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

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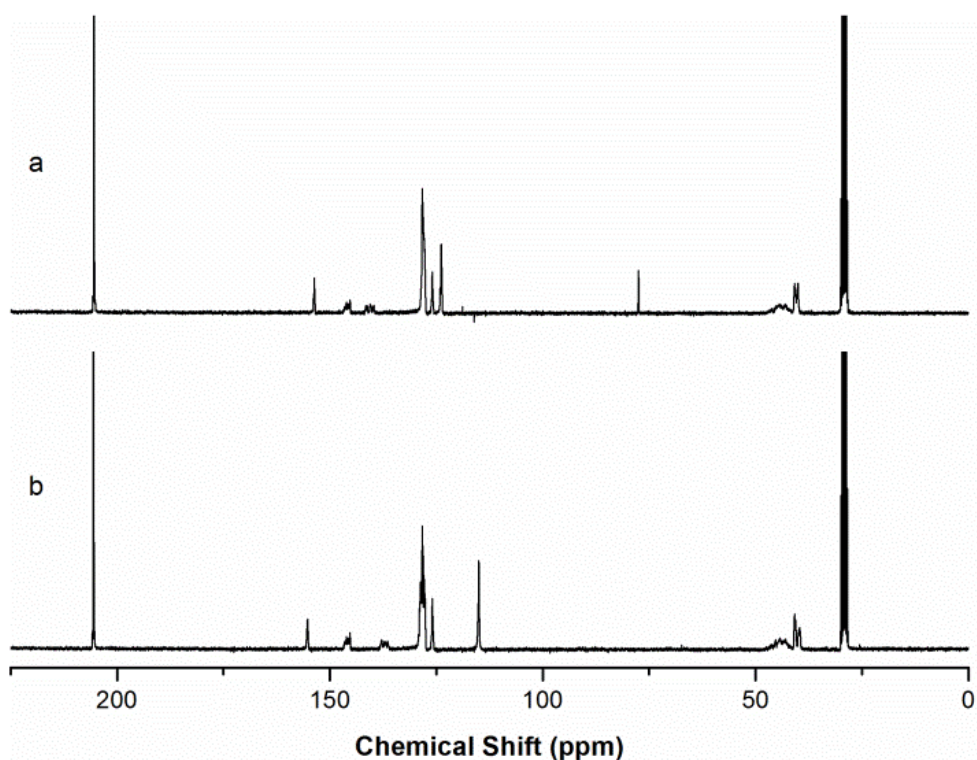
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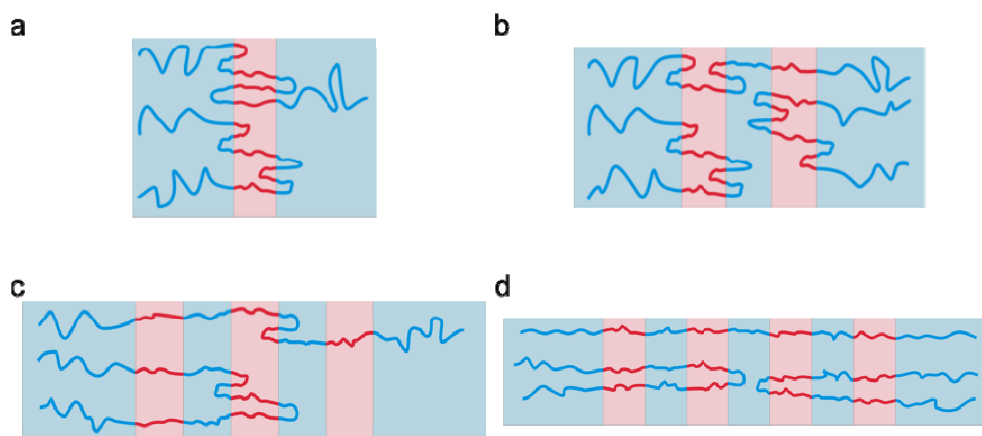
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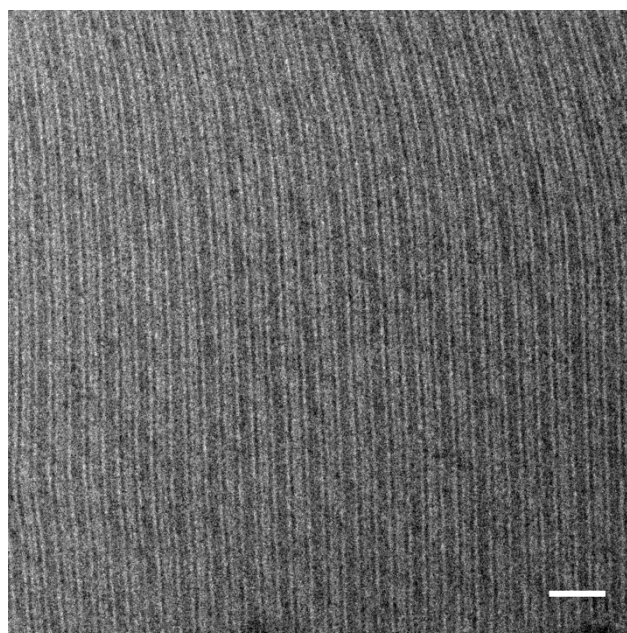
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**Fig. S1** <sup>13</sup>C-NMR spectra of a multiblock copolymer in acetone-d<sub>6</sub> (a) before and (b) after hydrolysis.



**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  $\text{g}\cdot\text{mol}^{-1}$  and  $\overline{M}_n$  (inner blocks) = 8,700  $\text{g}\cdot\text{mol}^{-1}$ , stained with  $\text{OsO}_4$ . Scale bar represents 100 nm.