Electronic Supplementary Material (ESI) for ChemComm.

Supporting information

Controlling interfacial interaction of supramolecular assemblies by light-responsive overcrowded alkene

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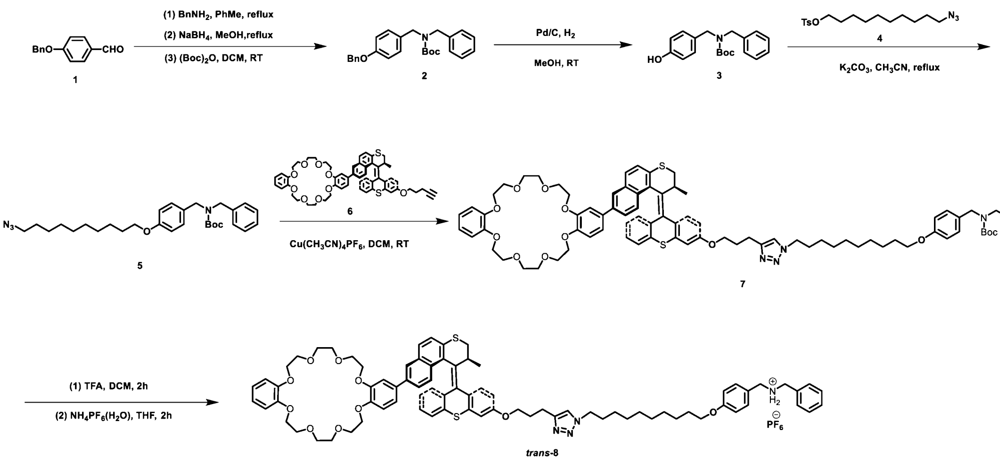
1. Experiments Section

1.1 General Methods and Details

Chemicals were purchased from Acros, Aldrich, TCI, Adamas, or Merck and used as received unless otherwise stated. Solvents were reagent grade, which were dried and distilled prior to use according to standard procedures. All reactions were carried out under an atmosphere of dry argon unless otherwise stated. ^{1}H NMR, ^{13}C NMR, ^{1}H-^{1}H COSY spectra were measured on a Brüker AV-400 and AV-600 spectrometer. The electronic spray ionization (ESI) mass spectra were tested on a LCT Premier XE mass spectrometer. UV-Vis absorption spectra were recorded on Varian Cary 100 spectrometer (1 cm, quartz cells). The UV light resource were obtained on a Perfect Light PL-LED 100. DLS were measured on MALV RN, ZETA SIZER, Model ZEN3600, 303K. SEM images were recorded on a 500-300000/GeminiSEM 500 apparatus. TEM images were recorded on a JEOL JEM-1400 apparatus, and the samples were prepared by casting dilute solution on copper sheet.

1.2 Synthesis of target monomer *trans*-8



Scheme S1. Synthetic route of *trans*-**8**

Compound **1**, Compound **4** and compound **6** were synthesized according to previous reports[1-3].

Synthesis of compound 2

A mixture of compound **1** (2.50 g, 11.79 mmol) and benzylamine (1.26 g, 11.79 mmol) in dry toluene (70 mL) was heated under reflux for 12 hours. The reaction mixture was cooled to room temperature and the solvent was removed under vacuum. The residue was dissolved in MeOH (45 mL), $NaBH_4$ (0.89 g, 23.53 mmol) was added under ice bath, and the mixture was stirred for 4 h. The solvent was evaporated under reduced pressure and the crude residue was partitioned between CH_2Cl_2 and 2 N NaOH solution. The aqueous layer was further extracted with CH_2Cl_2 and the organic layer was dried over anhydrous sodium sulfate. The obtained oil was stirred together with $(Boc)_2O$ (3.85 g, 17.68 mmol) in dry CH_2Cl_2 for 5 h at room temperature. The solvent was evaporated under reduced pressure and the crude residue was purified by column chromatography (SiO_2, PE/EA = 5/1) to afford compound **2** (3.60g, 76%) as a white oil. ^{1}H NMR (400 MHz, $CDCl_3$, 298K) δ (ppm): 7.38-7.45 (m, 4H), 7.35 (d, J = 1.6 Hz, 1H), 7.32 (d, J = 7.6 Hz, 2H), 7.07-7.29 (m, 5H), 6.94 (d, J = 8.8 Hz, 2H), 5.07 (s, 2H), 4.27-4.40 (m, 4H), 1.50 (s, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz, 298 K) δ (ppm): 158.21, 156.11, 137.15, 130.48, 129.51, 128.98, 128.71, 128.62, 128.09, 127.59, 127.29, 114.99, 80.12, 70.18, 49.20, 48.52, 28.60. HR-MS (ESI) (m/z): $[M + Na]^+$ calcd for $C_{26}H_{29}NO_3$, 426.2045, found 426.2043.

Synthesis of compound 3

A mixture of compound **2** (0.80 g, 2.0 mmol) and Pd/C (5%) (cat) in MeOH (30 mL) was stirred under H_2 (130 mL/min) atmosphere for 4 h. The resulting mixture was filtered over celite to remove Pd/C, and evaporated under reduced pressure to afford compound **3** (0.58 g, 93%) as a white oil. ^1H NMR (400 MHz, DMSO, 298 K) δ (ppm) : 9.35 (s, 1H), 7.20-7.35 (m, 5H), 7.03 (d, $J = 8.0$ Hz, 2H), 6.72 (d, $J = 8.0$ Hz, 2H), 4.10-4.30 (m, 4H), 1.39 (s, 9H). ^{13}C NMR (DMSO, 100 MHz, 298 K) δ (ppm): 156.54, 155.06, 128.90, 128.43, 128.11, 127.32, 127.02, 79.09, 48.60, 28.04. HR-MS (ESI) (m/z): $[M + Na]^+$ calcd for $C_{19}H_{23}NO_3$, 336.1576, found 336.1563.

Synthesis of compound 5

To a solution of compound **3** (300 mg, 0.96 mmol) in CH_3CN was added compound **4** (338 mg, 0.96 mmol) and potassium carbonate (397 mg, 2.87 mmol). The mixture was refluxed under argon atmosphere for 10 h, and the solvent was removed under vacuum. The residue was extracted with CH_2Cl_2 (3 × 50 mL) and washed with brine (50 mL), water (50 mL), then the organic layer was dried over anhydrous sodium sulfate and concentrated in vacuo to give the crude product, which was purified by column chromatography (SiO_2, PE/EA = 10/1) to afford compound **5** (408 mg, 86%) as a white oil. ^1H NMR (400 MHz, $CDCl_3$, 298 K) δ (ppm): 7.04-7.34 (m, 7H), 6.85 (d, $J = 8.8$ Hz, 2H), 4.26-4.39 (m, 4H), 3.94 (t, $J = 13.2$ Hz, 2H), 3.26 (t, $J = 14.0$ Hz, 2H), 1.74-1.81 (m, 2H), 1.56-1.62 (m, 2H), 1.50 (s, 9H), 1.32 (s, 12H). ^{13}C NMR ($CDCl_3$, 100 MHz, 298 K) δ (ppm): 158.55, 156.13, 129.93, 129.50, 128.95, 128.62, 128.08, 127.54, 127.28, 114.60, 80.11, 68.14, 51.61, 48.75, 29.90, 29.58, 29.54, 29.48, 29.41, 29.26, 28.96, 28.60, 26.84, 26.17. HR-MS (ESI) (m/z): $[M + Na]^+$ calcd for $C_{29}H_{42}N_4O_3$, 517.3155, found 517.3147.

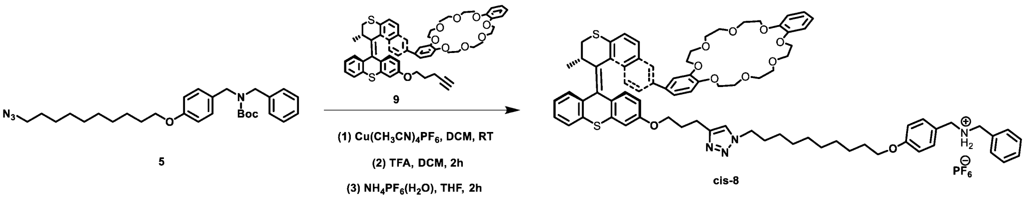
Synthesis of compound 7

To a solution of compound **5** (247 mg, 0.50 mmol) in dry CH_2Cl_2 was added compound **6** (468 mg, 0.50 mmol) and $Cu(CH_3CN)_4PF_6$ (187 mg, 0.50 mmol). The mixture was stirred at room temperature for 12 h. The solution was extracted with CH_2Cl_2 (3 × 50 mL), then the organic layer was dried over anhydrous sodium sulfate and concentrated in vacuo to give the crude product, which was purified by column chromatography (SiO_2, CH_2Cl_2/MeOH = 20/1) to afford compound **7** (630 mg, 88%) as a yellowish solid. ^1H NMR (400 MHz, $CDCl_3$, 298 K) δ (ppm): 7.64 (d, J = 1.2 Hz, 1H), 7.58 (d, J = 8.4 Hz, 1H), 7.51 (d, J = 8.8 Hz, 1H), 7.41 (d, J = 8.4 Hz, 1H), 7.31 (d, J = 8.8 Hz, 1H), 7.24 (s, 1H), 7.22 (s, 1H), 7.16-7.21 (m, 6H), 7.13 (d, J = 1.6 Hz, 1H), 7.10 (d, J = 2.0 Hz, 1H), 7.07 (d, J = 1.6 Hz, 1H), 6.96-7.06 (m, 7H), 6.84 (dd, J = 8.4 Hz, 1.6 Hz, 1H), 6.76 (d, J = 8.8 Hz, 2H), 6.60-6.65 (m, 1H), 6.31-6.36 (m, 2H), 4.20-4.31 (m, 14H), 4.00-4.05 (m, 3H), 3.85 (t, J = 13.2 Hz, 2H), 3.72 (s, 8H), 3.63-3.66 (m, 1H), 3.56 (s, 8H), 3.04 (dd, J = 11.2 Hz, 2.0 Hz, 1H), 2.87 (s, 2H), 2.15 (s, 2H), 1.81 (s, 2H), 1.65-1.72 (m, 2H), 1.41 (s, 9H), 1.21-1.26 (m, 12H), 0.72 (d, J = 6.8 Hz, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, 298 K) δ (ppm): 158.50, 157.63, 156.11, 148.28, 148.08, 147.64, 138.71, 138.27, 138.10, 137.46, 135.64, 135.57, 135.46, 135.18, 134.05, 132.24, 131.73, 131.20, 130.19, 129.86, 129.46, 129.13, 128.90, 128.60, 128.50, 128.05, 127.83, 127.51, 127.26, 126.55, 126.14, 125.58, 125.21, 125.18, 124.83, 124.57, 124.48, 124.08, 123.60, 123.46, 122.08, 116.74, 116.58, 116.36, 115.43, 115.39, 114.56, 113.25, 113.19, 80.09, 69.22, 69.01, 68.69, 68.50, 68.09, 67.43, 67.26, 49.09, 48.69, 48.38, 37.34, 32.03, 31.88, 31.60, 31.54, 30.36, 30.28, 30.24, 29.87, 29.80, 29.76, 29.53, 29.46, 29.43, 29.37, 29.08, 28.87, 28.57, 26.62, 26.27, 26.13, 22.80, 19.17, 14.24. HR-MS (ESI) (m/z): $[M + Na]^+$ calcd for $C_{85}H_{98}N_4O_{12}S_2$, 1453.6520, found 1453.6511.

