Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Supporting Information

Surfactant-Induced Chirality Transfer, Amplification and Inversion in the Cucurbit[8]uril-Viologen Host-Guest Supramolecular System

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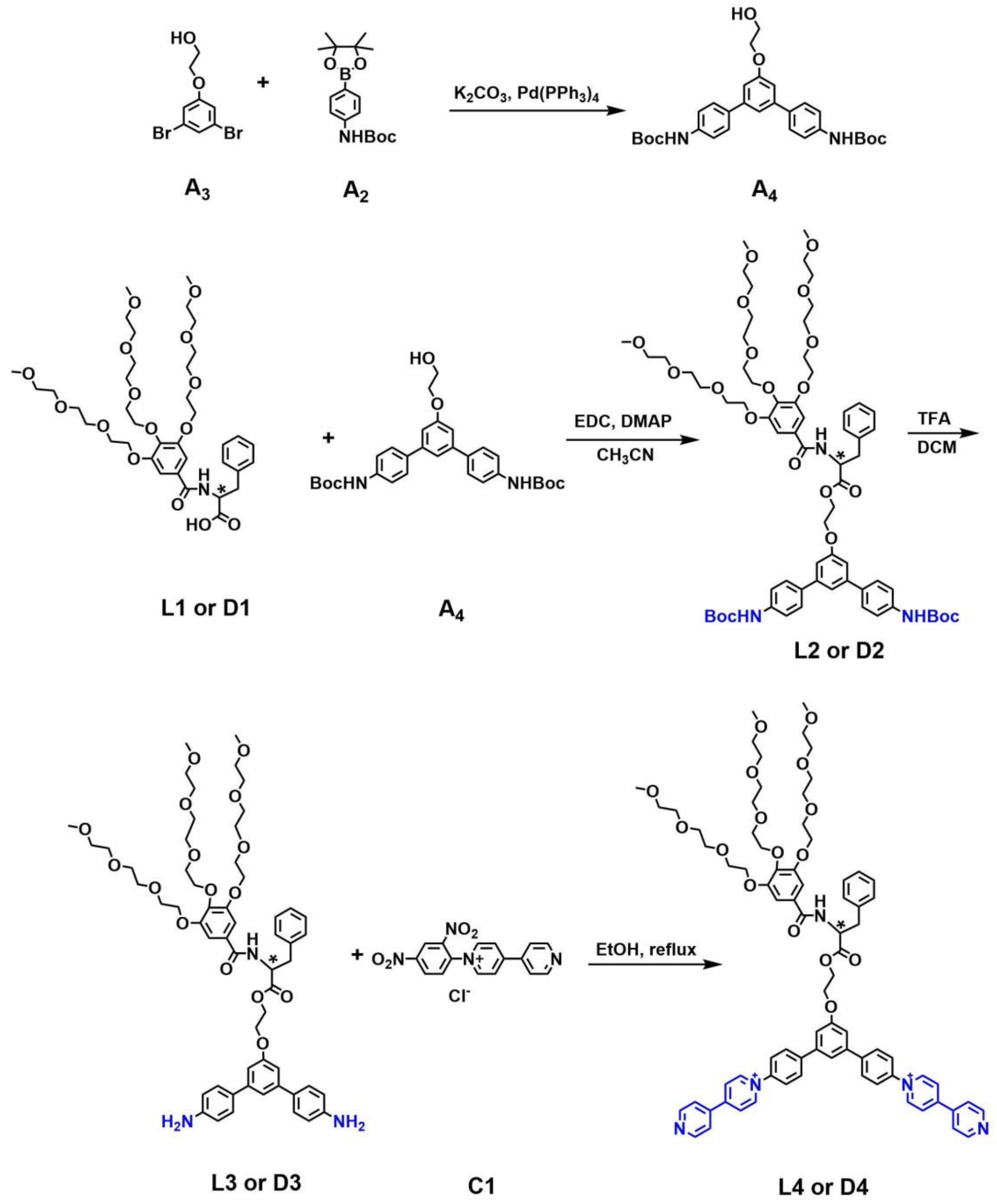
1. Synthetic procedures



Scheme S1. The pathway of synthesis of target chiral compounds of L4 or D4.

1.1 Preparation of compound A4

Compound A2 and A3 were synthesized according to the reported references.[1-2] A2 (6.703 g, 21 mmol), A3 (3.006 g, 8.38 mmol), anhydrous potassium carbonate (16.623

g, 120.27 mmol), deionized water (25 mL) and ultra-dry 1,4-dioxane (60 mL) were added into a flash (250 mL). After adding tetrakis(triphenylphosphine)palladium (964 mg, 0.834 mmol) under argon atmosphere, the mixture solution was refluxed at 120 ºC for 24 h followed by cooling to room temperature. Thin layer chromatography (TLC) confirmed that the reactants had reacted completely. After the reaction, the separatory funnel was used to remove the water phase from the reaction solution. The combined organic extracts were then dried with anhydrous Na_2SO_4 followed by filtration and concentration under vacuum. The residue was purified by silica gel chromatography with petroleum ether/ethyl acetate (10:1, v/v, Rf = 0.45) to afford 9.23 g (78%) of A4 as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$, 298 K), δ (ppm): 7.63-7.56 (m, 1H), 7.50-7.43 (m, 4H), 7.36 (d, J=8.5 Hz, 4H), 7.19 (s, 1H), 6.98(d, J=1.5 Hz, 2H), 6.54 (s, 2H), 4.15-4.08 (m, 2H), 3.93 (m, 2H), 1.46 (s, 18H) (Figure S23). ^{13}C NMR (100 MHz, $CDCl_3$, 298 K), δ (ppm): 158.35, 151.71, 141.60, 136.95, 134.61, 126.70, 117.76, 117.52, 110.66, 68.36, 60.50, 27.33 (Figure S24). HRMS (ESI) (m/z): $[M+Na]^+$ calcd for $C_{30}H_{36}N_2O_6Na$: 543.2471, found: 543.2469 (Figure S25).

1.2 Preparation of compound L2 and D2

Compound L1 and D1 were synthesized according to the reported reference.[3] L1 (2.226 g, 2.94 mmol), A4 (1.289 g, 2.47 mmol), 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (845 mg, 4.41 mmol), 4-dimethylaminopyridine (45 mg, 0.37 mmol), and ultradry dichloromethane (7 mL) were placed in a flask (50 mL). D1 (2.388 g, 3.16 mmol), A4 (1.815 g, 3.39 mmol), 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (1.81g, 9.44 mmol), 4-dimethylaminopyridine (47 mg, 0.38 mmol), and ultradry dichloromethane (12 mL) were placed in a flask (50 mL). The reaction was carried out overnight under the protection of argon, and the completion of the reaction was confirmed by TLC monitoring. After the reaction, the product was washed for 3 times with aqueous HCl (5%), $NaHCO_3$ (Saturated) and NaCl (Saturated). The combined dichloromethane solutions were dried with anhydrous $NaSO_4$, evaporated under vacuum, and purified by silica gel chromatography with ethyl acetate/methanol (20:1, v/v, Rf = 0.30) to afford 0.91 g (74%) of L2 a white solid and 1.52 g (37%) of D2 a white solid. L2: ^{1}H NMR (400 MHz, CD_3CN, 298 K), δ (ppm): 7.68 (s, 2H), 7.63-7.56 (m, 4H), 7.54-7.43 (m, 4H), 7.39 (s, 1H), 7.29-7.16 (m, 6H), 7.04 (d, *J*=1.5 Hz, 2H), 6.92 (s, 2H), 4.80 (m, 1H), 4.47 (m, 2H), 4.28 (m, 2H), 4.02 (m, 6H), 3.73-3.64 (m, 6H), 3.62-3.48 (m, 19H), 3.47-3.40 (m, 6H), 3.27 (m, 10H), 3.12 (m, 1H), 1.50 (s, 18H) (Figure S26). ^{13}C NMR (100 MHz, CD_3CN, 298 K), δ (ppm): 171.19, 165.99, 159.22, 152.65, 152.00, 142.07, 138.72, 134.28, 128.95, 128.09, 127.14, 126.42, 118.30, 111.08, 105.78, 79.31, 71.81, 71.27, 69.99, 69.89, 69.84, 69.78, 69.68, 68.92, 68.21, 65.93, 63.12, 57.57, 54.21, 36.51, 27.24 (Figure S27). HRMS (ESI) (m/z): $[M+Na]^+$ calcd for $C_{67}H_{91}N_3O_{20}Na$: 1280.6094, found: 1280.6094 (Figure S28). D2: ^{1}H NMR (400 MHz, CD_3CN, 298 K), δ (ppm): 7.78 (s, 2H), 7.58 (d, *J*=8.7 Hz, 4H), 7.48 (d, *J*=8.7 Hz, 4H), 7.38 (s, 1H), 7.35 (d, *J*=7.9 Hz, 1H), 7.29-7.20 (m, 4H), 7.17 (m, 1H), 7.03 (d, *J*=1.5 Hz, 2H), 6.93 (s, 2H), 4.81 (m, 1H), 4.45 (m, 2H), 4.31-4.19 (m, 2H), 4.07-3.96 (m, 6H), 3.72-3.64 (m, 6H), 3.60-3.48 (m, 19H), 3.48-3.40 (m, 7H), 3.26 (m, 11H), 3.12 (m, 1H), 1.49 (s, 18H) (Figure S29). ^{13}C NMR (100 MHz, CD_3CN, 298 K), δ (ppm): 171.23, 166.12, 159.15, 152.67, 151.94, 142.01, 138.69, 134.22, 128.93, 128.08, 127.09, 126.41, 118.29, 110.99, 105.75, 79.29, 71.79, 71.23, 69.95, 69.84, 69.79, 69.74, 69.63, 68.89, 68.14, 63.14, 57.57, 54.21, 36.50, 27.25 (Figure S30). HRMS (ESI) (m/z): $[M+K]^+$ calcd for $C_{67}H_{91}N_3O_{20}K$: 1296.5833, found: 1296.5852 (Figure S31).

1.3 Preparation of compound L3 and D3

L2 (4g, 3.22 mmol) and dichloromethane (20 mL) were added to a flask, and trifluorophosphoric acid (14.67 g/9.6 mL, 128.7 mmol) was slowly dripped into the reaction mixture. D2 (1.224 g, 0.984 mmol) and dichloromethane (12 mL) were added to the 50 mL flask, and trifluorophosphoric acid (4.489 g/3 mL, 39.4 mmol) was slowly dripped into the reaction solution. The mixture solution was stirred at room temperature for 18 h, and the solvent was evaporated under vacuum. The remaining residue was purified by silica gel chromatography to afford trifluoroacetate products of L3 (or D3).

Triethylamine was slowly added into the dichloromethane solution containing L3 (or D3) trifluoroacetate until the disappearance of the smoke, and the addition was accompanied by the formation of white precipitation. The product was extracted with dichloromethane, and the organic phase was washed with saturated salt water for 3 times. The combined dichloromethane solutions were dried with anhydrous $NaSO_4$ to get rid of water, and solution evaporated under vacuum to afford product L3 (3.136 g, yield 92%) as a yellow oil and D3 (0.84 g, yield 84%) as a yellow oil. L3: ^{1}H NMR (400 MHz, CD_3CN, 298 K), δ (ppm): 7.45-7.38 (m, 4H), 7.32-7.14 (m, 7H), 6.99-6.90 (m, 4H), 6.69 (m, 4H), 4.87-4.76 (m, 1H), 4.50-4.40 (m, 2H), 4.38-4.18 (m, 6H), 4.09-4.00 (m, 6H), 3.74-3.65 (m, 6H), 3.61-3.48 (m, 19H), 3.47-3.40 (m, 6H), 3.27 (m, 10H), 3.16-3.06 (m, 1H) (Figure S32).^{13}C NMR (100 MHz, CD_3CN, 298 K), δ (ppm): 171.24, 166.02, 159.13, 152.03, 147.58, 142.68, 129.12, 128.97, 128.10, 127.47, 126.43, 116.37, 114.30, 109.71, 105.80, 71.84, 71.27, 69.99, 69.87, 69.83, 69.79, 69.67, 68.93, 68.23, 65.76, 63.19, 57.58, 54.18, 36.57 (Figure S33). HRMS (ESI) (m/z): $[M+H]^+$ calcd for $C_{57}H_{76}N_3O_{16}$: 1058.5226, found: 1058.5219 (Figure S34). D3: ^{1}H NMR (400 MHz, CD_3CN, 298 K), δ (ppm): 7.47-7.39 (m, 4H), 7.30 (t, J=1.6 Hz, 1H), 7.27-7.15 (m, 6H), 6.94 (d, J=1.8 Hz, 4H), 6.74-6.65 (m, 4H), 4.81 (m, 1H), 4.46 (m, 2H), 4.37-4.11 (m, 6H), 4.08-4.01 (m, 6H), 3.83-3.65 (m, 7H), 3.63-3.49 (m, 20H), 3.48-3.40 (m, 7H), 3.27 (m, 11H), 3.12 (m, 1H) (Figure S35). ^{13}C NMR (100 MHz, CD_3CN, 298 K), δ (ppm): 171.22, 165.96, 159.12, 152.01, 147.53, 142.67, 136.99, 129.14, 128.96, 128.08, 127.47, 126.42, 114.29, 109.70, 105.73, 71.82, 71.26, 69.99, 69.82, 69.66, 68.91, 68.21, 65.77, 63.16, 57.56, 54.16, 36.54 (Figure S36). HRMS (ESI) (m/z): $[M+Na]^+$ calcd for $C_{57}H_{75}N_3O_{16}Na$: 1080.5045, found: 1080.5065 (Figure S37).

