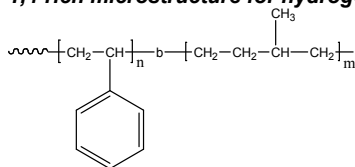


Sample Name: Poly(styrene-b-methyl butylene) obtained by the hydrogenation of

(Poly (styrene –b- isoprene rich in 1,4-addition))

Sample #: P9869-SMB

1,4-rich microstructure for hydrogenated polyisoprene block:

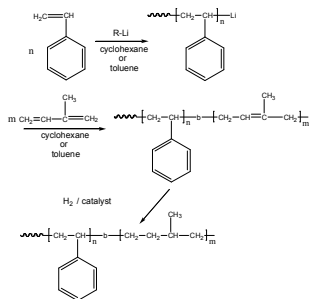


Composition:

Mn x 10 ³ S-b-MB	Mw/Mn (PDI)
6.8-b-18.2	1.07

Synthesis Procedure:

Poly(styrene-b-isoprene) is prepared by living anionic polymerization in non-polar solvent with sequence addition of styrene followed by isoprene and catalytic hydrogenation. The scheme of the reaction is illustrated below:



Characterization:

An aliquot of the anionic polystyrene block was terminated before addition of isoprene and analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI). The block copolymer composition was then calculated from ¹H-NMR spectroscopy by comparing the peak area of the vinylic isoprene (before hydrogenation) proton at about 5.1 ppm with the aromatic protons of polystyrene at about 6.3-7.2 ppm. Copolymer PDI is determined by SEC.

FTIR: After the hydrogenation the product was characterized by FTIR and the disappearance of absorbance at 890 and 841 cm⁻¹ indicate the quantitative hydrogenation.

HNMR of the product also confirm the quantitative hydrogenation (> 96%) of poly isoprene block.

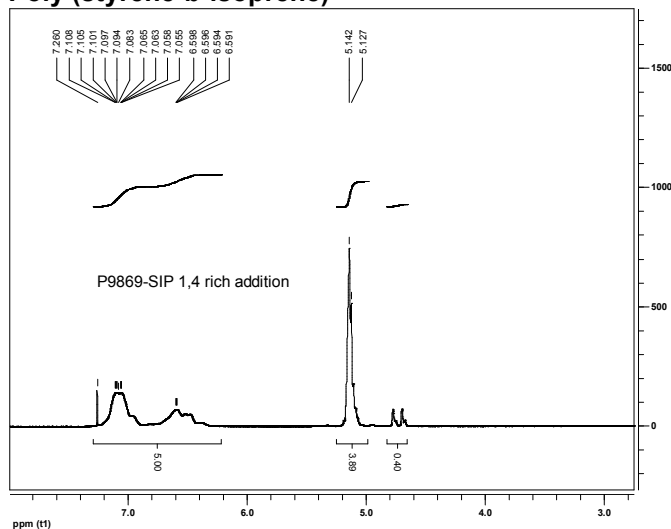
Solubility: Poly(styrene-b-hydrogenated isoprene) is soluble in THF, CHCl₃, toluene.

Purification

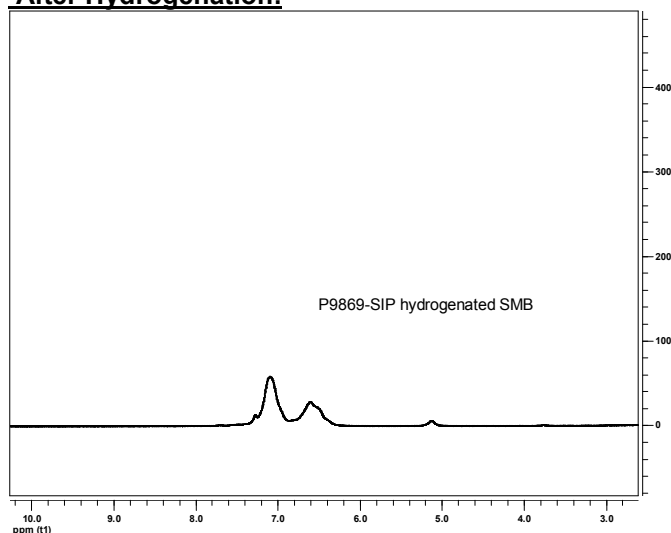
Purification of the obtained polymer was carried out rigorously as follows to ensure the removal of the catalyst side product:

1. Dissolved the polymer in warm toluene and Solution filtered and then passed through a column packed with basic silica at +40 °C.
2. Solution concentrated precipitated in ethanol.
3. Polymer redissolved in toluene and passed again through the silica packed column till get a light color solution. Polymer was recover and dissolved in benzene and filter through a filter paper and the solution freeze dried from benzene. Final dried under vacuum for 48h at 50°C.

¹H-NMR Spectrum of the block copolymer Poly (styrene-b-isoprene)

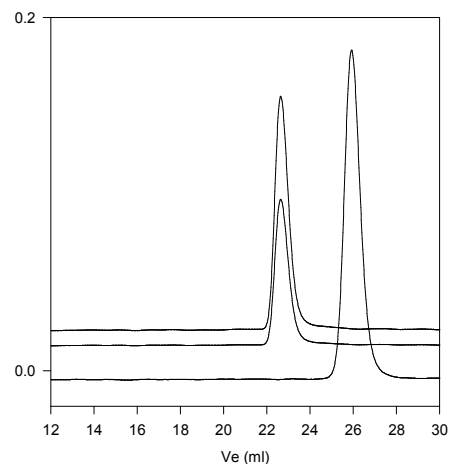


After Hydrogenation:



SEC of Sample of the block copolymer:

P9869-SIP and After Hydrogenation



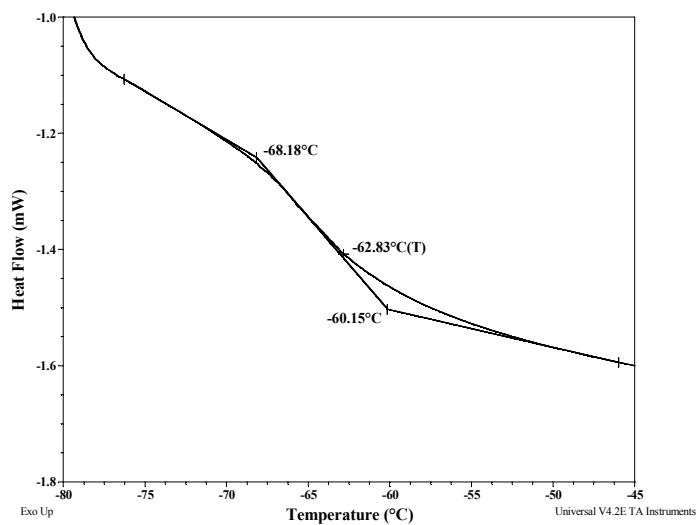
Size exclusion chromatography of polystyrene-b-polyisoprene, 1,4 addition

— Polystyrene, M_n=6800, Mw=7200 PI=1.05

— Block Copolymer:
PS-IP(6800)-b-PI(17,500), PI=1.07 (by H NMR)

after Hydrogenation: PS-b-MB: 6,800-b-18,200 Mw/Mn :1.07

DSC thermogram for Ip block:



DSC thermogram for PS block:

