Supporting Information

Cyclodextrin assisted two-component sonogel for visual humidity sensing

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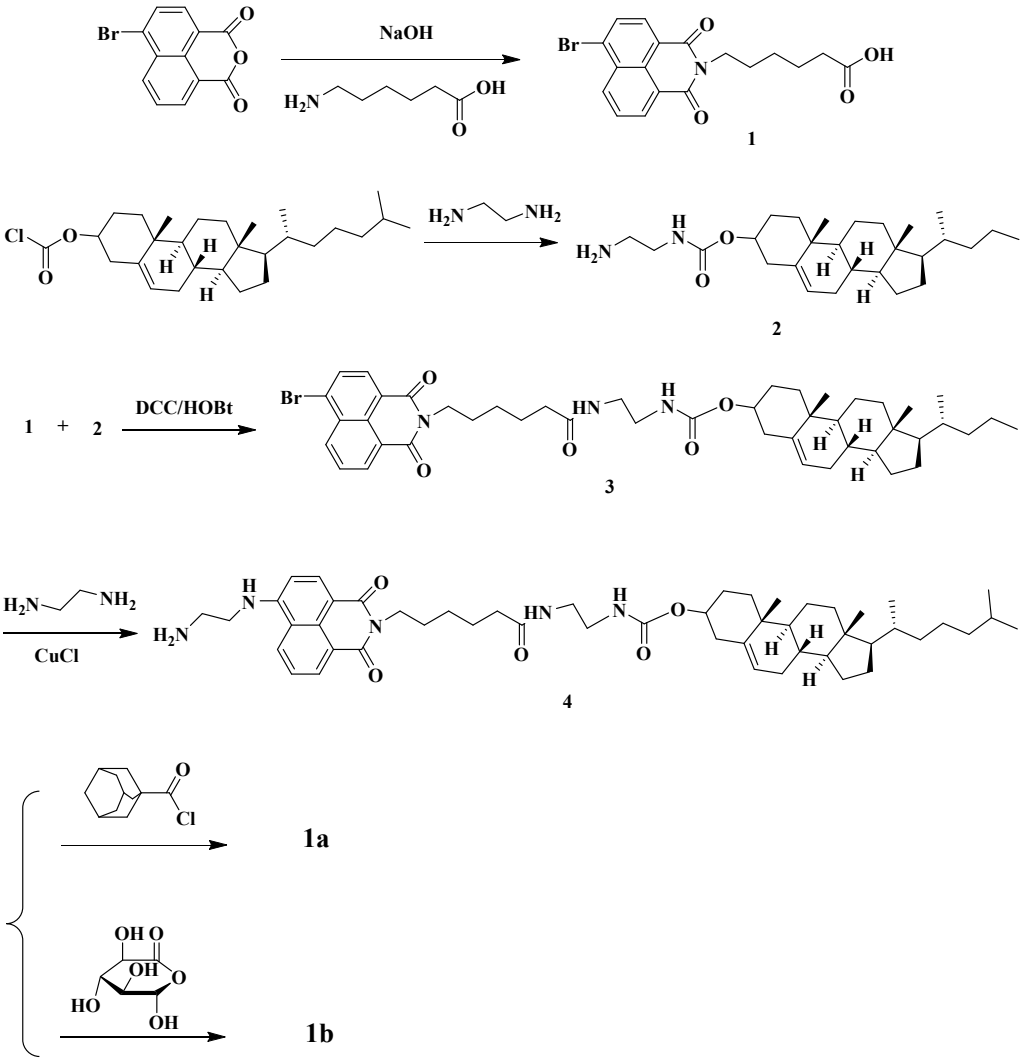
1. The synthesis procedures of 1a and 1b



Scheme S1 Synthesis procedures of **1a** and **1b**.