1.4 Preparation of compound L4 and D4

Compound C1 was synthesized according to the reported reference.[4] Under argon atmosphere, L3 (0.718 g, 0.678 mmol), C1 (490 mg, 1.51 mmol) and ethanol (16 mL) were placed in a flask (50 mL). D3 (0.852 g, 0.805 mmol), C1 (533 mg, 1.68 mmol) and ethanol (20 mL) were placed in a flask (50 mL). The mixture solution was refluxed at 90 ºC for 36 h followed by cooling to room temperature. The solvent evaporated under vacuum, and purified by using silica gel chromatography with dichloromethane/methanol (50:1, v/v, Rf = 0.20) to afford 0.726 g (80%) of L4 a yellowish solid and 0.638g (59%) of D4 a yellowish solid. L4: ^{1}H NMR (400 MHz, DMSO-d_6, 298 K), δ (ppm): 9.62 (d, J=6.7 Hz, 4H), 9.05 (d, J=7.5 Hz, 1H), 8.94-8.89 (m, 4H), 8.85 (d, J=6.6 Hz, 4H), 8.25 (d, J=8.4 Hz, 4H), 8.21-8.15 (m, 4H), 8.10 (d, J=8.6 Hz, 4H), 7.81 (s, 1H), 7.42 (s, 2H), 7.34 (d, J=7.2 Hz, 2H), 7.25 (t, J=7.5 Hz, 2H), 7.17 (d, J=2.9 Hz, 3H), 4.76-4.67 (m, 1H), 4.57-4.35 (m, 4H), 4.09 (m, 4H), 4.02 (m, 2H), 3.73 (m, 4H), 3.63 (m, 2H), 3.58 (m, 4H), 3.56-3.47 (m, 14H), 3.39 (m, 7H), 3.20 (m, 11H) (Figure S38). ^{13}C NMR (100 MHz, DMSO-d_6, 298 K), δ (ppm): 171.77, 153.05, 151.62, 151.08, 145.26, 142.20, 141.66, 140.53, 140.38, 137.67, 129.11, 128.64, 128.20, 126.45, 125.31, 125.13, 122.05, 113.43, 106.49, 71.75, 71.21, 69.91, 69.78, 69.65, 69.54, 68.88, 68.33, 57.98, 54.54, 36.24 (Figure S39). HRMS (ESI) (m/z): [M-2Cl]$^{2+}$ calcd for $C_{77}H_{87}N_5O_{16}/2$: 668.8068, found: 668.8057 (Figure S40). D4: ^{1}H NMR (400 MHz, DMSO-d_6, 298 K), δ (ppm): 9.68-9.54 (m, 4H), 8.98-8.91 (m, 4H), 8.89 (d, J=7.8 Hz, 1H), 8.87-8.80 (m, 4H), 8.30-8.23 (m, 4H), 8.22-8.16 (m, 4H), 8.12-8.03 (m, 4H), 7.83 (d, J=1.5 Hz, 1H), 7.44 (d, J=1.5 Hz, 2H), 7.35-7.29 (m, 2H), 7.26 (t, J=7.5 Hz, 2H), 7.20-7.14 (m, 1H), 7.12 (s, 2H), 4.70 (m, 1H), 4.56-4.37 (m, 4H), 4.08 (m, 4H), 4.02 (dd, J=5.8, 4.0 Hz, 2H), 3.73 (m, 4H), 3.67-3.61 (m, 2H), 3.61-3.44 (m, 18H), 3.42-3.37 (m, 7H), 3.20 (m, 11H) (Figure S41). ^{13}C NMR (100 MHz, DMSO-d_6, 298 K), δ (ppm): 171.83, 165.83, 159.52, 153.17, 151.69, 151.18, 151.15, 145.41, 145.39, 142.30, 141.78, 140.60, 140.47, 140.11, 137.70, 129.16, 128.68, 128.49, 128.26, 126.52, 125.31, 125.25, 125.17, 122.12, 118.63, 113.50, 106.52, 71.80, 71.27, 69.97, 69.84, 69.70, 69.59, 68.92, 68.36, 66.29, 63.13, 58.03, 54.53, 36.31(Figure S42). HRMS (ESI) (m/z): [M-2Cl]$^{2+}$ calcd for $C_{77}H_{87}N_5O_{16}/2$: 668.8068, found: 668.8069 (Figure S43).

2. Supplementary Figures

Table S1. pH value of different samples in aqueous solution.

	L4	L4/0.67SDS	L4/1SDS	L4/2SDS	L4/3SDS	L4/5SDS
pH	7.01	7.03	7.09	7.04	7.12	7.11
	L4/6.67 SDS	L4/1CB[8]	L4/0.67SDS /0.2CB[8]	L4/0.67SDS/ 0.4CB[8]	L4/0.67SDS /0.67CB[8]	L4/0.67SDS /1CB[8]
pH	7.10	6.94	6.97	6.89	6.94	6.88

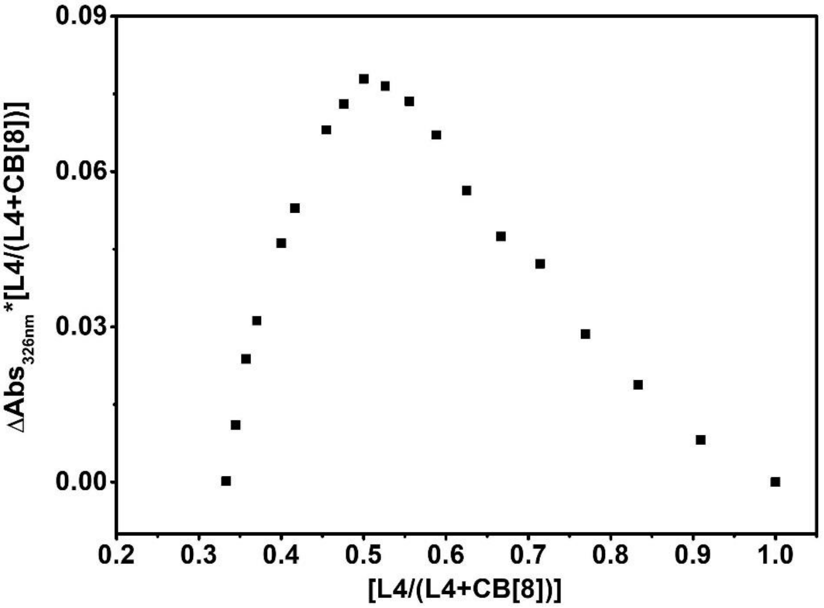


Figure S1. Job's plot based on the absorption shift of 326 nm in H_2O (L4 and CB[8]).

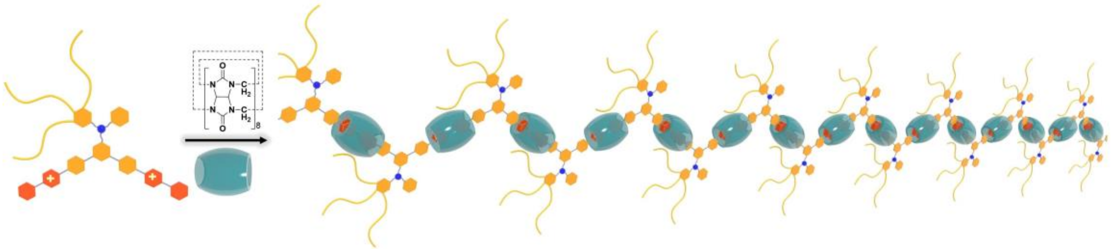


Figure S2. A schematic diagram of the possible assembly structures for L4/1CB[8] systems.

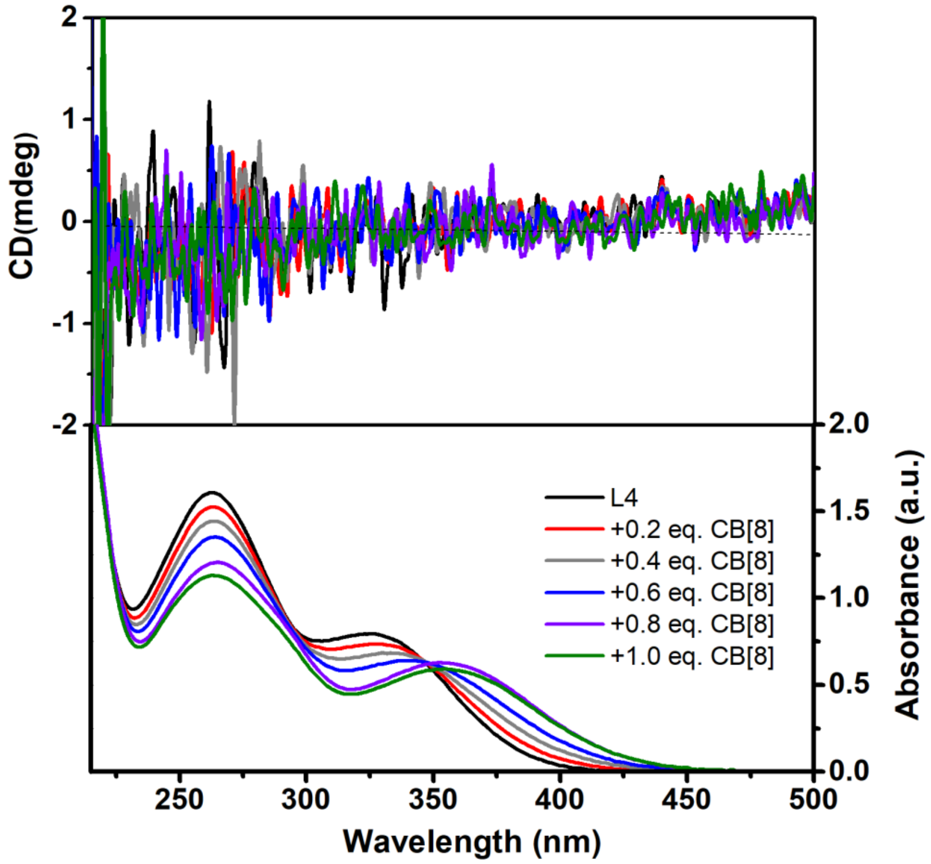


Figure S3. The CD spectra of L4 aqueous solution (0.05 mM) titrated by different amount of CB[8] (0.5 mM).

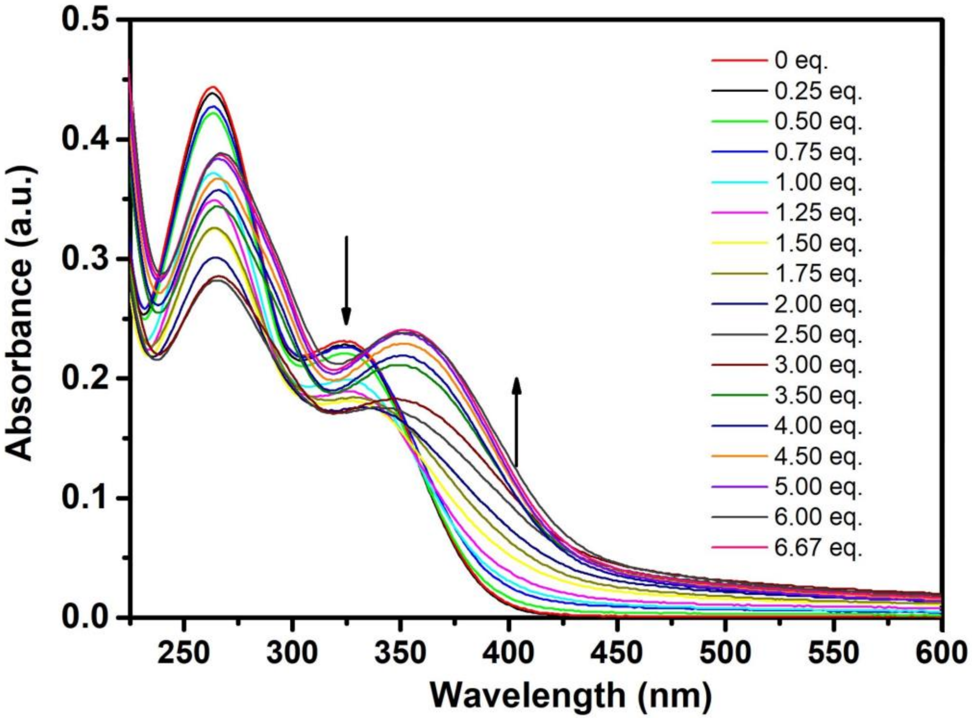


Figure S4. The UV-Vis absorption spectra of aqueous L4 solution (0.01 mM) titrated by different amount of SDS (0.01 M).

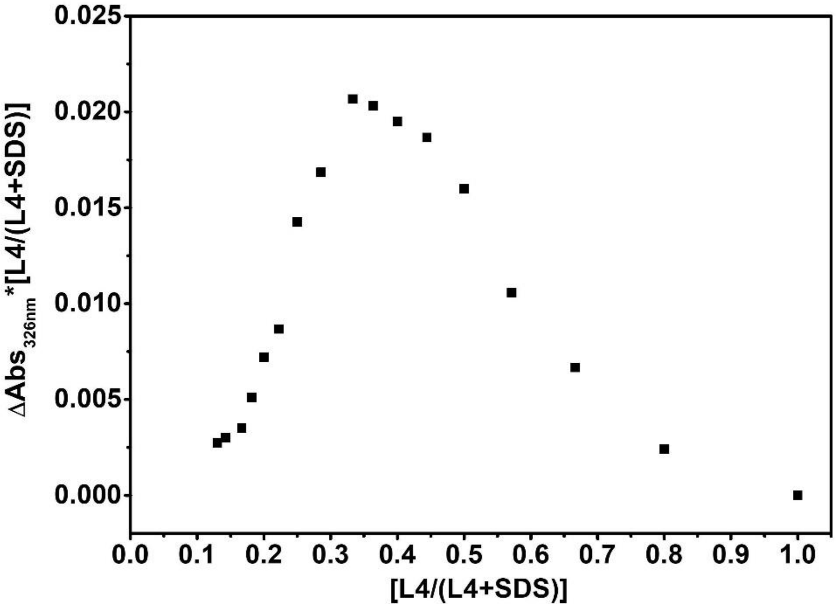


Figure S5. Job's plot based on the absorption shift of 326 nm in H_2O (L4 and SDS).

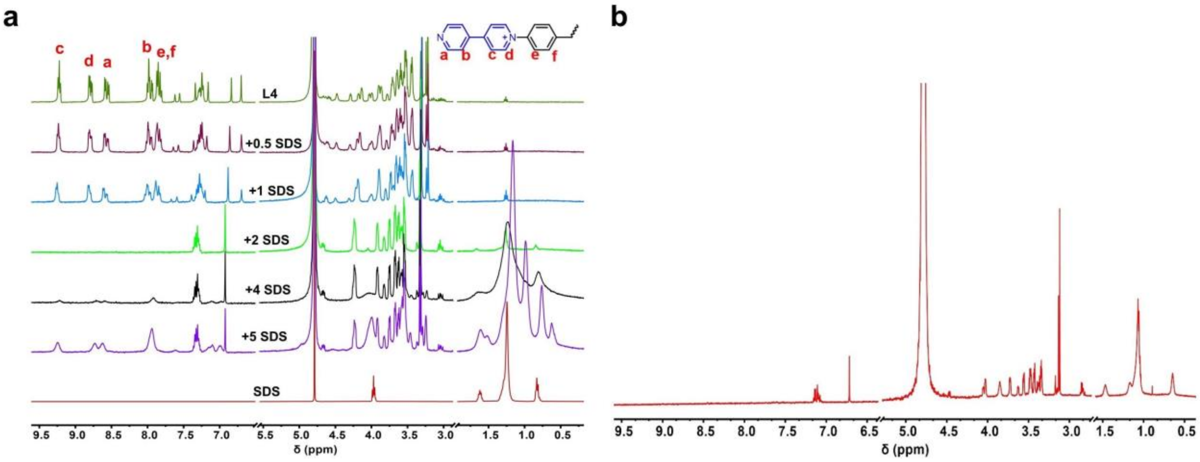


Figure S6. (a) ^{1}H NMR spectra L4 (1.0 mM) titrated by of SDS (0.1 M), D_2O as deuterated solvent, (b) Low temperature ^{1}H NMR spectra L4/2SDS at 278 K in D_2O.

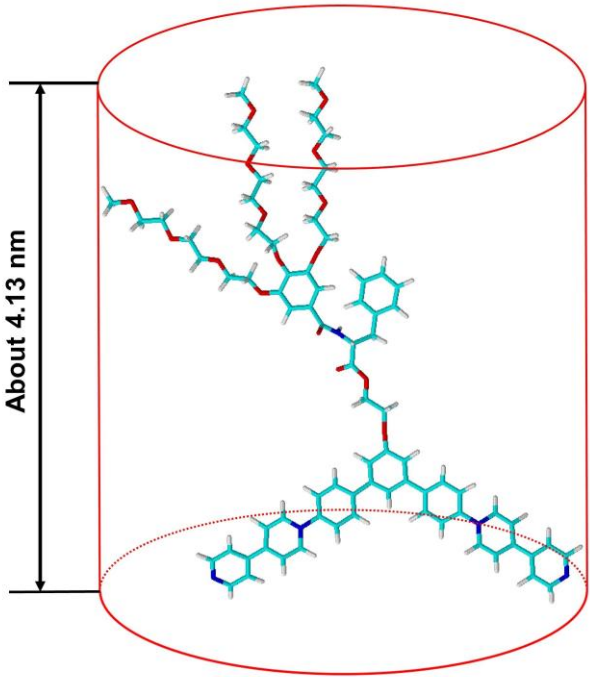


Figure S7. The length of L4 molecule was calculated and about 4.13 nm, estimated by Advanced Chemistry Development/ChemSketch molecular modeling software.

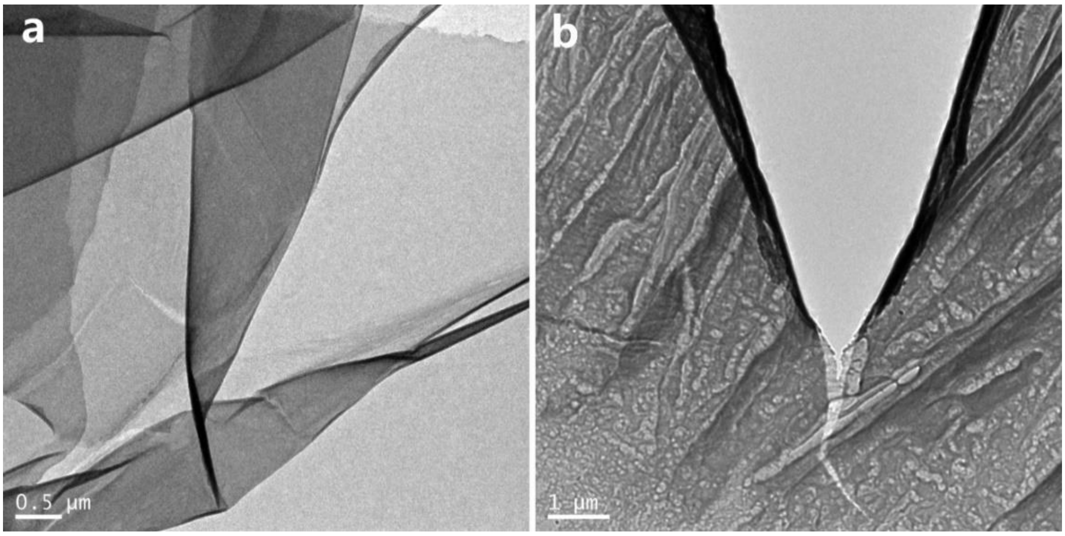


Figure S8. The TEM images of self-assembled structure of SDS obtained from the aqueous solution: (a) Smooth thin films of SDS from a dilute solution (0.01 mM), (b) Thick films of SDS with wrinkles and cracks from a concentrated solution (0.5 mM).

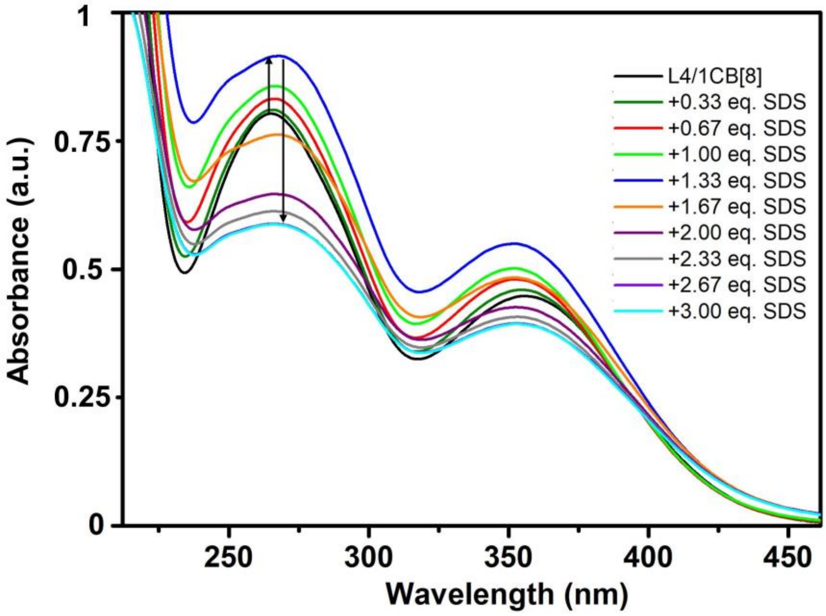


Figure S9. The UV-Vis spectra of L4/CB[8] ([L4] = 0.025 mM) titrated by SDS ([SDS] = 0.01 M), with the addition of SDS, the coassembled L4/CB[8] supramolecule dissociated (black trace to blue trace) and the generated L4 molecules coassembled with SDS gradually (blue trace to cyan trace).

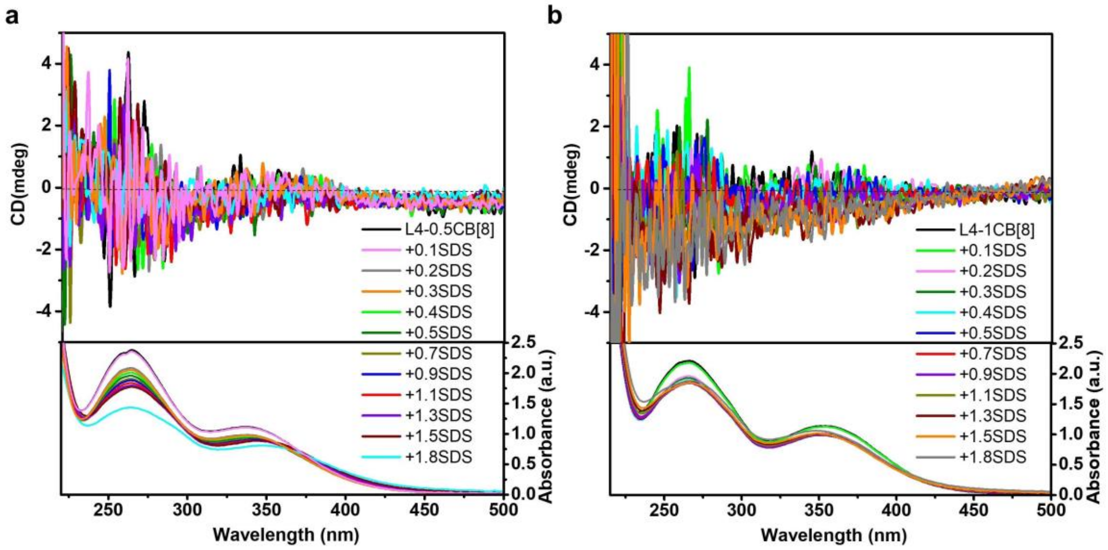


Figure S10. The CD spectra of L4/CB[8] binary supramolecular system titrated by SDS ([L4] = 0.05 mM), (a) L4: CB[8] = 1:0.5; (b) L4: CB[8] = 1:1.

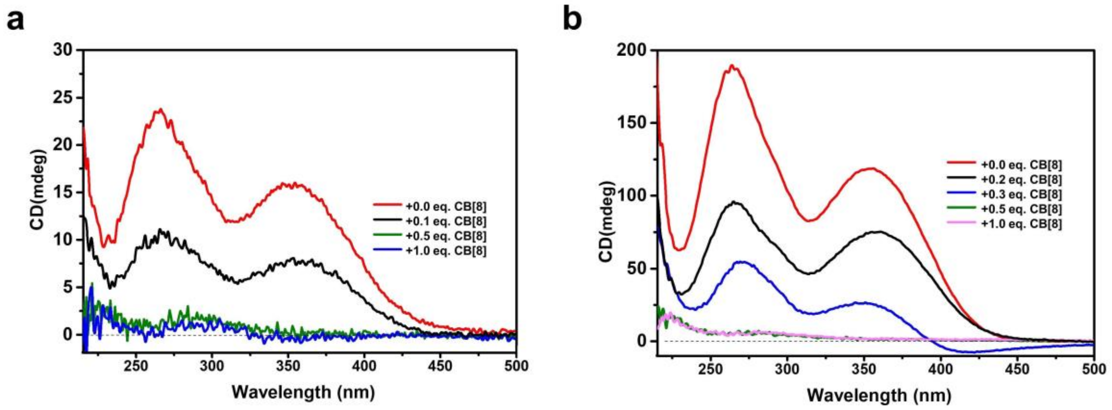


Figure S11. The CD spectra of L4/SDS binary supramolecular system titrated by CB[8] ([L4] = 0.05 mM), (a) L4:SDS = 1:2; (b) L4: SDS = 1:4.

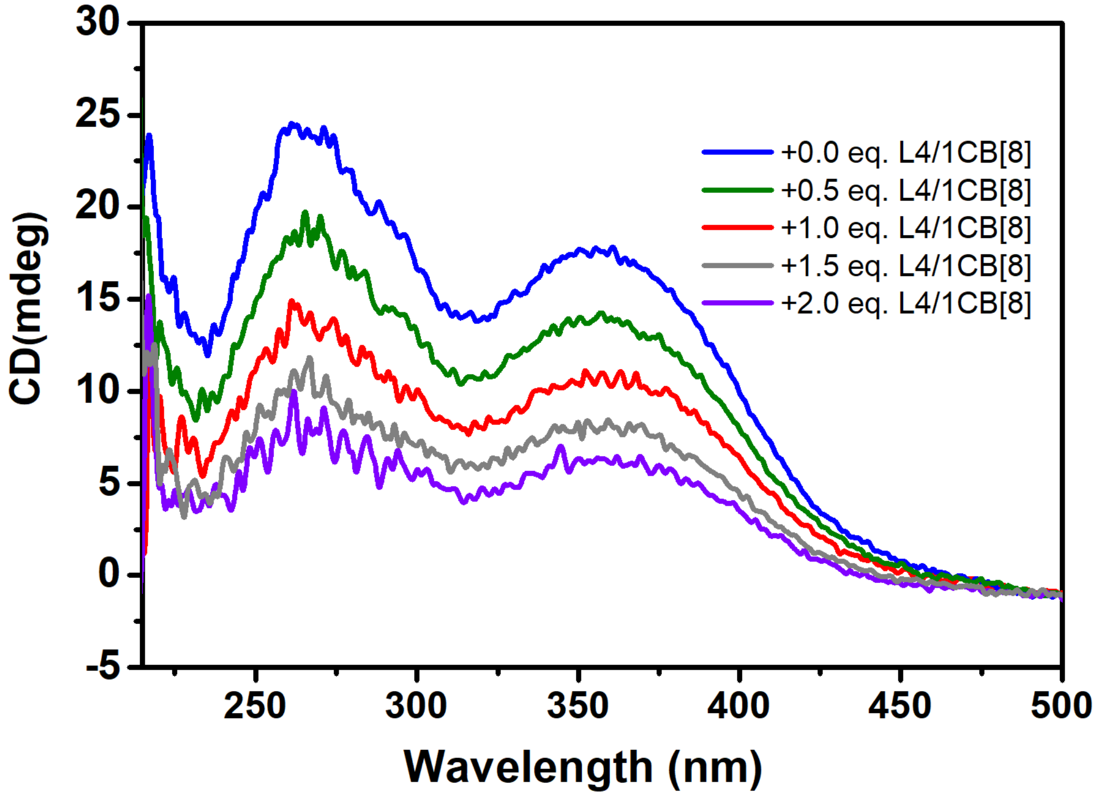


Figure S12. The CD spectra of mixture of two stable binary supramolecular systems of L4/CB[8] and L4/SDS ([L4] = 0.05 mM, L4/SDS was set as 1.0 *eq.*, and equivalent of L4/CB[8] varied)

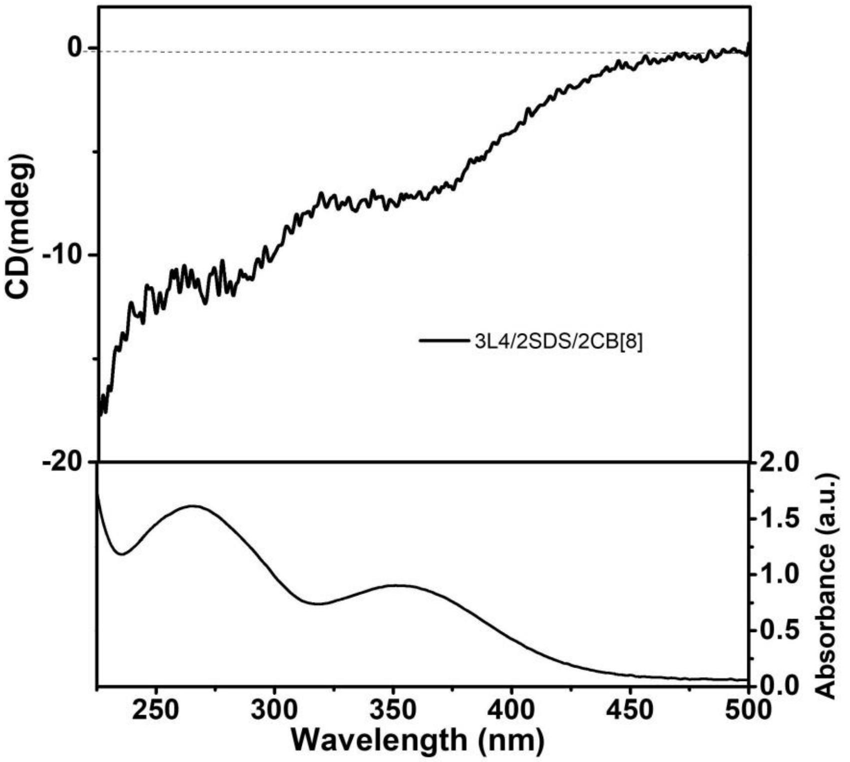


Figure S13. The CD spectrum of L4 ([L4] = 0.05 mM), SDS, and CB[8] which were mixed simultaneously with negative CD signals, the amount ratio of each component was set as 3:2:2 (1:0.67:0.67).

