Electronic Supplementary Material (ESI) for Polymer Chemistry.

Supplementary Information

"One-step" synthesis of poly(methacrylate)-*b*-polyester via "one organocatalyst, two polymerizations"

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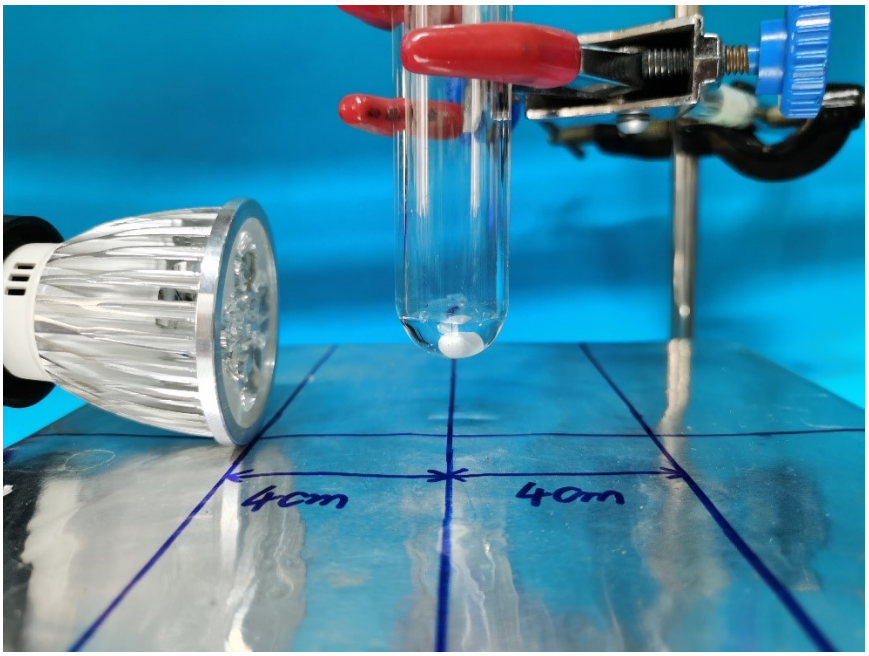

Figure S1 the picture of LED-light (the distance between the LED and the Schlenk tube is 4 centimeters).

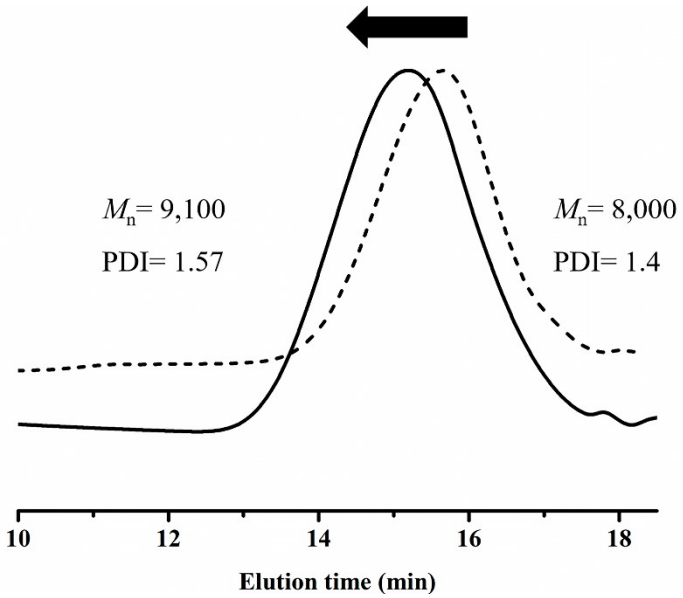

Figure S2 GPC traces of PMMA and block copolymer PMMA-*b*-PTMC (entry 1, Table 1).

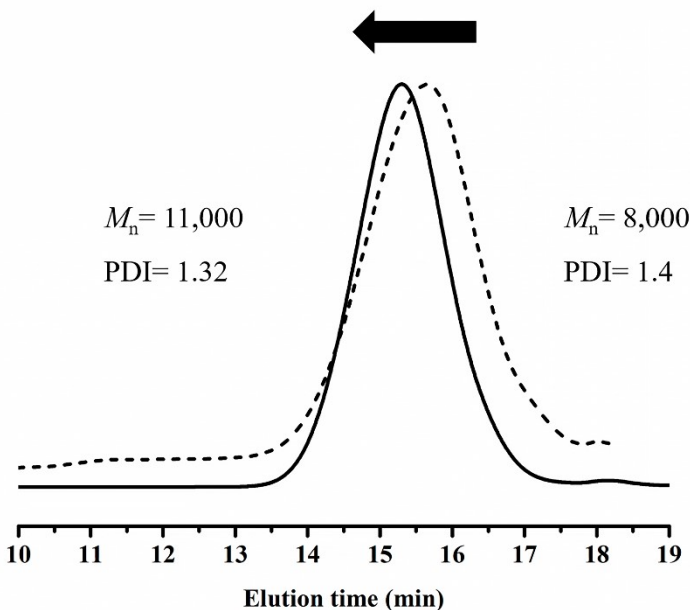

Figure S3 GPC traces of PMMA and block copolymer PMMA-*b*-PTMC (entry 2, Table 1).

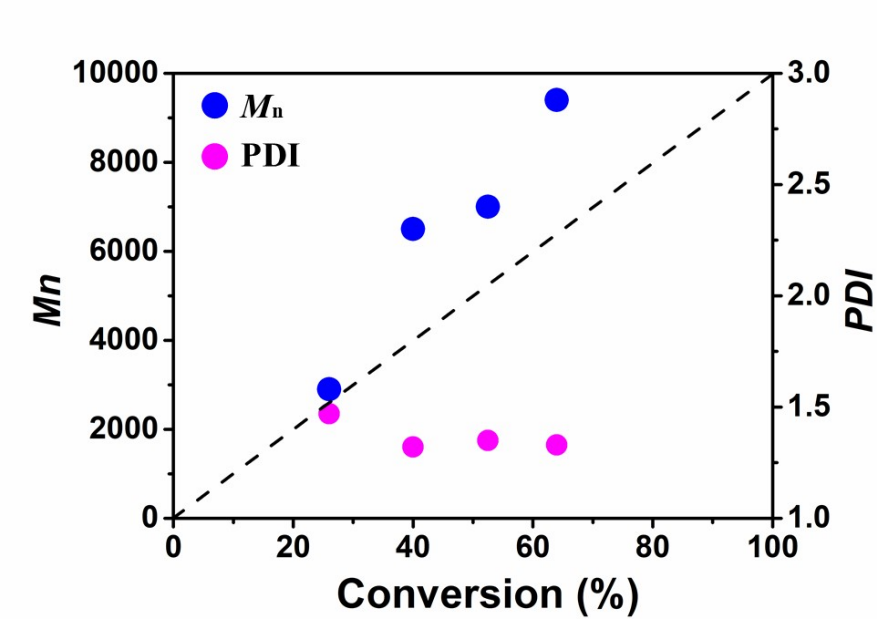

Figure S4 The number-average molecular weight ($M_{n,GPC}$) and PDI versus monomer conversion of block copolymer PMMA-*b*-PTMC.

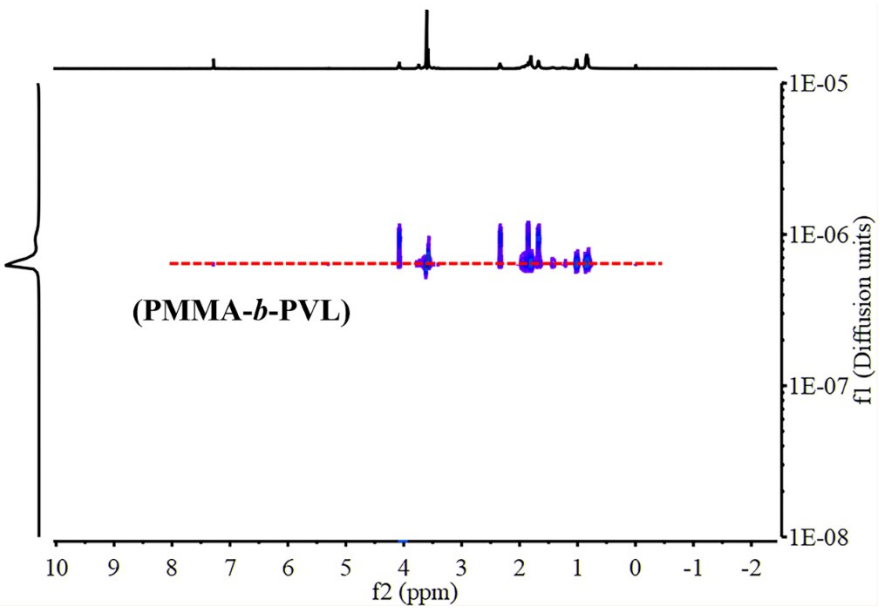

Figure S5 ^{1}H DOSY NMR spectrum of the block copolymer PMMA-*b*-PVL was synthesized via "one-pot, one step" (entry 4, Table 2).

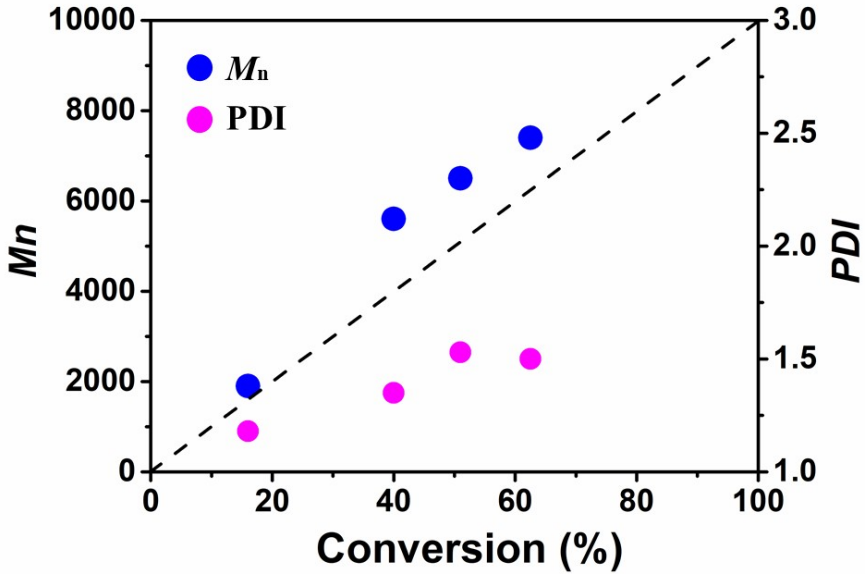

Figure S6 The number-average molecular weight ($M_{n,GPC}$) and PDI versus monomer conversion of block copolymer PMMA-*b*-PVL.

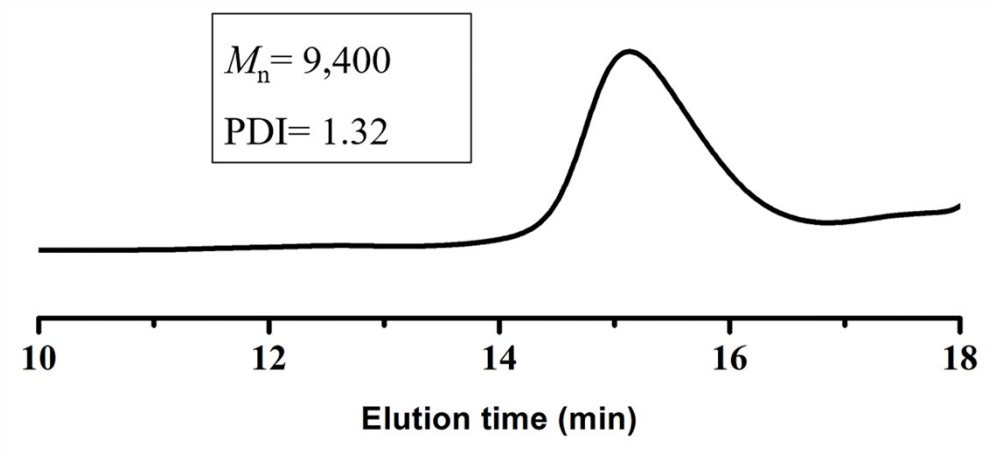

Figure S7 GPC trace of block copolymer PBzA-*b*-PTMC was synthesized via "one-pot, one step" (entry 5, Table 2).

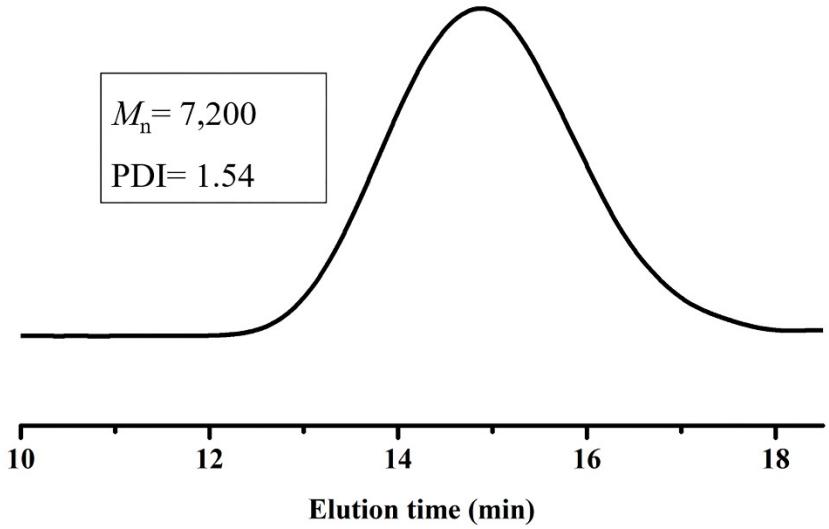

Figure S8 GPC trace of block copolymer PBzMA-*b*-PVL was synthesized via "one-pot, one step" (entry 6, Table 2).

