Electronic supplementary information for:

Multiresponsive of highly water-soluble poly(3-hexylthiophene)-*block*-poly(phenylisocyanide) block copolymers

Ming Su, Sheng-Yu Shi, Qian Wang, Na Liu,* Jun Yin, Chunhua Liu, Yunsheng Ding and Zong-Quan

Wu*

Department of Polymer Science and Engineering, School of Chemistry and Chemical Engineering, Hefei

University of Technology and Anhui Key Laboratory of Advanced Functional Materials and Devices,

Anhui Province, Hefei 230009, China

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Fig. S1 ¹H NMR spectrum of poly- 1_{30} in CDCl₃ at 25 °C (600 MHz).



Fig. S2 ¹H NMR spectrum of poly- $\mathbf{2}_{30}$ in CDCl₃ at 25 °C (600 MHz).



Fig. S3 ¹H NMR spectrum of poly($\mathbf{1}_{30}$ -*b*- $\mathbf{2}_{60}$) in CDCl₃ at 25 °C (600 MHz).



Fig. S4 FT-IR Spectrum of P3HT, PPI homopolymers and P3HT-*b*-PPI block copolymer at 25 °C using KBr pellets.



Fig. S5 Size exclusion chromatograms of Ni(II)-terminated P3HT macroinitiators and the resulting P3HT-*b*-PPI block copolymers. (a) run 2, (b) run 3, (c) run 4, and (d) run 5 in Table 1 in main text.



Fig. S6 UV-vis absorption spectra of P3HT, PPI, and P3HT-*b*-PPI block copolymer measured in CHCl₃

at 25 °C (c = 0.05 g/L).



Fig. S7 UV-vis spectra of P3HT-*b*-PPI block copolymer $poly(\mathbf{1}_{30}-b-\mathbf{2}_{60})$ in the mixed solvents of THF and methanol at 25 °C (c = 0.05 g/L).



Fig. S8 Photographs of P3HT-*b*-PPI block copolymer $poly(\mathbf{1}_{30}-b-\mathbf{2}_{60})$ in the mixed solvents of THF and methanol under UV light (365 nm) (c = 0.05 g/L).



Fig. S9 Fluorescent spectra of P3HT-*b*-PPI block copolymer $poly(1_{30}-b-2_{60})$ in the mixed solvents of THF and methanol with different volume ratios at 25 °C (c = 0.05 g/L).



Fig. S10 Dynamic light scattering curve of P3HT-*b*-PPI block copolymer $poly(1_{30}-b-2_{60})$ in THF (c = 0.1

g/L) measured at 25 °C.



Fig. S11 Proposed self-assembly structure for P3HT-*b*-PPI block copolymer $poly(1_{30}-b-2_{60})$ in water at

25 °C.



Fig. S12 Reversible emission changes of P3HT-*b*-PPI block copolymer $poly(\mathbf{1}_{30}-b-\mathbf{2}_{60})$ in CHCl₃ (c = 0.05 g/L) upon alternate addition of TFA (0.15 mM) and TEA (0.15 mM) at 25 °C with excitation at 365 nm.



Fig. S13 UV-vis spectra of P3HT homopolymer poly- $\mathbf{1}_{30}$ in CHCl₃ upon alternate additions of TFA and TEA at 25 °C (c = 0.05 g/L).



Fig. S14 1 H NMR spectrum of Me-TEG-Ts in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S15 ¹H NMR spectrum of compound **3** in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S16 1 H NMR spectrum of compound 4 in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S17 ¹H NMR spectrum of compound 5 in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S18 13 C NMR spectrum of compound 5 in CDCl₃ at 25 °C (150 MHz).



Fig. S19 ¹H NMR spectrum of compound **6** in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S20 13 C NMR spectrum of compound 6 in CDCl₃ at 25 °C (150 MHz).



Fig. S21 ¹H NMR spectrum of compound 7 in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S22 13 C NMR spectrum of compound 7 in CDCl₃ at 25 °C (150 MHz).



Fig. S23 ¹H NMR spectrum of monomer **2** in CDCl₃ at 25 $^{\circ}$ C (600 MHz).



Fig. S24 13 C NMR spectrum of monomer 2 in CDCl₃ at 25 °C (150 MHz).