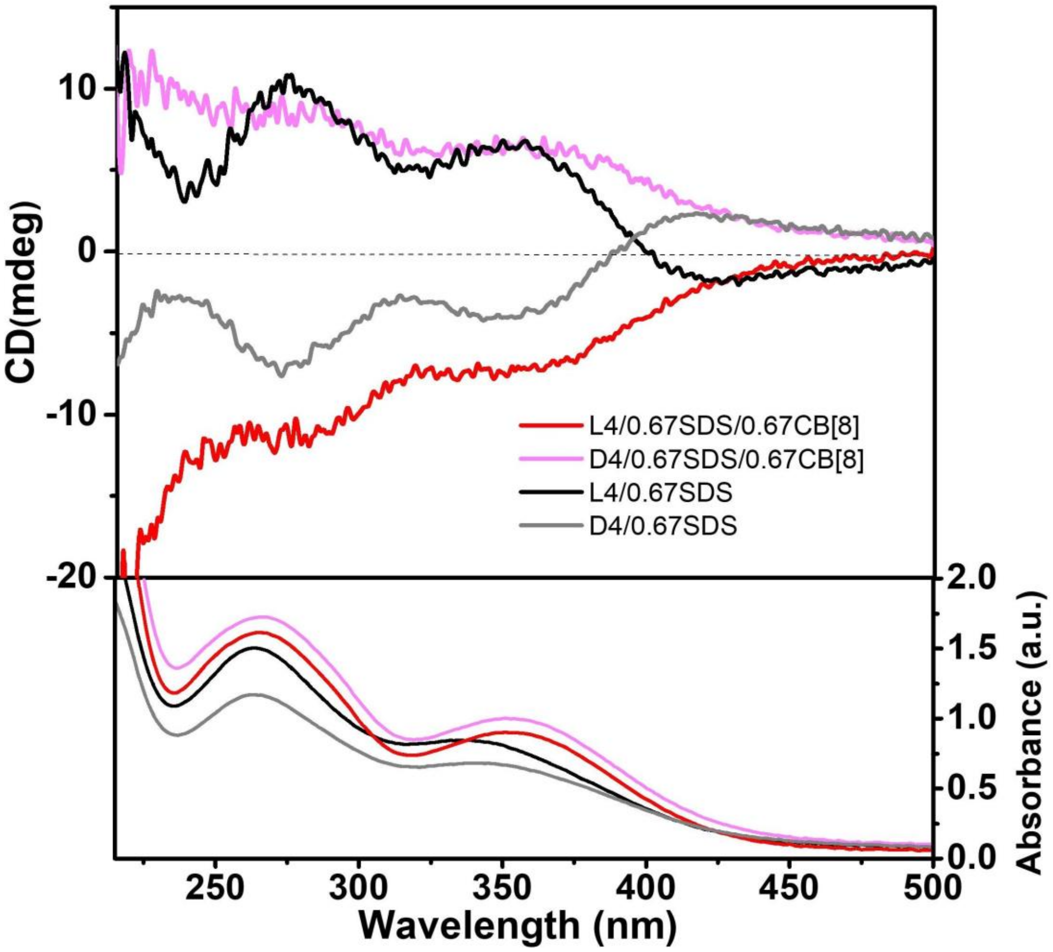


Figure S14. The CD spectra of L4/0.67SDS before (black line) and after (red line) the introduction of CB[8] ([L4] = 0.05 mM), D4/0.67SDS before (gray line) and after (pink line) the introduction of CB[8] ([L4] = 0.05 mM).

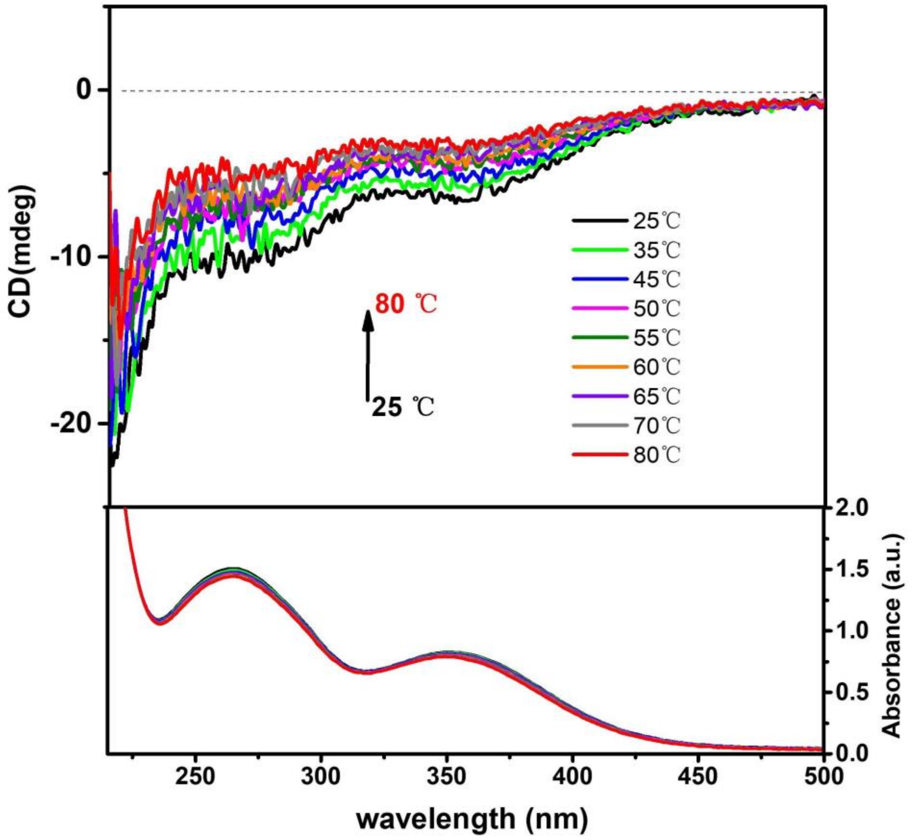


Figure S15. The CD spectra of the L4/0.67SDS/0.67CB[8] in H_2O ([L4] = 0.05 mM) at different temperatures.

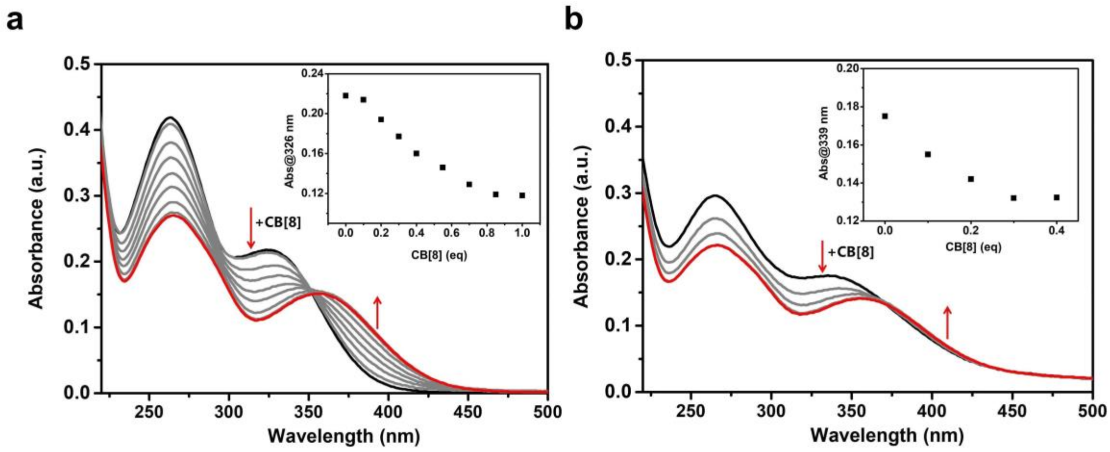


Figure S16. (a) the UV-Vis absorption spectra of L4/0.33SDS ([L4] = 0.01 mM) titrated by CB[8] ([CB[8]] = 0.1 mM, inset: the absorbance of L4/0.33SDS at 326 nm varies with different quantities of CB[8]). (b) the UV-Vis absorption spectra of L4/1.5SDS ([L4] = 0.01 mM) titrated by CB[8] ([CB[8]] = 0.1 mM, inset: the absorbance of L4/1.5SDS at 339 nm with different quantities of CB[8]).

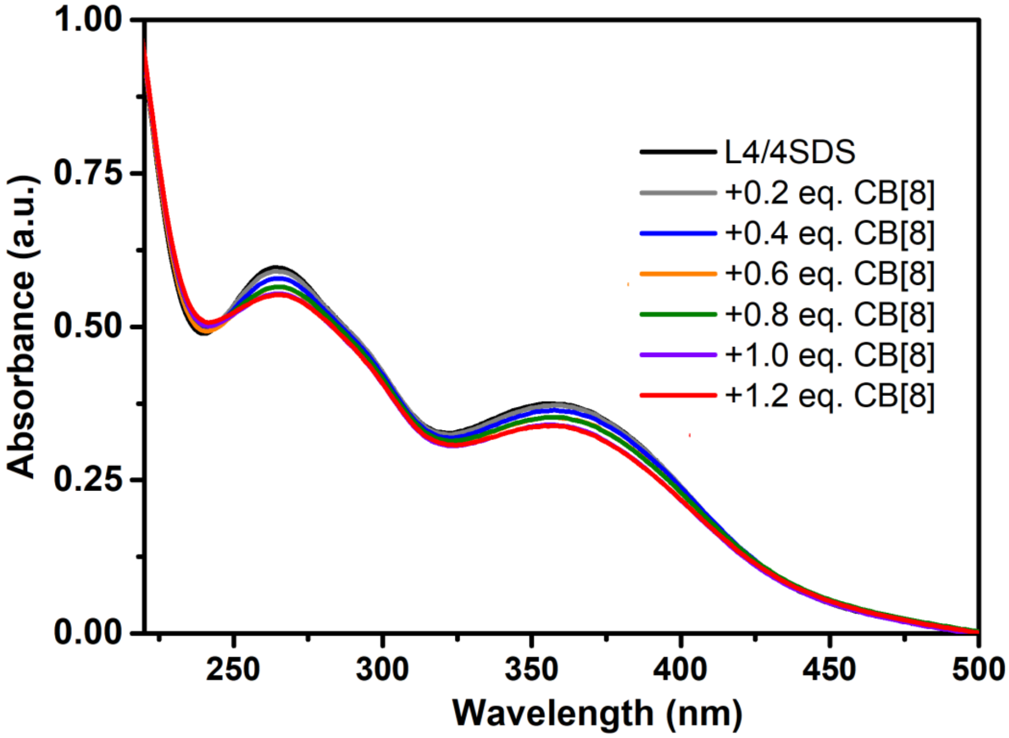


Figure S17. The UV-Vis absorption spectra of L4/4SDS ([L4] = 0.01 mM) titrated by CB[8] ([CB[8]] = 0.1 mM).

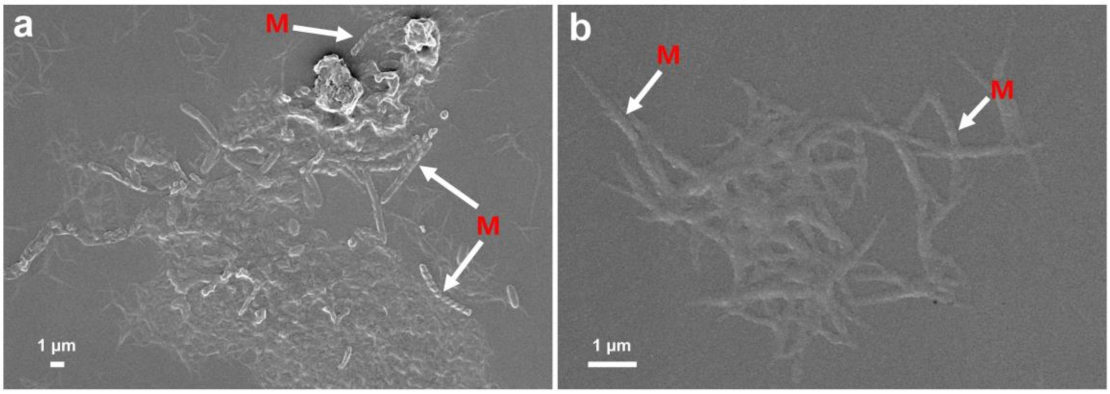


Figure S18. The FE-SEM images of L4/0.67SDS, showing M-type helical supramolecular structures obtained from H_2O solution ([L4] = 0.05 mM).

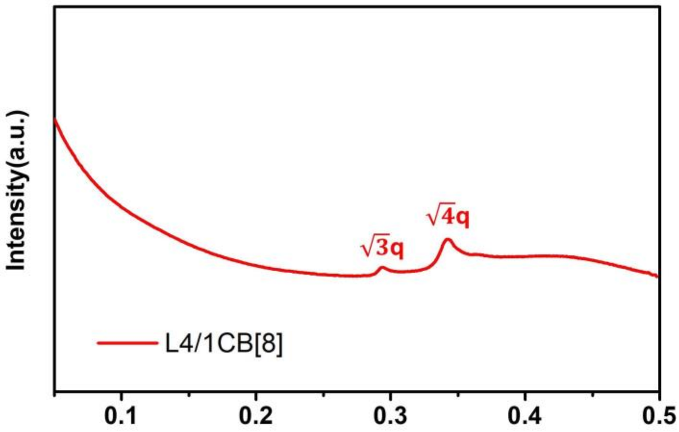


Figure S19. The SAXS pattern of stable binary L4/CB[8] supramolecules, showing a hexagonal stacking mode.

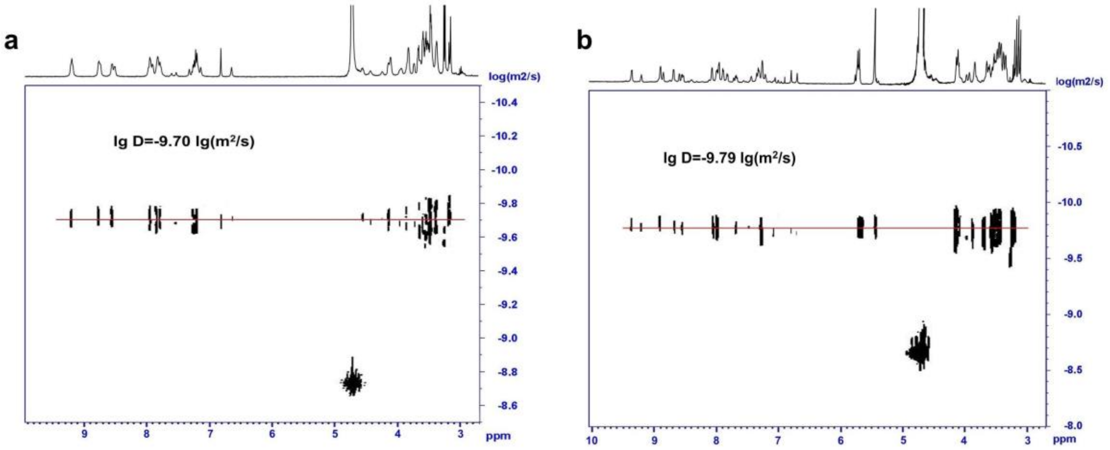


Figure S20. 2D-DOSY spectra (600 M, D_2O, 298 K) of (a) L4/0.67SDS and (b) L4/0.67SDS/0.67CB[8].

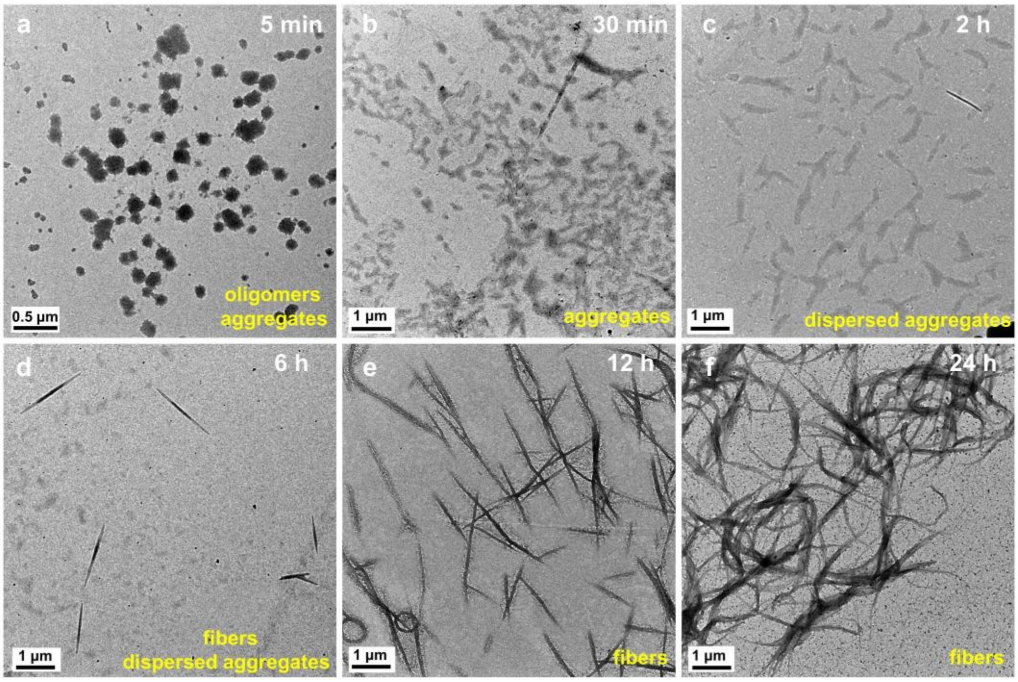


Figure S21. TEM images of binary L4/0.67SDS supramolecular structures at different periods. The upright time indicates the time after mixing L4 and SDS in water. (a) 5 min, (b) 30 min, (c) 2 h, (d) 6 h, (e) 12 h, and (f) 24 h.

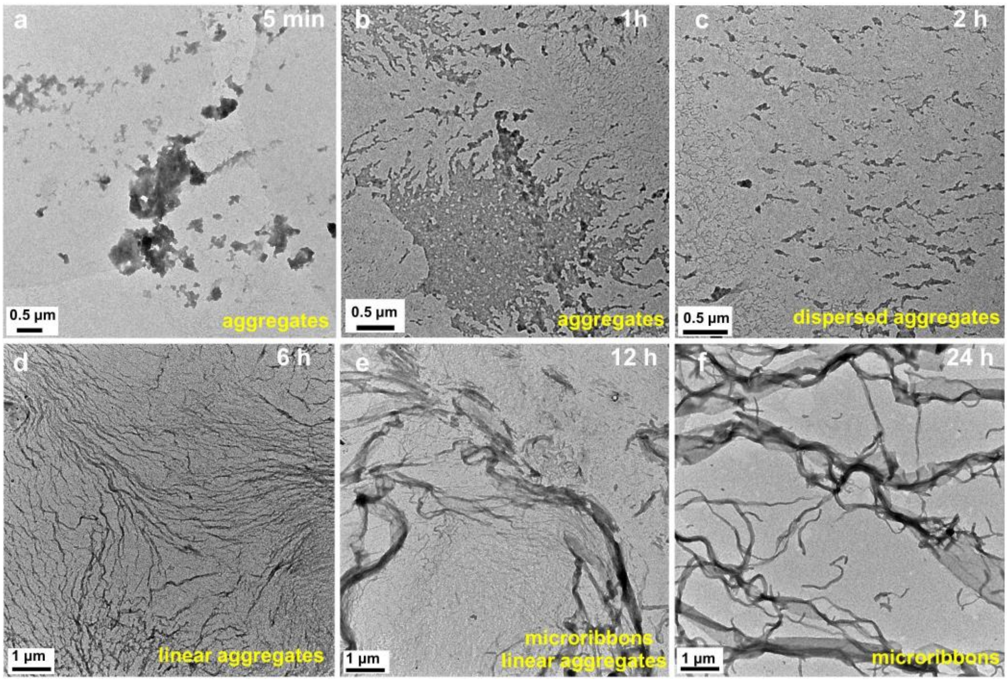


Figure S22. TEM images of ternary L4/0.67SDS/0.67CB[8] supramolecular structures at different periods. The upright time indicates the time after CB[8] was added to the aqueous solution. (a) 5 min, (b)1 h, (c)2 h, (d) 6 h, (e) 12 h, and (f) 24 h.

3. Additional Figures of Synthesis

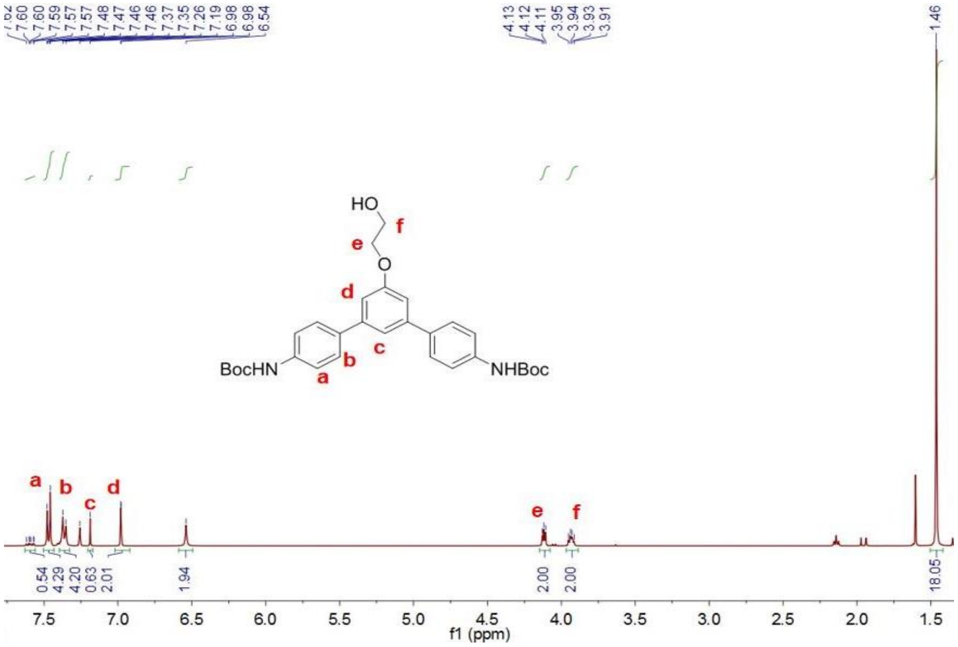


Figure S23. ^{1}H NMR spectrum (400 MHz, CDCl$_3$, 298 K) of compound A4.

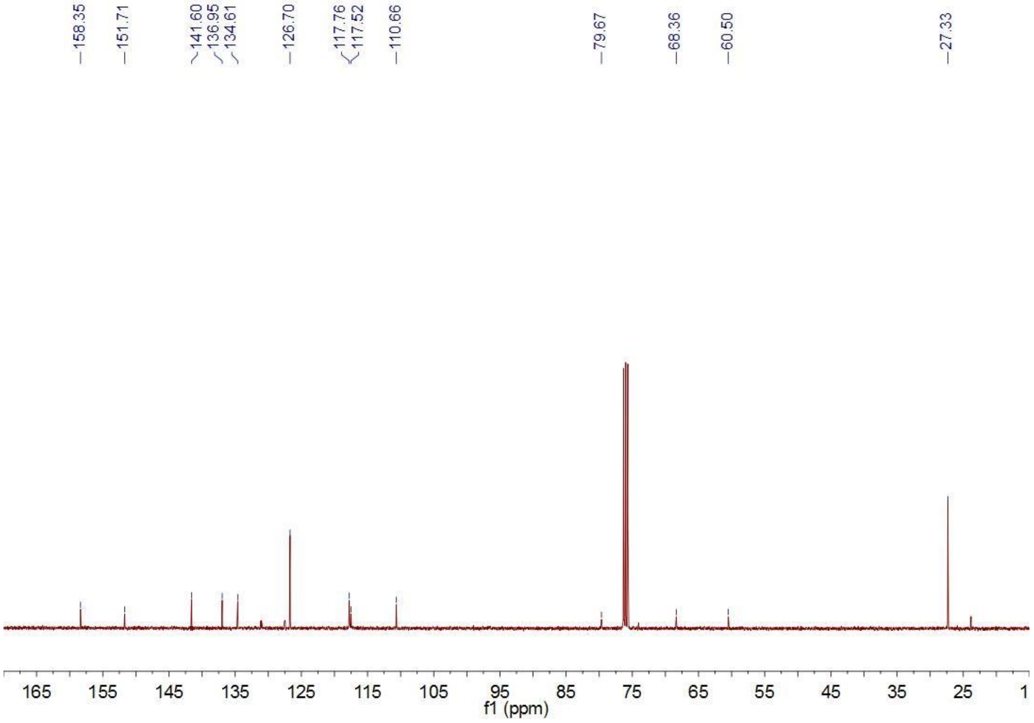
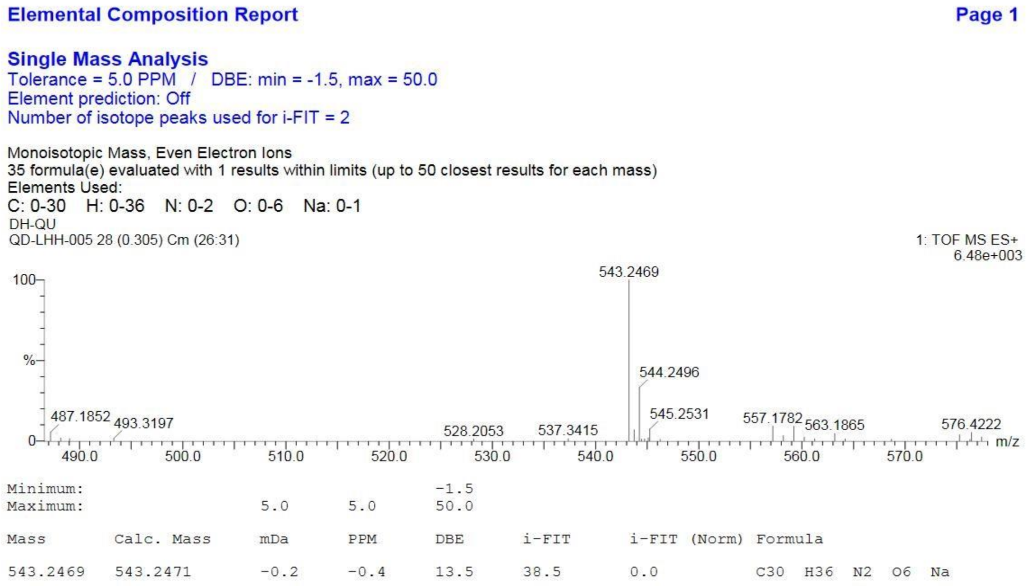


Figure S24. ^{13}C NMR spectrum (100 MHz, CDCl$_3$, 298 K) of compound A4.



Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
35 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-30 H: 0-36 N: 0-2 O: 0-6 Na: 0-1
DH-QU
QD-LHH-005 28 (0.305) Cm (26:31) 1: TOF MS ES+ 6.48e+003

```
Minimum:                                       -1.5
Maximum:                  5.0        5.0       50.0

Mass       Calc. Mass     mDa        PPM       DBE        i-FIT      i-FIT (Norm) Formula

543.2469   543.2471       -0.2       -0.4      13.5       38.5       0.0          C30  H36  N2  O6  Na
```

Figure S25. ESI-MS spectrum of compound A4.