Synthesis of compound *trans*-8

To a solution of compound **7** (286 mg, 0.20 mmol) in dry CH_2Cl_2 was added TFA (228 mg, 2.00 mmol). The mixture was stirred at room temperature under argon atmosphere for 3 h, and the solvent was removed under reduced pressure. Then to a solution of the residue in THF was added saturated aqueous solution of NH_4PF_6 (10 mL). The solvent was removed under reduced pressure, and the residue was extracted with CH_2Cl_2 (3 × 25 mL) and washed with deionized water. The organic layer was dried over anhydrous sodium sulfate and concentrated in vacuo to afford compound *trans*-**8** (263 mg, 89%) as a yellowish solid without further purification. ^{1}H NMR (400 MHz, DMSO, 298 K) δ (ppm) : 9.07 (s, 2H), 7.92 (d, J = 4.8 Hz, 2H), 7.75 (d, J = 8.8 Hz, 1H), 7.61 (d, J = 8.4 Hz, 1H), 7.35-7.51 (m, 10H), 7.31 (dd, J = 9.6 Hz, 0.8 Hz, 1H), 7.26 (d, J = 2.4 Hz, 1H), 7.23 (d, J = 1.2 Hz, 1H), 7.17 (dd, J = 8.4 Hz, 1.6 Hz, 1H), 6.93-7.02 (m, 6H), 6.86-6.89 (m, 2H), 6.77-6.81 (m, 1H), 6.50 (t, J = 14.8 Hz, 1H), 6.36 (t, J = 7.6 Hz, 1H), 4.29 (t, J = 14.0 Hz, 2H), 4.17-4.20 (m, 2H), 4.03-4.13 (m, 12H), 3.98-4.02 (m, 1H), 3.92-3.97 (m, 2H), 3.83-3.88 (m, 1H), 3.75-3.79 (m, 8H), 3.68 (d, J = 2.4 Hz, 8H), 3.15 (d, J = 12.0 Hz, 1H), 2.80 (t, J = 14.8 Hz, 2H), 2.04-2.11 (m, 2H), 1.74-1.81 (m, 2H), 1.65-1.71 (m, 2H), 1.23 (s, 12H), 0.65 (d, J = 6.8 Hz, 3H). ^{13}C NMR (DMSO, 100 MHz, 298 K) δ (ppm): 159.22, 157.27, 148.75, 148.47, 148.14, 146.01, 138.35, 136.48, 135.38, 135.29, 134.34, 133.59, 132.52, 131.88, 131.62, 131.39, 131.25, 130.29, 129.95, 129.21, 129.03, 128.73, 128.31, 127.96, 127.62, 126.63, 126.27, 125.71, 125.59, 124.50, 124.47, 124.11, 123.34, 121.91, 121.67, 121.15, 119.25, 114.51, 114.17, 114.02, 113.47, 112.84, 112.32, 70.42, 69.18, 68.91, 68.83, 68.69, 67.52, 67.06, 54.94, 49.81, 49.67, 49.18, 36.17, 31.32, 30.99, 29.70, 29.22, 29.05, 28.88, 28.80, 28.73, 28.61, 28.47, 28.34, 28.13, 25.84, 25.49, 22.12, 21.59, 18.59, 13.98. HR-MS (ESI) (m/z): $[M-PF_6]^+$ calcd for $C_{80}H_{91}F_6N_4O_{10}PS_2$ 1331.6177, found 1331.6166.

1.3 Synthesis of the reference compound *cis*-8



Scheme S2. Synthetic route of *cis*-**8**

To a solution of compound **5** (200 mg, 0.40 mmol) in dry CH_2Cl_2 was added compound **9** (375 mg, 0.40 mmol) and $Cu(CH_3CN)_4PF_6$ (150 mg, 0.40 mmol). The mixture was stirred at room temperature for 12 h. The solution was extracted with CH_2Cl_2 (3 × 50 mL), then the organic layer was dried over anhydrous sodium sulfate and concentrated in vacuo to give the crude product. The residue was dissolved in dry CH_2Cl_2 and TFA (456 mg, 4.00 mmol) was added. The mixture was stirred at room temperature under argon atmosphere for 3 h, and the solvent was removed under reduced pressure. Then to a solution of the residue in THF was added saturated aqueous solution of NH_4PF_6 (10 mL). The solvent was removed under reduced pressure, and the residue was extracted with CH_2Cl_2 (3 × 25 mL) and washed with deionized water. The organic layer was dried over anhydrous sodium sulfate and concentrated in vacuo to give crude product which was purified by column chromatography (SiO_2, CH_2Cl_2/MeOH = 20/1) to afford compound *cis*-**8** (484 mg, 82%) as a yellowish solid. ^{1}H NMR (400 MHz, DMSO, 298K) δ (ppm) : 9.00 (s, 2H), 7.94 (s, 1H), 7.75 (d, J = 8.8 Hz, 1H), 7.69 (t, J = 7.2 Hz, 2H), 7.53 (d, J = 8.8 Hz, 1H), 7.32-7.45 (m, 11H), 7.24 (s, 1H), 7.17 (d, J = 7.6 Hz, 1H), 6.92-7.00 (m, 6H), 6.85-6.88 (m, 2H), 7.53 (d, J = 8.8 Hz, 1H), 6.24 (d, J = 8.4 Hz, 1H), 6.07 (dd, J = 8.4 Hz, 2.4Hz, 1H), 4.17-4.23 (m, 4H), 4.02-4.09 (m, 12H), 3.91-3.96 (m, 2H), 3.84-3.89 (m, 2H), 3.76 (d, J = 3.6 Hz, 8H), 3.67 (s, 8H), 3.14 (d, J = 11.2 Hz, 1H), 1.63-1.80 (m, 6H), 1.42-1.52 (m, 2H), 1.09-1.20 (m, 12H), 0.65 (d, J = 6.4 Hz, 3H). ^{13}C NMR (DMSO, 100 MHz, 298 K) δ (ppm): 159.01, 156.65, 148.74, 148.46, 148.12, 135.65, 135.42, 135.29, 135.19, 134.74, 134.34, 132.68, 132.52, 131.38, 131.29, 131.18, 130.34, 130.30, 130.11, 129.68, 129.38, 129.05, 128.72, 128.64, 127.91, 127.65, 127.57, 127.53, 127.03, 126.72, 125.74, 124.58, 124.18, 121.13, 119.31, 114.45, 114.21, 114.05, 113.10, 112.41, 111.86, 70.85, 70.40, 70.38, 69.86, 69.15, 68.90, 68.83, 68.69, 67.48, 66.84, 49.09, 36.17, 31.27, 30.97, 29.62, 29.20, 29.18, 29.07, 29.02, 28.96, 28.94, 28.83, 28.75, 28.69, 28.62, 28.59, 28.30, 28.09, 26.54, 25.78, 25.69, 25.45, 25.10, 24.65, 22.08, 21.33, 21.05, 18.63, 13.94. HR-MS (ESI) (m/z): $[M-PF_6]^+$ calcd for $C_{80}H_{91}F_6N_4O_{10}PS_2$ 1331.6177, found 1331.6184.

2. Structural characterization of *trans*-8

2.1 ^{1}H-^{1}H COSY spectrum of trans-8 before adding DBU

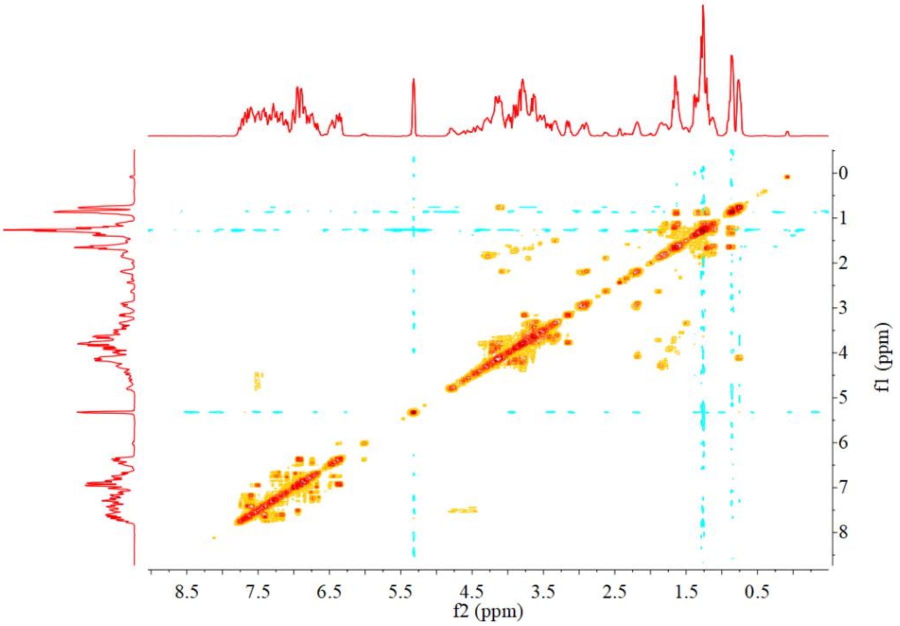


Figure S1 ^{1}H-^{1}H COSY spectrum of *trans*-**8** (600 MHz, CD_2Cl_2, 298 K)

