Electronic Supplementary Information for:

One pot synthesis of

poly(3-hexylthiophene)-*b*-poly(quinoxaline-2,3-diyl) rod-rod diblock copolymer and its tunable light emission properties

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Fig. S1 Size exclusion chromatograms of 3 synthesized using different feed ratios of 2 to macroinitiator 1 with $M_n = 5.5$ kDa, $M_w/M_n = 1.17$ (The feed ratio was indicated in legend).



Fig. S2 Size exclusion chromatograms of macroinitiators 1b–1e, and their respective block copolymers 3b–3e (see Fig. 1a of main text for SEC traces of 1a and 3a).



Fig. S3 Absorption spectra of P3HT, poly(quinoxaline-2,3-diyl) and P3HT-*b*-poly(quinoxaline-2,3-diyl) block copolymer (**3a**) in CHCl₃ at room temperature (c = 0.05 mg/mL).



Fig. S4 FT-IR spectra of P3HT, poly(quinoxaline-2,3-diyl) and P3HT-*b*-poly(quinoxaline-2,3-diyl) block copolymer (**3a**) measured at room temperature (KBr).



Fig. S5 Concentration dependent absorption spectra of P3HT-*b*-poly(quinoxaline-2,3-diyl) block copolymer (3a) in 1/4 mixture of chloroform and methanol at room temperature (v/v).



Fig. S6 Plot of absorption intensity at 550 nm of **3a** as a function of the concentration (solvent: chloroform/methanol = 1/4, v/v).



Fig. S7 Reversible emission intensity changes of P3HT-*b*-poly(quinoxaline-2,3-diyl) block copolymer **3a** in 3/1 and 1/4 mixtures of chloroform and methanol at room temperature (c = 0.05 mg/mL).



Fig. S8 ¹H NMR (600 MHz) spectroscopy of 4,5-dimethylbenzene-1,2-diamine measured in CDCl₃ at 25 °C.



Fig. S9 ¹H NMR (600 MHz) spectroscopy of 1,2-diisocyano-4,5-dimethylbenzene (2) measured in CDCl₃ at 25 °C.



Fig. S10 ¹³C NMR (150 MHz) spectroscopy of 1,2-diisocyano-4,5-dimethylbenzene measured in CDCl₃ at 25 °C.



Fig. S11 FT-IR spectrum of 1,2-diisocyano-4,5-dimethylbenzene (**2**) measured at room temperature (KBr).



Fig. S12 ¹H NMR (600 MHz) spectroscopy of P3HT measured in CDCl₃ at 25 °C.



Fig. S13 ¹H NMR (600 MHz) spectroscopy of P3HT-*b*-poly(quinoxaline-2,3-diyl) measured in CDCl₃ at 25 °C.



Fig. S14 ¹H NMR (600 MHz) spectroscopy of poly(quinoxaline-2,3-diyl) measured in CDCl₃ at 25 °C.



Fig. S15 Height (a) and phase (b) AFM images of the self-assembled structure of 3c.