly(butadiene-b-ethylene oxide)

Poly butadiene rich in 1,2 or 1,4 microstructure

Sample #: P10944-BdEOBiotin

(poly butadiene block rich in 1,2 microstructure)

Structure of 1,2-rich microstructure:



Composition:

Mn x 10 ³ Bd-b-EO-Biotin	Mw/Mn (PDI)	% 1,2 addition Butadiene
2.5-b-1.3	1.04	89
NH2 Functionality	>99%	
Biotin Functionality	>99% ±10%	Integration from _NH-CO- CH2 CH2 at 2.28ppm

Synthesis Procedure:

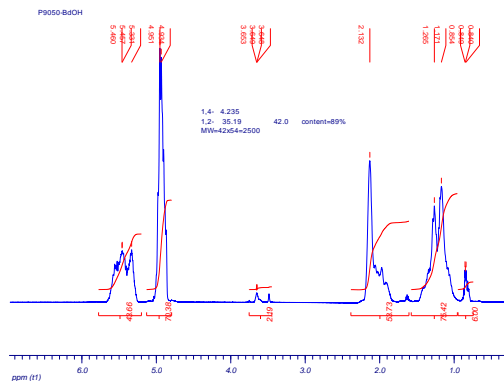
Poly(butadiene(1,4 addition or 1,2 addition)-b-ethylene oxide) can be prepared by the different routes as reported in the literature (ref: *Macromolecules* 1996, 29, 6994). The direct synthesis of diblock copolymer using lithium counter ion in the presence of **Phosphazene Base** *t*-BuP₄ is interesting as reported in *Macromolecules*, **32** (8), 2783 - 2785, 1999. These polymers can also be successfully synthesized using the different end functionalized polymers as investigated in our lab. These methodologies are proprietary.

Characterization:

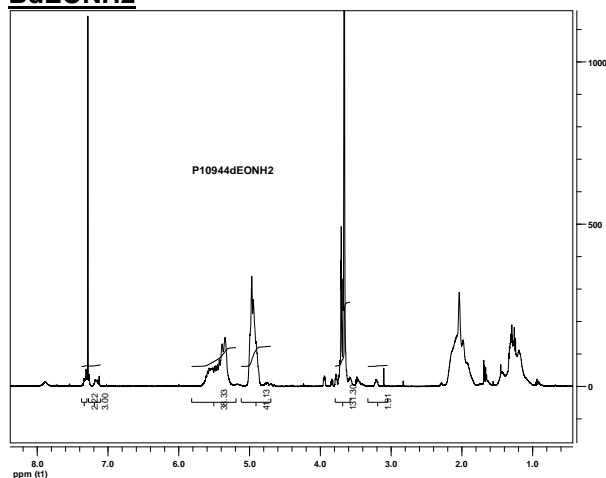
Polybutadiene polymer was analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI). The final block copolymer composition was calculated from ¹H-NMR spectroscopy by comparing the peak area of the vinylic butadiene protons between about 5.0-5.4 ppm with the ethylene oxide protons at 3.6 ppm. Block copolymer PDI is determined by SEC. Note: The ¹H-NMR of 1,2-polybutadiene is composed of 1 proton signal at 5.4 ppm and 2 proton signals at 5.0 ppm. Signals due to vinylic 1,4-polybutadiene are also present at 5.4 ppm.

Amino Titration: the degree of functionality was confirmed by titration with HClO₄ using crystal violet as the indicator.

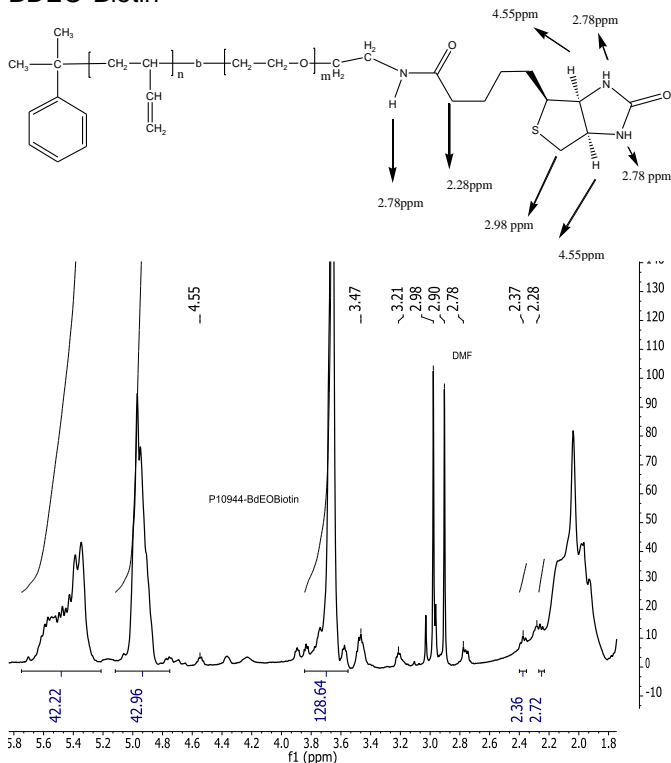
¹H NMR spectrum of the sample at different steps:
PBd-EO



