Sample Name:

Deuterated Polystyrene (d₈)- 4 vinyl pyridiene (protonated)

Sample #: P10339-dPS4VP

Structure:

$$\begin{array}{c|c} & & & & \\ \hline \\ & & \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ \end{array} \begin{array}{c|c} & & \\ \end{array} \begin{array}{c|c} & & \\ \hline \\ \end{array} \begin{array}{c|c} & & \\ \hline \end{array} \begin{array}{c|c} & & \\ \end{array} \begin{array}{c|c}$$

Composition:

Mn x 10 ³ (dPS-b-4VP)	PDI
65.0-b-19.0	1.18
Tg for dPS block	104°C
Tg for 4Vp block	149 oC

Synthesis Procedure:

Deuterated poly(styrene-b-4-vinyl pyridiene) diblock copolymer is prepared by living anionic polymerization.

Characterization:

The molecular weight and polydispersity index (PDI) of the block copolymer are characterized by size exclusion chromatography (SEC). composition of the block copolymer was calculated from ¹H-NMR by comparing the peak area of the phenyl polystyrene protons between 6.4 to 7.2 ppm (indicating about 1% protonated fraction) and the ethylene oxide protons at 3.65 ppm. This is given an approximate analysis. The yield of the polymer from the theoretical amount of deuterated styrene and protonated pyridiene monomer calculate also the compositions required.

Thermal analysis

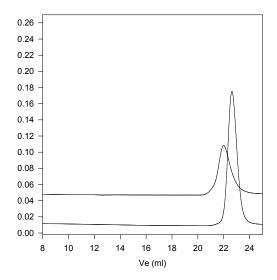
Thermal analysis of the samples was carried out on a TA Q100 differential scanning calorimeter at a heating rate of 10° C/min.The midpoint of the slope change of the heat flow plot of the second heating scan was considered as the glass transition temperature (Γ_g).

Solubility:

The polymer is soluble in THF (at 35°C), CHCl₃, benzene, toluene, dioxane.

SEC of the product:

P10339-dPS4VP

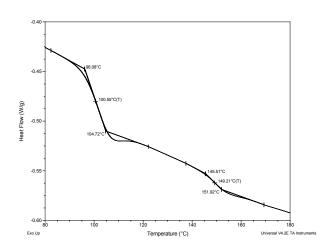


Size exclusion chromatography of P(dPS-b-4VP) in DMF at 40 oC:

____ dPS block: M_n =65,000, M_w =68000, PI=1.05

—— Block Copolymer dPS-4VP (65,000)-b-4VP(19,000), PI=1.18

DSC thermogram for dPS block:



References for further information:

- 1. S. K. Varshney, R. Fayt, Ph. Teyssie, and J.P. Hautekeer US Patent 5,264,527 (1993)
- 2. S. K. Varshney, Jian-Xin Zhang. US Patent 7009,033 B3 2006.