Electronic Supplementary Material (ESI) for Polymer Chemistry.
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Supporting Informations

Diffusion-Ordered SpectroscopY NMR DOSY: A All-In-One Tool to Simultaneously Follow Side Reactions,Mechanism Livingness and Molar Masses of PolyMethylMethacrylate by Nitroxide Mediated Polymerization

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Monomer conversion by ^{1}H NMR.

The monomer to polymer conversion for the polymerization of MMA was calculated using ^{1}H NMR spectra, via comparison between integral of polymer peak and monomer peak, using the signals from the methoxy (O–CH3) (δ 3.75 ppm) hydrogens of the unreacted monomer and the methoxy (O–CH3) (δ 3.60 ppm) of the polymer.

$$\% \text{ MMA conversion} = [\int 3.6 / (\int 3.6 + \int 3.75)] \times 100$$

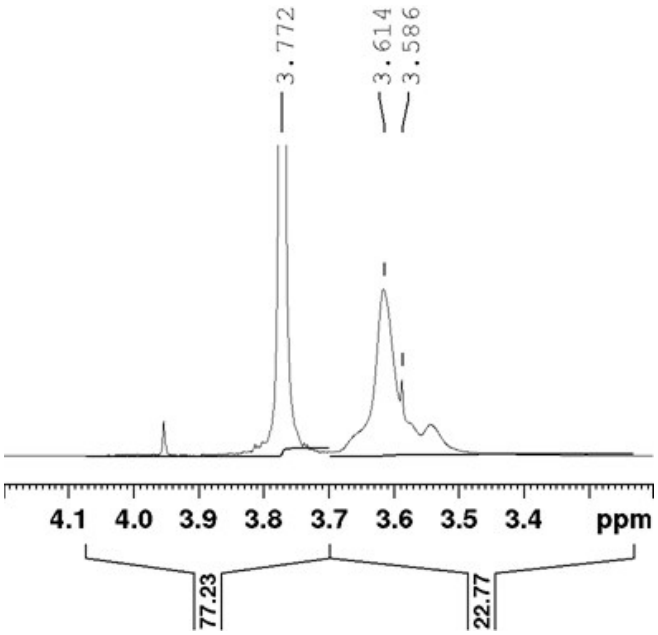

Fig. SI.1. ^{1} H NMR spectrum (CDCl3, 400 MHz) of PMMA

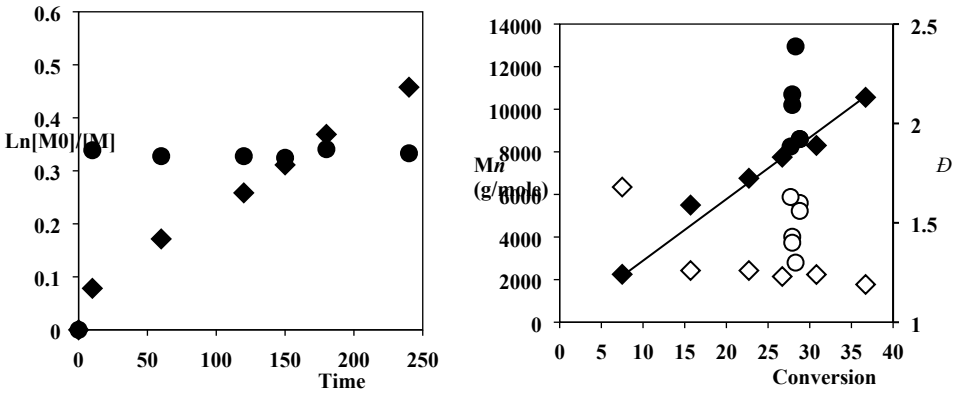

Fig. SI.2. Bulk polymerizations ofMMA and MMA-*co*-S at 80°C : (left) variation of Ln [M]0/[M] *vs.* time and (right) evolution ofMn_{SEC} and *Đvs.* conversion (pure MMA(℘,ᘓ) and (MMA-*co*-S) (◆,❋)).

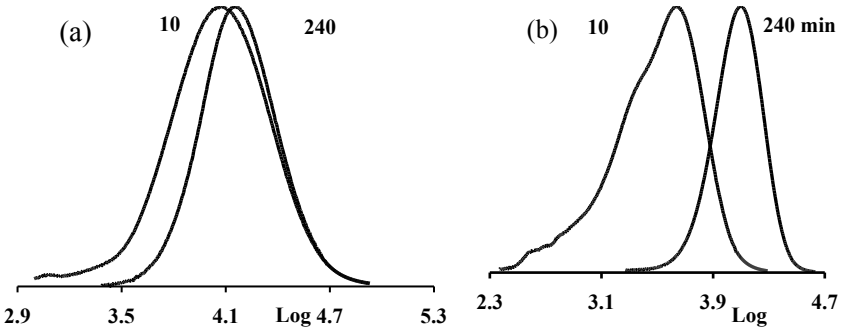

Fig.SI.3. Size exclusion chromatograms superposition of (a) PMMA and (b) P(MMA-*co*-S) depending of time of polymerization.

