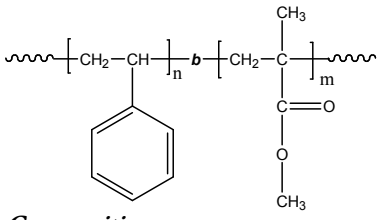
Sample Name:

Poly(styrene-b-methyl methacrylate) (polymethylmethacrylate rich in syndiotactic contents > 78%)

Sample #: **P4622P-SMMA (purified)**

Structure:



Composition:

Mn x 10^3 S-b-MMA	PDI
20.0-b-80.0	1.08
T_g for PS block: 107°C	T_g for PS block: 133°C

***Synthesis Procedure*:**

Poly(styrene-b-methyl methacrylate) is prepared by living anionic polymerization in THF at –78 °C using sec.BuLi initiator in the presence of LiCl. Polystyrene macroanions were end capped with a unit of diphenyl ethylene (DPE) before adding methylmethacrylate (MMA) monomer. For further details please see our published articles.[1-5]

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 that of aromatic protons of polystyrene at 6.3-7.2 ppm. Copolymer PDI is determined by SEC.

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.

Solubility:

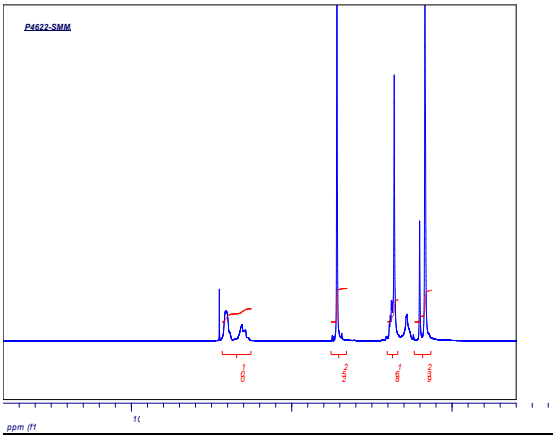
Poly(methyl methacrylate) is soluble in THF, $CHCl_3$, toluene and dioxane. The polymer precipitates from hexanes, methanol and ethanol.

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

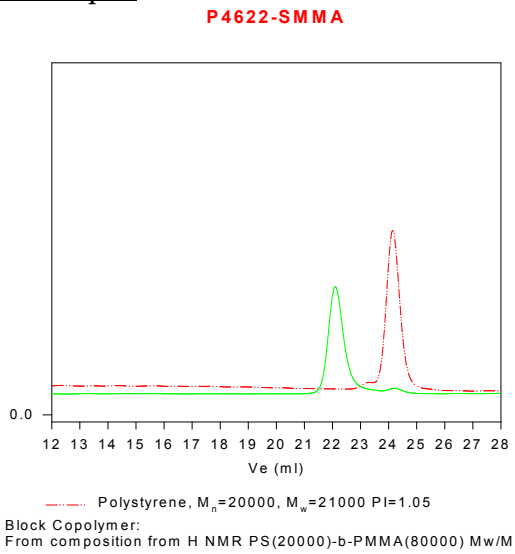
After Soxhlet the polymer using cyclohexane to remove any traces amount of homopolystyrene.:

1. Dissolved the polymer in CHCl3 and wash with de-ionized distilled water to remove the any soluble organic catalyst side product.
2. Polymer extracted from water with chloroform.
3. Polymer solution in CHCl3 was dried over anhydrous sodium sulfate.
4. Solution filtered and than passed through a column packed with basic Al_2O_3 .
5. Solution concentrated on rota-evaporator
6. Solution precipitated in cold methanol and redissolved in dioxane and freeze dried.
7. Final dried under vacuum for 48h at 50°C.

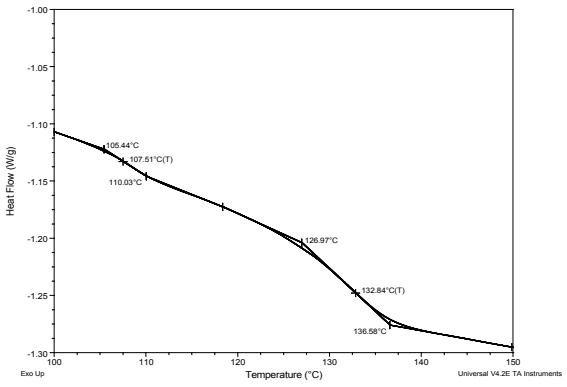
^{1}H-NMR Spectrum of the Polymer:



SEC of Sample :



Thermogram of the sample



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.*