2.2 ^{1}H-^{1}H COSY spectrum of *trans*-8 after adding DBU

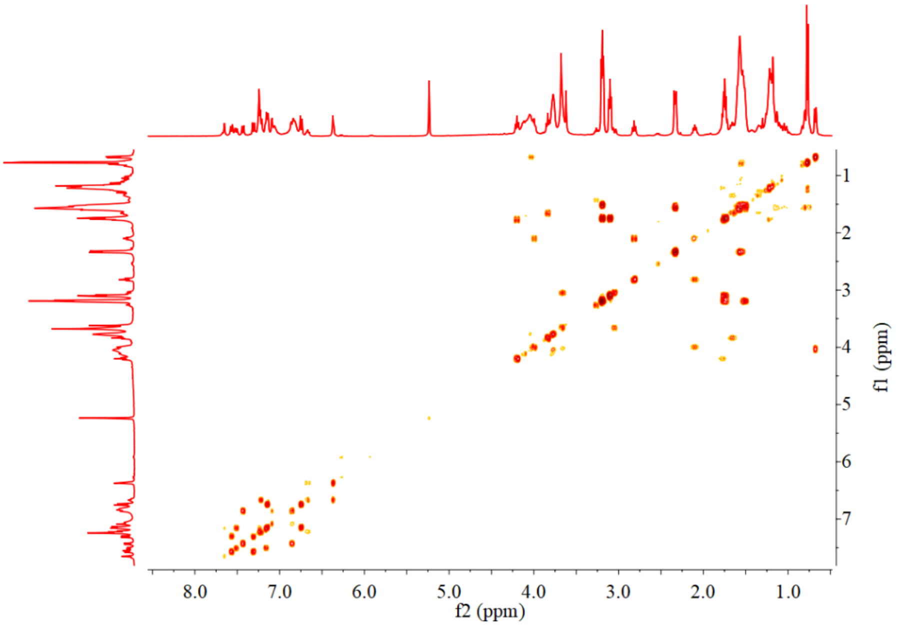


Figure S2. ^{1}H-^{1}H COSY spectrum of *trans*-**8** after adding one equiv DBU (400 MHz, CD_2Cl_2, 298 K)

3. Photo- and thermal-isomerization behaviors of *trans*-8 in dichloromethane-d_2

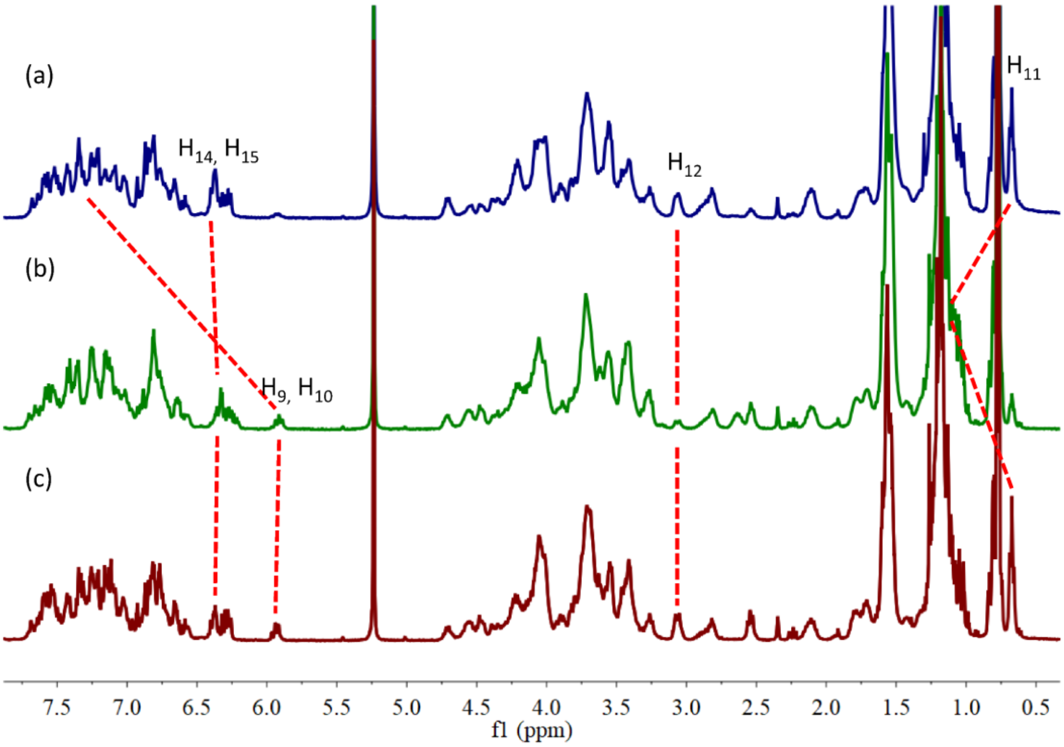


Figure S3. ^{1}H NMR spectrum (600 MHz, 20 mM, CD_2Cl_2, 298 K). (a) *trans*-**8** before UV irradiation; (b) *trans*-**8** after UV irradiation (365 nm) for 2 h; (c) *trans*-**8** after UV irradiation (365 nm) for 2 h and then heated 60^{0}C for 24 h

4. UV-vis absorption spectra and DLS data of *trans*-8

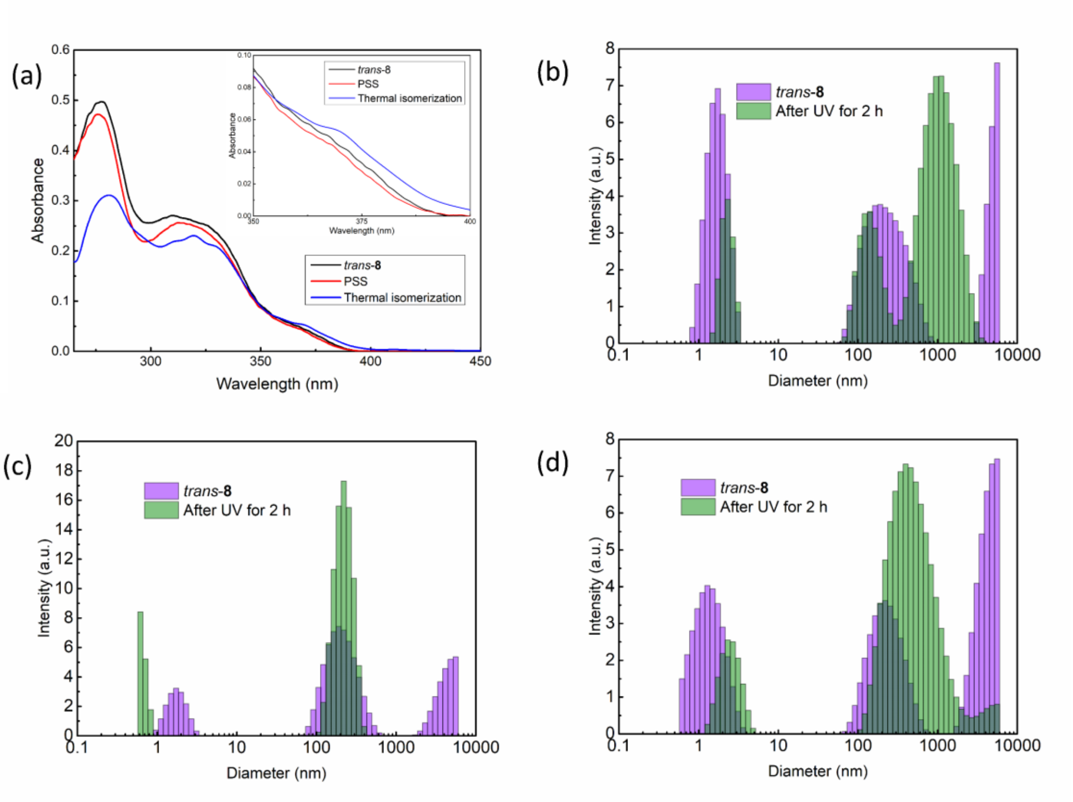


Figure S4. (a) UV-vis absorption spectra (DMSO, 298 K) of *trans*-**8** (black line), after UV irradiation (photoisomerization, red line) and heating (thermal isomerization, blue line). DLS data of *trans*-**8** and after UV for 2 h in different solvents: (b) THF (1.00 mM); (c) acetone (0.01 mM); (d) DCM (0.01 mM)

5. SEM images of *trans*-8 before and after UV irradiation in tetrahydrofuran

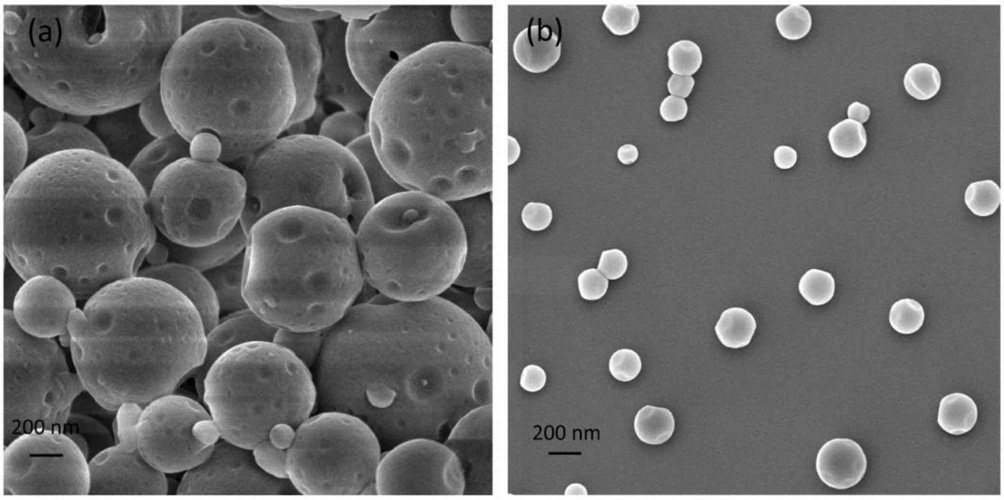


Figure S5. SEM images of *trans*-**8** aggregates in THF (c = 1 × 10^{-3} M): (a) before UV (365 nm) irradiation; (b) after UV (365 nm) irradiation for 2 h.

