Electronic Supplementary Information for

Reversible fabrication and self-assembly of gemini supra-amphiphile driven by dynamic covalent bond

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Fig. S1 Variation in size distribution of the **A-C** mixed solutions with the addition of **B**; The concentration of **C**: (a) 40 mM; (b) 50 mM; (c) 90 mM; (d) 100 mM.





Fig. S2 ESI-MS profiles of (a) the **A-C** mixed solution and (b) the **A-B-C** mixed solution with 60 mM **C**; (c) ESI-MS profile of the **A-C** mixed solution with 80 mM **C**; ESI-MS profiles of (d,e) the bottom phase and (f) upper phase of the **A-B-C** mixed solution with 80 mM **C**.



Fig. S3 (a) ¹³C NMR spectrum of the precipitate and (b) ¹H NMR spectrum of the clear solution obtained via CO_2 treatment.



Fig. S4 Size distribution of the clear solution separated from precipitate after CO_2 treatment.