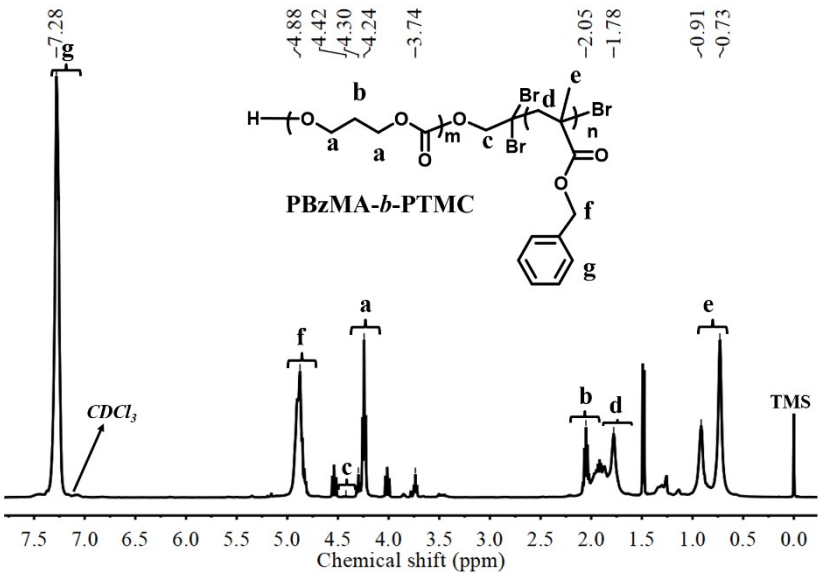

Figure S9 [1]H NMR spectrum of block copolymer PBzMA-*b*-PTMC was synthesized via "one-pot, one step" (entry 5, Table 2).

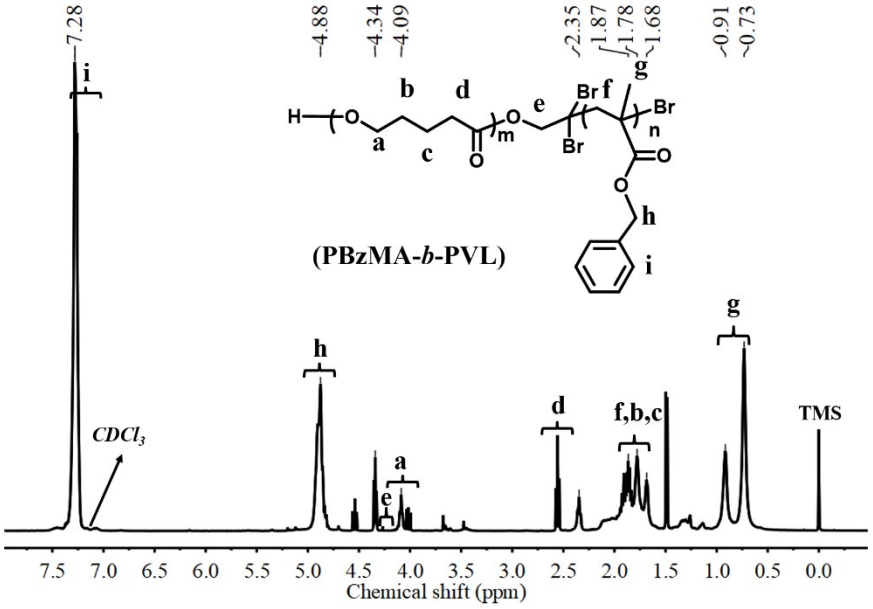

Figure S10 [1]H NMR spectrum of block copolymer PBzMA-*b*-PVL was synthesized via "one-pot, one step" (entry 6, Table 2).

Table S1 Photo-ATRP of MMA only or ROP of TMC only.

Entry	Monomer	[M]/[TBE] /[THS] (mM)	*t* (h)	Conv. (%)
1	MMA	50/1/1	2	8
			4	18
			6	35
			8	41
			10	51
2	TMC	50/1/1	4	7
			6	15
			8	20
			10	27

Table S2 Photo-ATRP of MMA only or ROP of δ-VL only.

Entry	Monomer	[M]/[TBE] /[THS] (mM)	*t* (h)	Conv. (%)
1	MMA	50/1/0.5	4	20
			6	27
			8	36
			10	52
2	δ-VL	50/1/0.5	4	9
			6	18
			8	27
			10	40