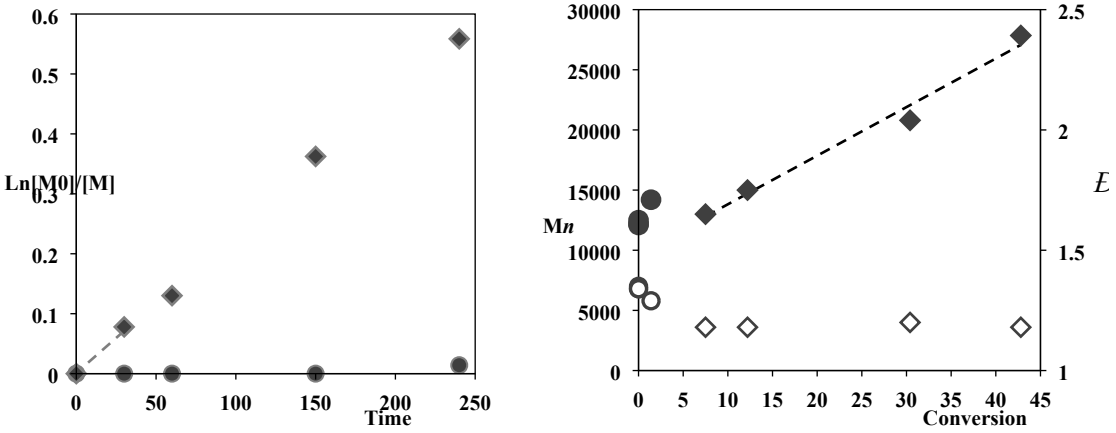

Fig. SI.4.BA Bulk polymerizations and chains extensionfrom PMMA and P(MMA-*co*-S) at 120°C : (Left) Variation of $Ln[M]_0/[M]$ *vs.* time and (Right) Evolution of the M*n*(black) and

Đ(Mw/Mn)(white) *vs.* conversion for PMMA-*b*-PBA (ଌ,ଓ) and P(MMA-c*o*-S)-*b*-PBA (◆,❋) syntheses.

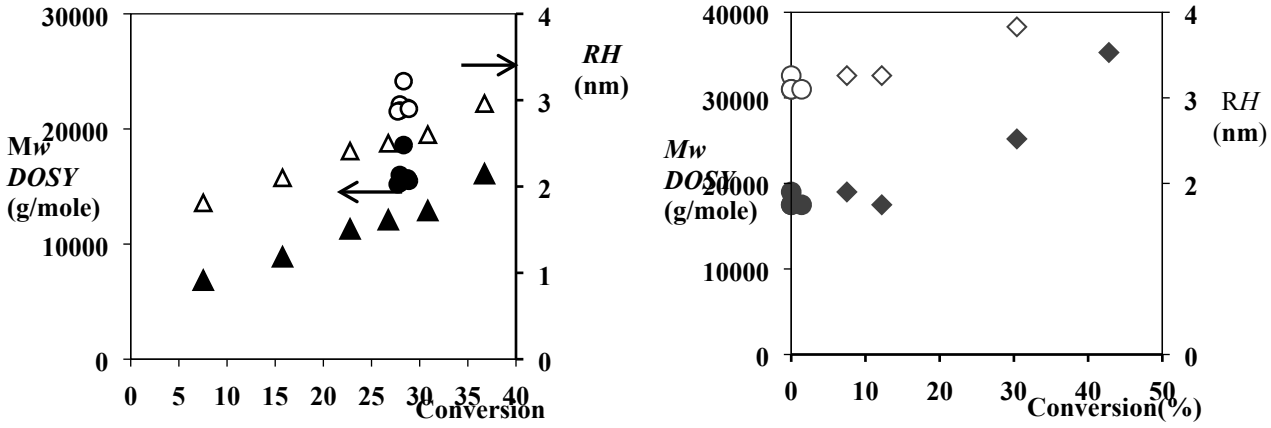

Fig. SI-5.$M_{w\,DOSY}$(black) and R_H(white) values by DOSY *vs.* polymerization conversion (left) for PMMA (ଌ,ଓ) and (MMA-*co*-S) (◆,❋) and (right) PMMA-*b*-PBA (ଌ,ଓ)and P(MMA-co-S)-*b*-BA (◆,❋)diblockcopolymers