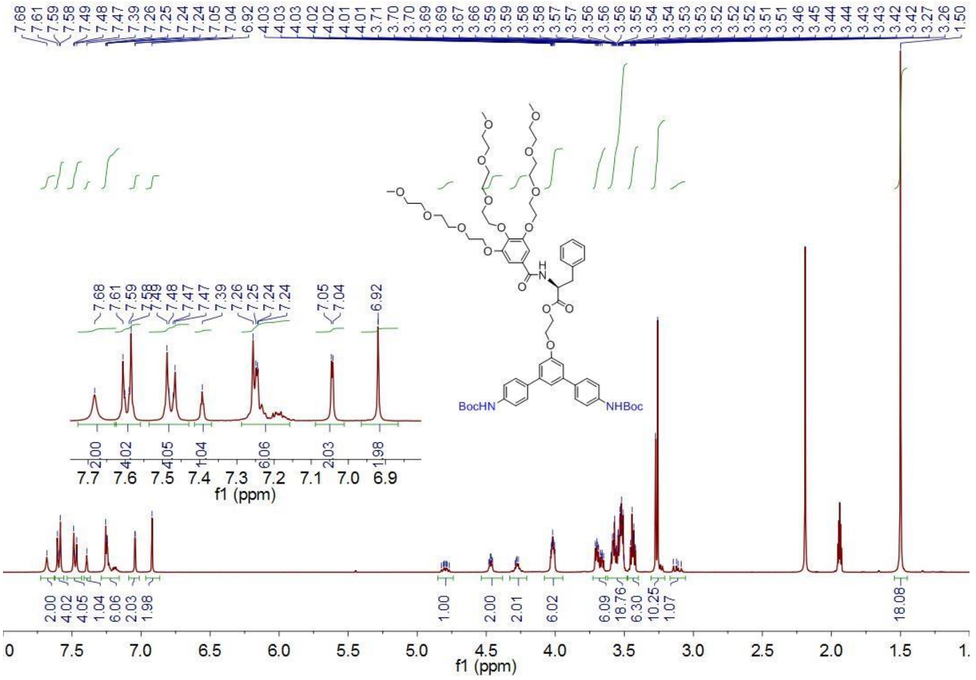


Figure S26. ^{1}H NMR spectrum (400 MHz, CD_3CN, 298 K) of compound L2.

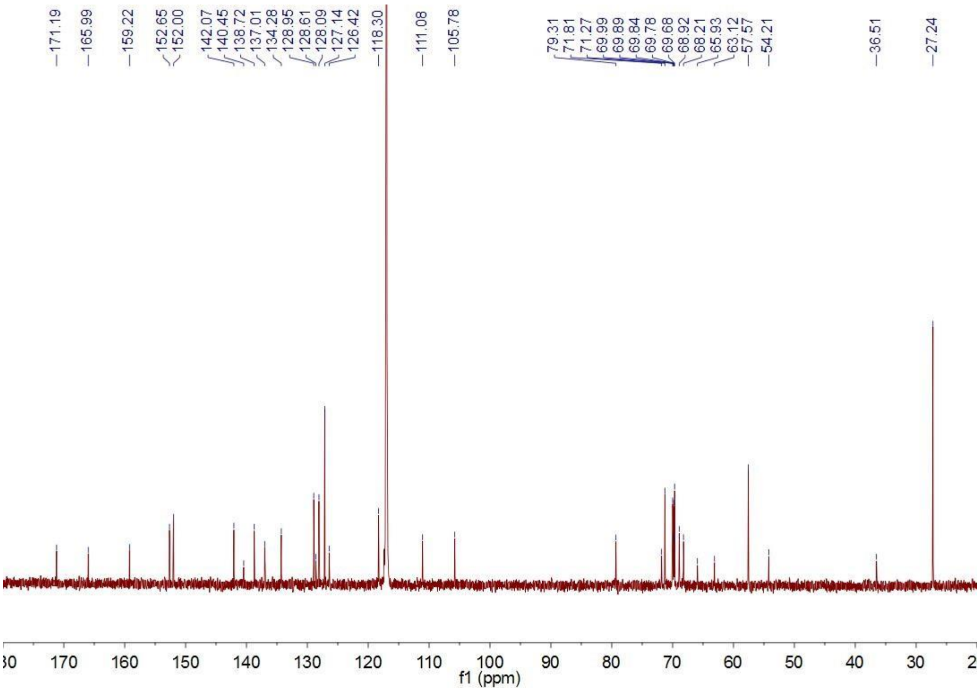
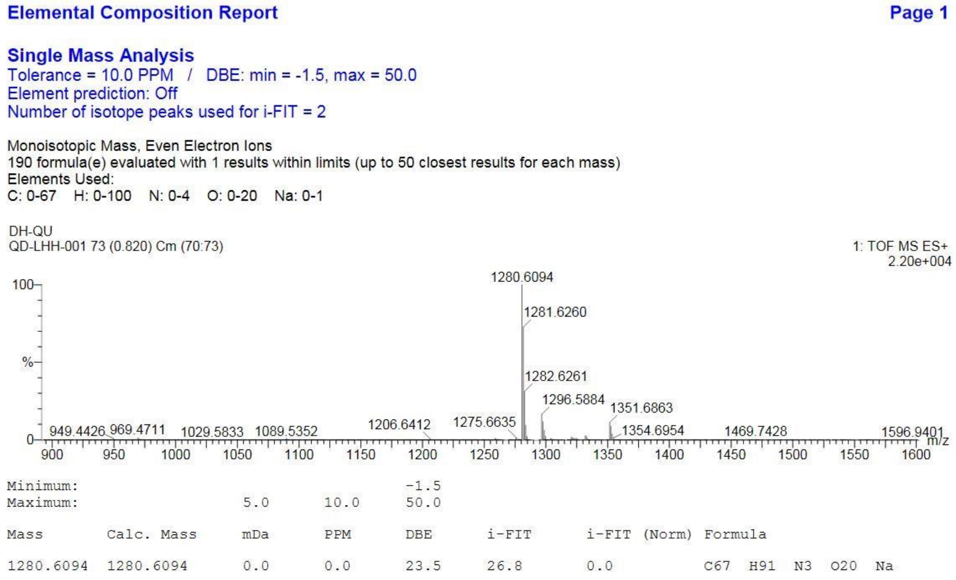


Figure S27. ^{13}C NMR spectrum (100 MHz, CD_3CN, 298 K) of compound L2.



Elemental Composition Report

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Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
190 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-67 H: 0-100 N: 0-4 O: 0-20 Na: 0-1

DH-QU
QD-LHH-001 73 (0.820) Cm (70:73)

1: TOF MS ES+
2.20e+004

```
Minimum:                               -1.5
Maximum:                  5.0    10.0  50.0

Mass       Calc. Mass     mDa    PPM   DBE     i-FIT    i-FIT (Norm) Formula

1280.6094  1280.6094      0.0    0.0   23.5    26.8     0.0          C67  H91  N3  O20  Na
```

Figure S28. ESI-MS spectrum of compound L2.

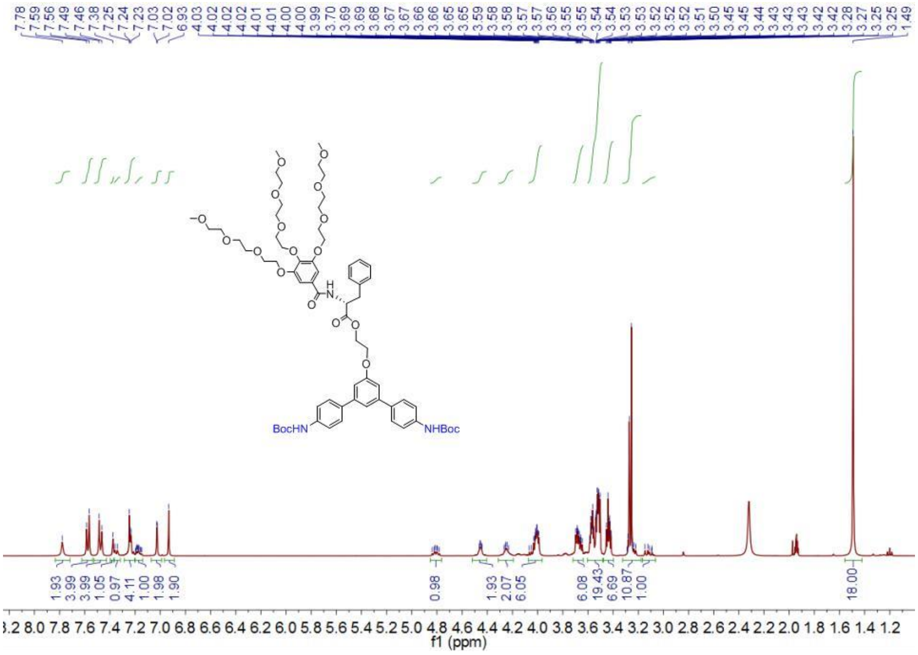


Figure S29. ^{1}H NMR spectrum (400 MHz, CD_3CN, 298 K) of compound D2.

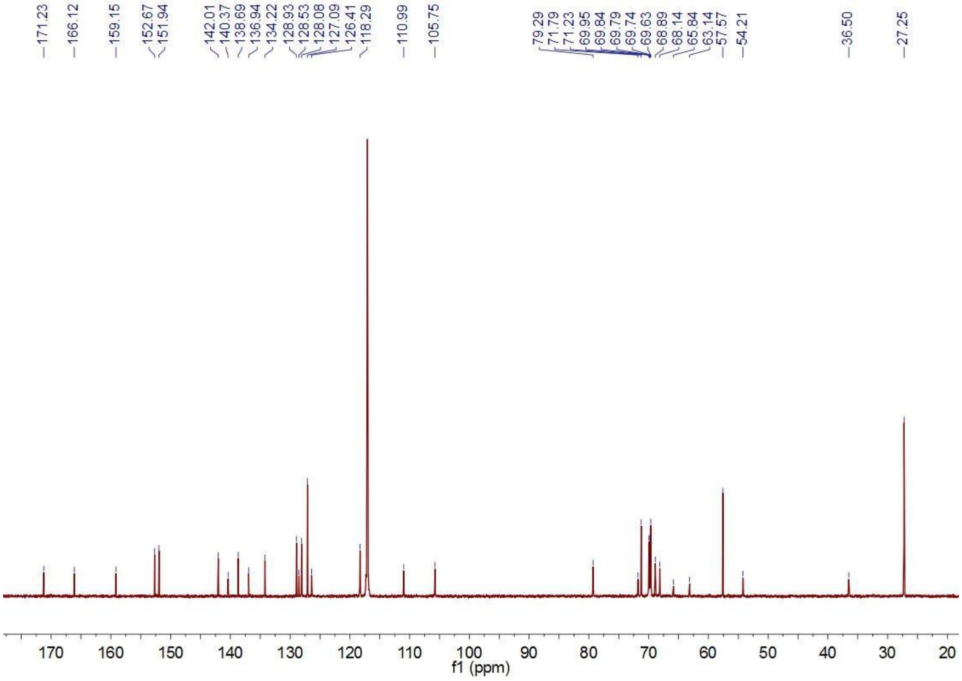
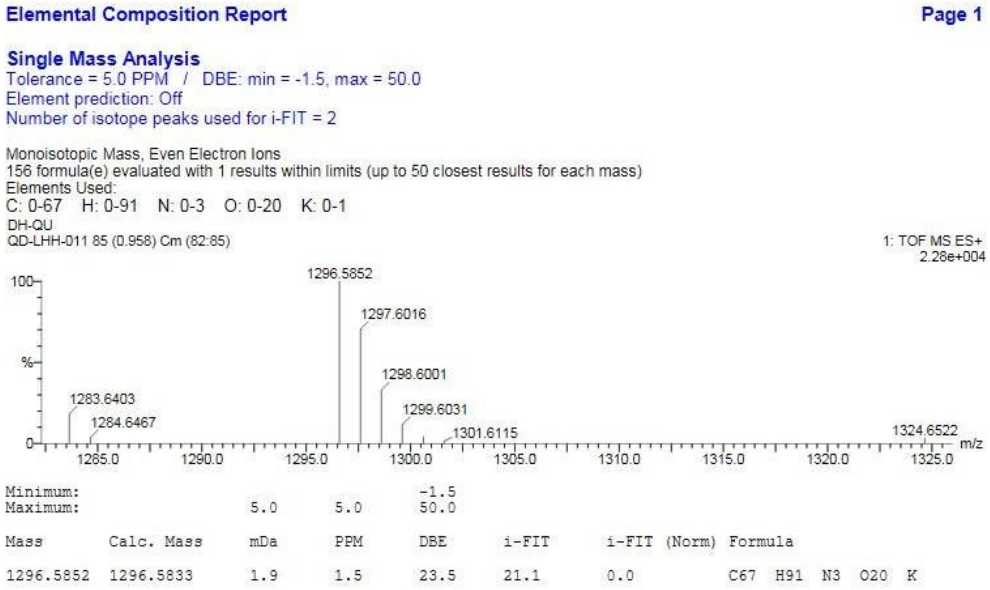


Figure S30. ^{13}C NMR spectrum (100 MHz, CD_3CN, 298 K) of compound D2.



Elemental Composition Report

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Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
156 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-67 H: 0-91 N: 0-3 O: 0-20 K: 0-1
DH-QU
QD-LHH-011 85 (0.958) Cm (82:85)

1: TOF MS ES+
2.28e+004

```
Minimum:                                  -1.5
Maximum:                   5.0     5.0    50.0

Mass        Calc. Mass     mDa     PPM     DBE      i-FIT     i-FIT (Norm) Formula

1296.5852   1296.5833      1.9     1.5     23.5     21.1      0.0          C67  H91  N3  O20  K
```

Figure S31. ESI-MS spectrum of compound D2.

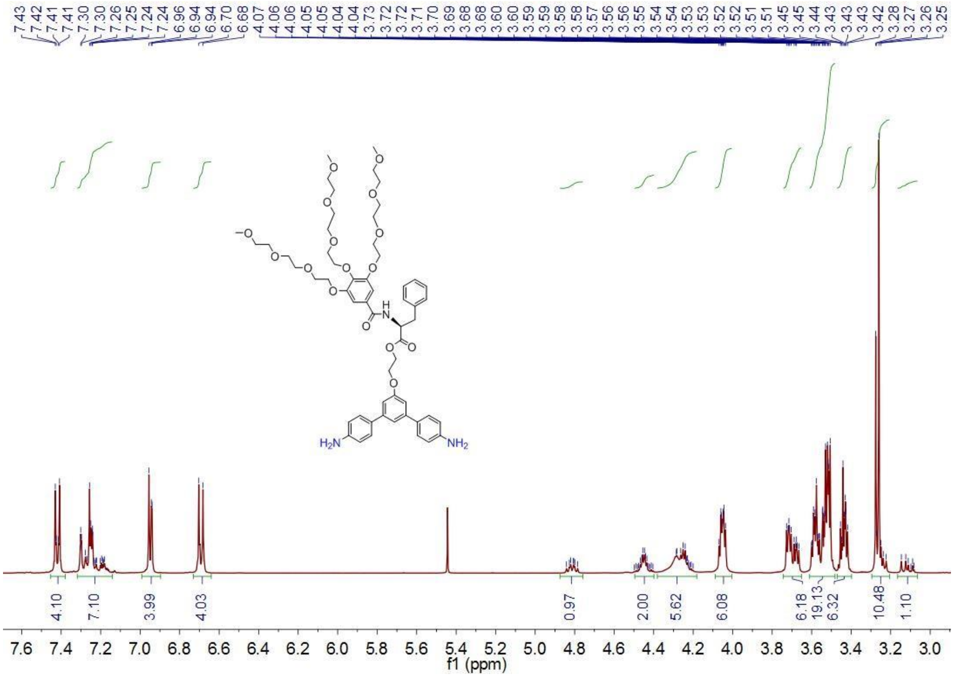


Figure S32. ^{1}H NMR spectrum (400 MHz, CD_3CN, 298 K) of compound L3.

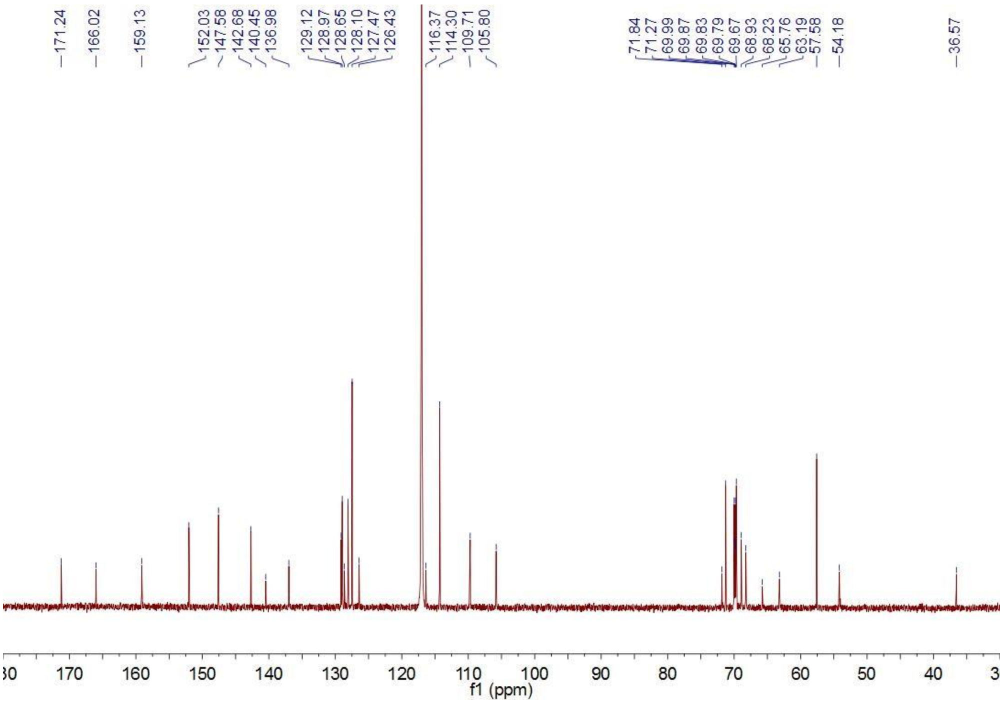
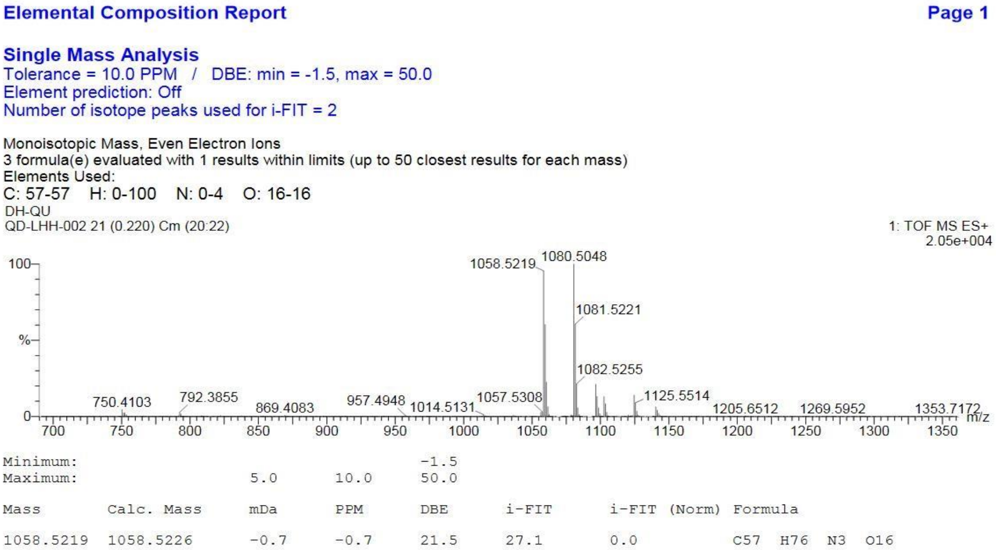


Figure S33. ^{13}C NMR spectrum (100 MHz, CD_3CN, 298 K) of compound L3.



Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
3 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 57-57 H: 0-100 N: 0-4 O: 16-16
DH-QU
QD-LHH-002 21 (0.220) Cm (20:22)

1: TOF MS ES+
2.05e+004

```
Minimum:                                        -1.5
Maximum:                     5.0        10.0    50.0

Mass       Calc. Mass       mDa      PPM      DBE       i-FIT      i-FIT (Norm) Formula

1058.5219  1058.5226       -0.7     -0.7     21.5      27.1       0.0          C57  H76  N3  O16
```

Figure S34. ESI-MS spectrum of compound L3.

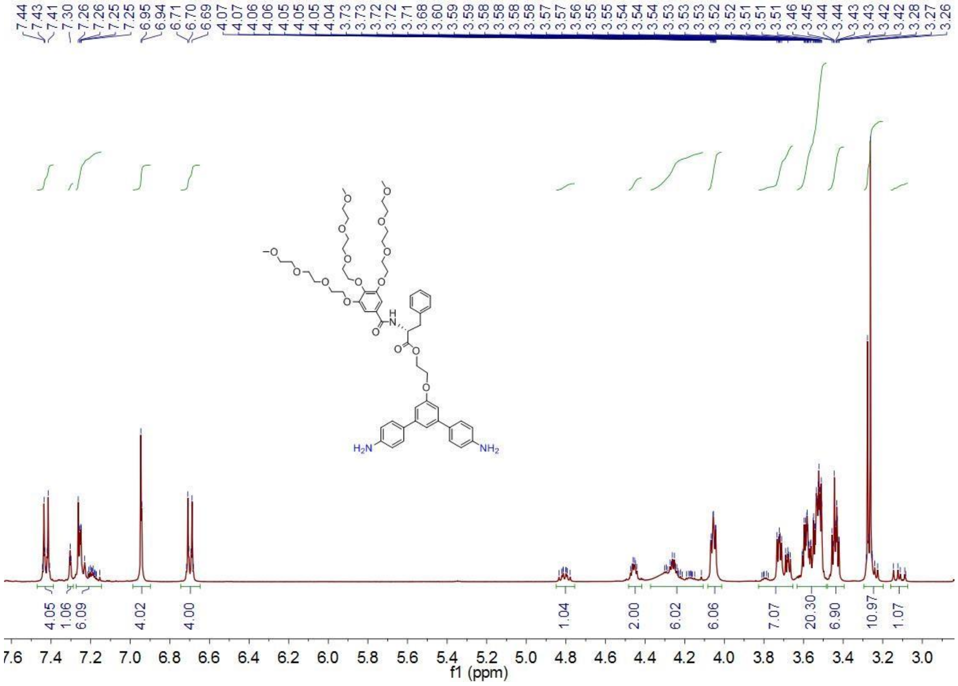


Figure S35. ^{1}H NMR spectrum (400 MHz, CD_3CN, 298 K) of compound D3.

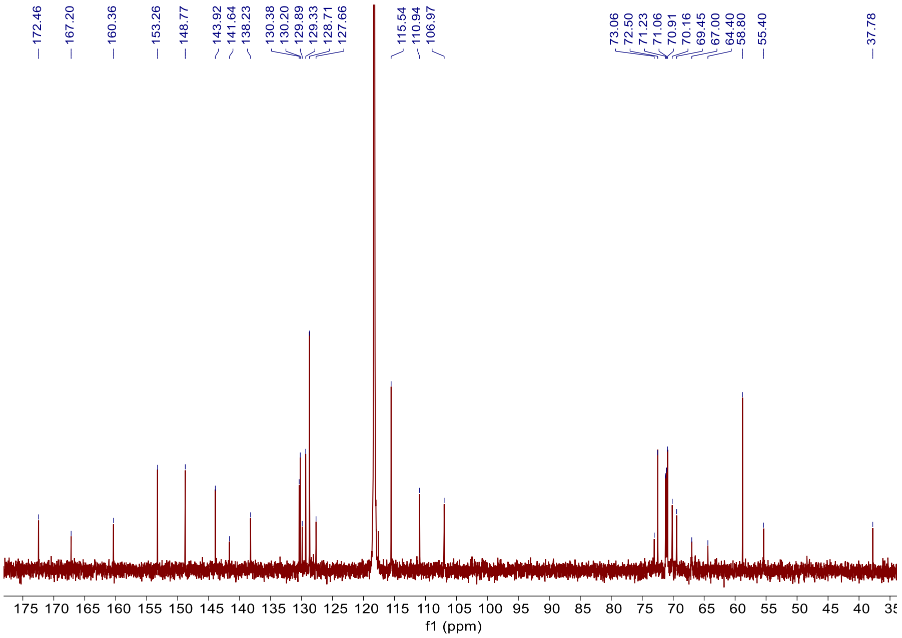
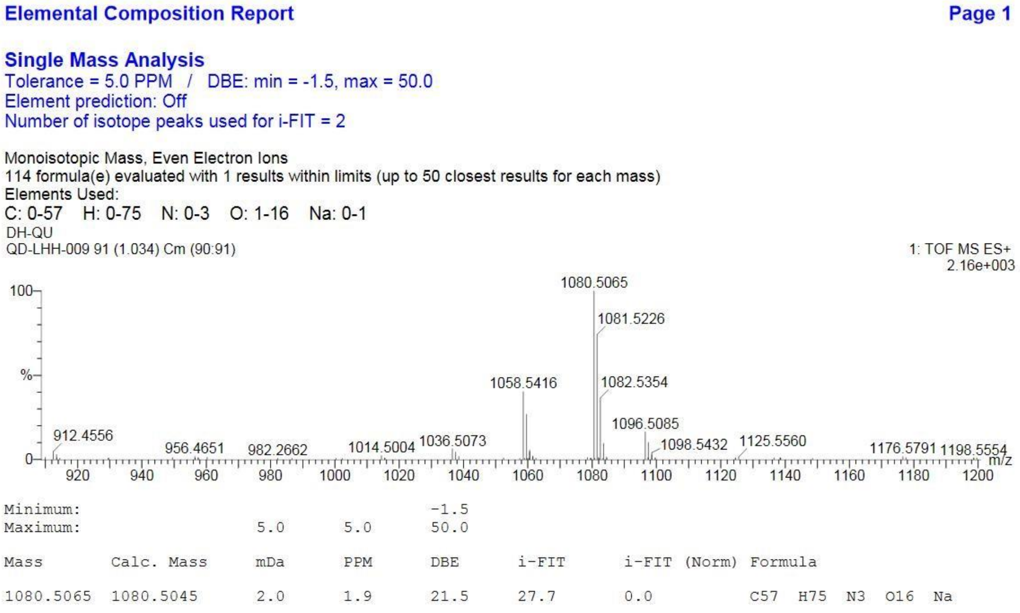


Figure S36. ^{13}C NMR spectrum (100 MHz, CD_3CN, 298 K) of compound D3.



Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
114 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-57 H: 0-75 N: 0-3 O: 1-16 Na: 0-1
DH-QU
QD-LHH-009 91 (1.034) Cm (90:91)

1: TOF MS ES+
2.16e+003

```
Minimum:                                    -1.5
Maximum:                 5.0       5.0      50.0

Mass       Calc. Mass    mDa       PPM      DBE      i-FIT      i-FIT (Norm)  Formula

1080.5065  1080.5045     2.0       1.9      21.5     27.7       0.0           C57  H75  N3  O16  Na
```

Figure S37. ESI-MS spectrum of compound D3.

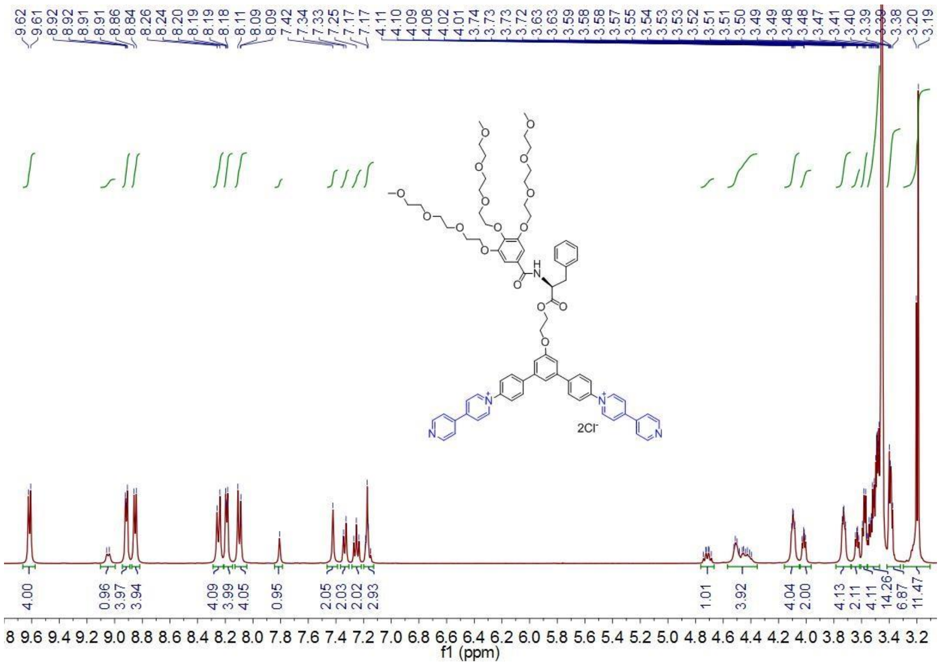


Figure S38. ^{1}H NMR spectrum (400 MHz, DMSO-d_6, 298 K) of compound L4.