6. TEM images of *trans*-8 before and after UV irradiation in acetone

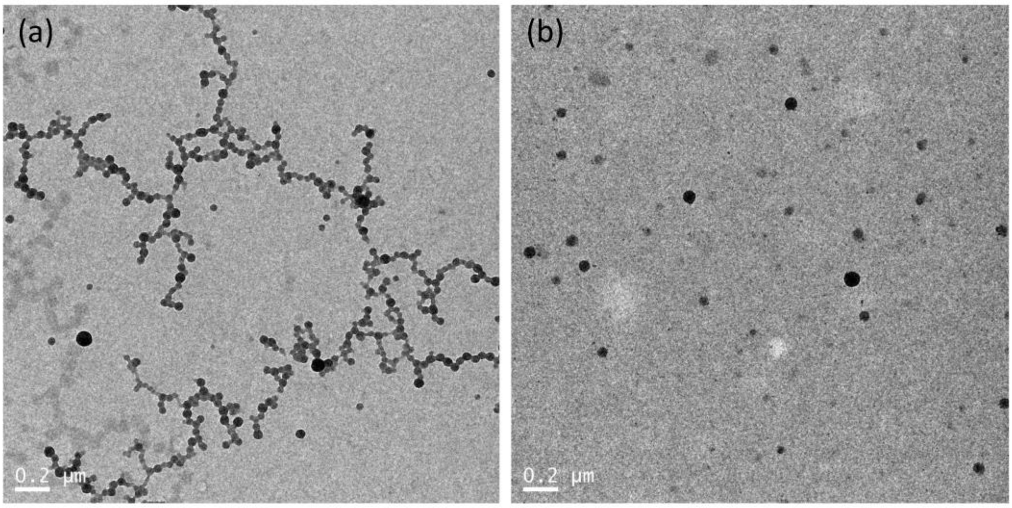


Figure S6. TEM images of *trans*-**8** aggregates in acetone (c = 1 × 10^{-5} M) : (a) before UV (365 nm) irradiation (b) after UV (365 nm) irradiation for 2 h.

7. Solubility of *trans*-8 in different solvents

Table S1. Solubility of *trans*-**8** in different solvents

Solvent	Solubility
THF	15 mg mL^{-1}
CH_3OCH_3	228 mg mL^{-1}
DCM	> 510 mg mL^{-1}

8. TEM and SEM images of *cis*-8 before and after UV irradiation in tetrahydrofuran

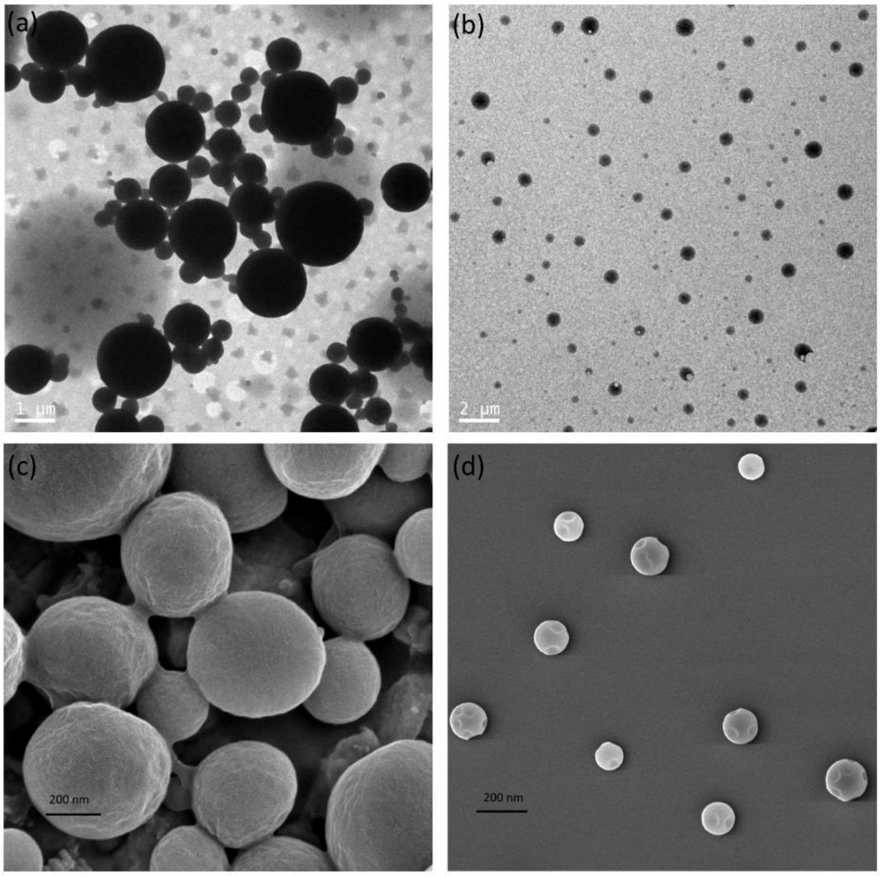


Figure S7. TEM images of *cis*-**8** aggregates (c = 1 × 10^{-3} M): (a) before UV (365nm) irradiation; (b) after UV (365 nm) irradiation for 2 h. SEM images of *cis*-**8** aggregates (c = 1 × 10^{-3} M): (c) before UV (365 nm) irradiation; (d) after UV (365 nm) irradiation for 2 h.

9. pH-responsive behavior of *trans*-8

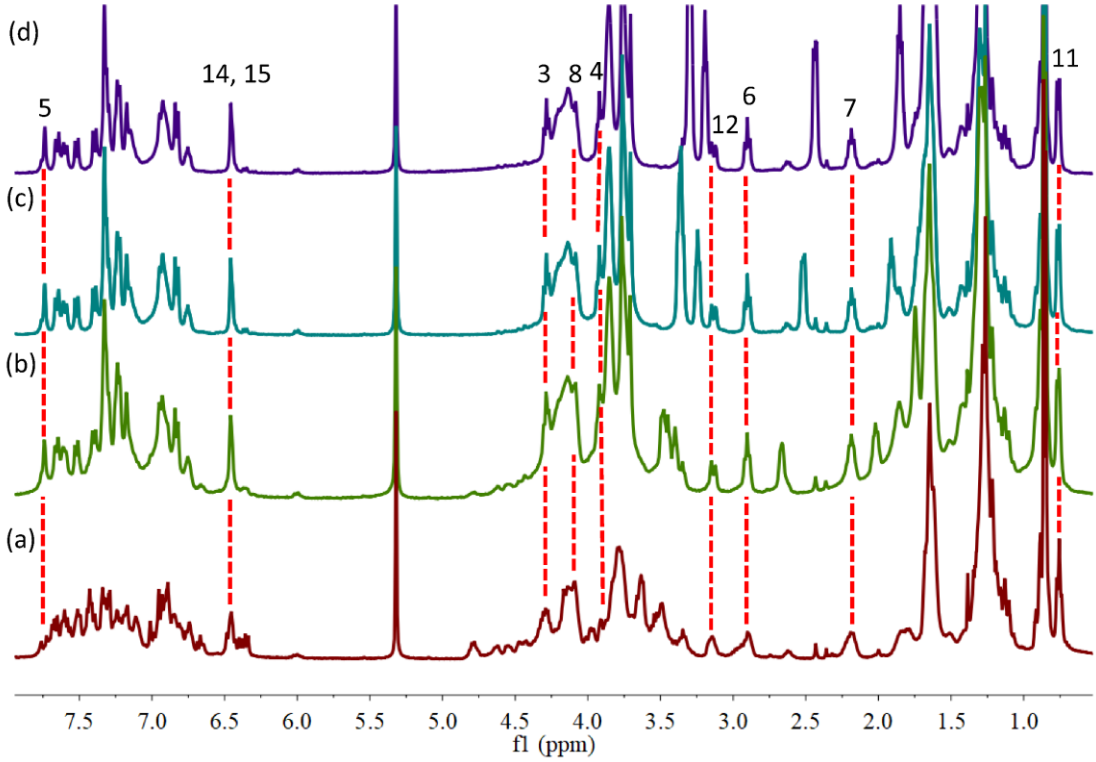


Figure S8. ^{1}H NMR spectrum (600 MHz, CD_2Cl_2, 298 K) of 20 mM *trans*-**8** upon adding DBU: a) 0 equiv; b) 0.5 equiv; c) 1.0 equiv; d) 1.5 equiv.

10. References

(1) N. Erdeljac, G. Kehr, M. Ahlqvist, L. Knerr, R. Gilmour, *Chem. Commun.*, 2018, **54**, 12002-12005.

(2) H. Li, Z. X. Zhu, A. C. Fahrenbach, B. M. Savoie, C. F. Ke, J. C. Barnes, J. Y. Lei, Y. L. Zhao, L. M. Lilley, T. J. Marks, M. A. Ratner, J. F. Stoddart, *J. Am. Chem. Soc.*, 2013, **135**, 456-467.

(3) J. J. Yu, L. Y. Zhao, Z. T. Shi, Q. Zhang, G. London, W. J. Liang, C. Gao, M. M. Li, X. M. Cao, H. Tian, B. L. Feringa, D. H. Qu, *J. Org. Chem.*, 2019, **84**, 5790-5802.

11. Appendix

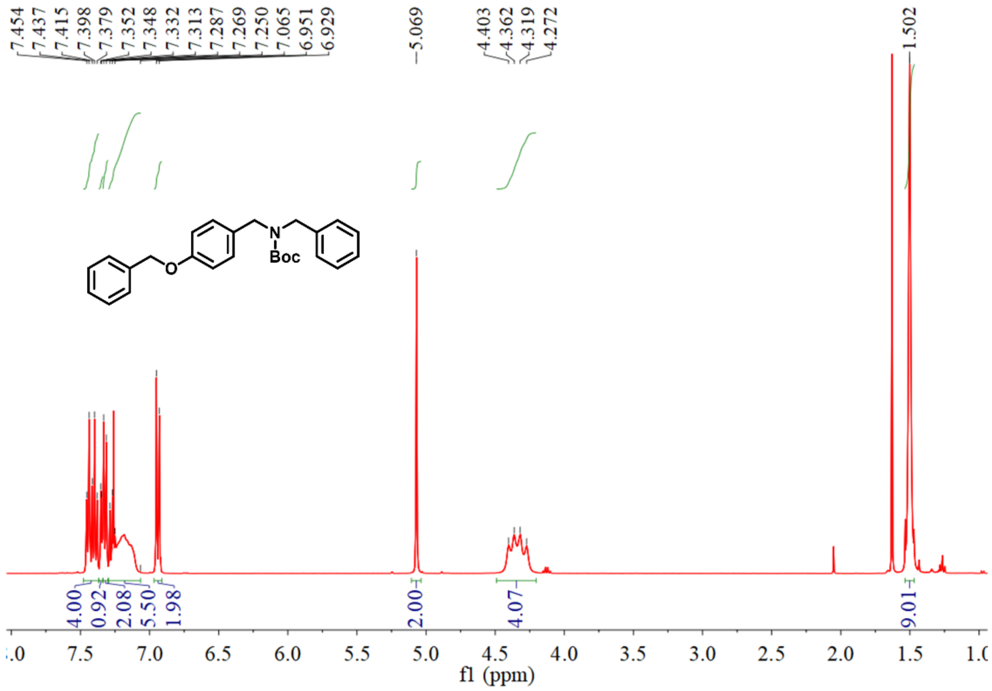


Figure S9. ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 298 K) of compound **2**

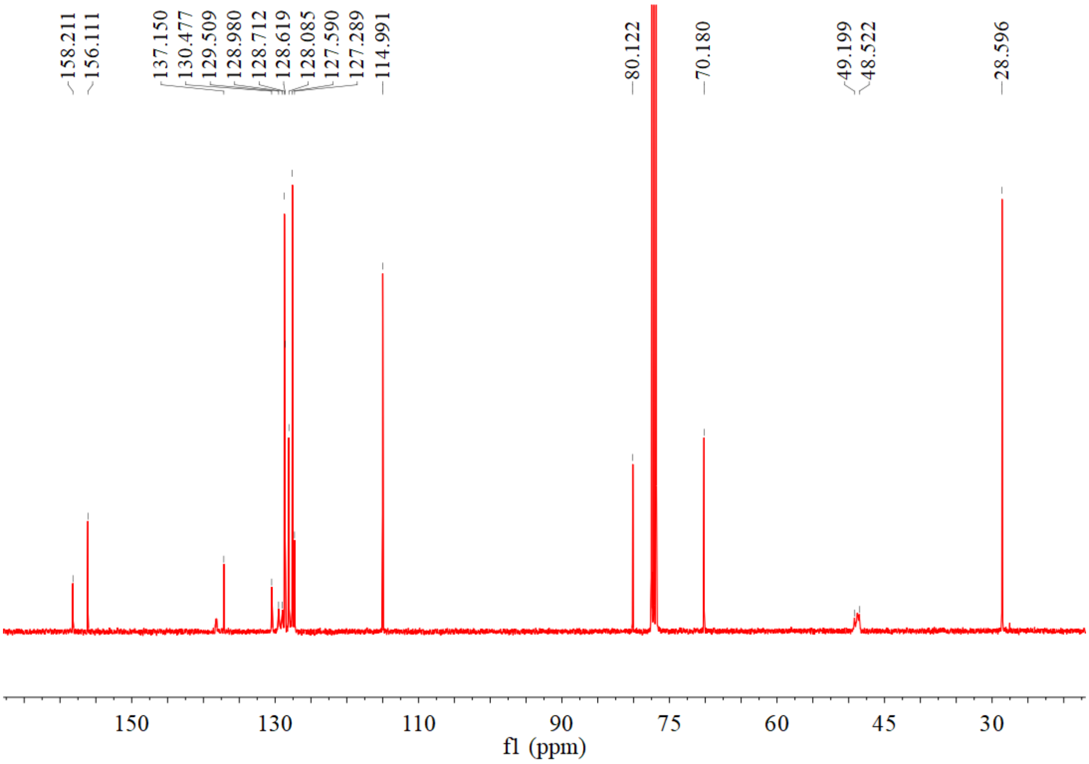


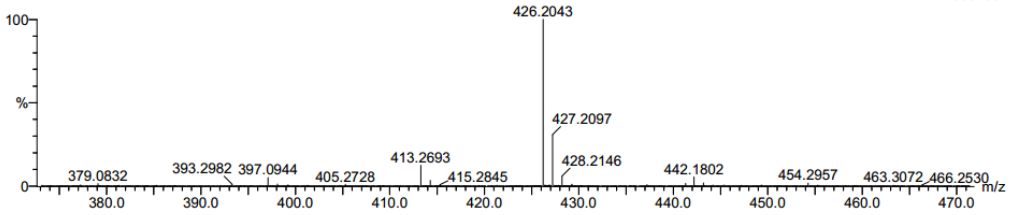
Figure S10. ^{13}C NMR spectrum (100 MHz, $CDCl_3$, 298 K) of compound **2**

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
209 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 25-29 H: 0-99 N: 0-4 O: 1-11 Na: 0-1

QH-QU
QD-ZT-038 38 (0.425) Cm (30:38) 1: TOF MS ES+ 2.65e+004



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Minimum:                                  -1.5
Maximum:                 5.0      5.0     50.0

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

426.2043    426.2045     -0.2     -0.5    12.5     231.2      0.0          C26  H29  N  O3  Na
```

Figure S11. ESI-MS spectrum of compound **2**

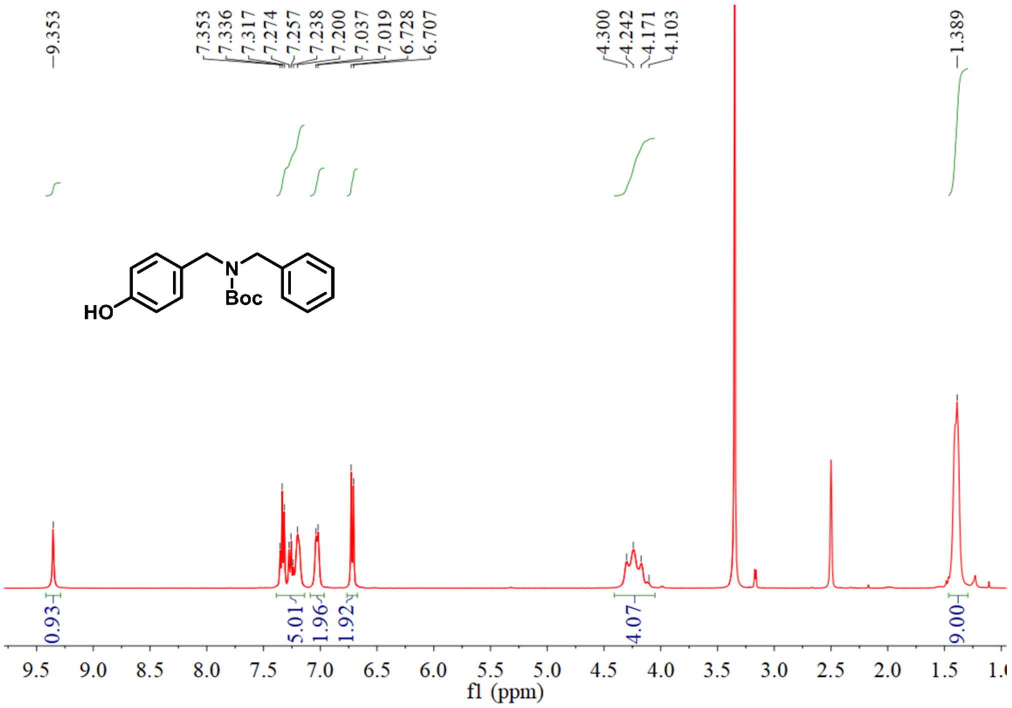


Figure S12. ^{1}H NMR spectrum (400 MHz, DMSO, 298 K) of compound **3**

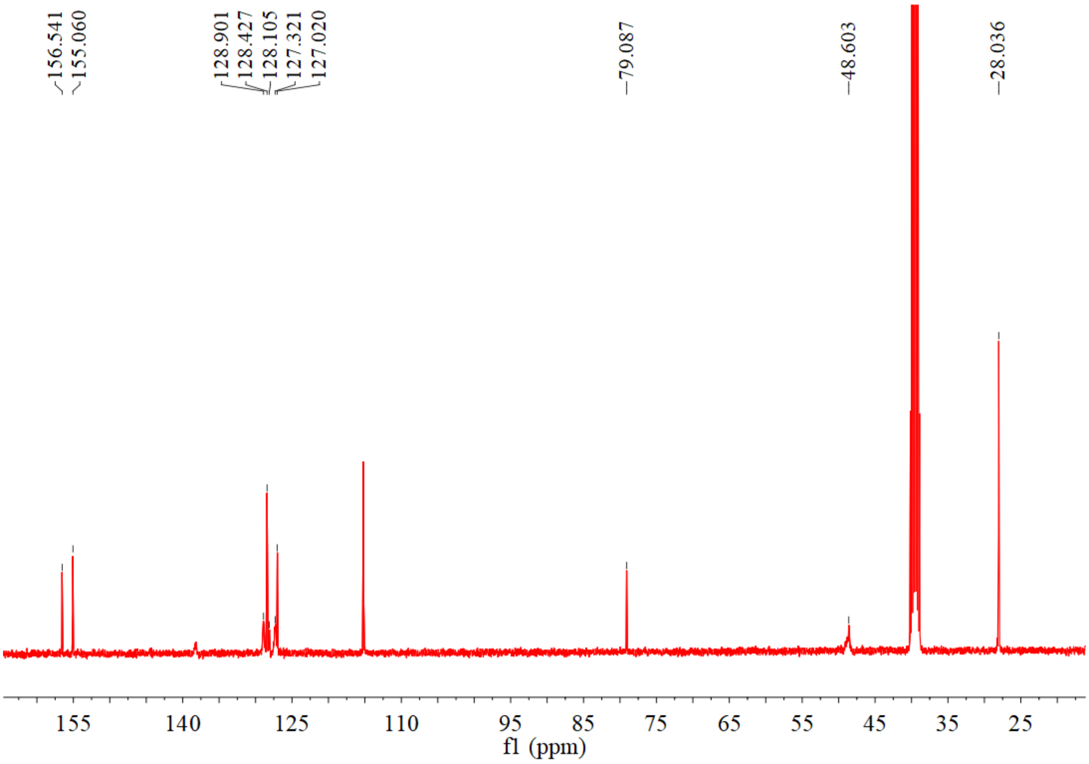
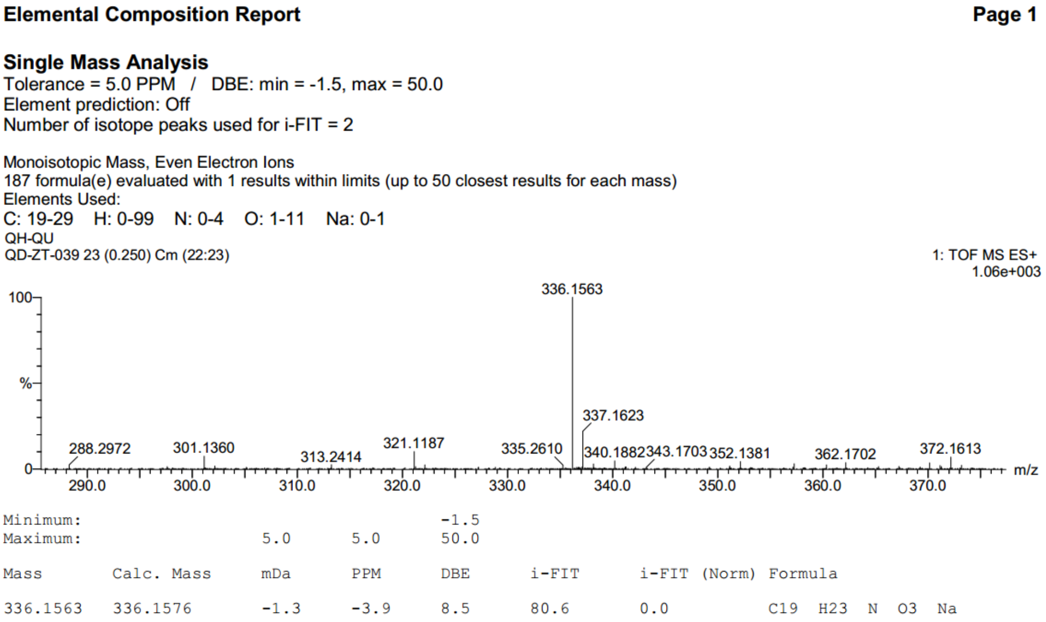


Figure S13. ^{13}C NMR spectrum (100 MHz, DMSO, 298 K) of compound **3**



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
187 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 19-29 H: 0-99 N: 0-4 O: 1-11 Na: 0-1
QH-QU
QD-ZT-039 23 (0.250) Cm (22:23)

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

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

336.1563   336.1576       -1.3    -3.9    8.5     80.6     0.0          C19  H23  N  O3  Na
```

Figure S14. ESI-MS spectrum of compound **3**

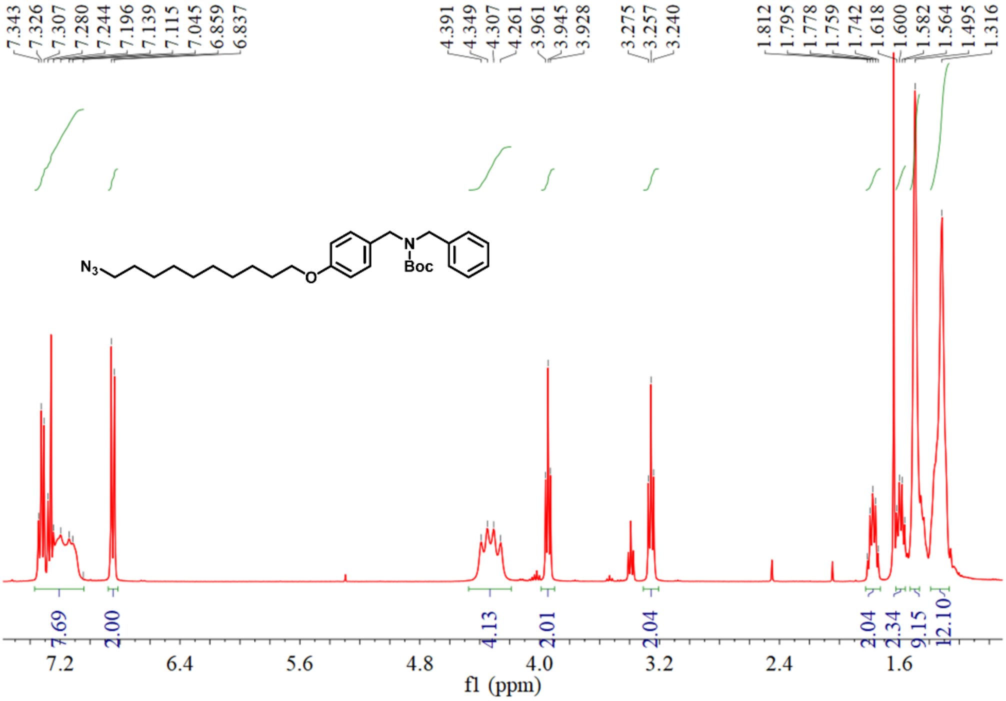


Figure S15. ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 298 K) of compound **5**

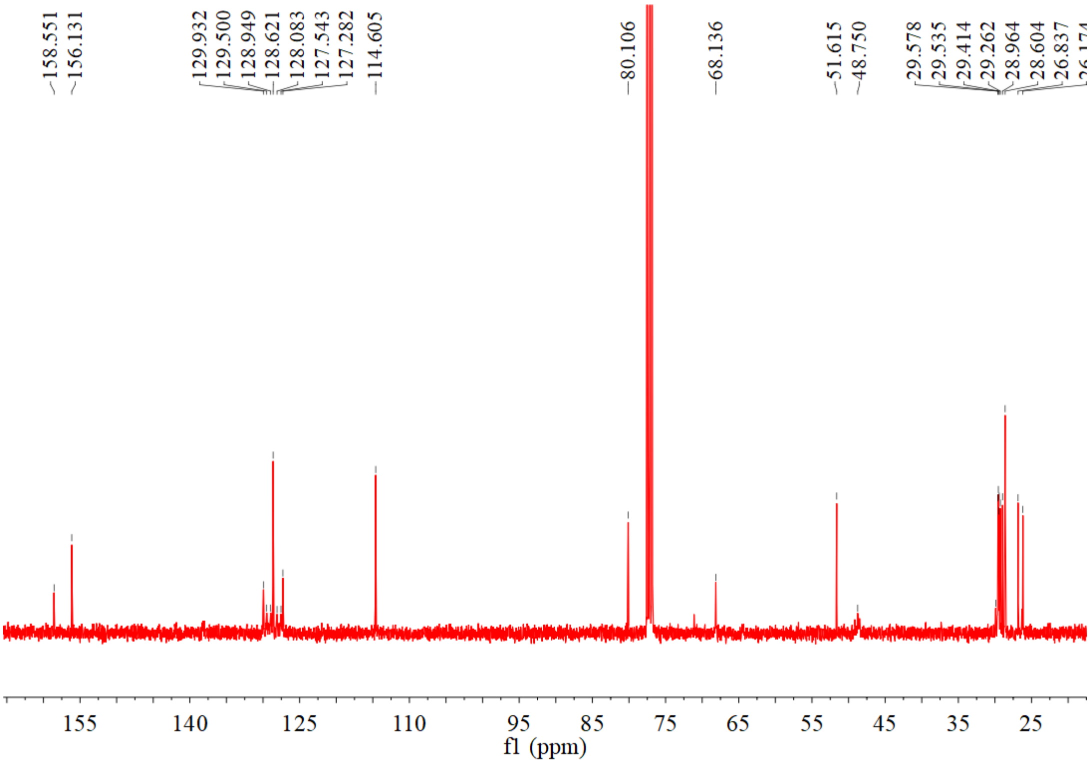
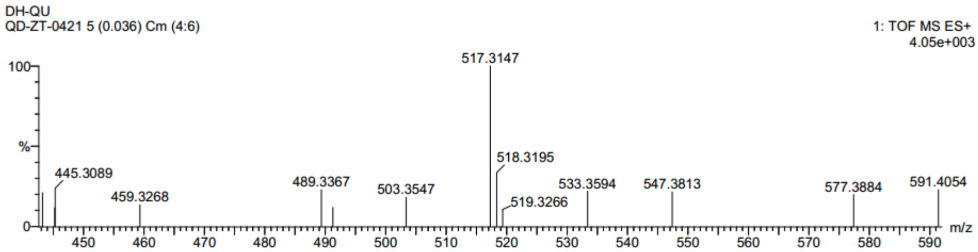


Figure S16. ^{13}C NMR spectrum (100 MHz, $CDCl_3$, 298 K) of compound **5**

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: 0-29 H: 0-42 N: 0-4 O: 0-3 Na: 0-1



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

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

517.3147   517.3155      -0.8      -1.5    10.5      8.8        0.0          C29  H42  N4  O3  Na
```

Figure S17. ESI-MS spectrum of compound **5**

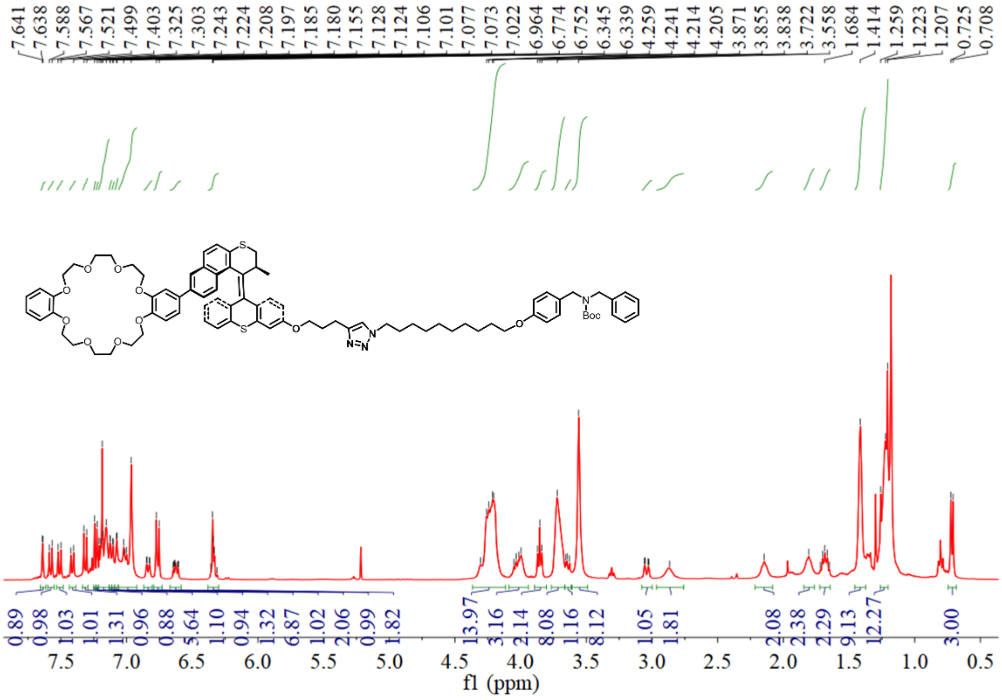


Figure S18. ^{1}H NMR spectrum (400 MHz, $CDCl_3$, 298 K) of compound **7**

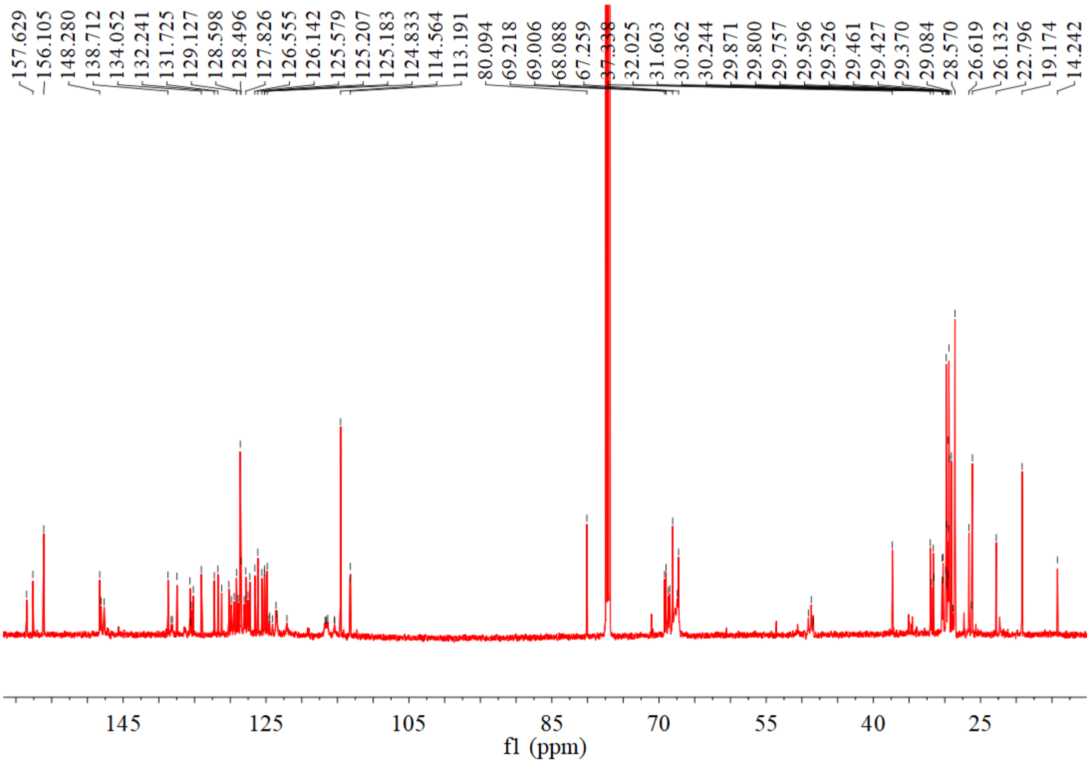
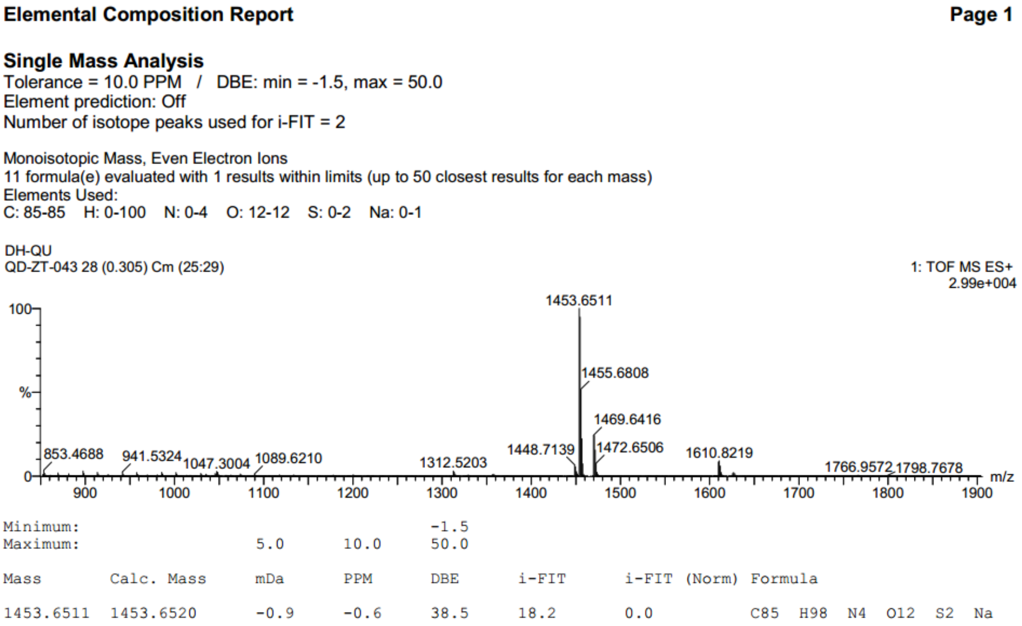


Figure S19. ^{13}C NMR spectrum (100 MHz, $CDCl_3$, 298 K) of compound **7**



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
11 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 85-85 H: 0-100 N: 0-4 O: 12-12 S: 0-2 Na: 0-1

DH-QU
QD-ZT-043 28 (0.305) Cm (25:29)

1: TOF MS ES+
2.99e+004

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

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

1453.6511  1453.6520       -0.9     -0.6     38.5     18.2       0.0          C85  H98  N4  O12  S2  Na
```