The synthesis of **1-4** could be seen in our previous work. [32]

Synthesis of **1a**

Compound **4** (1 mmol, 823 mg) and 1-adamantanecarboxylic acid chloride (1 mmol, 199 mg) were stirred in CH_2Cl_2 for 24 hours, and the mixture was concentrated and purified by chromatography using CH_2Cl_2 and MeOH (50:1) as an eluent, resulting in a yellow solid (424 mg, 43%). Mp: 146-148 °C. ^{1}H NMR (DMSO-d_6, 500 M): 0.59 (s, 3H), 0.84 (d, 3H, J = 3 Hz), 0.85-0.86 (d, 3H, J = 3Hz), 0.87-2.27 (m, 55H), 2.97-3.10

(m, 6H), 3.38-3.42 (m, 4H), 3.96-3.99 (t, 2H, *J* = 7.5 Hz), 4.26-4.32 (m, 1H), 5.25 (s, 1H), 6.87-6.89 (d, 1H, *J* = 9 Hz), 7.68-7.71 (1H, t, *J* = 8.5 Hz), 8.25-8.27 (1H, d, *J* = 8.5 Hz), 8.42-8.44 (d, 1H, *J* = 7Hz), 8.57-8.59 (d, 1H, *J* = 8.5 Hz). ^{13}C NMR (125 M, $CDCl_3$, δ), 7.8, 9.4, 12.1, 19.1, 19.5, 21.1, 23.0, 23.2, 23.9, 24.3, 25.6, 26.9, 27.9, 28.1, 28.2, 28.3, 28.4, 31.8, 35.7, 36.1, 36.2, 36.5, 36.6, 36.7, 37.1, 37.9, 42.3, 43.4, 46.0, 49.9, 56.1, 56.5, 73.5, 104.2, 108.4, 120.7, 122.3, 122.5, 124.8, 128.9, 129.9, 131.2, 134.7, 140.2, 151.3, 156.4, 163.4, 164.2, 172.8, 178.5. Modi-Tof mass calc. for $[C_{61}H_{87}N_5O_6Na]^+$ ($M+Na^+$): 1008.6554; Found: 1008.6552.

Synthesis of 1b

Compound **4** (1 mmol, 823 mg) and D-(+)-Gluconic acid δ-lactone (445 mg, 2.5 mmol) were refluxed in ethanol for 24 hours, then the precipitate was washed with water and recrystallized with ethanol for twice, yellow solid was obtained (300 mg, 30%). Mp: 227-229 °C. ^{1}H NMR (DMSO-d_6, 500 M): 0.60 (s, 3H), 0.84 (d, 3H, *J* = 2.5 Hz), 0.85-0.86 (d, 3H, *J* = 2.5 Hz), 0.86-2.41 (m, 47H), 2.99-3.07 (m, 6H), 3.36-3.46 (m, 6H), 3.97-3.96 (d, 2H, *J* = 3.5 Hz), 4.04-4.05 (d, 2H, *J* = 3.5 Hz), 4.27-4.35 (m, 1H), 5.27 (m, 1H), 6.90-6.91 (d, 1H, *J* = 8.5 Hz), 7.68-7.71 (t, 1H, *J* = 8 Hz), 8.26-8.27 (d, 1H, *J* = 8.5 Hz), 8.43-8.44 (d, 1H, *J* = 7 Hz), 8.61-8.63 (d, 1H, *J* = 8 Hz). ^{13}C NMR (125 M, $CDCl_3$, *δ*): 12.2, 14.4, 19.1, 19.5, 21.1, 23.0, 23.3, 23.9, 24.4, 25.6, 26.9, 28.0, 28.1, 28.3, 28.5, 31.9, 35.3, 38.9, 39.2, 42.4, 43.4, 50.0, 56.1, 56.6, 60.9, 63.9, 70.7, 72.1, 72.9, 73.6, 74.1, 104.4, 108.5, 120.7, 122.4, 122.5, 128.9, 129.9, 131.2, 134.7, 140.3, 151.2, 156.4, 163.5, 164.3, 172.8, 174.1. Modi-Tof mass calc. for $[C_{56}H_{83}N_5O_{11}Na]^+$ ($M+Na^+$): 1024.5987; Found: 1024.5270.

Table S1 Gelation properties of **1b** (25 mg/mL) and **1b**/α-CD (with molar ratio of 1:1).

Solvent	H-C (**1b**)	S (**1b**)	S (**1b**/α-CD)	H-C (**1b**/α-CD)
methanol	P	I	I	P
ethanol	P	I	I	P
isopropanol	P	I	I	P
2-butanol	P	I	I	P
tert-butanol	P	I	I	P
n-butanol	P	I	G	I
toluene	P	I	I	P
ethyl acetate	P	I	I	P
THF	P	I	I	P
1,4-dioxane	P	I	I	P
glycol monomethyl ether	P	I	I	P

Note: P, precipitate; I, insoluble; G, gel.

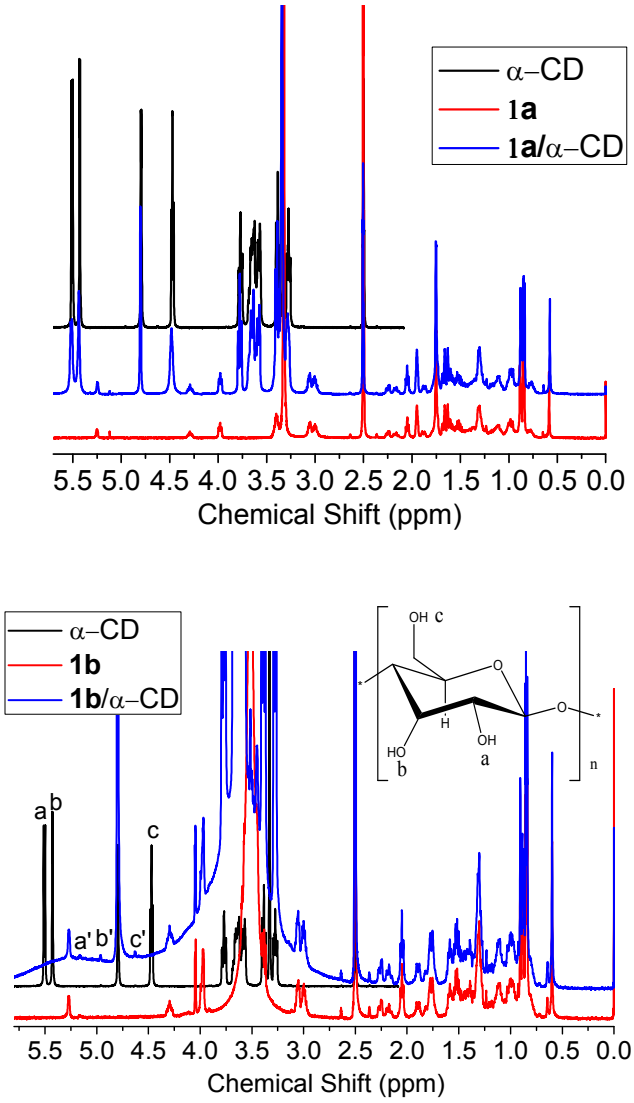


Figure S1 ^{1}H NMR spectra of **1a**, α-CD, and **1a**/α-CD; **1b**, α-CD, and **1b**/α-CD (molar ratio 1:1).

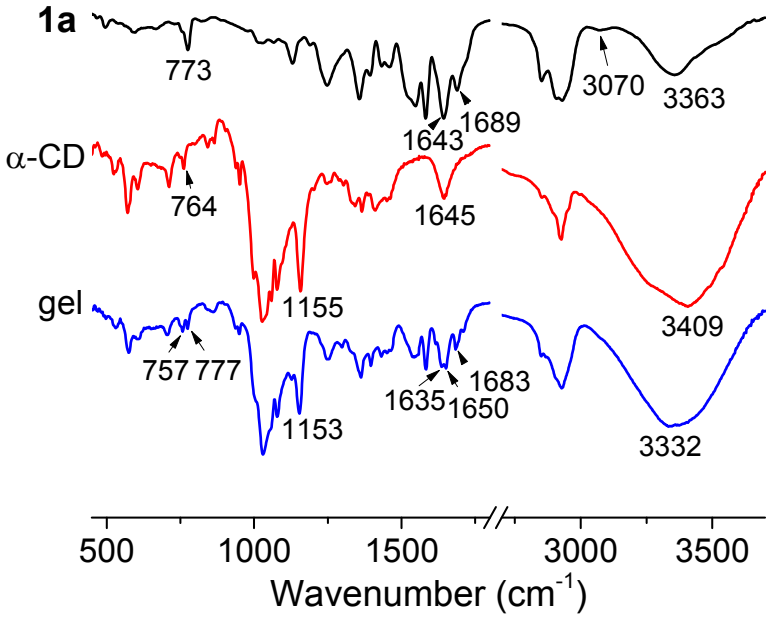


Figure S2 IR spectra of powder of **1a,** α-CD and xerogel of **1a**/α-CD (with molar ratio of 1:1).

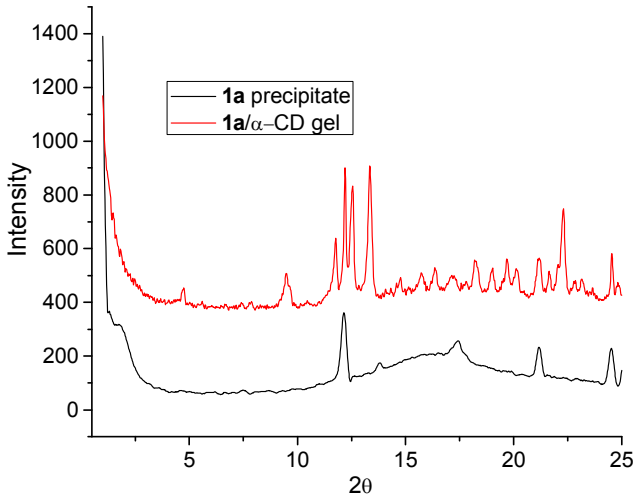


Figure S3 XRD spectra of **1a** precipitate and **1a**/α-CD gel.

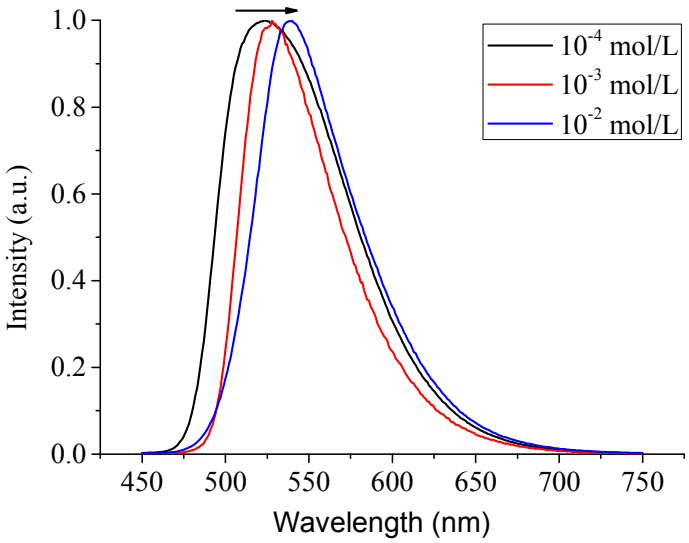


Figure S4 Fluorescence emission spectra of **1a** solution (10^{-4} ~10^{-2} M).

