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## An amphiphilic water-soluble biphen[3]arene with tunable lower critical solution temperature behavior

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## **Electronic Supplementary Information**

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1. <sup>1</sup>H NMR, <sup>13</sup>C NMR and mass spectra of amphiphilic water-soluble biphen[3] arene H



*Figure S1.* <sup>1</sup>H NMR spectrum (400 MHz, dichloromethane- $d_2$ , 293 K) of **H**.



Figure S2. <sup>13</sup>C NMR spectrum (100 MHz, dichloromethane-d<sub>2</sub>, 293 K) of H.



*Figure S3.* Low-resolution electrospray ionization mass spectrum (LRESI-MS) of **H**. Assignment of the main peak: m/z 767.3 [M + Na+ K]<sup>2+</sup>, 1510.4 [M + K]<sup>+</sup>.



*Figure S4.* High-resolution electrospray ionization mass spectrum (HRESI-MS) of **H**. Assignment of the main peak: m/z 1493.7648 [M + Na]<sup>+</sup>.



Figure S5. UV-vis absorption spectrum of H (0.500 mM) in aqueous solution.

3. Transmission electron microscopy (TEM) studies of **H** below and above its LCST



*Figure S6.* TEM images of aqueous solutions: (a) amphiphilic macrocycle **H** dried at 10 °C; (b) amphiphilic macrocycle **H** dried at 30 °C.  $[\mathbf{H}] = 2.00 \text{ mM}$ .

4. Dynamic light scattering (DLS) results of H



*Figure S7.* DLS result of **H** (2.00 mM) in aqueous solution at 10 °C. The average hydrodynamic diameter of **H** was  $\sim$ 59 nm, indicating that **H** forms assembled structure.



*Figure S8.* DLS result of **H** (2.00 mM) in aqueous solution at 30 °C. The average hydrodynamic diameter of **H** increased to ~296 nm above  $T_{cloud}$ , indicating that **H** forms large aggregate structure.

5. <sup>1</sup>*H* NMR study of **H** in the presence and absence of  $K^+$ 



*Figure S9.* <sup>1</sup>H NMR spectrum (D<sub>2</sub>O, 293 K, 500 MHz) of **H**: (a) in the absence of  $K^+$ ; (b) in the presence of equimolar  $K^+$ . [**H**] = 5.00 mM.