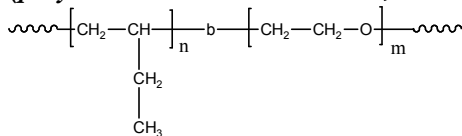


Sample Name: Poly[(ethylene-co-butene)-b-ethylene oxide] (Hydrogenated Poly(1,2-butadiene))

Sample #: P8944-EBEO

(poly butadiene block rich in 1,2 microstructure)



Composition:

Mn x 10 ³ EB-b-EO	Mw/Mn (PDI)	% 1,2 addition Butadiene
21.7-b-4.0	1.08	>88
% Hydrogenation over 97%		

Synthesis Procedure:

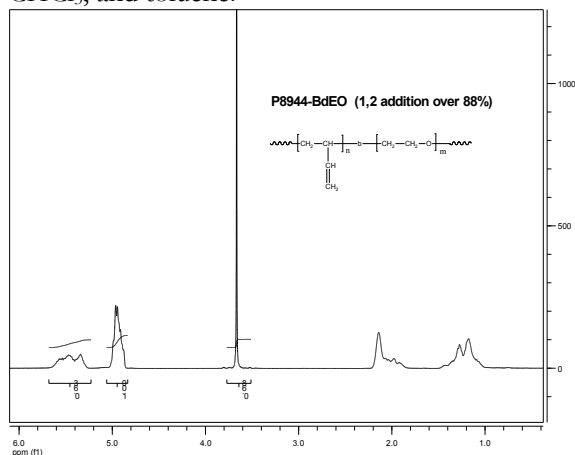
Poly(butadiene(1,4 addition or 1,2 addition)-b-ethylene oxide) can be prepared by the different routes as reported in the literature (ref: *Macromolecules* 1996, 29, 6994). The direct synthesis of diblock copolymer using lithium counter ion in the presence of **Phosphazene Base t-BuP₄** is interesting 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:

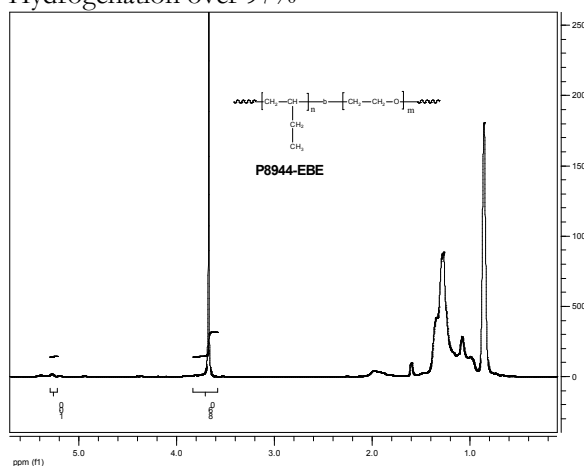
The final block copolymer composition was calculated from ¹H-NMR spectroscopy by comparing the peak area of the vinylic butadiene protons between about 5.0-5.4 ppm with the ethylene oxide protons at 3.6 ppm. Block copolymer PDI is determined by SEC. Note: The ¹H-NMR of 1,2-polybutadiene is composed of 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.

Solubility:

Poly[(ethylene-co-butene)-b-ethylene oxide] (Hydrogenated Poly(1,2-butadiene)) is soluble in THF, CHCl₃, and toluene.

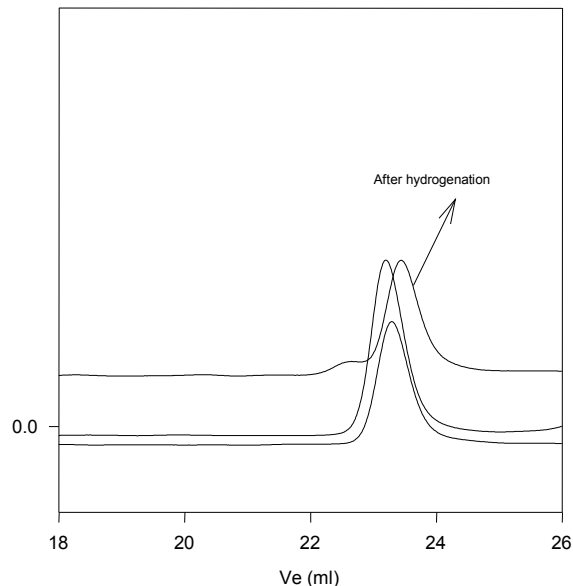


¹H NMR of the Polymer after Hydrogenation:
Hydrogenation over 97%



SEC profile of the block copolymer

P8944-Bd ^{1,2 rich} **EO precursor for P8944-EBEO**



Size Exclusion Chromatogram of Poly(butadiene-b-ethylene oxide)

