Supplementary Information

## Synthesis of block copolymers using end-functionalized polyacetylenes as

## macroinitiators

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Fig. S1 <sup>1</sup>H NMR (400 MHz) spectrum of poly(1a) measured in CDCl<sub>3</sub>.



Fig. S2 IR spectrum of poly(1a).



Fig. S3 SEC curve of poly(1a) (Sample: Table 1, run 1).



Fig. S4 SEC curve of poly(1a) (Sample: Table 1, run 2).



Fig. S5 <sup>1</sup>H NMR (400 MHz) spectrum of poly(1b) measured in CDCl<sub>3</sub>.



Fig. S6 IR spectrum of poly(1b).



Fig. S7 SEC curve of poly(1b) (Sample: Table 1, run 5).



Fig. S8 <sup>1</sup>H NMR (400 MHz) spectrum of poly(1c) measured in CDCl<sub>3</sub>.



Fig. S9 IR spectrum of poly(1c).



Fig. S10 SEC curve of poly(1c) (Sample: Table 1, run 7).



Fig. S11 <sup>1</sup>H NMR (400 MHz) spectrum of Si coupled poly(1a) measured in CDCl<sub>3</sub>.



Fig. S12 SEC curve of poly(1a) before Si coupling.



Fig. S13 SEC curve of poly(1a) after Si coupling.



Fig. S14 <sup>1</sup>H NMR (400 MHz) of poly(1a)-*block*-poly(2a) measured in CDCl<sub>3</sub>.



Fig. S15 IR spectrum of poly(1a)-block-poly(2a).



Fig. S16 SEC curve of poly(1a)-*block*-poly(2a).



Fig. S17 <sup>1</sup>H NMR (400 MHz) spectrum of poly(1a)-*block*-poly(2b) measured in CDCl<sub>3</sub>.



Fig. S18 IR spectrum of poly(1a)-*block*-poly(2b).



Fig. S19 SEC curve of poly(1a)-*block*-poly(2b).



Fig. S20 <sup>1</sup>H NMR (400 MHz) spectrum of poly(1b)-*block*-poly(2a) measured in CDCl<sub>3</sub>.



Fig. S21 IR spectrum of poly(1b)-block-poly(2a).



Fig. S22 SEC curve of poly(1b)-*block*-poly(2a).



Fig. S23 <sup>1</sup>H NMR (400 MHz) spectrum of poly(1c)-*block*-poly(2b) measured in CDCl<sub>3</sub>.



Fig. S24 IR spectrum of poly(1c)-*block*-poly(2b).



Fig. S25 SEC curve of poly(1c)-*block*-poly(2b).



Fig. S26 <sup>1</sup>H NMR (400 MHz) spectrum of poly(2a) measured in CDCl<sub>3</sub>.



Fig. S27 IR spectrum of poly(2a).