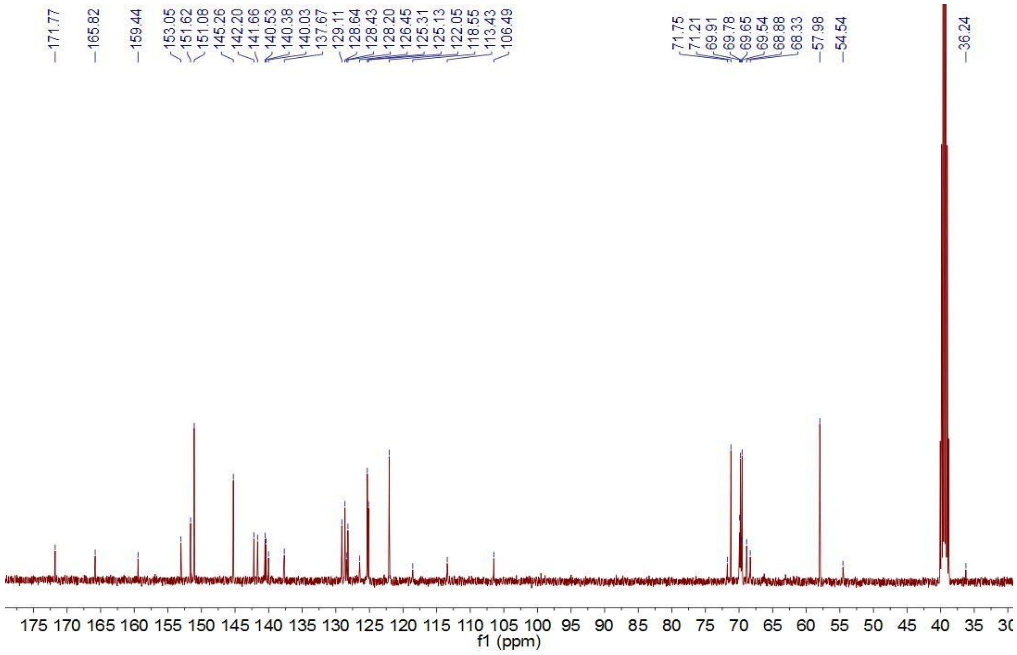


Figure S39. ^{13}C NMR spectrum (100 MHz, DMSO-d$_6$, 298 K) of compound L4.

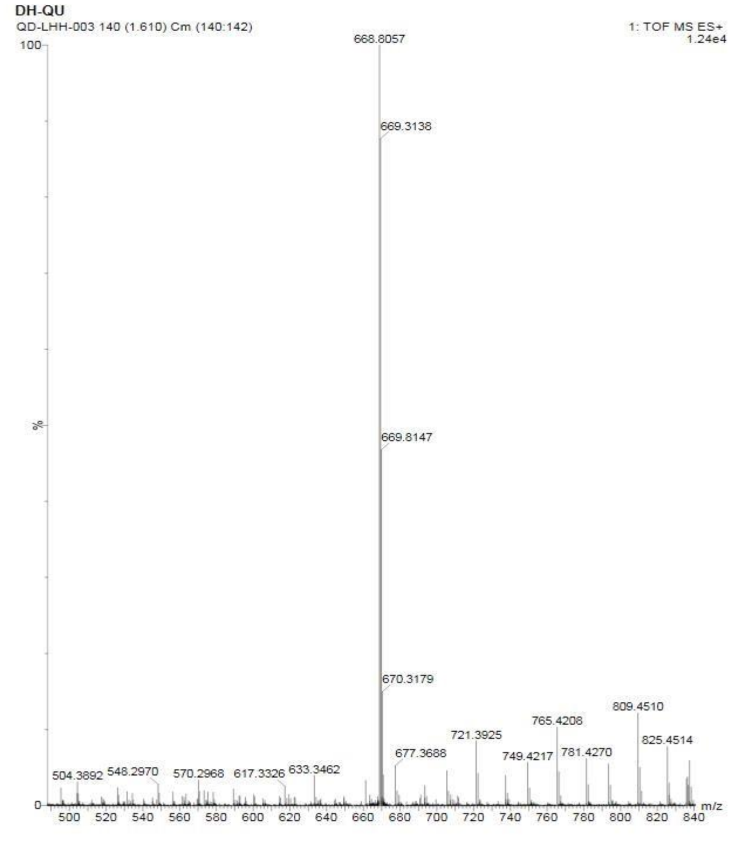


Figure S40. ESI-MS spectrum of compound L4.

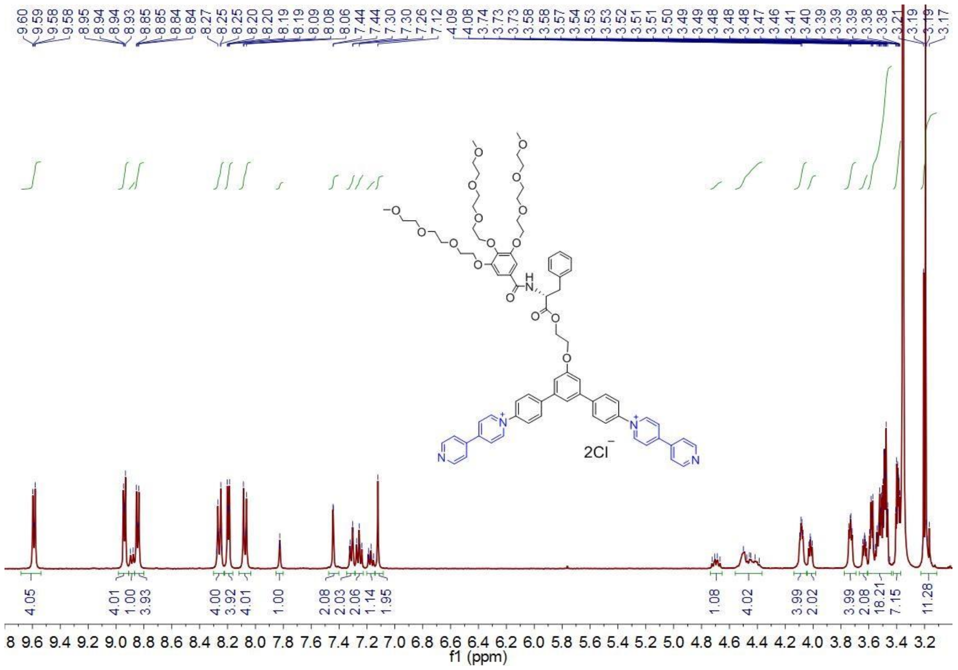


Figure S41. ^{1}H NMR spectrum (400 MHz, DMSO-d$_6$, 298 K) of compound D4.

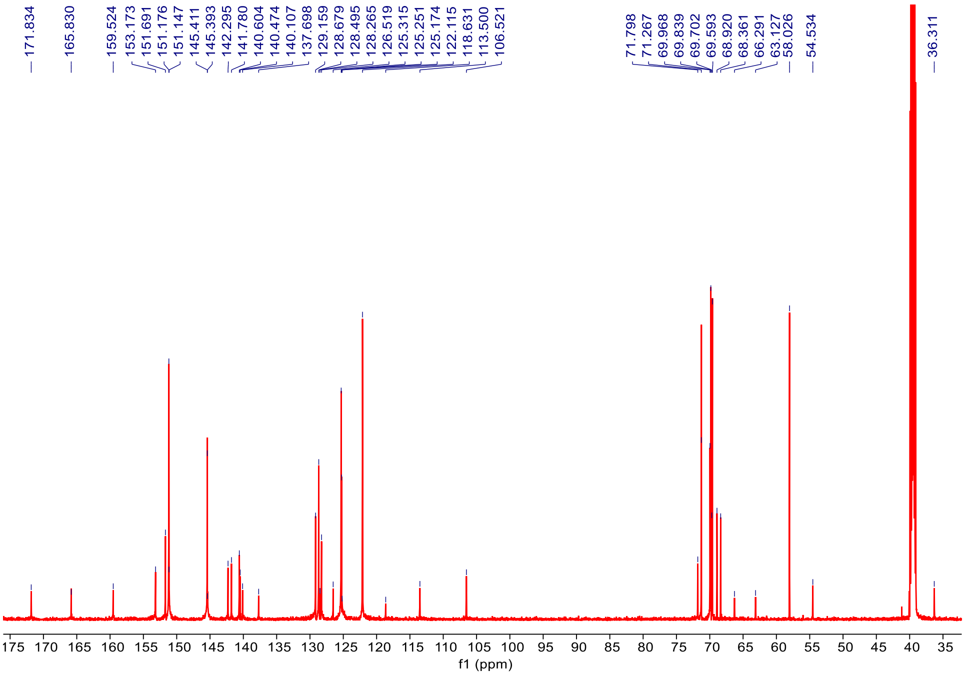


Figure S42. ^{13}C NMR spectrum (100 MHz, DMSO-d$_6$, 298 K) of compound D4.

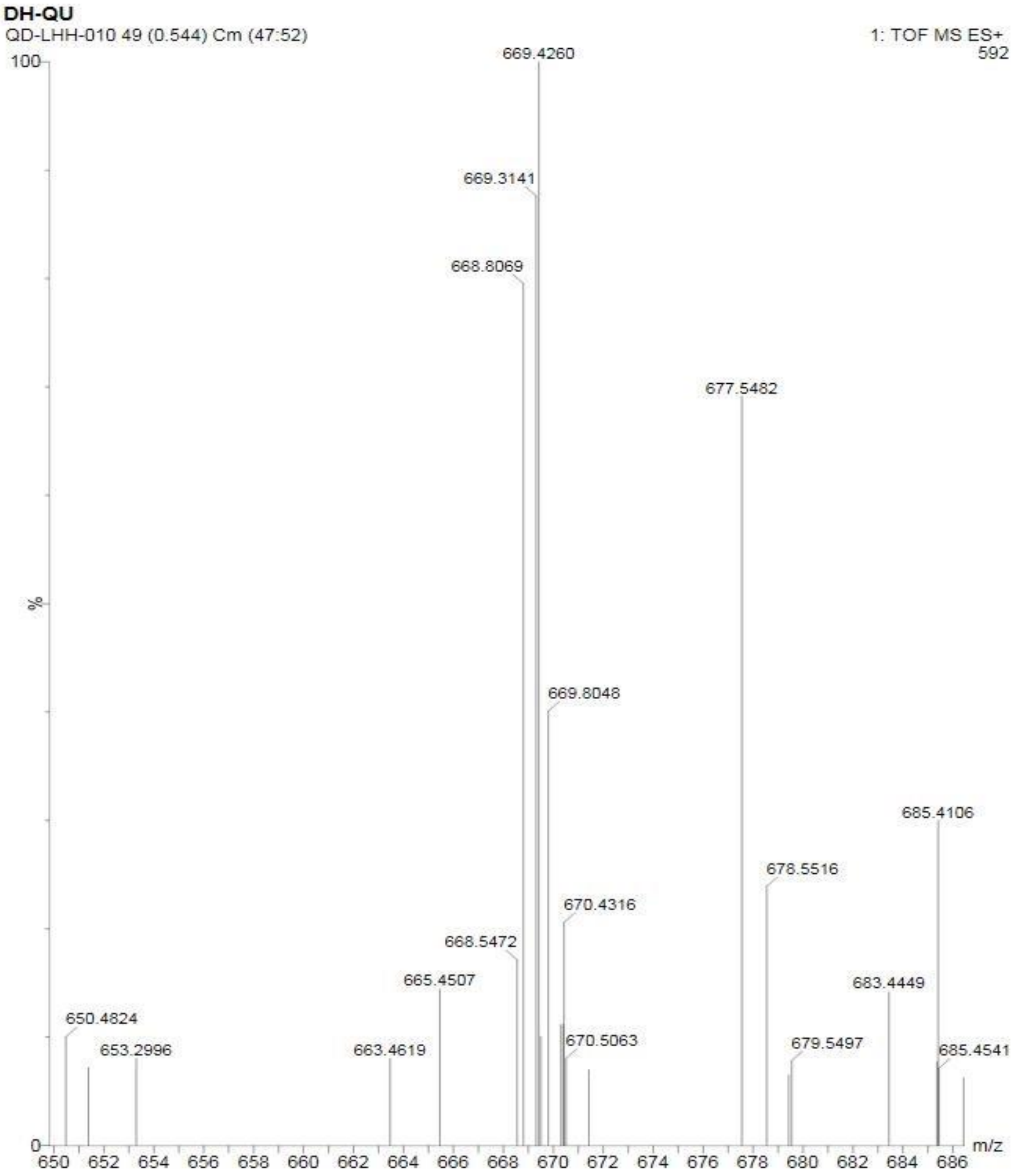


Figure S43. ESI-MS spectrum of compound D4.

4. References

1. T. Kikuchi, S. Sato and M. Fujita, *J. Am. Chem. Soc.*, 2010, **132**, 15930-15932.
2. C. Chen, P. Pan, Z. Deng, D. Wang, Q. Wu, L. Xu, T. Hou and S. Cui, *Bioorg. Med. Chem. Lett.*, 2019, **29**, 912-916.
3. S. Zhang, H. J. Sun, A. D. Hughes, R. O. Moussodia, A. Bertin, Y. Chen, D. J. Pochan, P. A. Heiney, M. L. Klein and V. Percec, *Proc. Natl. Acad. Sci. U. S. A.*, 2014, **111**, 9058-9063.
4. L. Cao, G. Fang and Y. Wang, *Langmuir*, 2017, **33**, 980-987.