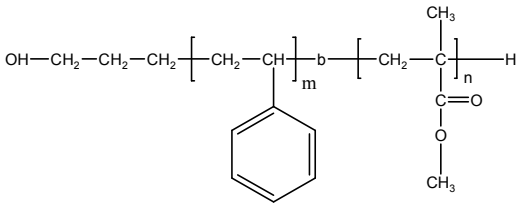
Sample Name: **Hydroxy terminated Poly(styrene-b-methyl methacrylate)**
(polymethylmethacrylate rich in syndiotactic contents > 78%) purified through column of Al2O3
Sample #: **P9211E- HOSMMA**

Structure:

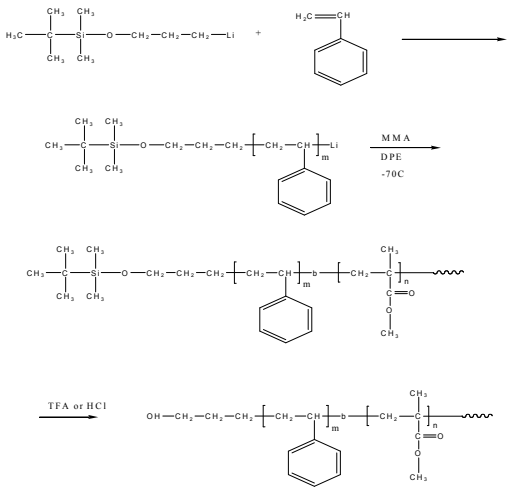


Composition:

Mn x 10^3 S-b-MMA	PDI
26.0-b-66.0	1.29
T_g for PS block: 107°C	T_g for PMMA block: 130°C

Synthesis Procedure:

HO terminated Poly(styrene-b-methyl methacrylate) is prepared by living anionic polymerization in THF at –78 °C using tert.butyl dimethyl siloxy propyl lithium as initiator in the presence of LiCl. Polystyrene macroanions were end capped with a unit of diphenyl ethylene (DPE) before adding methylmethacrylate (MMA) monomer.



Characterization:

An aliquot of the anionic polystyrene block was terminated before addition of MMA and analyzed by size exclusion chromatography (SEC) to obtain the molecular weight and polydispersity index (PDI). The final block copolymer composition was calculated from ^{1}H-NMR spectroscopy by comparing the peak area of the poly(methyl methacrylate) protons (eg. –OCH_3 at 3.6ppm) with the of aromatic protons of polystyrene at 6.3-7.2 ppm. Copolymer PDI is determined by SEC.

Solubility:

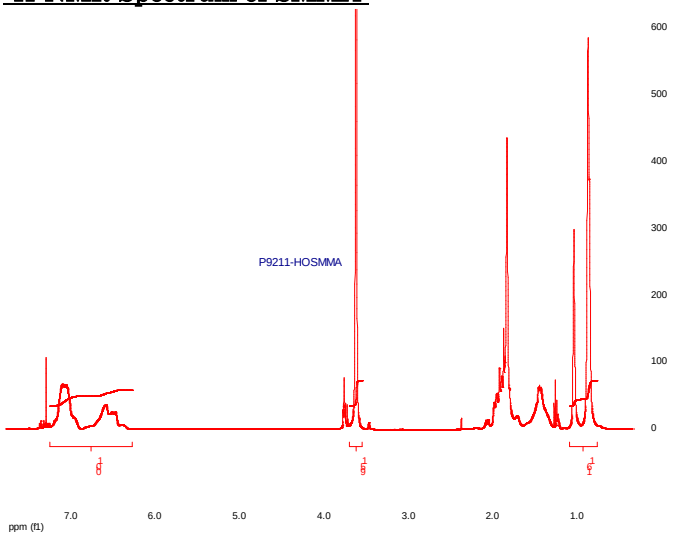
Poly(styrene-b-methyl methacrylate) is soluble in THF, toluene, dioxane and $CHCl_3$. This polymer readily precipitates from methanol, ethanol, hexanes and water.

Purification:

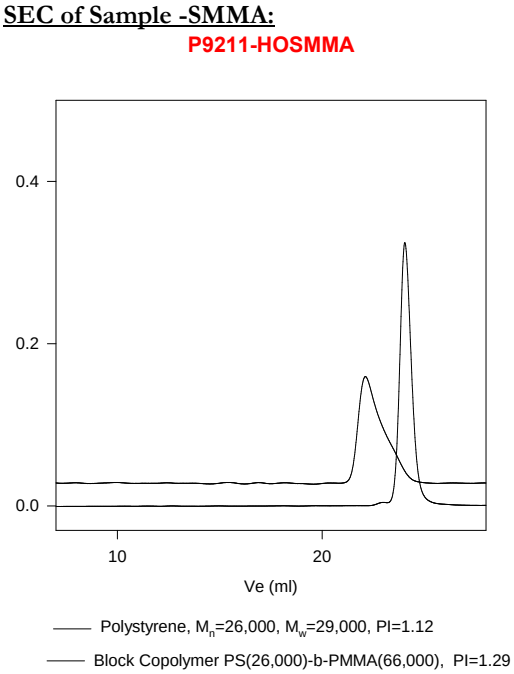
Purification of the obtained polymer was carried out rigorously as follows to ensure the removal of the catalyst side product:

1. Polymer first soxhlet in Cyclohexane to remove trace amount of homopolystyrene fraction if any present.
2. Dissolved the polymer in $CHCl_3$ and wash with de-ionized distilled water to remove any soluble organic catalyst side product.
3. Polymer extracted from water with chloroform.
4. Polymer solution in $CHCl_3$ was dried over anhydrous sodium sulfate.
5. Solution filtered and than passed through a column packed with basic Al_2O_3.
6. Solution concentrated on rota-evaporator
7. Solution precipitated in cold methanol and redissolved in dioxane and freeze dried.
8. Final dried under vacuum for 48h at 50°C.

^{1}H-NMR Spectrum of SMMA:



SEC of Sample -SMMA:



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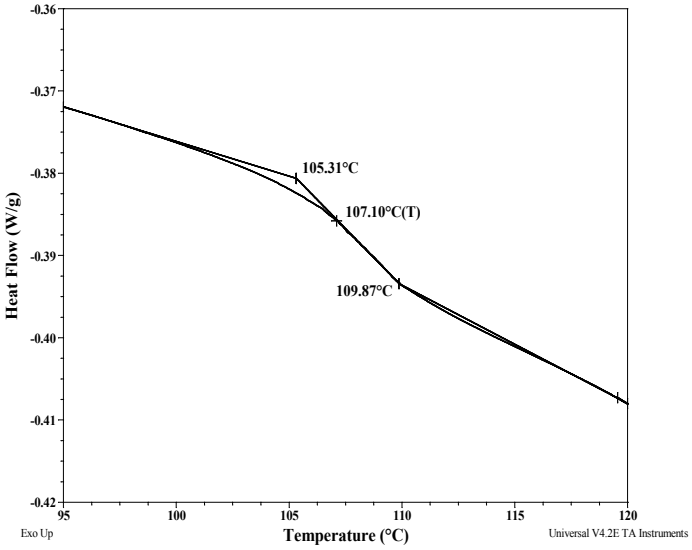
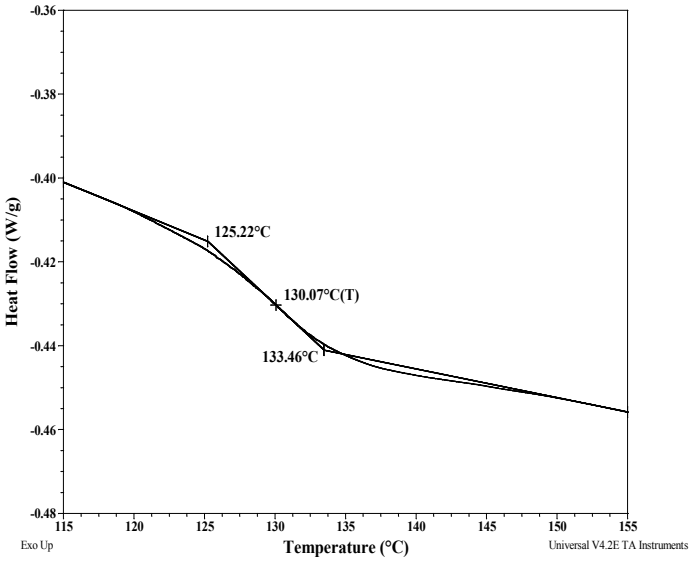
Thermal Analysis of the sample P9211-OHSMMA:

Thermal analysis of the samples was carried out using a differential scanning calorimeter (TA Q100) at a heating rate of 15°C/min. The inflection glass transition temperature (T_g) of the sample has been considered.

Thermogram for PS block:



Thermogram for MMA block:



References for further information:

1. S. K. Varshney, R. Fayt, Ph. Teyssie, and J.P. Hautekeer US Patent 5,264,527 (1993)
2. Ph. Teyssie, Ph. Bayard, R. Jerome, S. K. Varshney, and J. S. Wang, *35th IUPAC International Union of Pure & Applied Chemistry International Symposium on Macromolecules" 1994, 67.*
3. Ph. Teyssie, R. Fayt, J. P. Hautekeer, C. Jacobs, R. Jerome, L. Leemans and S. K. Varshney *Makromolekular Chemie, Macromol. Symp., 1990, 32,61-73.*
4. S. K. Varshney, J. P. Hautekeer, R. Fayt, R. Jerome, and Ph.Teyssie *Macromolecules, 1990, 23, 2618-2622.*
5. R. Jerome, R. Forte, S. K. Varshney, R. Fayt, and Ph. Teyssie "The Anionic Polymerization of Alkylacrylates:A Challenge" in the Recent Advances in Mechanistic and Synthetic Aspects of Polymerization: M. Fontanaille and A. Guyot Ed., NATO ASI Series *C 215,101 (1987), CA Vol. 108, 12, 094992.*