BdEONH2

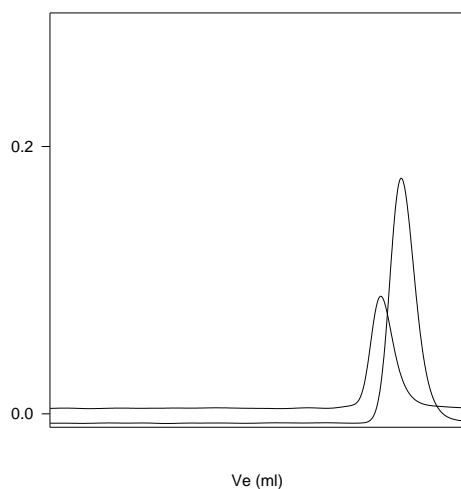


BDEO-Biotin

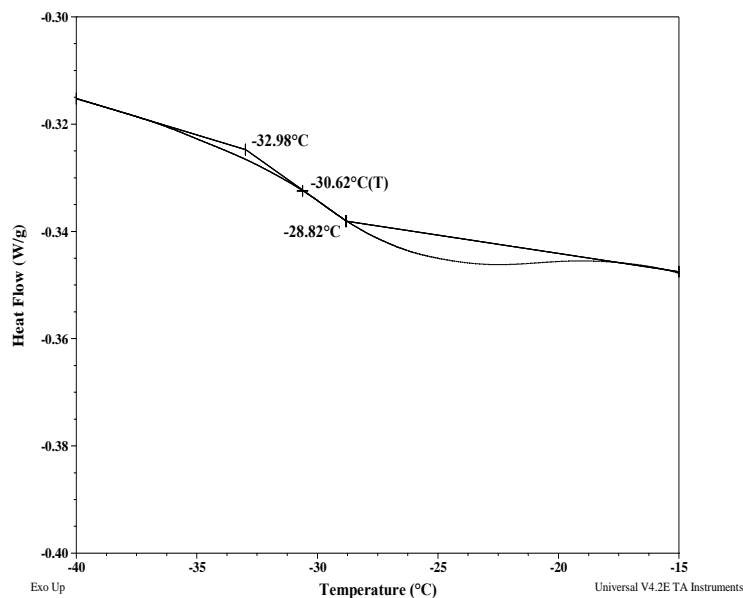


SEC profile of the BDEO before converting Biotin

P10944-BdEO



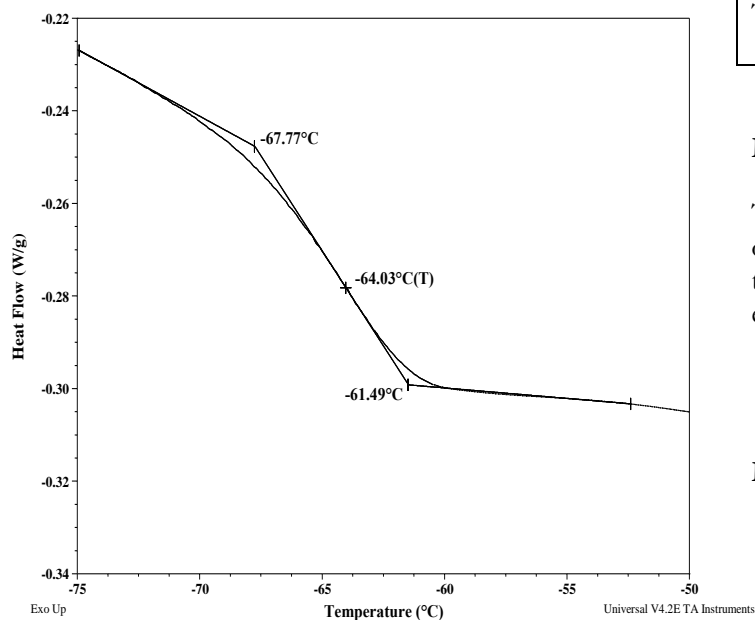
Size exclusion chromatography of poly(butadiene-b-ethylene oxide):
Poly BD (1,2 addition) 2500-b-EO 1300 Mw/Mn 1.04
Composition by HNMR



Thermal analysis of the sample P10944-BdEO – precursor for P10944 BdEO-biotin

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).

Thermogram for PEO block:



Thermogram for PBd block:

Thermal analysis results at a glance

For Bd block		
T_g : -31°C	T_m : -	T_c : -
For PEO block		
T_g : -64°C	T_m : 48°C	T_c : Not found

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.

Melting curve for PEO block:

