Electronic Supplementary Information for

Controllable hierarchical self-assembly of gemini supraamphiphiles: effect of spacer length

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Fig. S1 ESI-MS profiles of the [M-*n*-M]Br₂/SDBS mixed solutions.

Fig. S2 Surface tension of the upper phase at $R_2 = 1.0$ with diluted multiple at 25 °C.

Fig. S3 Surface tension of the upper phase at $R_3 = 0.5$ with diluted multiple at 25 °C.

Fig. S4 2D ¹H-¹H COSY spectrum of the β -CD/[M-10-M]Br₂ mixed solution in D₂O.





Fig. S1 Positive ion (a-c, e-h) and negative ion (d) MS spectra of the $[M-n-M]Br_2/SDBS$ mixed solutions. (a) The $[M-2-M]Br_2/SDBS$ mixed solution at $R_1 = 0.5$; (b) the bottom and (c,d) upper phases of the $[M-6-M]Br_2/SDBS$ mixed solution at $R_2 = 0.5$; (e) the bottom and (f) upper phases of the $[M-6-M]Br_2/SDBS$ mixed solution at $R_2 = 1.0$. (g) the bottom and (h) upper phases of the $[M-10-M]Br_2/SDBS$ mixed solution at $R_3 = 0.5$.



Fig. S2 Surface tension of the upper phase at $R_2 = 1.0$ with diluted multiple at 25 °C.



Fig. S3 Surface tension of the upper phase at $R_3 = 0.5$ with diluted multiple at 25 °C.



Fig. S4 2D ¹H-¹H COSY spectrum of the β -CD/[M-10-M]Br₂ mixed solution in D₂O.