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Supporting Information of Manuscript Entitled with

Preparation and self-assembly of two-length-scale A-*b*-(B-*b*-A)_n-*b*-B multiblock copolymers

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Fig. S1 ¹³C-NMR spectra of a multiblock copolymer in acetone-d₆ (a) before and (b) after hydrolysis.

Fig. S2 Possible lamellar self-assembled structures with 1, 3, 5 and 7 thin internal layers, respectively, for an $A-b-(B-b-A)_6-b-B-b-A$ block copolymer consisting of 7 short inner blocks and two long outer blocks.

Fig. S3 Bright-field TEM image of a $S-b-(pHS-b-S)_3-b-pHS$ octablock copolymer, where $\overline{M}_n$ (outer blocks) = 26,000 g·mol⁻¹ and $\overline{M}_n$ (inner blocks) = 8,700 g·mol⁻¹, stained with OsO₄. Scale bar represents 100 nm.