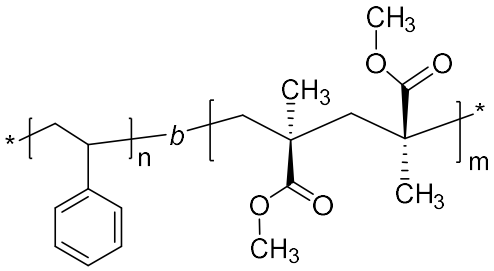
Sample Name: **Poly(styrene-b-methyl methacrylate)**

(PMMA block is predominantly syndiotactic, 80%)

Sample # **P9758A-SMMA**

Structure:



Composition:

$M_n \times 10^3$ (g/mol) [PS–*b*–PMMA]	M_w/M_n
63.7–*b*–64.3	1.02

T_{g1} (PS block)	T_{g2} (PMMA block)
107 °C	130 °C

Synthesis Procedure:

Poly(styrene-*block*-methyl methacrylate) was prepared by living anionic polymerization of styrene in THF at -78 °C using sec-BuLi initiator in presence of LiCl. Polystyrene macroanions were end-capped with a unit of diphenyl ethylene (DPE) before adding methyl-methacrylate (MMA) monomer.

For further details, please, see our publications [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 of the first block (PS). The final composition (ratio between PS and PMMA blocks in the diblock copolymer) was calculated from proton NMR spectroscopy by comparing the peak area of the poly(methyl methacrylate) protons (-OCH_3 at 3.6 ppm) with the of aromatic protons of polystyrene (at 6.3–7.2 ppm). Polydispersity of the diblock copolymer was determined by SEC of the final product.

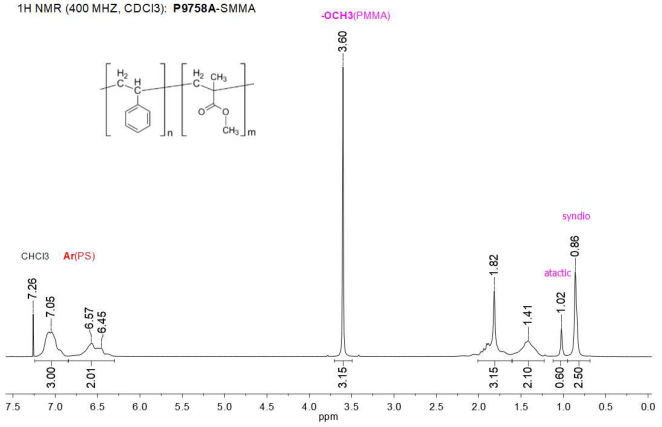
Tacticity of the PMMA block was calculated by NMR.

Thermal analysis was performed on TA Instruments Q100 differential scanning calorimeter (DSC) under a nitrogen atmosphere. The glass transition temperature (T_g) of the polymer was measured at a scan rate of 10°C/min shortly after creating thermal history of the sample.

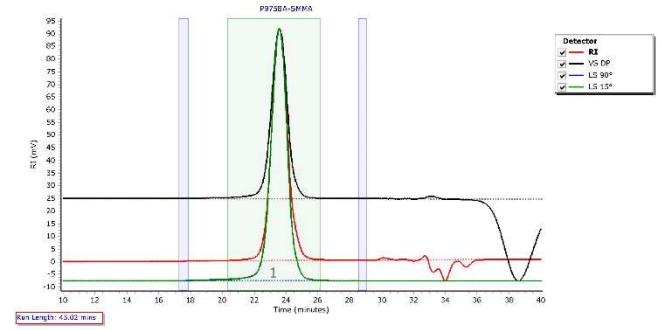
Solubility:

Poly(styrene–b–methyl methacrylate) is soluble in THF, toluene, dioxane, and chloroform. The diblock copolymer precipitates from methanol, ethanol, hexanes, and water.

^{1}H NMR spectrum of PS-PMMA in chloroform-*d*:



SEC chromatogram of PS-PMMA in THF:



Triple Analysis

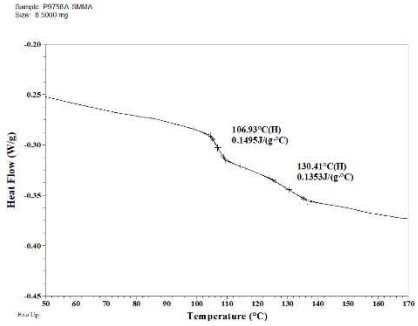
Molecular Weight Averages

Peak	Mp (g/mol)	Mn (g/mol)	Mw (g/mol)	Mz (g/mol)	Mz+1 (g/mol)	Mv (g/mol)	PD
Peak 1	130060	128031	131042	134275	137996	133924	1.024

Processing Parameters

Concentration options	Calculate Sample Properties from Entered Sample Concentration
Entered sample concentration (mg/mL)	9.175
Calculated dn/dc (mL/g)	0.131
MW calculation method	Use all angles
Log MI-v-RT curve fit options	Set the fit limits using the limits at peak width of 10 %.

DSC thermogram of PS-PMMA (2 heating scan, 10°C/min):



References:

1. S.K.Varshney, R.Fayt, Ph.Teyssie, J.P.Hautekeer; *US Patent* (1993) 5, 264, 527.
2. Ph.Teyssie, Ph.Bayard, R.Jerome, S.K.Varshney, 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, S.K.Varshney; *Makromolekular Chemie, Macromol. Symp.* 1990, 32, 61–73.
4. S.K.Varshney, J.P.Hautekeer, R.Fayt, R.Jerome, Ph.Teyssie; *Macromolecules* 1990, 23, 2618–2622.
5. R.Jerome, R.Forte, S.K.Varshney, R.Fayt, Ph.Teyssie; *"The Anionic Polymerization of Alkylacrylates:A Challenge" in Recent Advances in Mechanistic and Synthetic Aspects of Polymerization*: M. Fontanaille and A. Guyot Ed., *NATO ASI Series C* (1987) 215, 101, CA Vol. 108, 12, 094992.