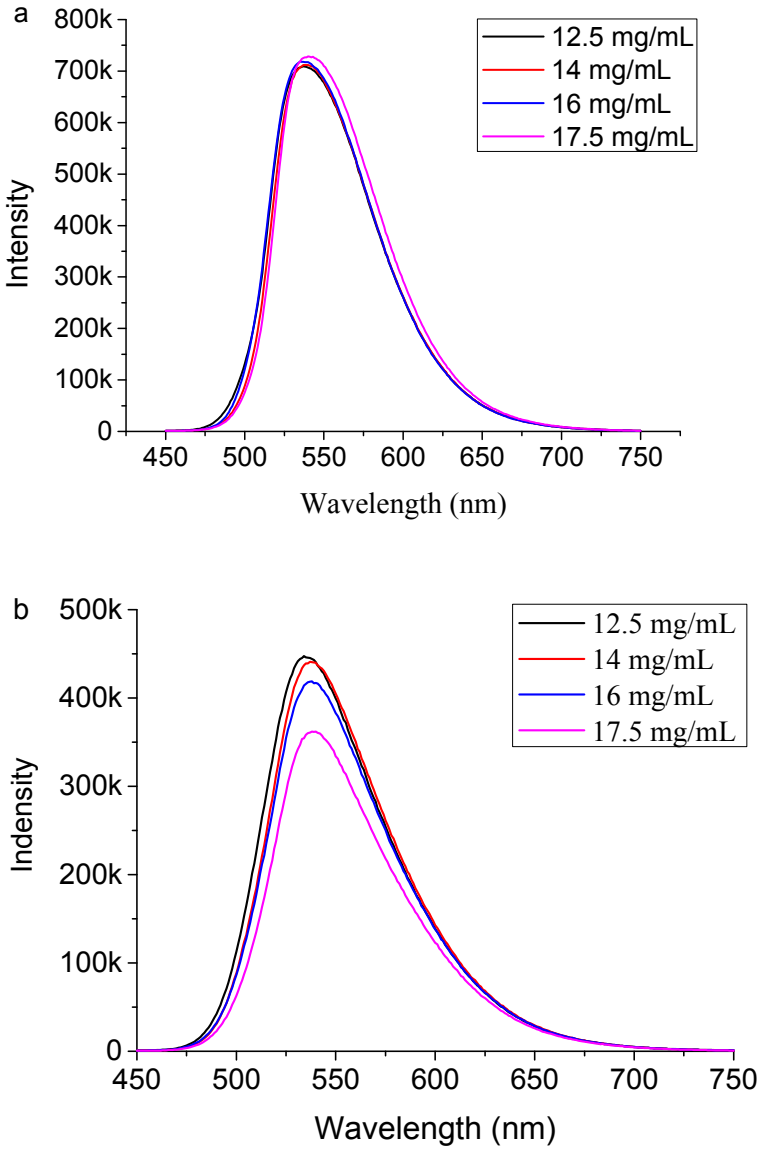


Figure S5 Fluorescent emission spectra of (a) the gel of **1a**/α-CD and (b) **1a** solution with different concentration.

