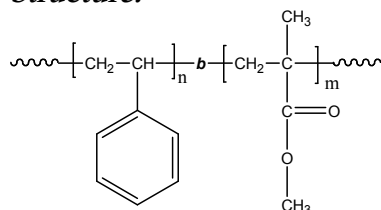


**Sample Name:** Poly(styrene-*b*-methyl methacrylate)  
(polymethylmethacrylate rich in syndiotactic contents > 80%)

**Sample #:** P11429-SMMA Electronic Grade

**Structure:**



**Composition:**

Mn x 10 <sup>3</sup> S-b-MMA	PDI
31.0-33.0	1.08
T <sub>g</sub> for PS block: 103°C	T <sub>g</sub> for PMMA block: 103°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.<sup>1-5</sup>

#### 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 <sup>1</sup>H-NMR spectroscopy by comparing the peak area of the poly(methyl methacrylate) protons (eg. -OCH<sub>3</sub> 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 10°C/min. The inflection glass transition temperature (T<sub>g</sub>) of the sample has been considered.

#### Solubility:

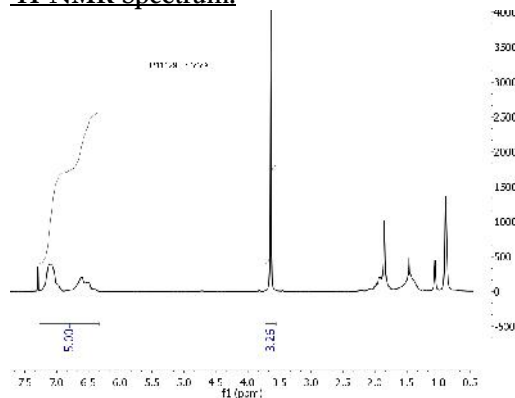
Poly(styrene-*b*-methyl methacrylate) is soluble in THF, toluene, dioxane and CHCl<sub>3</sub>. This polymer readily precipitates from methanol, ethanol, hexanes and water.

#### Purification:

Purification of the obtained polymer was carried out rigorously as follows to ensure the removal of the catalyst side product: After Soxhlet the polymer using cyclohexane to remove any traces amount of homopolystyrene:

1. Dissolved the polymer in CHCl<sub>3</sub> 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 CHCl<sub>3</sub> was dried over anhydrous sodium sulfate.
4. Solution filtered and than passed through a column packed with basic Al<sub>2</sub>O<sub>3</sub>.
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.

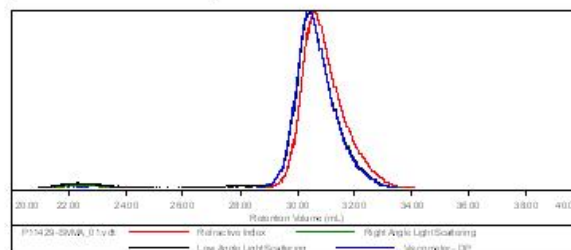
#### <sup>1</sup>H-NMR Spectrum:



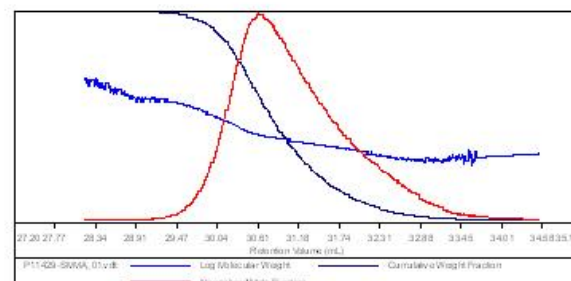
#### SEC of Sample

Sample ID: P11429-SMMA

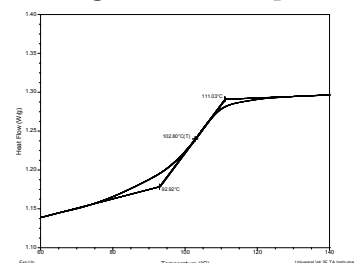
Concentration (mg/mL)	4.7984
Sample chide (mL/g)	0.1390
Method File	P0801-4pr-2013-0000.ucm
Column Set	3x PL 1113-6300
System	System 1



Sample	Mn	Mw	Mp	Mw/Mn	PDI
P11429-SMMA_01.vit	64,686	69,915	70,425	1.081	0.4652



#### 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 Makromolekulare 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.