

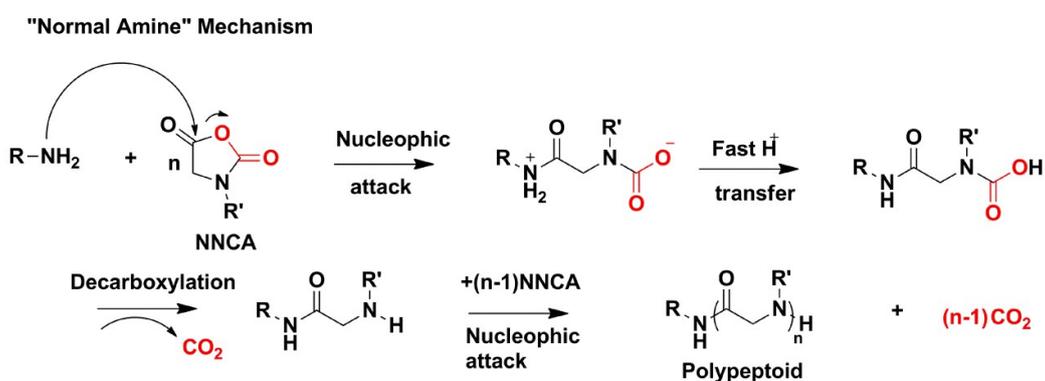
## Supporting Information

### Amphiphilic Star-Shaped Poly(sarcosine)-*block*-poly( $\epsilon$ -caprolactone) Diblock Copolymers: One-Pot Synthesis, Characterization, and Solution Properties

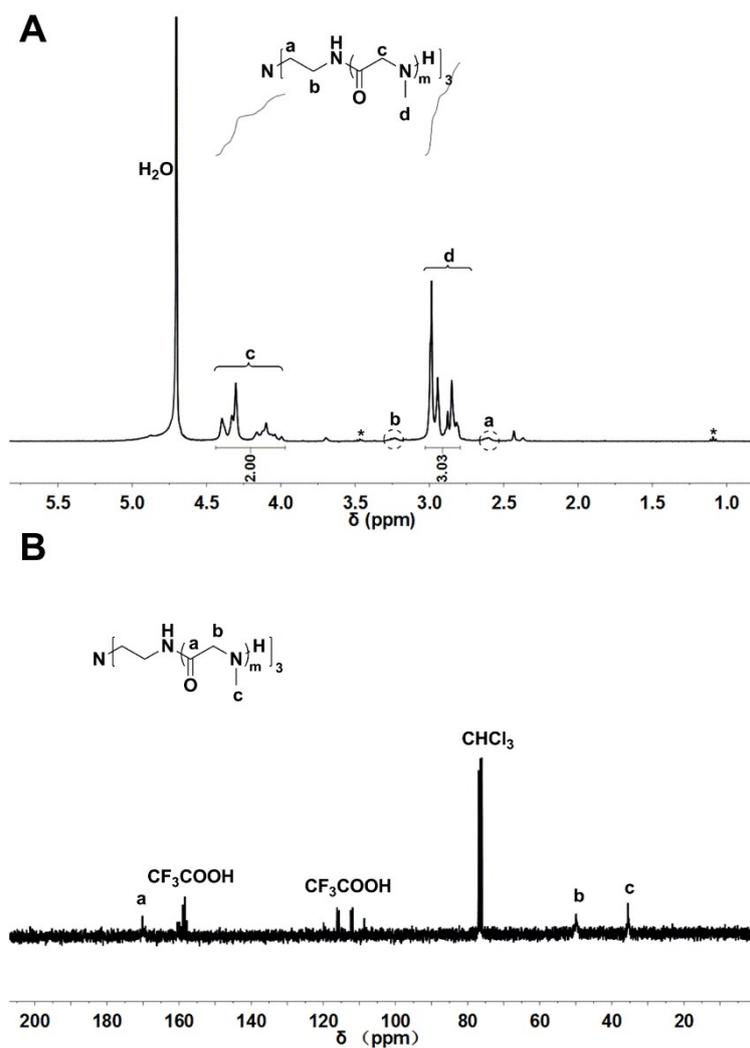
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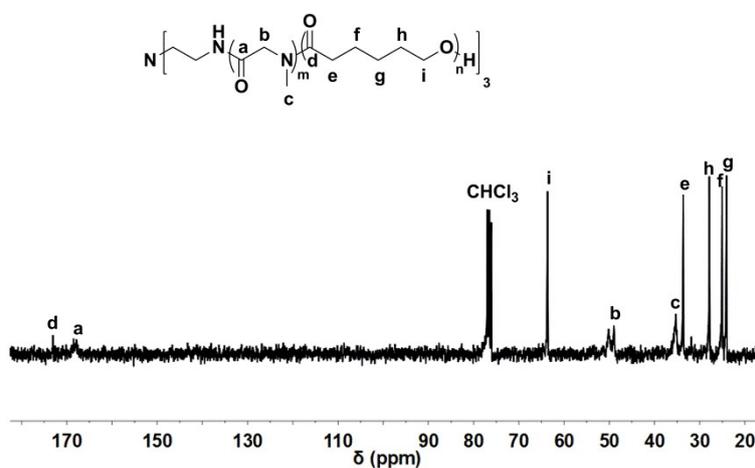
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**Scheme S1.** The "normal amine" mechanism for the ring-opening polymerization of NNCA initiated by primary amines.<sup>1</sup>