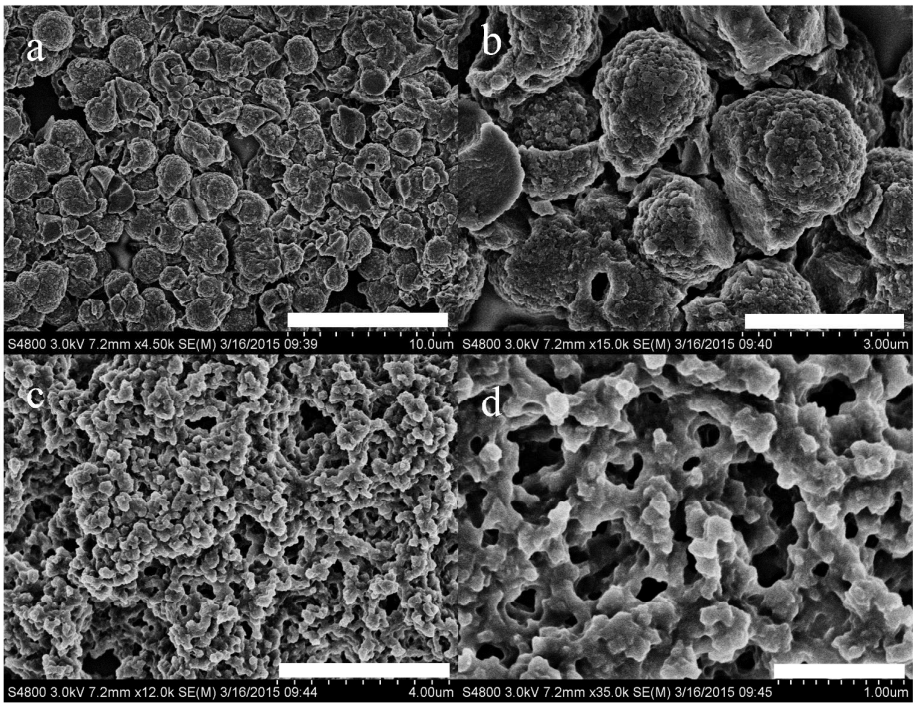


Figure S6 SEM images of **1a** assembly. a) **1a** precipitate from propanol (16.5 mg/mL); b) magnification of a); c) **1a** precipitate from propanol after triggered by sonication for 5 min; d) magnification of c).

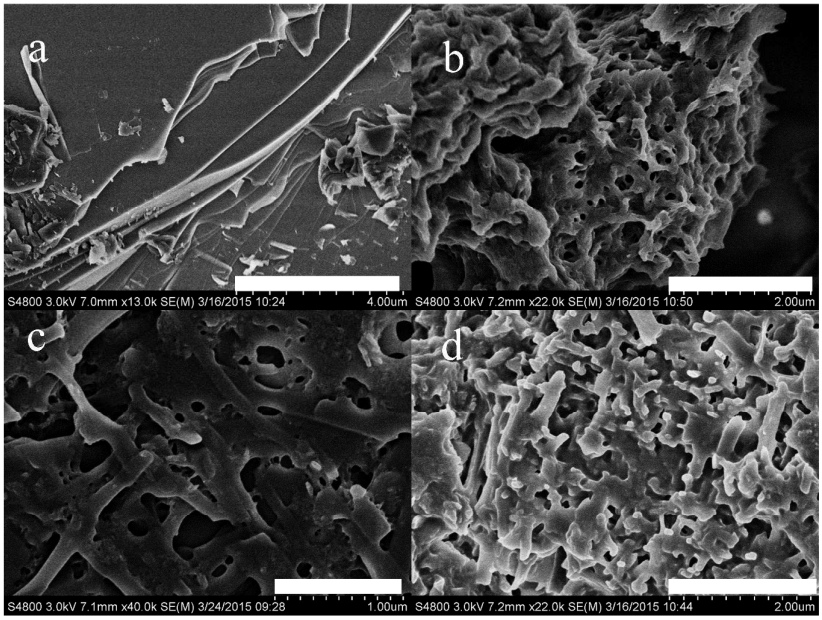


Figure S7 SEM images of **1b** assembly. a) **1b** precipitate from butanol (25 mg/mL); b) and c) **1b** precipitate from butanol then treated by sonication for 5 min; d) **1b**/α-CD xerogel (25 mg/mL, with ratio of 1:1).

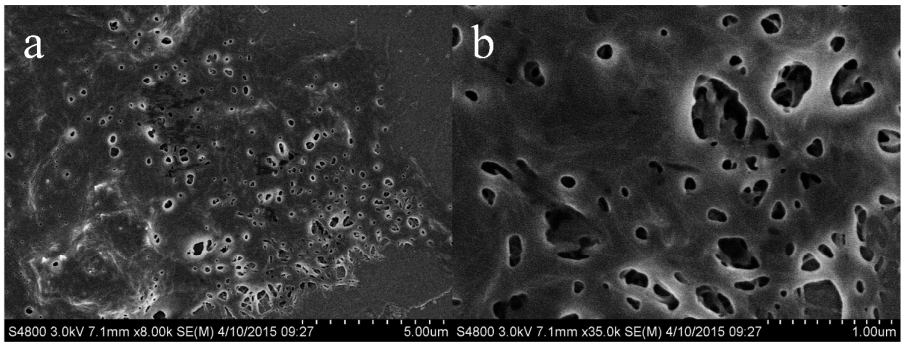


Figure S8 SEM images of α-CD assembly (25 mg/mL) from propanol when treated with sonication for 5 min.