— Polybutadiene: M_n=21000, M_w=22000, M_w/M_n=1.06

— PBd-b-PEO: M_n PBd(21000)-PEO(4000), M_w/M_n=1.08

The Mn of PEO is calculated from NMR results,

After Hydrogenation of Poly butadiene block: Mn 21700-b-4000

*: GPC profile after the hydrogenation indicating the Mw/Mn : 1.08

Purification of the Polymer:

We have used Wilkinson catalyst for the Hydrogenation. This catalyst always leaves behind the finger prints of the polymer matrix therefore the polymer after purification is light brown in color. The polymers solution was passed several times through silica packed column to remove any trace amount of the insoluble impurities. The obtained polymer was freeze dried from 1,4 dioxane solution.

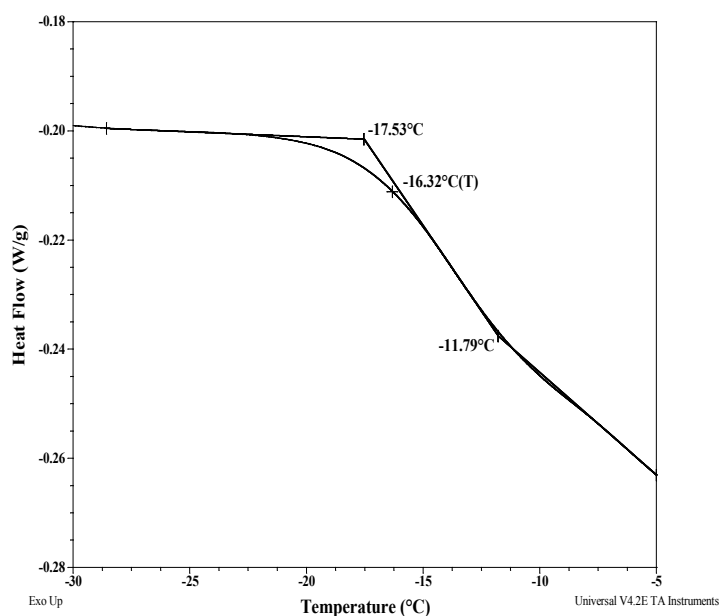
Thermal analysis of the sample P8944-BdEO

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).

Thermal analysis results at a glance

For Bd block		
T_g : -16°C	T_m : -	T_c : -
For PEO block		
T_g : Not distinct	T_m : 52°C	T_c : -30°C

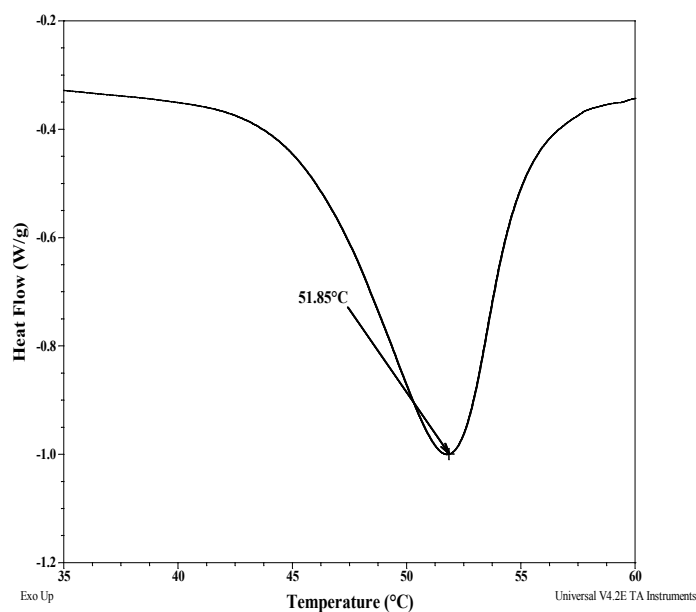
Thermogram for PBd block



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:



Crystallization curve For PEO block