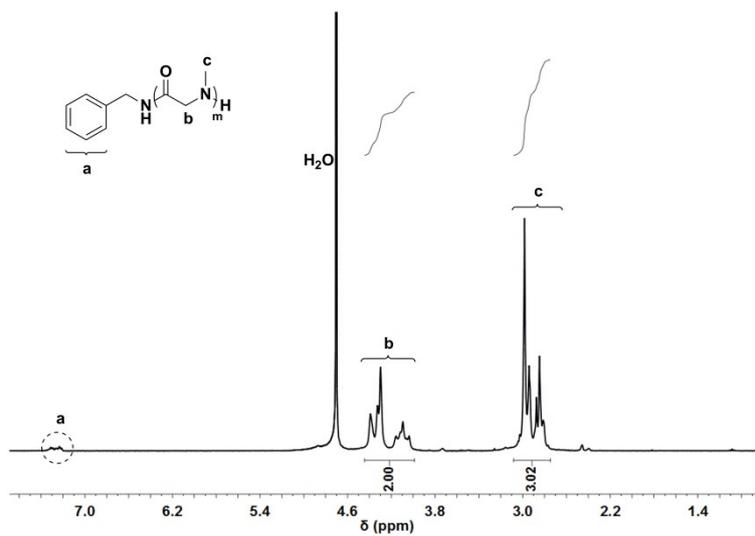


**Figure S1.** (A)  $^1\text{H}$  spectrum of a star-shaped PSar sample in  $\text{D}_2\text{O}$ . Asterisk refers to the residual diethyl ether in sample. (B)  $^{13}\text{C}$  NMR spectrum of a star-shaped PSar sample in  $\text{CDCl}_3/\text{CF}_3\text{COOH}$ .