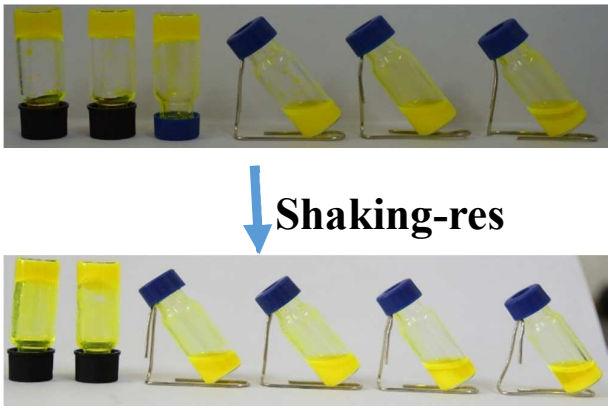


Figure S9 Photos of gels or suspensions of **1a** (16. 5 mg/mL) with different ratio of α-CD from left to right: 1:0.2; 1:0.6; 1:1; 1:1.2; 1:1.6; 1:2.

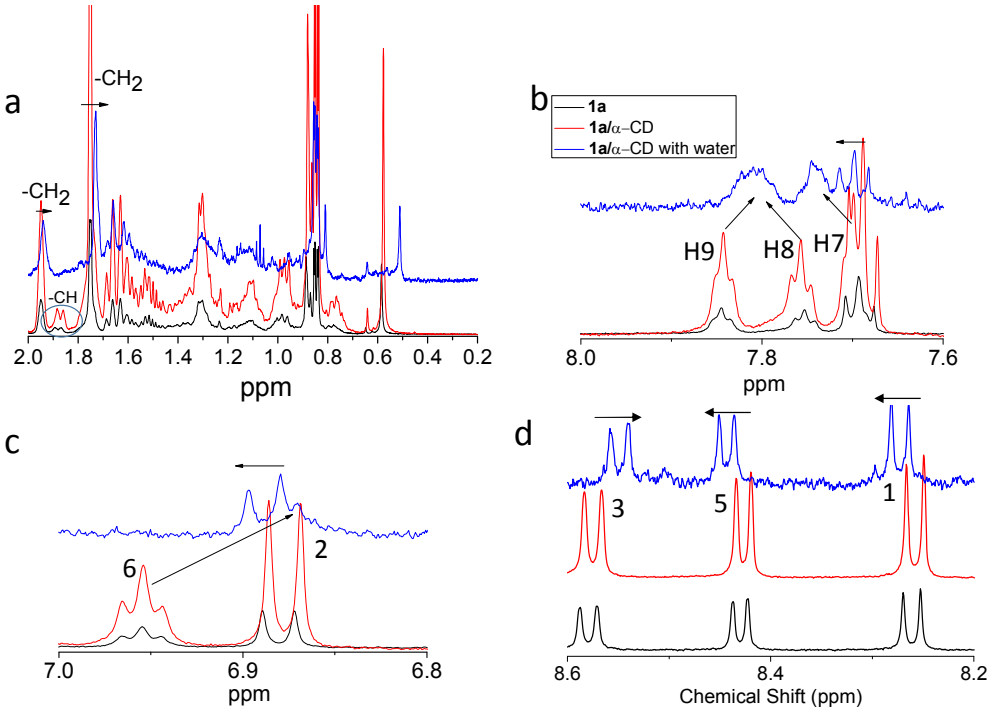


Figure S10 ^{1}H NMR spectra of **1a** (black), **1a**/α-CD (with molar ratio of 1:1) (red), and then upon the addition of 30 μL water (blue).