Figure S20. ESI-MS spectrum of compound **7**

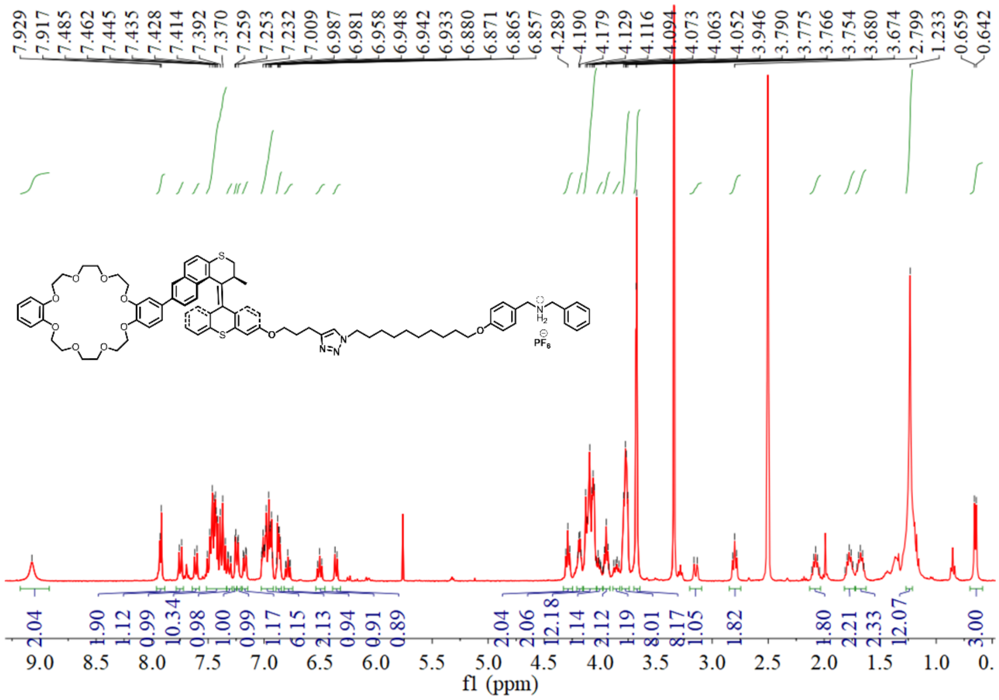


Figure S21. ^{1}H NMR spectrum (400 MHz, DMSO, 298 K) of *trans*-**8**

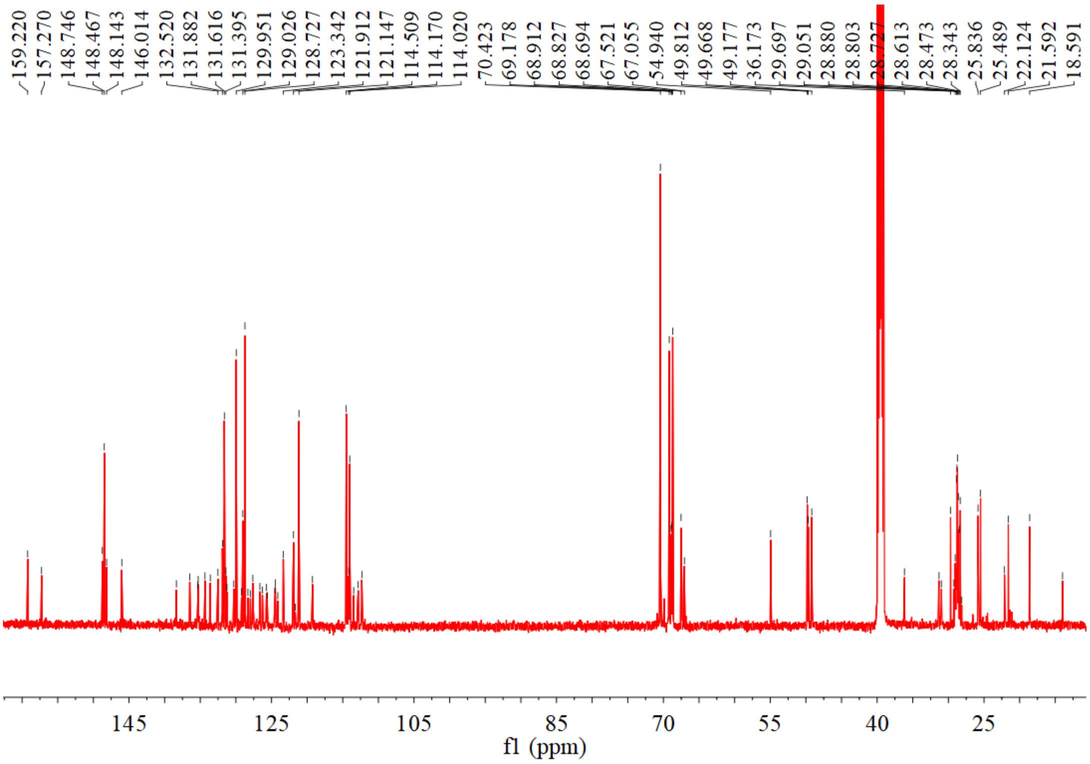
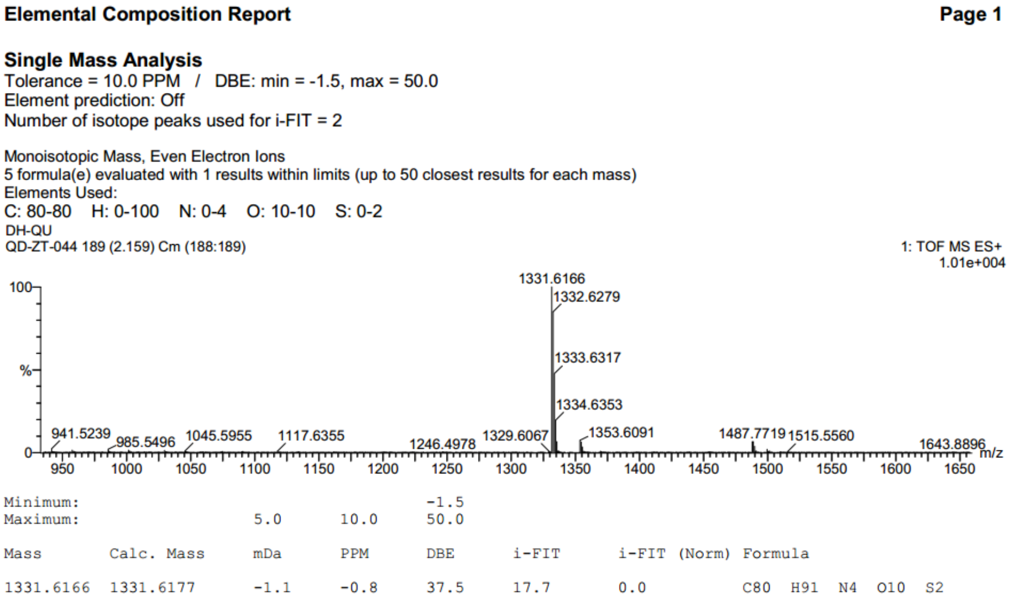


Figure S22. ^{13}C NMR spectrum (100 MHz, DMSO, 298 K) of *trans*-**8**



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
5 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 80-80 H: 0-100 N: 0-4 O: 10-10 S: 0-2
DH-QU
QD-ZT-044 189 (2.159) Cm (188:189)

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

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

1331.6166   1331.6177      -1.1      -0.8   37.5     17.7       0.0          C80   H91   N4   O10   S2
```

Figure S23. ESI-MS spectrum of *trans*-**8**

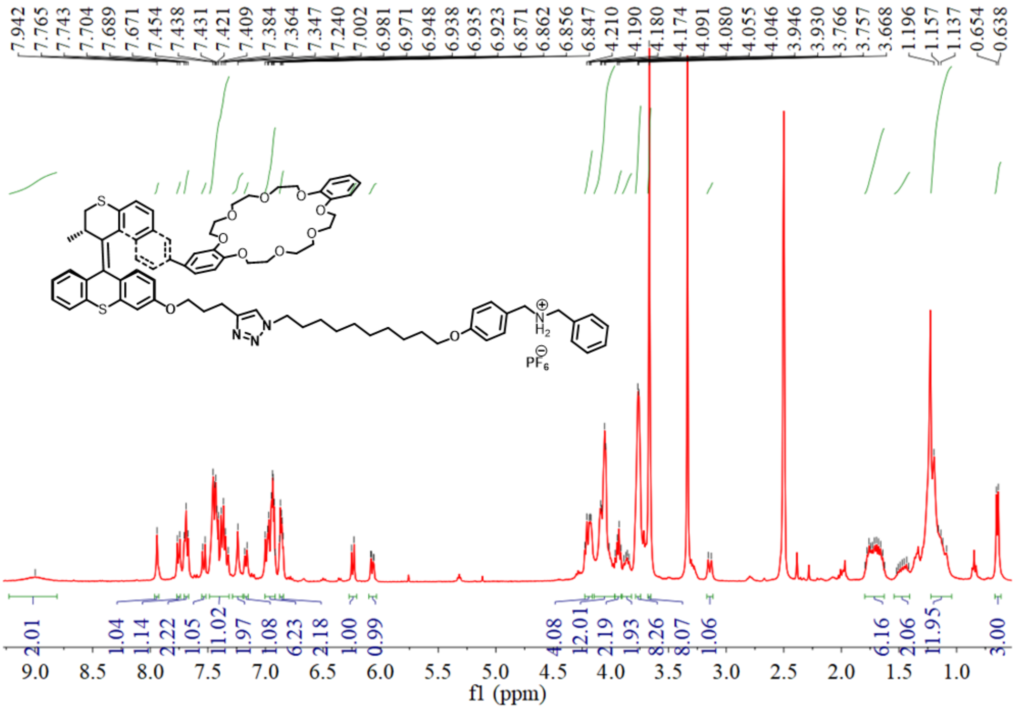


Figure S24. ^{1}H NMR spectrum (400 MHz, DMSO, 298 K) of *cis*-**8**

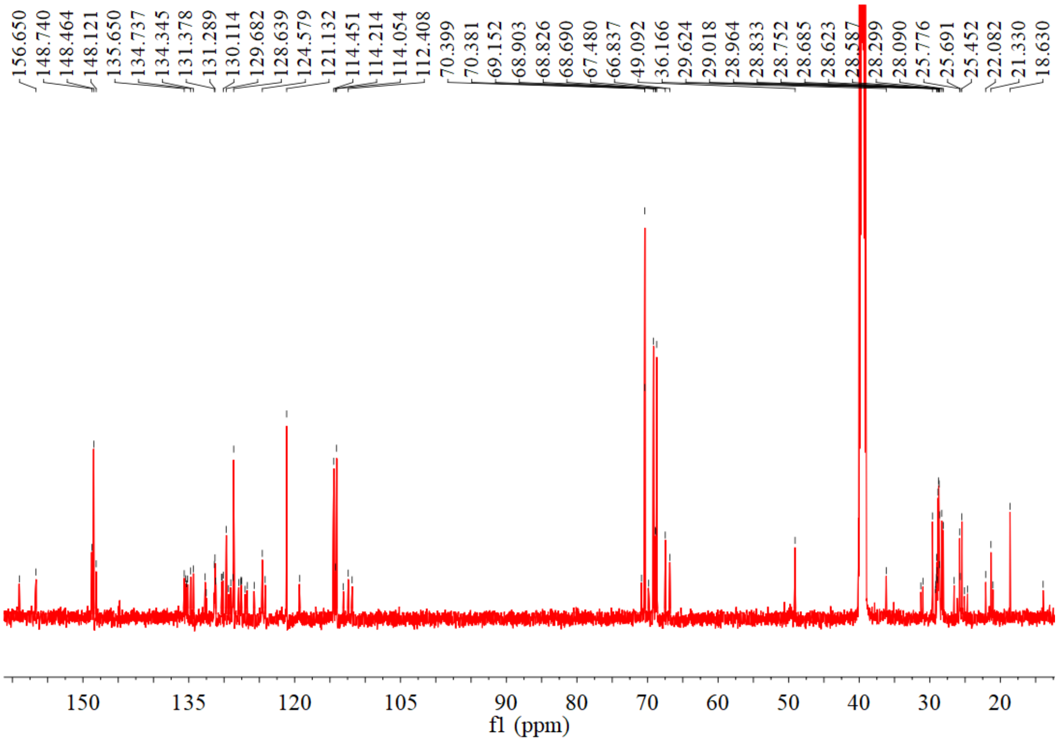
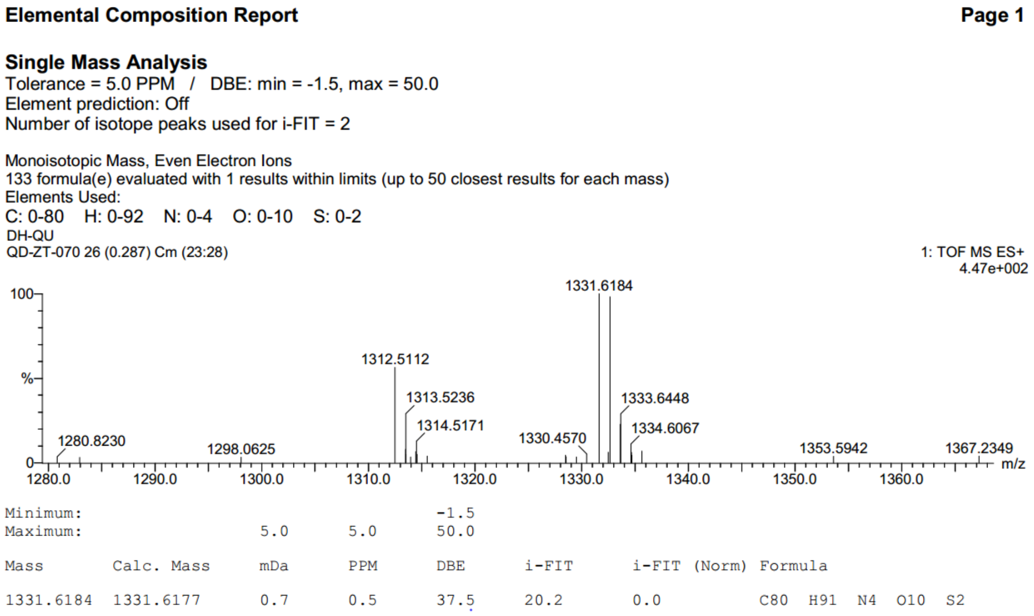


Figure S25. ^{13}C NMR spectrum (100 MHz, DMSO, 298 K) of *cis*-**8**



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
133 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-80 H: 0-92 N: 0-4 O: 0-10 S: 0-2
DH-QU
QD-ZT-070 26 (0.287) Cm (23:28)

1: TOF MS ES+
4.47e+002

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

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

1331.6184  1331.6177       0.7      0.5     37.5     20.2       0.0          C80  H91  N4  O10  S2
```

Figure S26. ESI-MS spectrum of *cis*-**8**