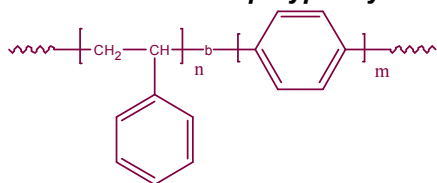


**Sample Name:** Poly(styrene-b-phenylene)  
(Polyphenylene rich in 1,4-addition)

**Sample #:** P6504-SPP  
*para-rich microstructure for polyphenylene block:*

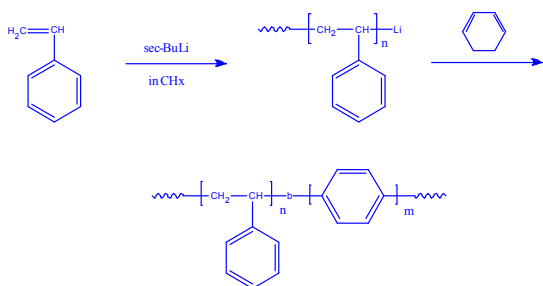


#### Composition:

| Mn x 10 <sup>3</sup><br>S-b-PP      | Mw/Mn (PDI)                         |
|-------------------------------------|-------------------------------------|
| 5.7-2.9                             | 1.14                                |
| T <sub>g</sub> for PS block: 120 °C | T <sub>g</sub> for PP block: 180 °C |

#### Synthesis Procedure:

Poly(styrene-b-phenylene) is prepared by dehydrogenation of poly(styrene-b-1,4-cyclohexadiene), which was synthesized via polymerization in non-polar solvent with sequence addition of styrene followed by 1,3-cyclohexadiene. The scheme of the reaction is illustrated below:



#### Characterization:

An aliquot of the anionic polystyrene block was terminated before addition of cyclohexadiene and analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI). The block copolymer composition was then calculated from <sup>1</sup>H-NMR spectroscopy by comparing the peak area of the vinylic diene proton at 5.7 ppm with the aromatic protons of polystyrene at 6.3-7.2 ppm. Copolymer PDI is determined by SEC. The aromatization of cyclohexadiene was confirmed by NMR with disappearance of peak at 5.7 ppm.

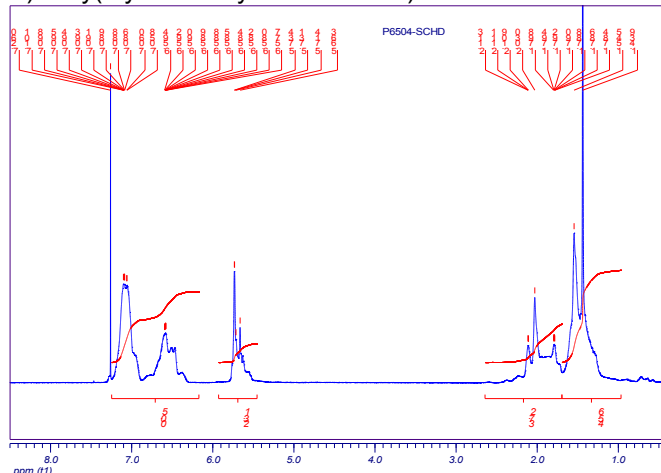
Thermal analysis of the samples was carried out using a differential scanning calorimeter (TA Q100) at a heating rate of 10°C/min. The inflection glass transition temperature (T<sub>g</sub>) has been considered.