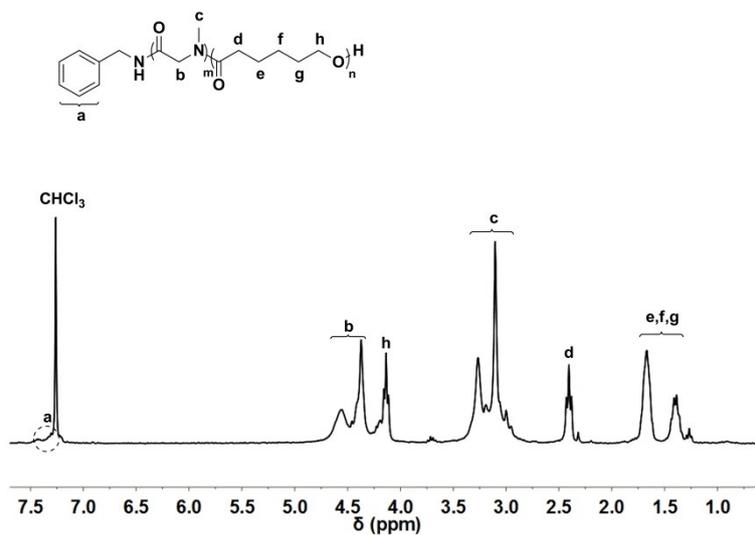


**Figure S2.**  $^{13}\text{C}$  NMR spectrum of a star-shaped poly(sarcosine)-*b*-poly( $\epsilon$ -caprolactone)

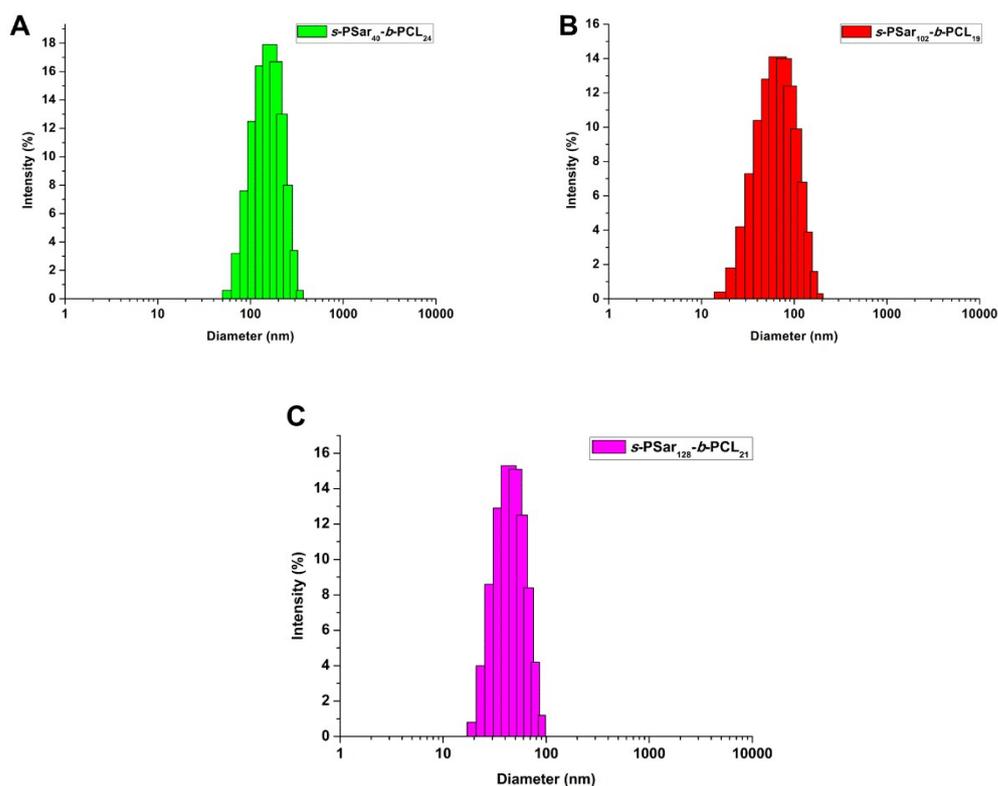
diblock copolymer sample in  $\text{CDCl}_3$ .



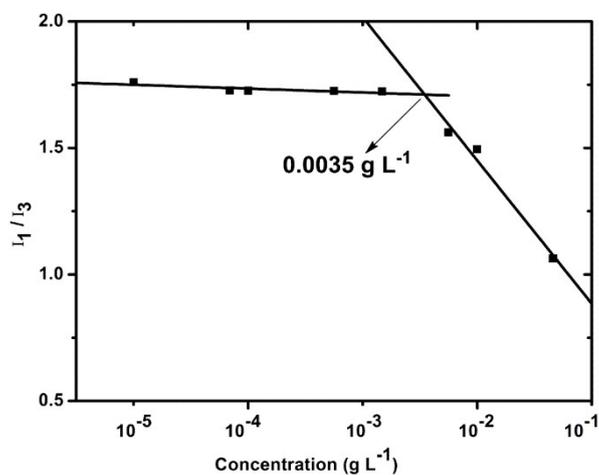
**Figure S3.**  $^1\text{H}$  NMR spectrum of linear poly(sarcosine) in  $\text{D}_2\text{O}$ .



**Figure S4.**  $^1\text{H}$  NMR spectrum of linear poly(sarcosine)-*b*-poly( $\epsilon$ -caprolactone) diblock copolymer in  $\text{CDCl}_3/\text{TFA}$ .



**Figure S5.** Particles size distributions in aqueous solution of (A) *s*-PSar<sub>40</sub>-*b*-PCL<sub>24</sub>, (B) *s*-PSar<sub>102</sub>-*b*-PCL<sub>19</sub>, (C) *s*-PSar<sub>128</sub>-*b*-PCL<sub>21</sub>.



**Figure S6.** The ratio  $I_1/I_3$  of pyrene emission spectra for the *s*-PSar<sub>40</sub>-*b*-PCL<sub>24</sub> copolymer as a function of copolymer concentrations (measured at 25 °C).

## References

- (a) Zhang, D. H.; Lahasky, S. H.; Guo, L.; Lee, C. U.; Lavan, M., Polypeptoid Materials: Current Status and Future Perspectives. *Macromolecules* **2012**, *45* (15), 5833-5841; (b) Luxenhofer, R.; Fetsch, C.; Grossmann, A., Polypeptoids: A perfect match for molecular definition and macromolecular engineering? *J. Polym. Sci. Pol. Chem.* **2013**, *51* (13), 2731-2752; (c) Klinker, K.; Barz, M., Polypept(o)

ides: Hybrid Systems Based on Polypeptides and Polypeptoids. *Macromol. Rapid Commun.* **2015**, *36* (22), 1943-1957; (d) Hadjichristidis, N.; Iatrou, H.; Pitsikalis, M.; Sakellariou, G., Synthesis of Well-Defined Polypeptide-Based Materials via the Ring-Opening Polymerization of alpha-Amino Acid N-Carboxyanhydrides. *Chem. Rev.* **2009**, *109* (11), 5528-5578.