#### Solubility:

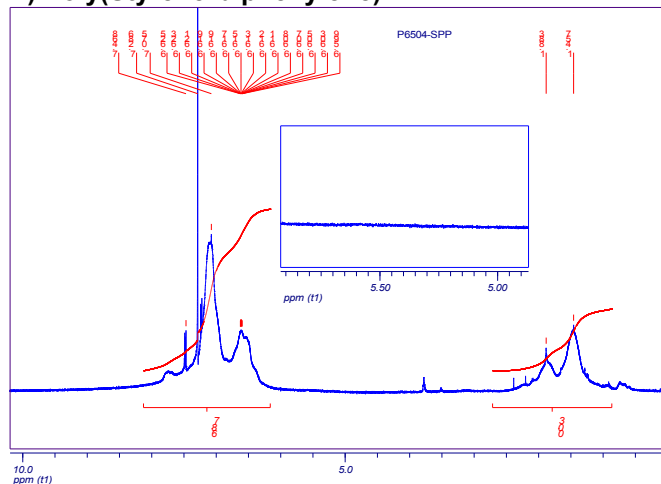
Poly(styrene-b-phenylene) is soluble in THF, toluene, and CHCl<sub>3</sub>. This polymer readily precipitates from methanol, ethanol, and water.

#### <sup>1</sup>H-NMR Spectrum of the block copolymer:

##### A) Poly(styrene-b-cyclohexadiene)

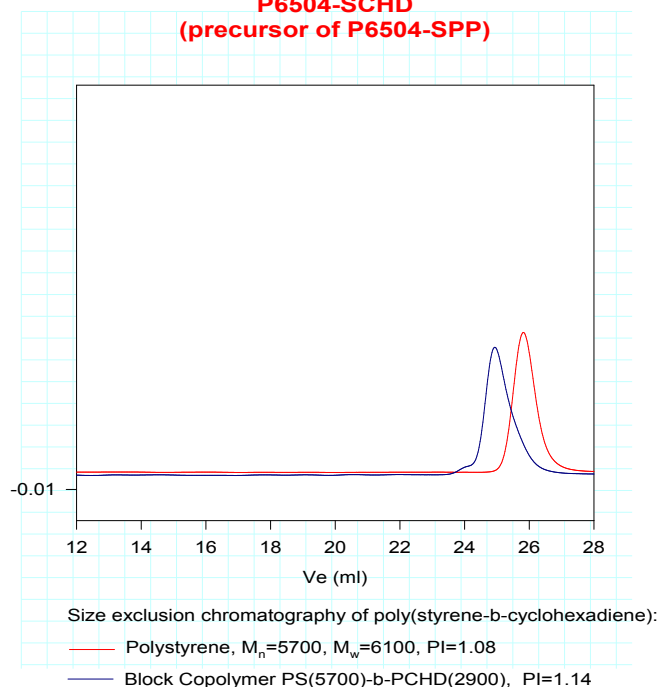


##### B) Poly(Styrene-b-phenylene)

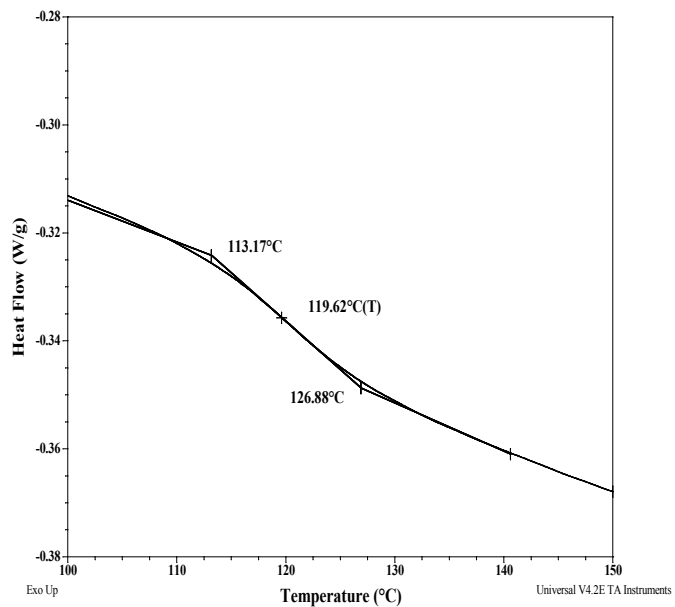


#### SEC of Sample of the block copolymer:

##### P6504-SCHD (precursor of P6504-SPP)



### DSC thermogram for polystyrene block:



### DSC thermogram for polyphenylene block:

