

General methods. All reactions were carried out under a dry nitrogen atmosphere. All solvents were dried before use following standard procedures. Unless otherwise indicated, all starting materials were obtained from commercial suppliers and were used without further purification. Analytical thin-layer chromatography (TLC) was performed on 0.2 mm silica coated on glass plates with FB254B indicator. The ^1H NMR spectra were recorded on 500, or 300 MHz spectrometers in the indicated solvents. Chemical shifts are expressed in parts per million (δ) using residual solvent protons as internal standard (chloroform: δ 7.26 ppm; DMSO: δ 2.50 ppm). The ^{13}C NMR spectra were recorded on 300 MHz spectrometer in the indicated solvents. Chemical shifts are expressed in parts per million using CDCl_3 as internal standard (chloroform: δ 77.0 ppm).

Compound 1a. To a stirred solution of compound **5** (0.16 g, 1.00 mmol) and triethylamine (0.3 mL, 3.00 mmol) in 1,4-dioxane (10 mL) was added di-*tert*-butyl dicarbonate (0.44 g, 2.00 mol). The mixture was stirred for 30 h and then concentrated with a rotavapor. The resulting residue was triturated with chloroform (20 mL) and the organic phase washed successively with saturated sodium bicarbonate solution (10 mL \times 2), water (10 mL) and brine (10 mL), and dried over sodium sulfate. Upon removal of the solvent, the resulting crude product was recrystallized from ethyl acetate to give **1a** as a white solid (0.27 g, 75%). ^1H NMR (400 MHz, CDCl_3): δ 7.78 (s, 1H), 7.66 (d, J = 8.8 Hz, 1H), 7.32 (dd, J_1 = 8.8 Hz, J_2 = 2.0 Hz, 1H), 6.62 (s, 1H), 1.55 (s, 9H). ^{13}C NMR (125 MHz, CDCl_3): δ 152.7, 136.4, 134.6, 128.4, 126.6, 117.8, 113.9, 80.6, 28.4. MS (ESI): m/z 376.2 $[\text{M}+\text{NH}_4]^+$. HRMS (MALDI-FT): calcd for $\text{C}_{20}\text{H}_{26}\text{N}_2\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 381.1785. Found: 381.1779.

Compound 1f. To a stirred solution of compounds **6**¹ (0.15 g, 0.19 mmol) and **7**¹ (0.58 g, 0.42 mmol) in DMF (10 mL) and DIEA (0.22 mL, 1.26 mmol) was added HATU (0.19 g, 0.50 mmol). The mixture was stirred for 11 h and then concentrated with a rotavapor. The resulting residue was triturated in chloroform (10 mL) and the organic phase washed with water (5 mL \times 2) and brine (5 mL), and dried over sodium sulfate. Upon removal of the solvent in vacuo, the crude product was subjected to flash chromatography ($\text{CH}_2\text{Cl}_2/\text{EtOAc}/\text{MeOH}$ 4:6:1) to give **1f** as pale yellow solid (0.52 g, 76%). ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 10.75-10.69 (m, 10H), 9.57 (s, 2H), 8.71-8.69 (m, 5H), 8.44 (s, 8H), 8.33 (s, 2H), 8.21 (s, 10H), 8.00 (s, 2H), 7.93-7.75 (m, 22H), 7.50-7.47 (m, 2H), 3.71 (s, 20H), 3.57-3.34 (m, 100H), 3.21 (s, 15H), 3.13 (s, 15H), 1.51 (s, 18H). ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$): δ 171.4, 165.1, 153.0, 136.9, 136.6, 135.2, 134.1, 133.8, 129.4, 128.1, 127.2, 120.3, 119.3, 118.6, 114.5, 80.2, 71.7, 71.6, 70.3, 70.1, 69.9, 69.0, 68.1, 58.8, 58.6, 49.8, 45.0, 29.6, 28.3. MS (MALDI-TOF): m/z 3297.5 $[\text{M}-2(\text{CH}_3)_2\text{C}=\text{CH}_2-2\text{CO}_2+\text{Na}]^+$, 3313.4 $[\text{M}-2(\text{CH}_3)_2\text{C}=\text{CH}_2-2\text{CO}_2+\text{K}]^+$. HRMS: calcd for $\text{C}_{175}\text{H}_{215}\text{N}_{17}\text{O}_{45}\text{Na}$ $[\text{M}-2(\text{CH}_3)_2\text{C}=\text{CH}_2-2\text{CO}_2+\text{Na}]^+$: 3297.4950. Found: 3297.4946.

Compound 1g. To a solution of compounds **7** (0.50 g, 0.36 mmol) and **8**² (0.24 g, 0.16 mmol) in DMF (10 mL) and DIEA (0.2 mL, 1.1 mmol) was added HATU (0.18 g, 0.48 mmol). Stirring was continued for 11 h and then solution was concentrated with a rotavapor. After workup, the resulting residue was subjected to flash chromatography ($\text{CH}_2\text{Cl}_2/\text{EtOAc}/\text{MeOH}$ 4:6:1) to give **1g** as a pale yellow solid (0.52 g, 78%). ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 10.75-10.68 (m, 10H), 10.14 (s, 2H), 9.56 (s, 2H), 8.71-8.69 (m, 6H), 8.44 (s, 9H), 8.35-8.32 (m, 3H), 8.21-8.19 (m, 12H), 8.00 (s, 2H), 7.92-7.71 (m, 25H), 7.54-7.47 (m, 3H), 3.71 (s, 24H), 3.58-3.33 (m, 120H), 3.21 (s, 18H), 3.13 (s, 18H), 1.51 (s, 18H). ^{13}C NMR (125 MHz, $\text{DMSO}-d_6$): δ 171.6, 165.1, 153.0, 136.8, 135.2, 134.2, 133.8, 128.0, 127.2, 120.5, 119.3, 118.6, 116.8, 80.2, 71.7, 71.6, 70.3, 70.1, 69.9,

69.0, 68.1, 58.8, 58.7, 49.8, 44.9, 29.6, 28.3. MS (MALDI-TOF): m/z 3920.8 [M-2(CH₃)₂C=CH₂-2CO₂+Na]⁺. HRMS: calcd for C₂₀₈H₂₅₆N₂₀O₅₄Na [M-2(CH₃)₂C=CH₂-2CO₂+Na]⁺: 3920.7793. Found: 3920.7790.

Compound 2a. A solution of compound **9**¹ (0.84 g, 1.66 mmol), n-butylamine (0.52 mL, 5.16 mmol), HATU (1.28 g, 3.34 mmol), DIEA (1.32 mL, 7.72 mmol) in DMF (20 mL) was stirred for 24 h and then concentrated with a rotavapor. After workup, the crude product was subjected to column chromatography (CH₂Cl₂/MeOH 30:1) to give **2a** as a pale yellow oil (1.06 g, 64 %). ¹H NMR (300 MHz, CDCl₃): δ 8.29 (s, 1H), 8.07 (s, 2H), 6.83 (br, 2H), 3.77 (s, 4H), 3.67-3.41 (m, 24H), 3.35-3.31 (m, 6H), 1.64-1.55 (m, 4H), 1.47-1.34 (m, 4H), 0.96 (t, $J = 7.2$ Hz, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 171.1, 166.0, 137.4, 135.0, 128.4, 126.3, 73.6, 71.8, 70.5, 70.3, 69.9, 69.3, 68.1, 58.9, 49.9, 44.9, 39.9, 31.6, 20.1, 13.7. MS (MALDI-TOF): m/z 612.5 [M+H]⁺, 634.6 [M+Na]⁺. HRMS (MALDI-FT): calcd for C₃₁H₅₃N₃O₉Na [M+Na]⁺: 634.3672. Found: 634.3674.

Compound 11. A solution of compound **10**¹ (1.08 g, 2.10 mmol), EDCI (0.52 g, 2.73 mmol), HOBt (85 mg, 0.63 mmol) and n-butylamine (0.28 mL, 2.73 mmol) in dichloromethane was stirred at room temperature for 23 h and then washed with diluted hydrochloric acid (1 N, 10 mL), water (10 mL×2) and brine (10 mL), and dried over sodium sulfate. Upon removal of the solvent, the resulting residue was subjected to column chromatography (CH₂Cl₂/MeOH 30:1) to give **11** as a colorless oil (0.92 g, 76%). ¹H NMR (300 MHz, CDCl₃): δ 8.51 (t, $J = 1.5$ Hz, 1H), 8.18 (t, $J = 1.5$ Hz, 1H), 8.15 (t, $J = 1.2$ Hz, 1H), 6.85 (t, $J = 5.4$ Hz, 1H), 3.94 (s, 3H), 3.78 (s, 4H), 3.67-3.38 (m, 22H), 3.35-3.27 (m, 6H), 1.65-1.56 (m, 2H), 1.47-1.35 (m, 2H), 0.98-0.93 (t, $J = 7.2$ Hz, 3H). ¹³C NMR (500 MHz, CDCl₃): δ 170.7, 165.9, 165.8, 137.5, 135.3, 130.7, 130.6, 130.1, 129.0, 71.9, 70.6, 70.4, 70.3, 70.2, 69.3, 68.2, 58.9, 52.4, 49.9, 45.0, 40.0, 31.7, 20.2, 13.8. MS (MALDI-TOF): m/z 571.1 [M+H]⁺, 593.1 [M+Na]⁺. HRMS (MALDI-FT): calcd for C₂₈H₄₆N₂O₁₀Na [M+Na]⁺: 593.3060. Found: 593.3045.

Compound 12. To a stirred solution of compound **11** (0.84 g, 1.48 mmol) in THF (12 mL) and methanol (4 mL) was added lithium hydroxide hydrate (94 mg, 2.24 mmol). The mixture was stirred for 12 h and then diluted hydrochloric acid was added to pH = 6. The mixture was concentrated with a rotavapor. The resulting residue was triturated with ethyl acetate (20 mL). The organic phase was washed with water (10 mL) and brine (10 mL), and dried over sodium sulfate. Evaporation of the solvent in vacuo afforded **12** as a colorless oil (0.80 g, 98%). ¹H NMR (300 MHz, CDCl₃): δ 8.46 (s, 1H), 8.26 (s, 1H), 8.18 (s, 1H), 7.23-7.21 (m, 2H), 3.79 (s, 4H), 3.67-3.42 (m, 22H), 1.64-1.59 (m, 2H), 1.45-1.37 (m, 2H), 0.95 (t, $J = 7.5$ Hz, 3H). ¹³C NMR (500 MHz, CDCl₃): δ 171.1, 167.4, 166.2, 136.8, 135.4, 131.2, 130.8, 130.6, 129.4, 71.9, 70.4, 70.2, 69.2, 68.2, 59.0, 58.8, 50.0, 45.1, 40.1, 31.6, 20.2, 13.8. MS (ESI): m/z 555.3 [M-H]⁺. HRMS (MALDI-FT): calcd for C₂₇H₄₄N₂O₁₀Na [M+Na]⁺: 579.2888. Found: 579.2870.

Compound 2b. A solution of compound **12** (0.40 g, 0.72 mmol), EDCI (0.19 g, 1.01 mmol) and HOBt (11 mg, 0.08 mmol) in dichloromethane (10 mL) was stirred for 1 h and diamine **5** (46 mg, 0.29 mmol) was added. The solution was stirred for 10 h and then diluted with dichloromethane (10 mL). The solution was washed with diluted hydrochloric acid (0.5 N, 10 mL), saturated sodium bicarbonate (10 mL), water (10 mL) and brine (10 mL), and dried over sodium sulfate. Upon removal of the solvent under reduced pressure, the resulting residue was subjected to column chromatography (CH₂Cl₂/EtOAc/MeOH 10:10:1 to 8:8:1 to 5:5:1) to afford **2b** as a pale yellow gummy solid (0.28 g, 79%). ¹H NMR (300 MHz, CDCl₃): δ 9.33 (s, 2H), 8.42 (s, 2H), 8.19 (s, 4H), 8.04 (s, 2H), 7.78-7.67 (m, 4H), 7.22 (t, $J = 5.1$ Hz, 2H), 3.77 (s, 8H), 3.66-3.41 (m, 44H), 3.32 (s,

6H), 3.25 (s, 6H), 1.64-1.56 (m, 4H), 1.45-1.38 (m, 4H), 0.96 (t, $J = 6.9$ Hz, 6H). ^{13}C NMR (125 MHz, CDCl_3): δ 171.2, 166.2, 165.0, 137.2, 136.3, 135.4, 135.2, 134.1, 128.9, 128.7, 128.2, 126.9, 120.0, 117.4, 71.8, 70.5, 70.3, 70.2, 70.1, 70.0, 69.2, 68.2, 58.8, 49.9, 45.0, 40.0, 31.6, 20.2, 13.8. MS (MALDI-TOF): m/z 1256.8 $[\text{M}+\text{Na}]^+$, 1272.9 $[\text{M}+\text{K}]^+$. HRMS (MALDI-FT): calcd for $\text{C}_{64}\text{H}_{94}\text{N}_6\text{O}_{18}\text{Na}$ $[\text{M}+\text{Na}]^+$: 1257.6555. Found: 1257.6547.

Compound 2c. To a stirred solution of compound **12** (0.26 g, 0.47 mmol), HATU (0.14 g, 0.88 mmol), DIEA (0.18 mL, 1.05 mmol) in DMF (10 mL) was added compound **6** (0.17 g, 0.21 mmol). The mixture was stirred for 10 h and then concentrated in vacuo. The resulting slurry was triturated with dichloromethane (20 mL). After workup, the resulting crude product was subjected to column chromatography ($\text{CH}_2\text{Cl}_2/\text{EtOAc}/\text{MeOH}$ 10:10:1 to 7.5:7.5:1 to 5:5:1) to give **2c** as a pale yellow solid (0.27 g, 68%). ^1H NMR (500 MHz, CDCl_3): δ 9.66 (s, 2H), 9.36 (s, 2H), 8.52 (s, 1H), 8.37 (s, 2H), 8.23 (s, 2H), 8.15 (s, 2H), 8.07 (s, 2H), 8.04 (s, 2H), 7.96 (s, 2H), 7.73-7.68 (m, 8H), 7.36 (br, 2H), 3.75 (s, 12H), 3.62-3.18 (m, 82H), 1.60-1.58 (m, 4H), 1.41-1.39 (m, 4H), 0.95 (t, $J = 7.3$ Hz, 6H). ^{13}C NMR (125 MHz, CDCl_3): δ 171.1, 166.3, 165.2, 165.1, 137.2, 137.0, 136.5, 136.4, 135.6, 135.2, 134.2, 129.1, 129.0, 128.5, 128.3, 127.4, 126.9, 120.0, 117.6, 71.9, 71.8, 70.6, 70.4, 70.3, 70.1, 70.0, 69.3, 68.2, 58.9, 58.8, 49.9, 45.1, 40.1, 31.6, 20.2, 13.8. MS (MALDI-TOF): m/z 1879.3 $[\text{M}+\text{Na}]^+$, 1895.7 $[\text{M}+\text{K}]^+$. HRMS: calcd for $\text{C}_{97}\text{H}_{135}\text{N}_9\text{O}_{27}\text{Na}$ $[\text{M}+\text{Na}]^+$: 1880.9359. Found: 1880.9360.

Compound 3a. A solution of compound **13** (1.80 g, 2.43 mmol), n-butylamine (0.27 mL, 2.68 mmol), HATU (1.20 g, 3.16 mmol) and DIEA (1.25 mL, 7.29 mmol) in DMF (30 mL) was stirred for 24 h and then concentrated under reduced pressure. The resulting slurry was triturated with dichloromethane (50 mL). The solution was washed successively with water (25 mL \times 2) and brine (25 mL), and dried over sodium sulfate. Upon removal of the solvent, the crude product was purified by column chromatography ($\text{CH}_2\text{Cl}_2/\text{MeOH}$ 20:1) to give **3a** as a pale yellow solid (1.55 g, 80%). ^1H NMR (300 MHz, CDCl_3): δ 9.37 (s, 1H), 8.41 (s, 1H), 8.17 (s, 1H), 8.12 (s, 1H), 8.01 (s, 1H), 7.76 (s, 1H), 7.71-7.64 (m, 3H), 7.45 (d, $J = 9.3$ Hz, 1H), 7.20 (t, $J = 5.5$ Hz, 1H), 7.04 (s, 1H), 3.75 (s, 4H), 3.63-3.41 (m, 22H), 3.31 (s, 3H), 3.24 (s, 3H), 1.60-1.56 (m, 2H), 1.55 (s, 9H), 1.40-1.38 (m, 2H), 0.93 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (125 MHz, DMSO): δ 171.1, 166.2, 164.9, 152.9, 137.2, 136.6, 136.4, 135.4, 135.3, 134.4, 128.8, 128.6, 128.3, 128.2, 127.3, 126.9, 119.2, 118.5, 116.9, 114.3, 80.5, 71.8, 70.5, 70.3, 70.1, 70.0, 69.9, 69.2, 68.0, 58.8, 58.7, 49.9, 44.9, 40.0, 31.5, 28.3, 20.1, 13.7. MS (MALDI-TOF): m/z 819.4 $[\text{M}+\text{Na}]^+$. HRMS: calcd for $\text{C}_{42}\text{H}_{60}\text{N}_4\text{O}_{11}\text{Na}$ $[\text{M}+\text{Na}]^+$: 819.4151. Found: 819.4124.

Compound 3b. A solution of compound **7** (0.32 g, 0.24 mmol), n-butylamine (0.05 mL, 0.49 mmol), HATU (0.16 g, 0.43 mmol) and DIEA (0.12 mL, 0.71 mmol) in DMF (10 mL) was stirred for 24 h and then concentrated in vacuo. The resulting slurry was triturated in dichloromethane (25 mL). After workup, the crude product was subjected to column chromatography ($\text{CH}_2\text{Cl}_2/\text{EtOAc}/\text{MeOH}$ 7.5:7.5:1) to give **3b** as a pale yellow solid (0.33 g, 88%). ^1H NMR (500 MHz, CDCl_3): δ 9.67 (s, 1H), 9.55 (s, 1H), 9.39 (s, 1H), 8.51 (s, 1H), 8.36 (s, 1H), 8.19-8.14 (m, 3H), 8.07 (s, 3H), 7.94 (s, 1H), 7.73-7.68 (m, 8H), 7.48 (d, $J = 8.9$ Hz, 1H), 7.34 (s, 1H), 7.04 (s, 1H), 3.74 (s, 8H), 3.60-3.17 (m, 54H), 1.59-1.56 (m, 2H), 1.54 (s, 9H), 1.40-1.37 (m, 2H), 0.93 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (125 MHz, CDCl_3): δ 171.1, 171.1, 166.3, 165.1, 165.0, 153.0, 137.1, 136.7, 136.6, 136.4, 136.3, 135.8, 135.6, 135.4, 135.2, 134.4, 134.1, 129.1, 128.9, 128.5, 128.3, 127.4, 126.9, 120.0, 119.2, 117.5, 116.8, 80.5, 71.9, 71.8, 70.6, 70.4, 70.2, 70.1, 70.0, 69.2, 68.1, 58.9, 58.7, 49.9, 45.1, 40.0, 31.6, 28.4, 20.2, 13.8. MS (MALDI-TOF): m/z 1442.6 $[\text{M}+\text{Na}]^+$.

HRMS: calcd for $C_{75}H_{101}N_7O_{20}Na$ $[M+Na]^+$: 1442.6977. Found: 1442.6994.

Compound 14. A solution of compounds **6** (0.14 g, 0.18 mmol), **7** (0.24 g, 0.18 mmol), HATU (96 mg, 0.25 mmol) and DIEA (0.09 mL, 0.53 mmol) in DMF (5 mL) was stirred for 11 h and then concentrated with a rotavapor. The resulting slurry was triturated with chloroform (10 mL). After workup, the solvent was removed and the resulting crude product was subjected to column chromatography ($CH_2Cl_2/EtOAc/MeOH$ 8:2:1) to afford **14** as a pale yellow solid (0.16 g, 42 %). 1H NMR (300 MHz, $DMSO-d_6$): δ 10.59-10.57 (m, 4H), 10.52 (s, 1H), 10.38 (s, 1H), 9.33 (s, 1H), 8.73 (s, 1H), 8.70-8.69 (m, 2H), 8.42-8.40 (m, 4H), 8.32 (s, 1H), 8.22-8.18 (m, 6H), 8.07 (s, 1H), 7.98 (s, 1H), 7.79-7.77 (m, 8H), 7.69-7.43 (m, 7H), 6.84 (dd, $J_1 = 8.7$ Hz, $J_2 = 1.8$ Hz, 1H), 6.79-6.76 (m, 1H), 5.07 (br, 2H), 3.72 (s, 12H), 3.59-3.31 (m, 60H), 3.23 (s, 9H), 3.16 (s, 9H), 1.51 (s, 9H). ^{13}C NMR (500 MHz, $DMSO$): δ 171.2, 168.1, 165.1, 153.0, 145.1, 137.0, 136.7, 136.5, 136.2, 135.3, 134.3, 133.9, 129.3, 128.7, 128.2, 127.3, 125.2, 120.1, 119.3, 118.7, 117.6, 117.0, 114.5, 108.3, 80.4, 71.8, 71.7, 70.3, 70.2, 70.0, 69.0, 68.3, 58.8, 58.7, 49.9, 45.2, 28.4. MS (MALDI-TOF): m/z 2151.5 $[M+Na]^+$, 2167.6 $[M+K]^+$. HRMS (MALDI-FT): calcd for $C_{114}H_{141}N_{11}O_{29}Na$ $[M+Na]^+$: 2150.9792. Found: 2150.9789.

Compound 4. To a stirred solution of compound **15** (34 mg, 0.12 mmol), HATU (76 mg, 0.20 mmol) and DIEA (0.10 mL, 0.56 mmol) in DMF (2 mL) was added **14** (81 mg, 0.038 mmol). Stirring was continued for 15 h and then the solvent was evaporated with a rotavapor. After workup, the crude product was purified by column chromatography ($CH_2Cl_2/EtOAc/MeOH$ 6:4:1) to afford compound **4** as a yellow solid (82 mg, 90%). 1H NMR (300 MHz, $DMSO-d_6$): δ 10.75-10.69 (m, 6H), 10.16 (s, 1H), 9.58 (s, 1H), 8.68 (s, 3H), 8.47-7.71 (m, 37H), 7.54 (d, $J = 8.1$ Hz, 1H), 7.48 (d, $J = 9.0$ Hz, 1H), 3.71 (s, 12H), 3.57-3.30 (m, 60H), 3.21 (s, 9H), 3.12 (s, 9H), 2.16-2.14 (m, 2H), 1.51 (s, 9H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.9, 171.3, 165.2, 153.1, 137.0, 136.0, 135.3, 134.3, 133.9, 131.4, 130.9, 129.9, 129.5, 128.8, 128.2, 127.5, 127.4, 126.6, 125.8, 125.0, 124.9, 124.8, 123.4, 120.2, 80.4, 71.8, 71.7, 70.5, 70.4, 70.2, 70.0, 69.0, 68.2, 58.8, 58.7, 49.9, 45.1, 36.7, 32.7, 28.4, 27.3. MS (MALDI-TOF): m/z 2424.2 $[M+Na]^+$. HRMS: calcd for $C_{134}H_{155}N_{11}O_{30}Na$ $[M+Na]^+$: 2421.0835. Found: 2421.0834.

References

- 1) Y.-X. Xu, G.-T. Wang, X. Zhao, X.-K. Jiang and Z.-T. Li, *Langmuir*, 2009, **25**, 2684.
- 2) Y.-X. Xu, X. Zhao, X.-K. Jiang and Z.-T. Li, *J. Org. Chem.*, 2009, **74**, 7267.

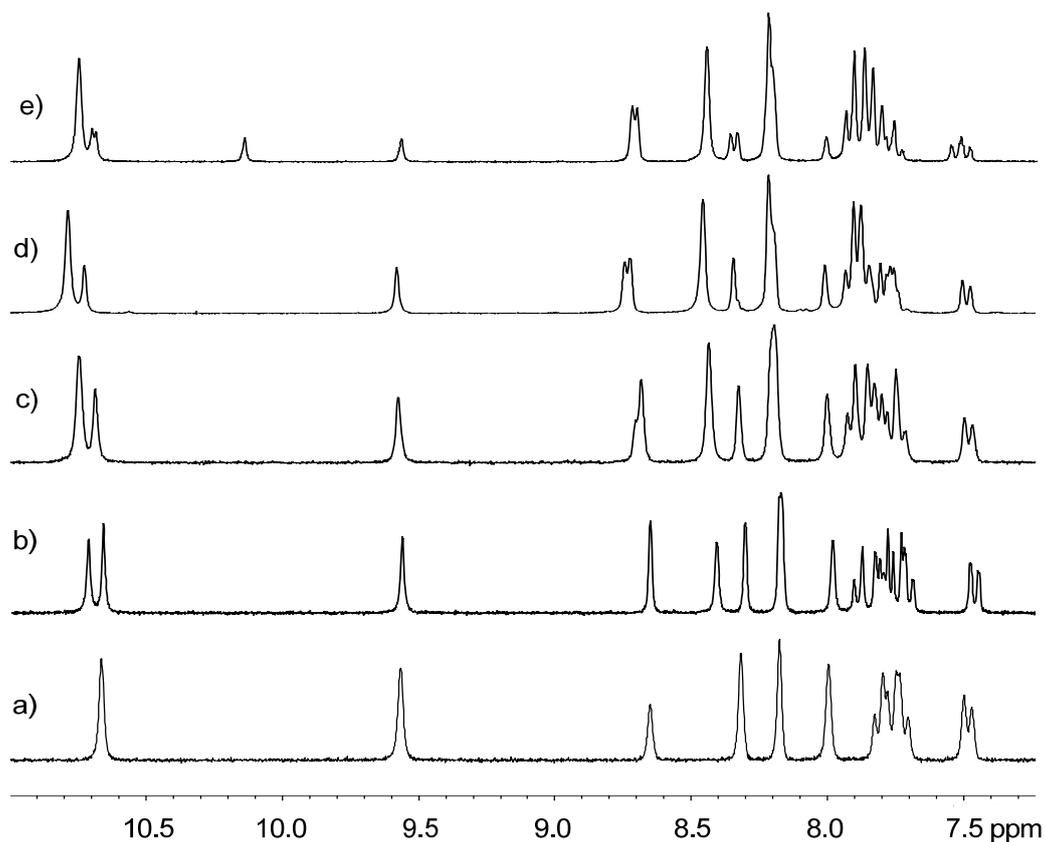


Figure S1. Partial ^1H NMR spectra (400 MHz) of compounds a) **1b**, b) **1c**, c) **1d**, d) **1e**, and e) **1g** in $\text{DMSO-}d_6$ (6.0 mM) at 25 °C.

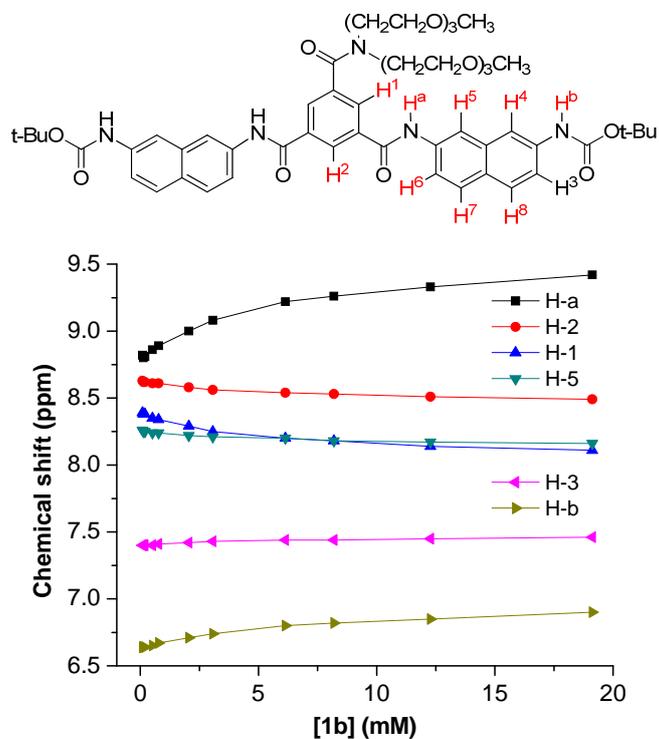


Figure S2. The plot of chemical shifts of the ^1H NMR signals of **1b** versus $[\mathbf{1b}]$ in CDCl_3 at 25 °C.

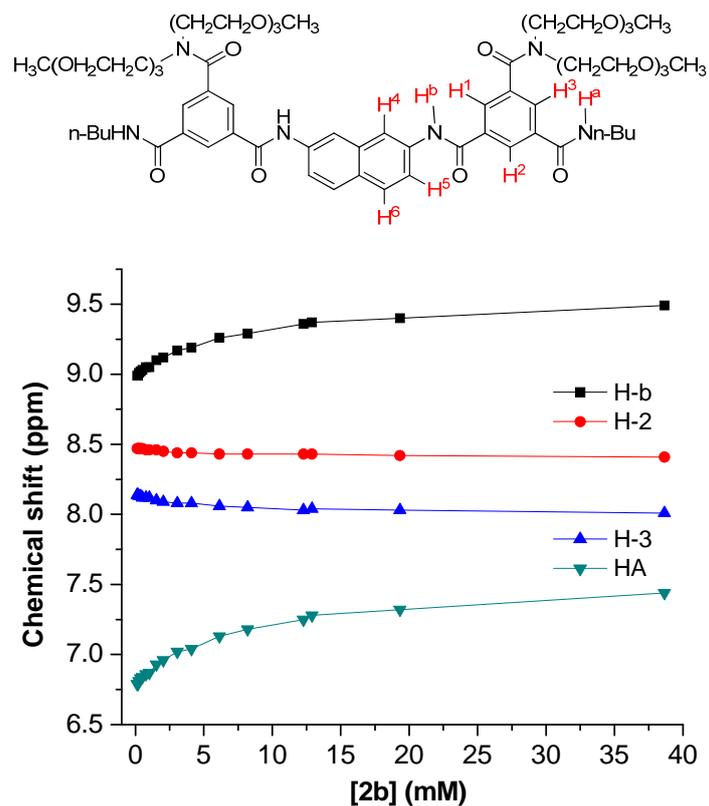


Figure S3. The plot of chemical shifts of the ¹H NMR signals of **2b** versus [**2b**] in CDCl₃ at 25 °C.

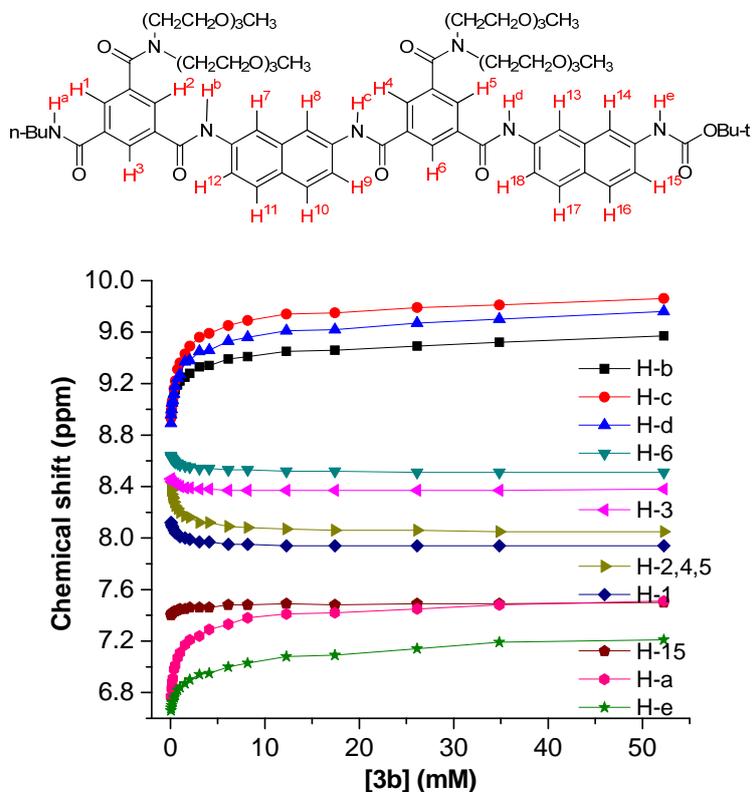


Figure S4. The plot of chemical shifts of the ¹H NMR signals of **3b** versus [**3b**] in CDCl₃ at 25 °C.

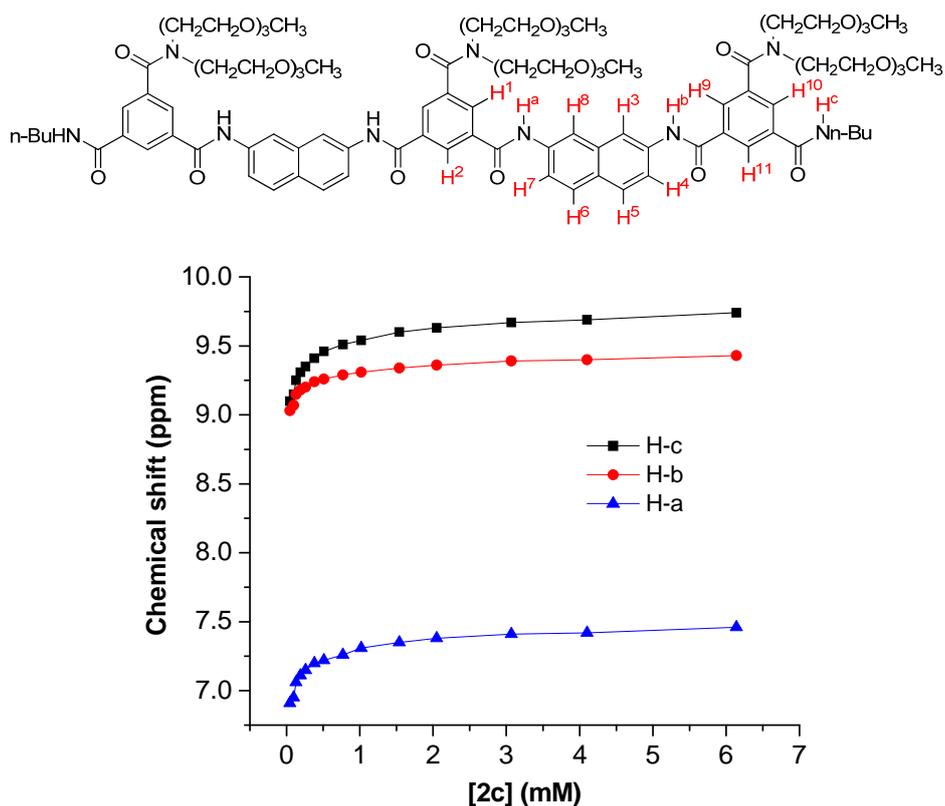


Figure S5. The plot of chemical shifts of the ¹H NMR signals of **2c** versus [2c] in CDCl₃ at 25 °C.

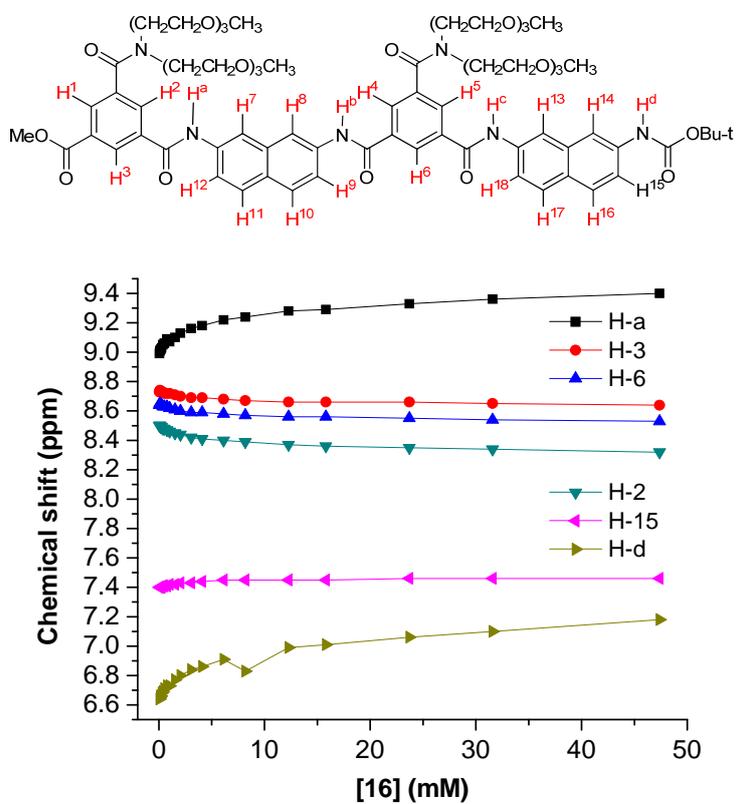


Figure S6. The plot of chemical shifts of the ¹H NMR signals of **16** versus [16] in CDCl₃ at 25 °C.

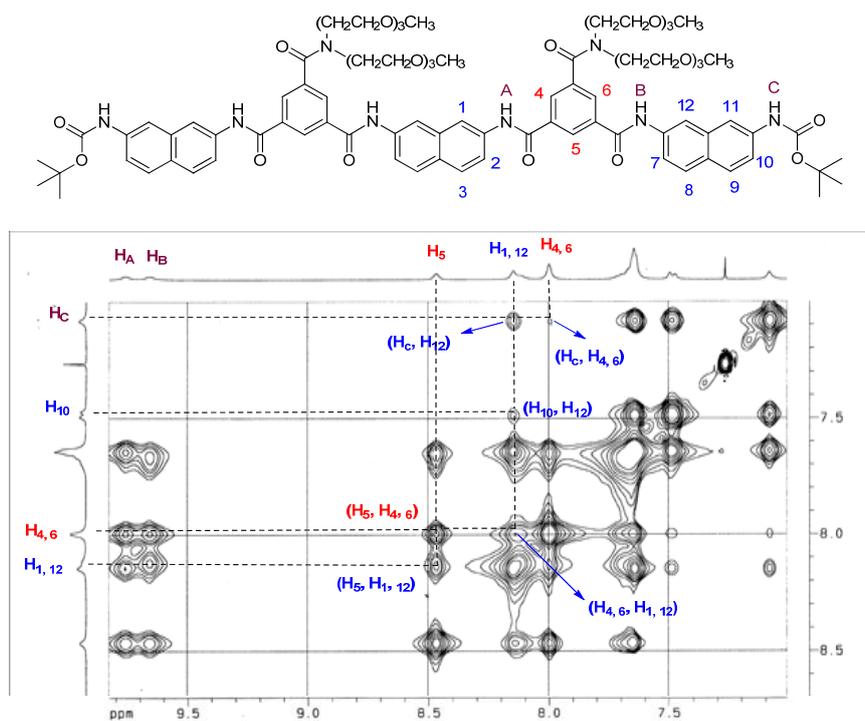


Figure S7. Partial ¹H NMR NOESY spectrum (400 MHz) of **1c** (6.0 mM) in CDCl₃ (mixing time = 1s).

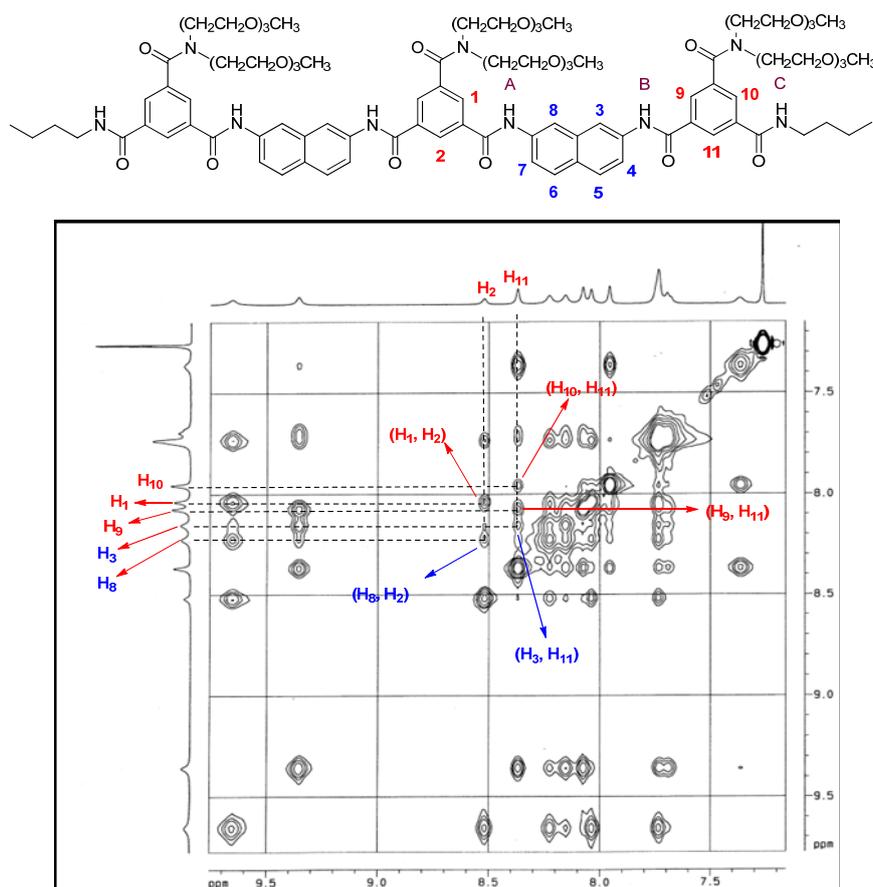


Figure S8. Partial ¹H NMR NOESY spectrum (400 MHz) of **2c** (6.0 mM) in CDCl₃ (mixing time = 1s).

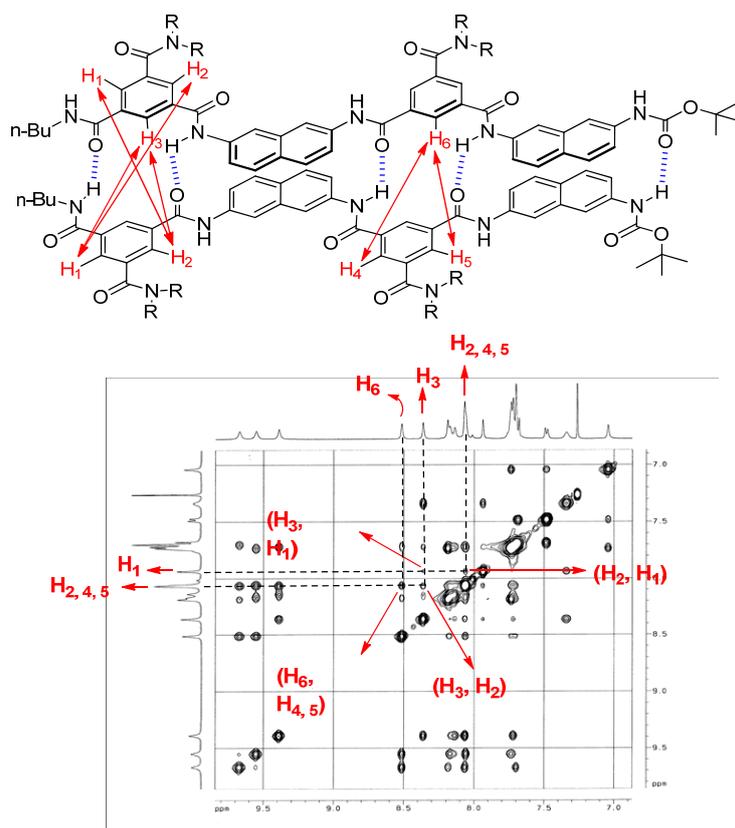


Figure S9. Partial intermolecular NOE connections observed for oligomer **3b** (6.0 mM in CDCl₃, 500 MHz) at 23 °C (mixing time: 1 s).

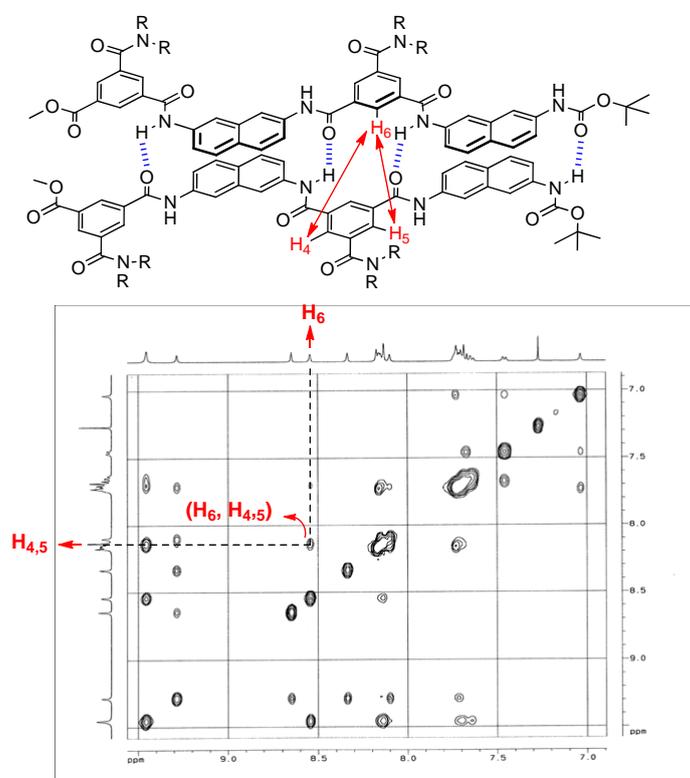


Figure S10. Partial intermolecular NOE connections observed for oligomer **16** (6.0 mM in CDCl₃, 500 MHz) at 23 °C (mixing time: 1 s).

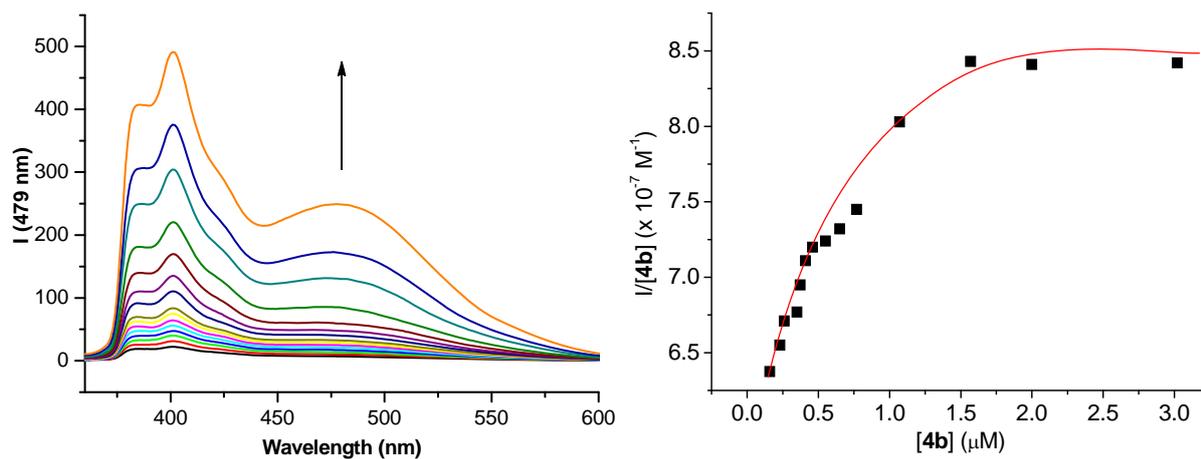


Figure S11. Concentration dependent fluorescence spectra of **4b** in chloroform at 25 °C.

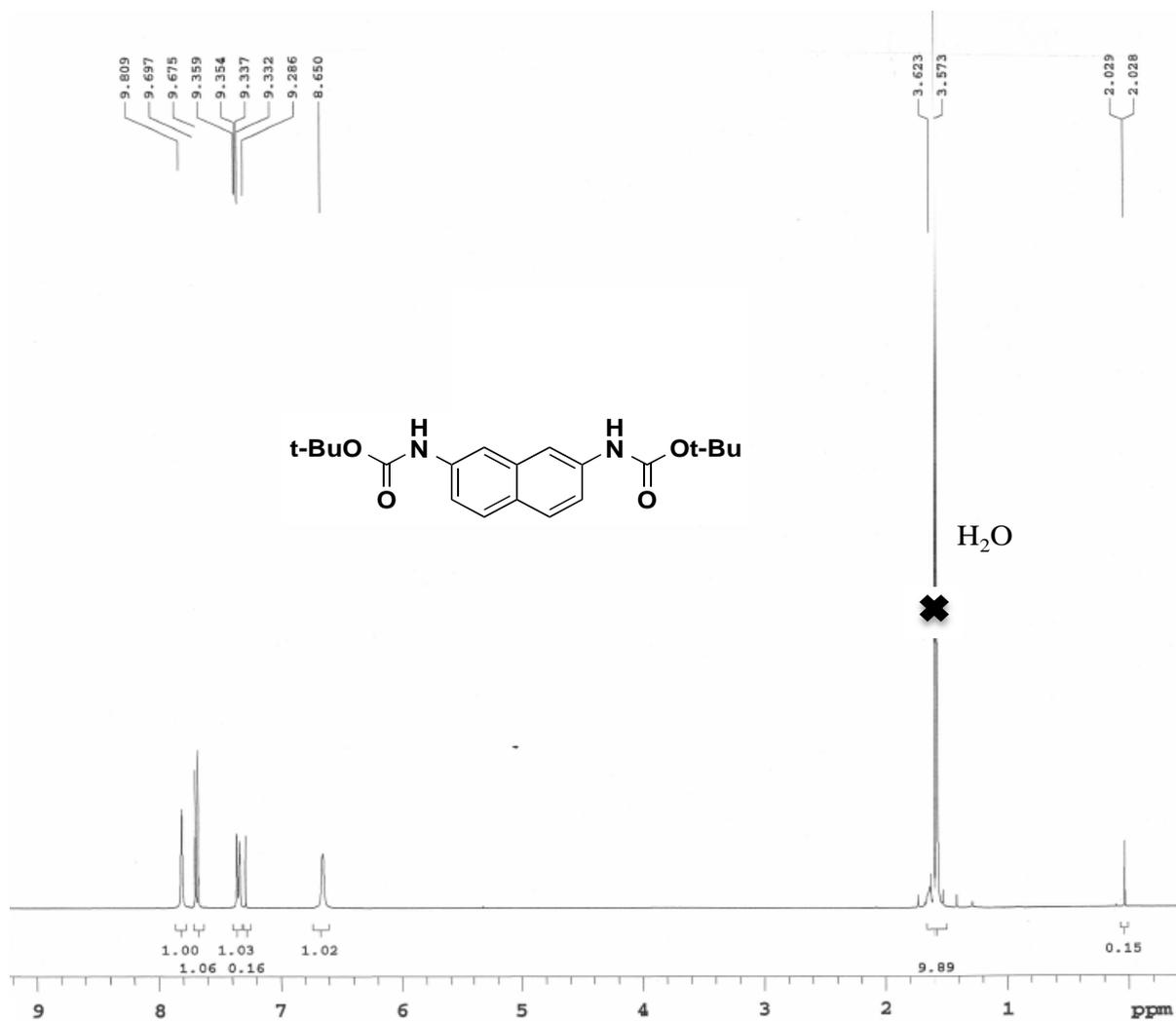


Figure S12. ^1H NMR spectrum (500 MHz) of **1a** in CDCl_3 .

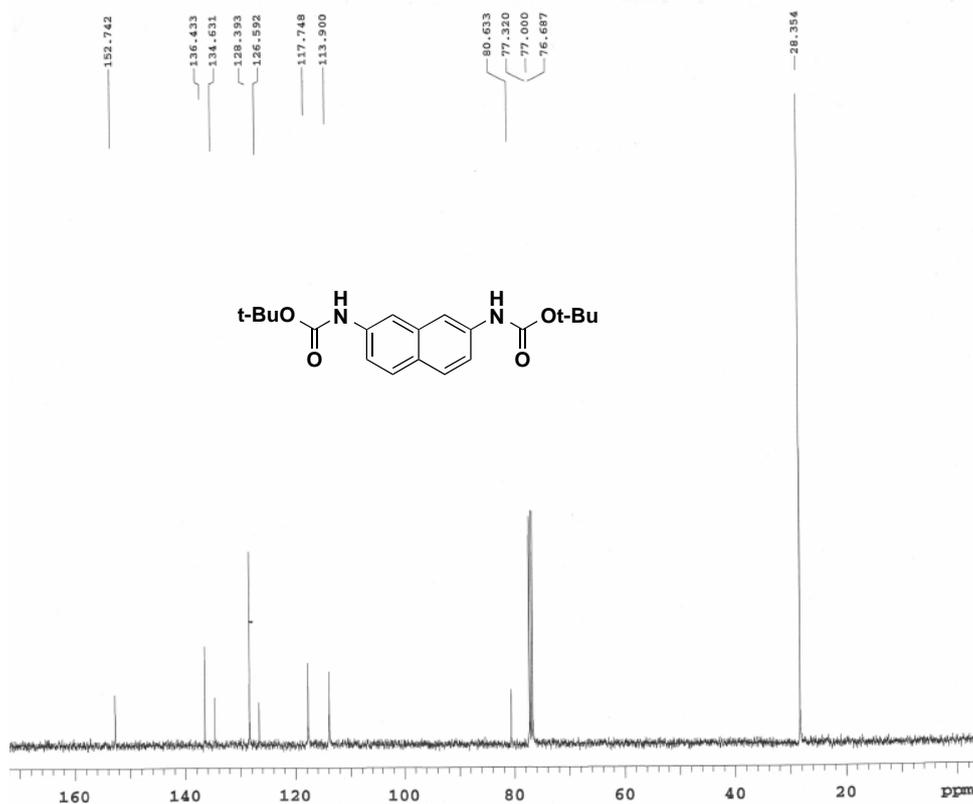


Figure S13. ^{13}C NMR spectrum (125 MHz) of **1a** in CDCl_3 .

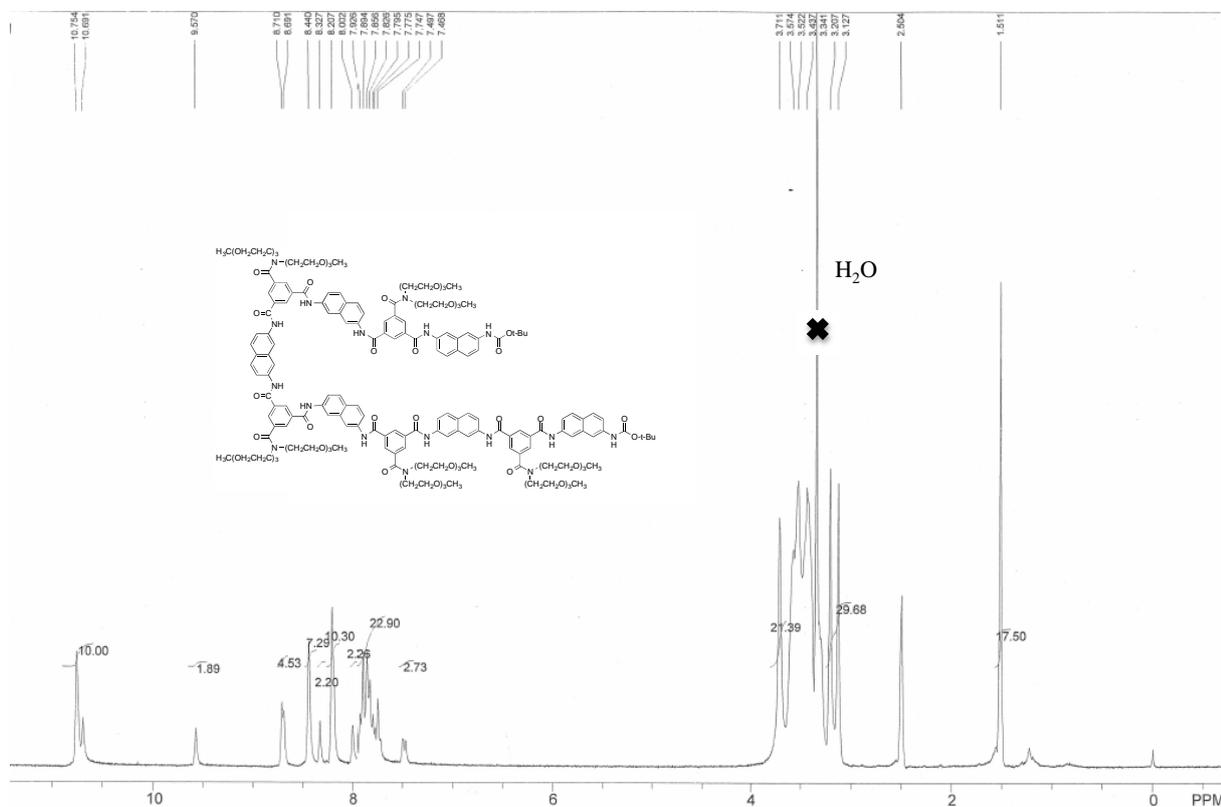


Figure S14. ^1H NMR spectrum (500 MHz) of **1f** in DMSO-d_6 .

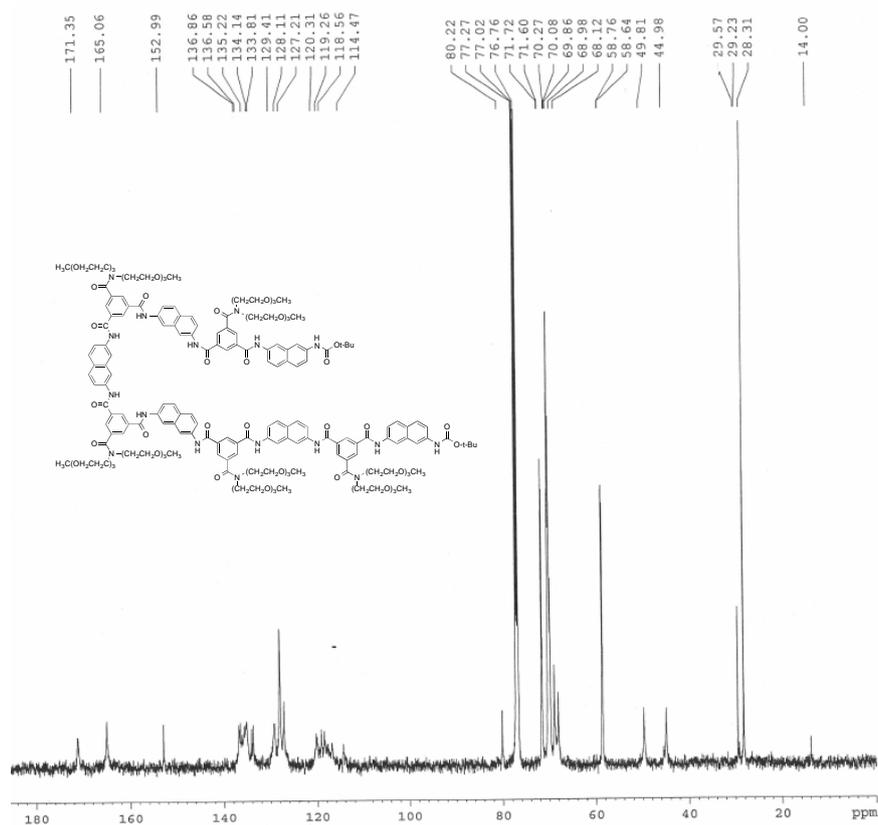


Figure S15. ^{13}C NMR spectrum (125 MHz) of **1f** in CDCl_3 .

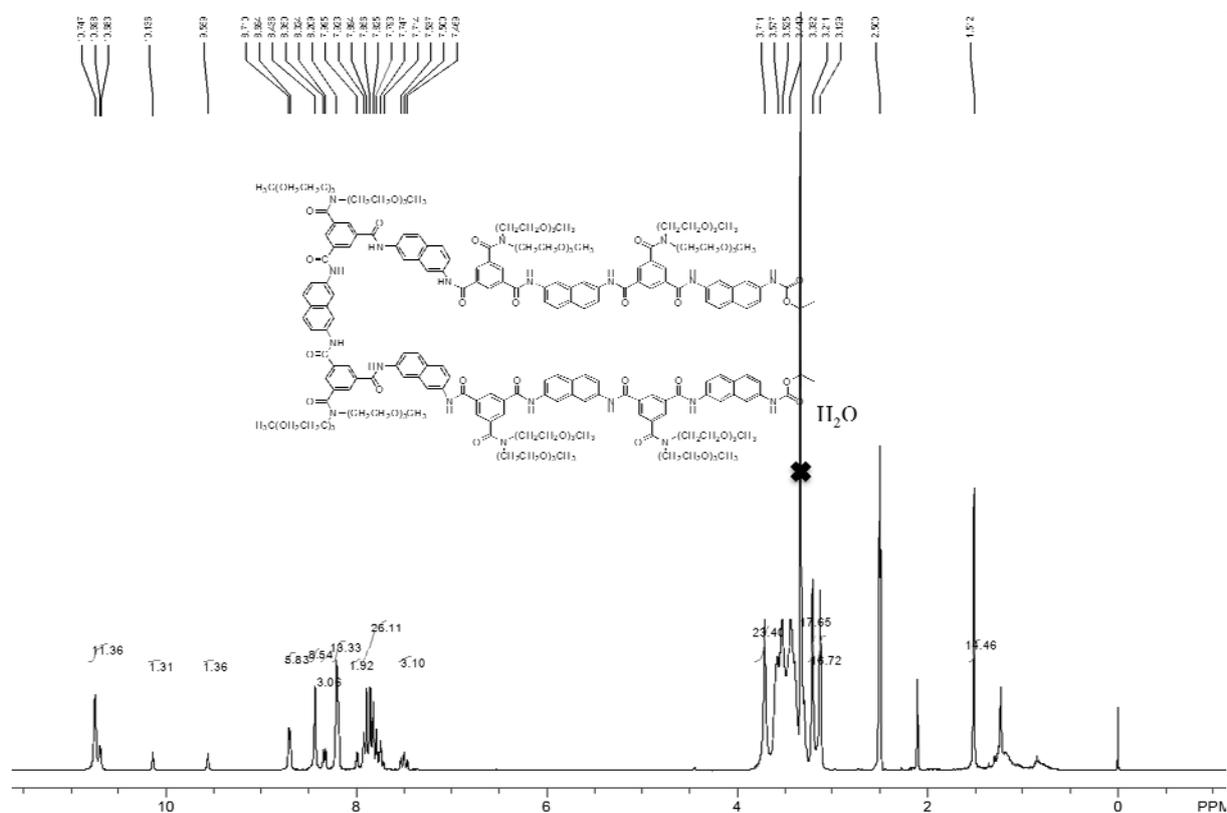


Figure S16. ^1H NMR spectrum (300 MHz) of **1g** in DMSO-d_6 .

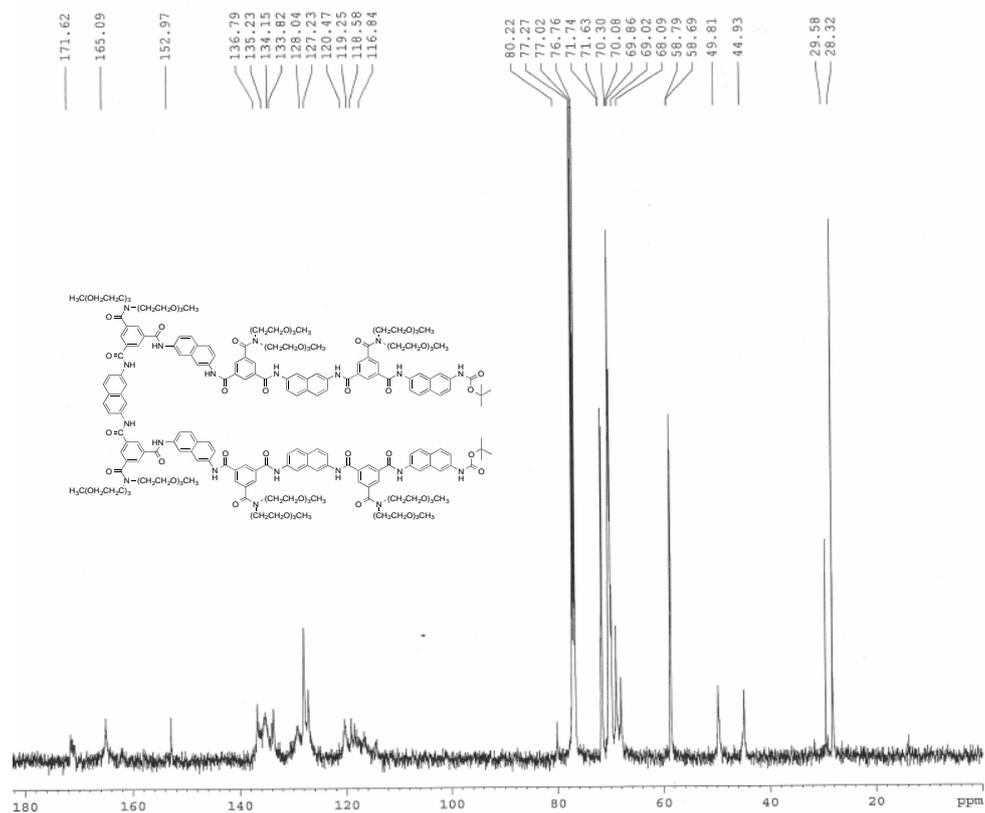


Figure S17. ^{13}C NMR spectrum (125 MHz) of **1g** in CDCl_3 .

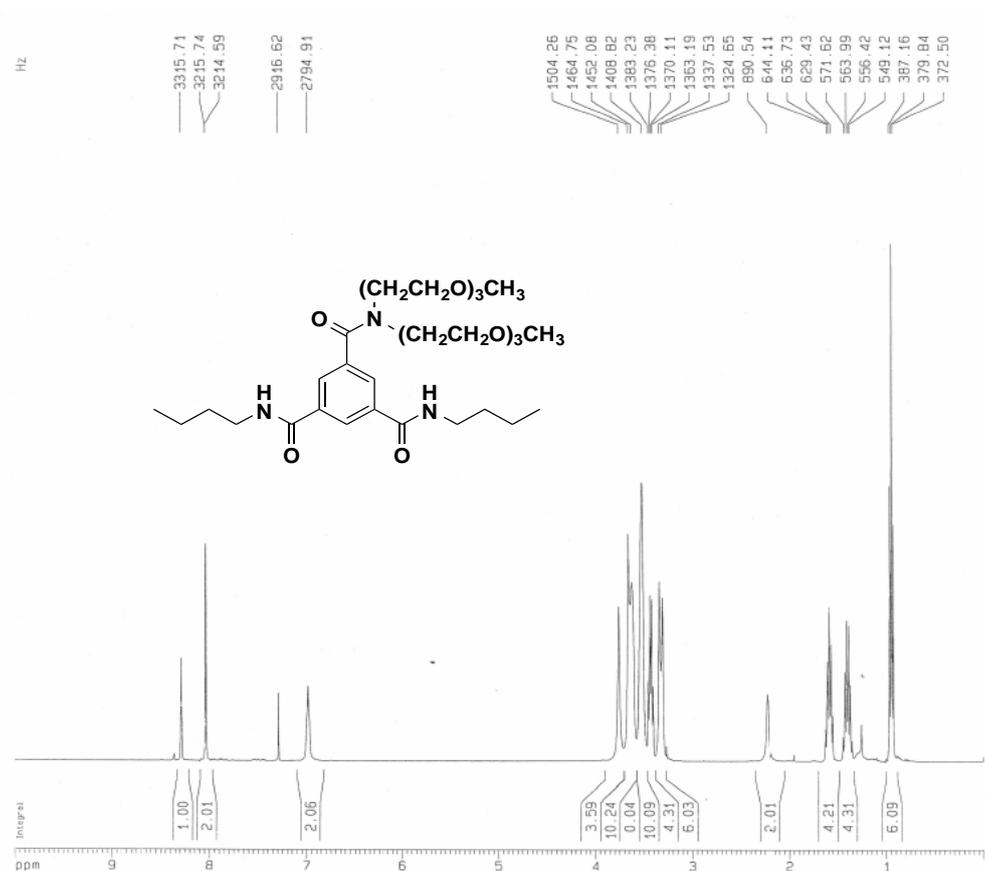


Figure S18. ^1H NMR spectrum (400 MHz) of **2a** in CDCl_3 .

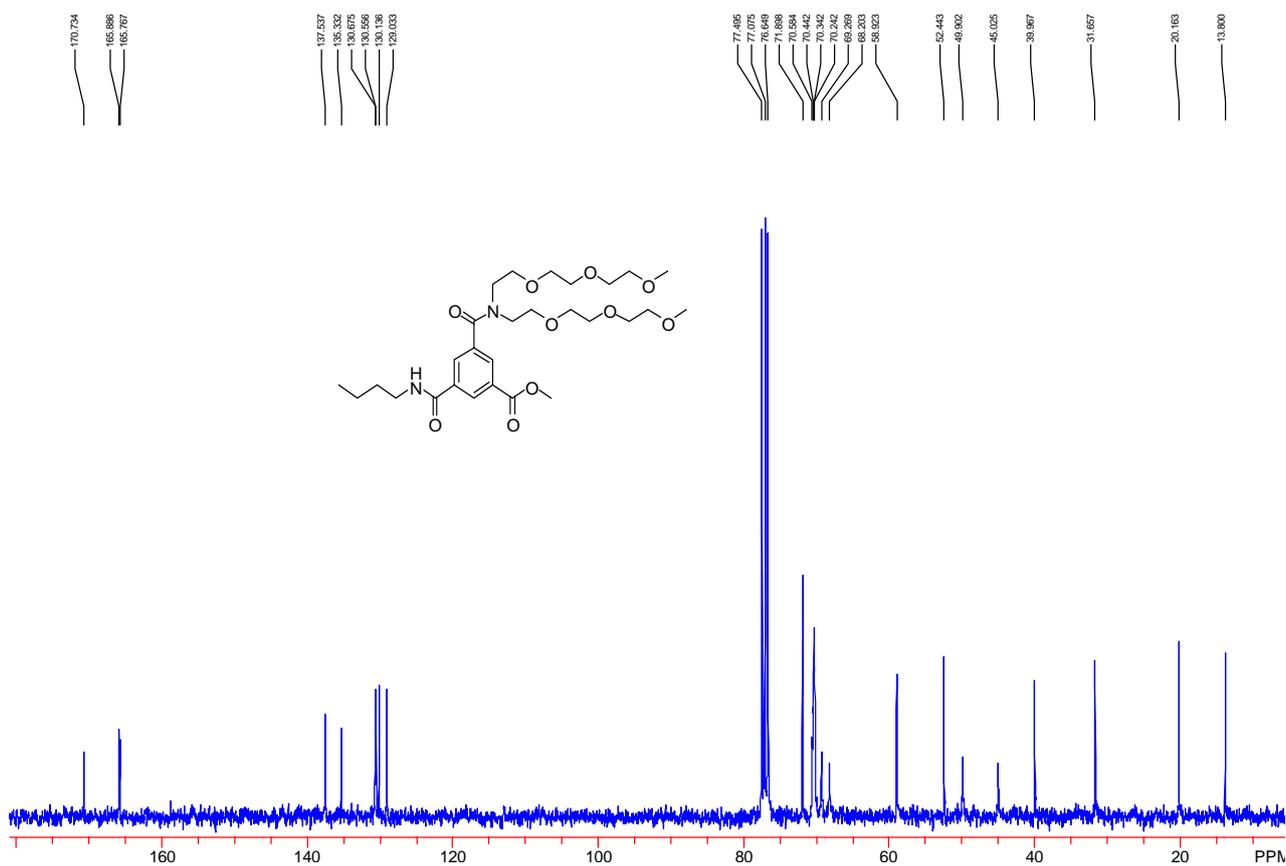


Figure S21. ^{13}C NMR spectrum (100 MHz) of **11** in CDCl_3 .

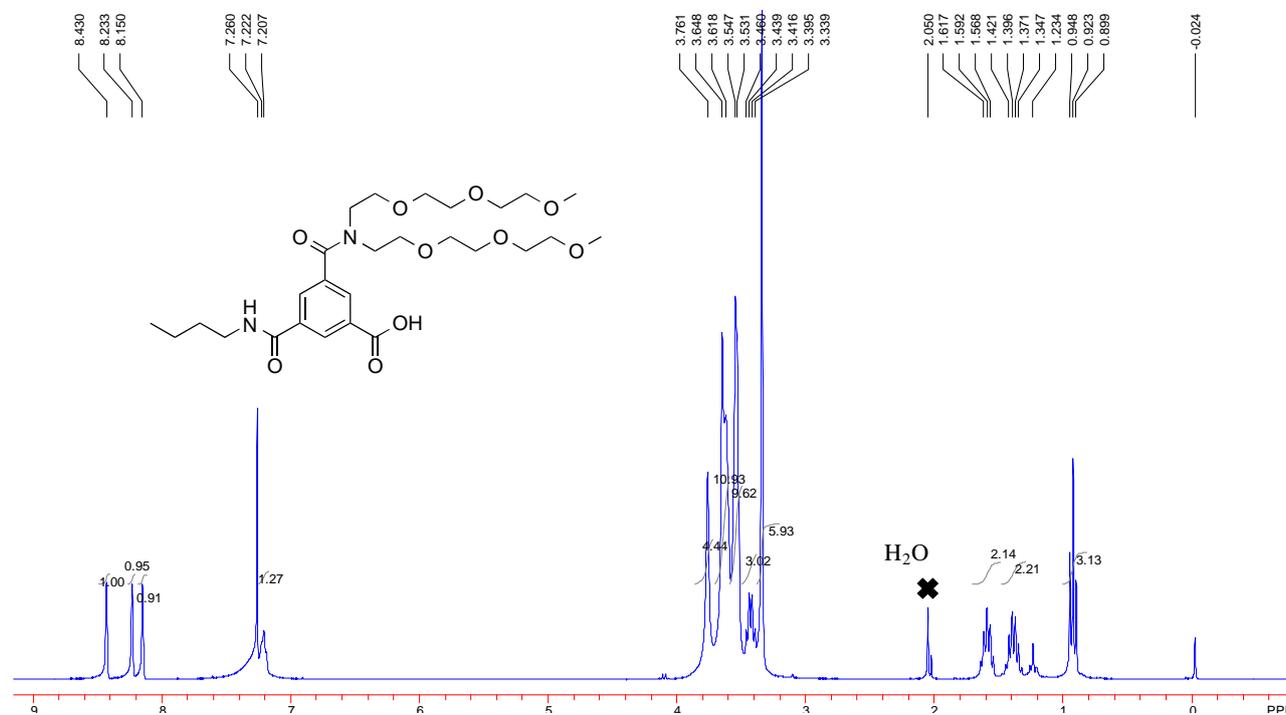


Figure S22. ^1H NMR spectrum (300 MHz) of **12** in CDCl_3 .

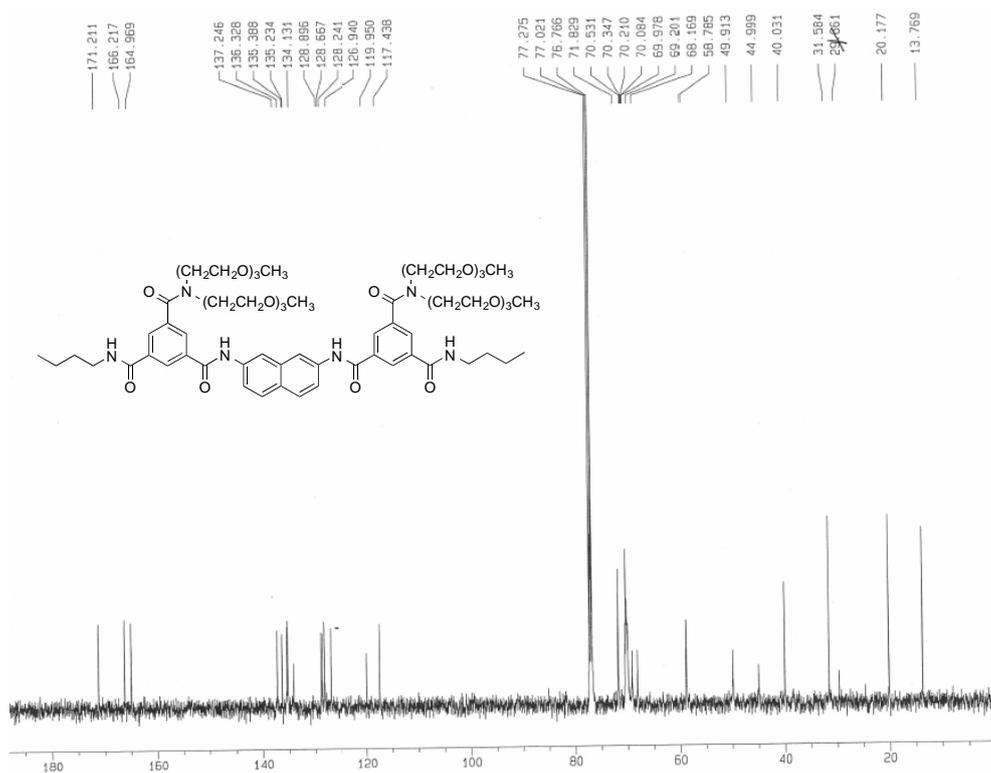


Figure S25. ^{13}C NMR spectrum (100 MHz) of **2b** in CDCl_3 .

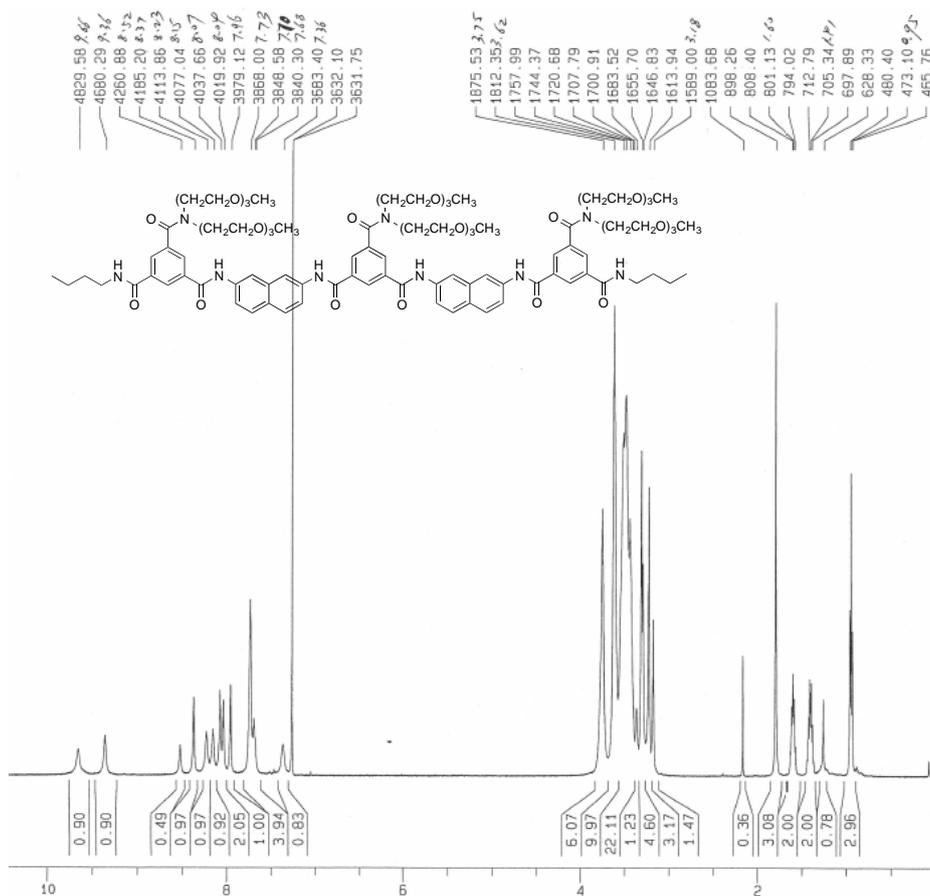


Figure S26. ^1H NMR spectrum (500 MHz) of **2c** in CDCl_3 .

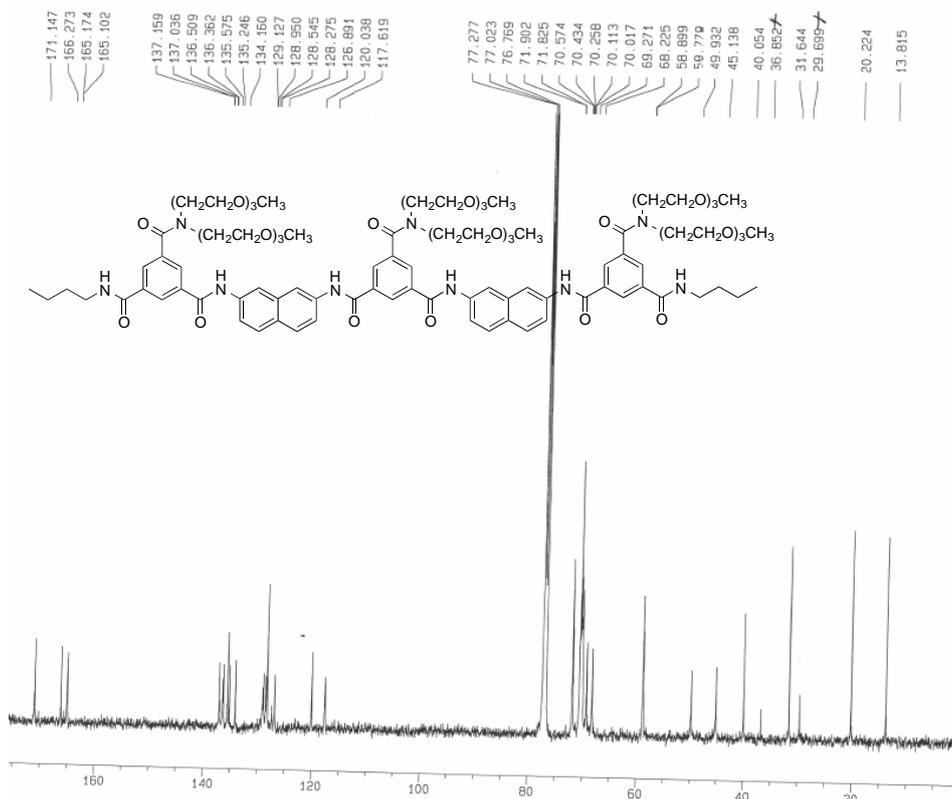


Figure S27. ¹³C NMR spectrum (125 MHz) of **2c** in CDCl₃.

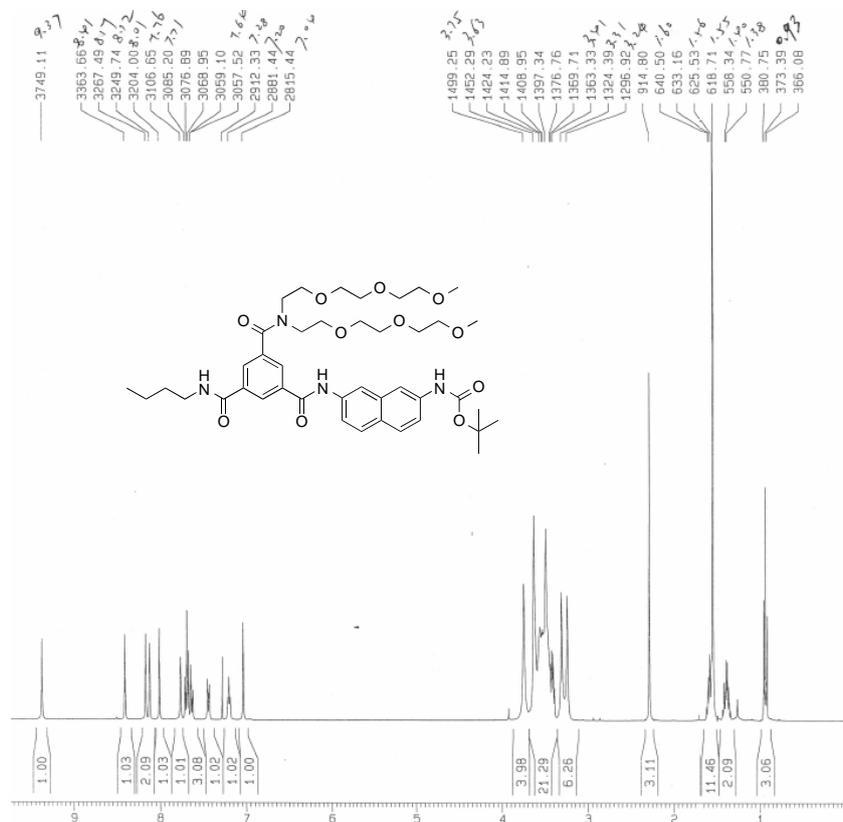


Figure S28. ¹H NMR spectrum (400 MHz) of **3a** in CDCl₃.

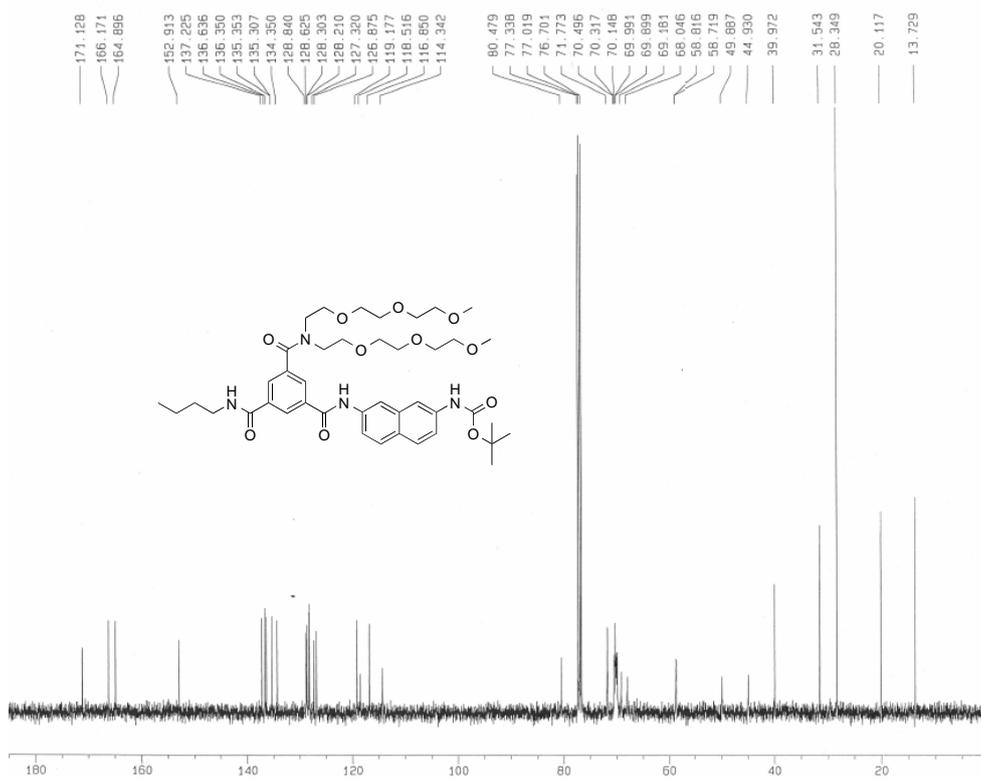


Figure S29. ^{13}C NMR spectrum (100 MHz) of **3a** in CDCl_3 .

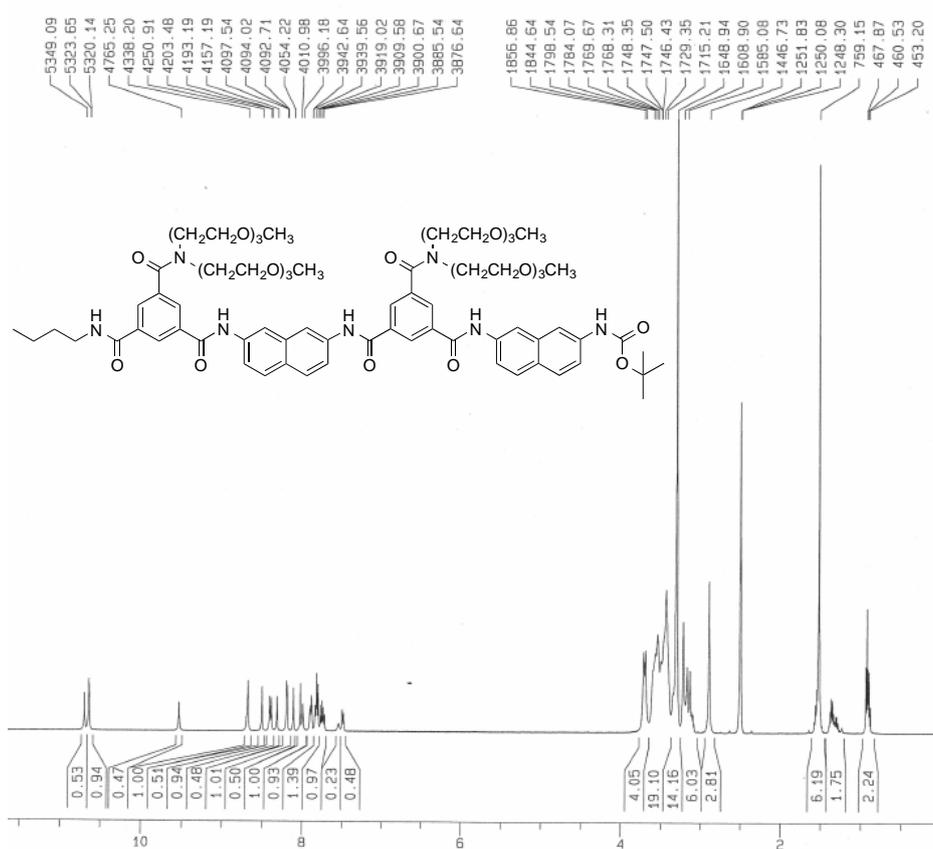


Figure S30. ^1H NMR spectrum (500 MHz) of **3b** in CDCl_3 .

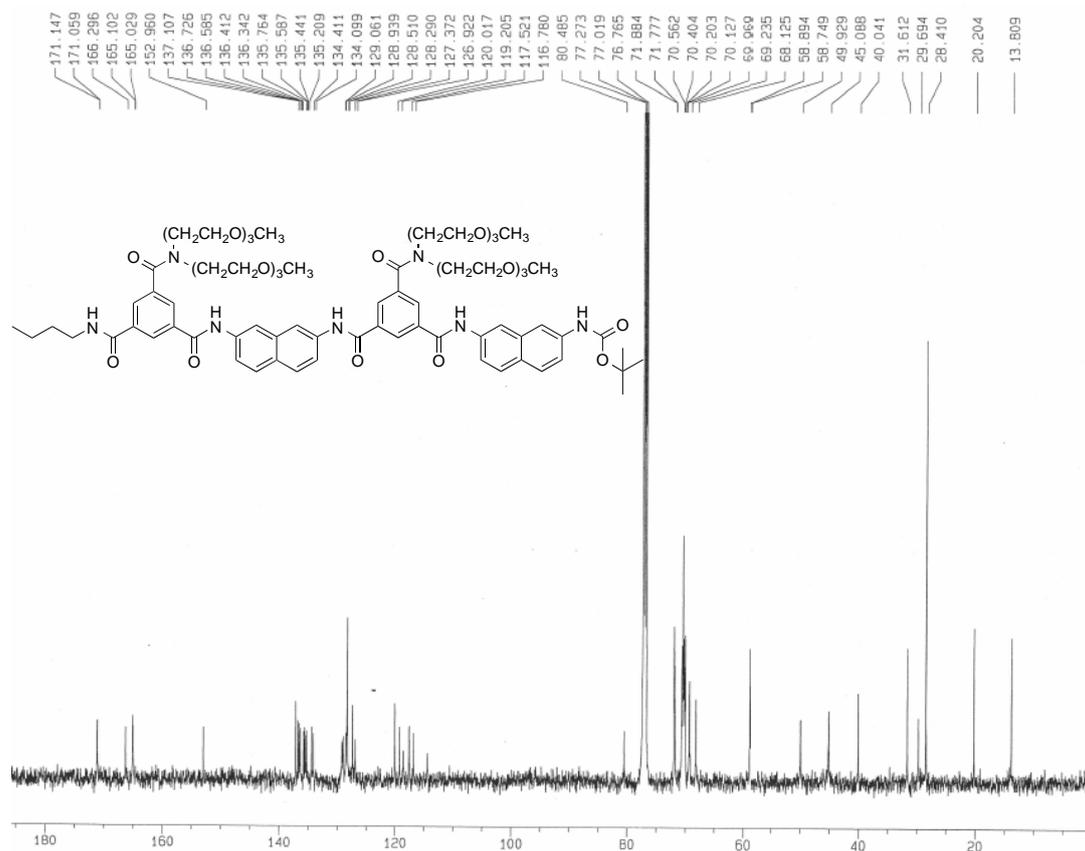
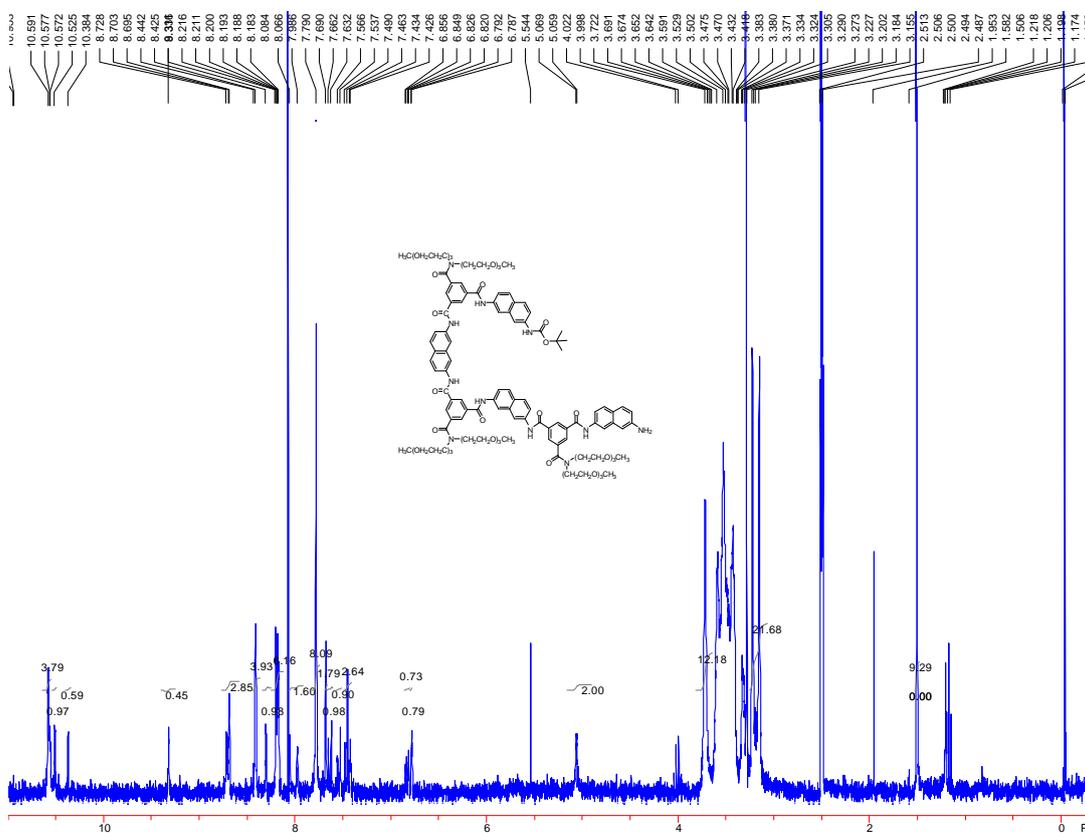


Figure S31. ^{13}C NMR spectrum (125 MHz) of **3b** in CDCl_3 .



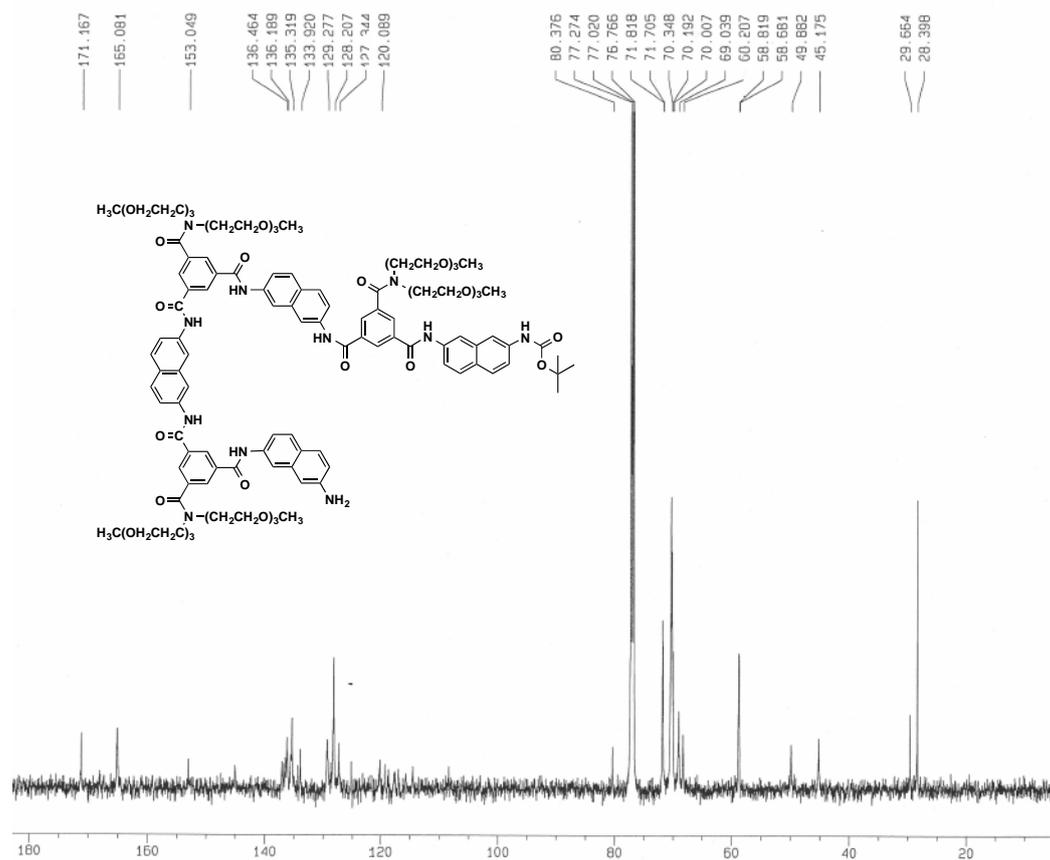


Figure S33. ^{13}C NMR spectrum (125 MHz) of **14** in CDCl_3 .

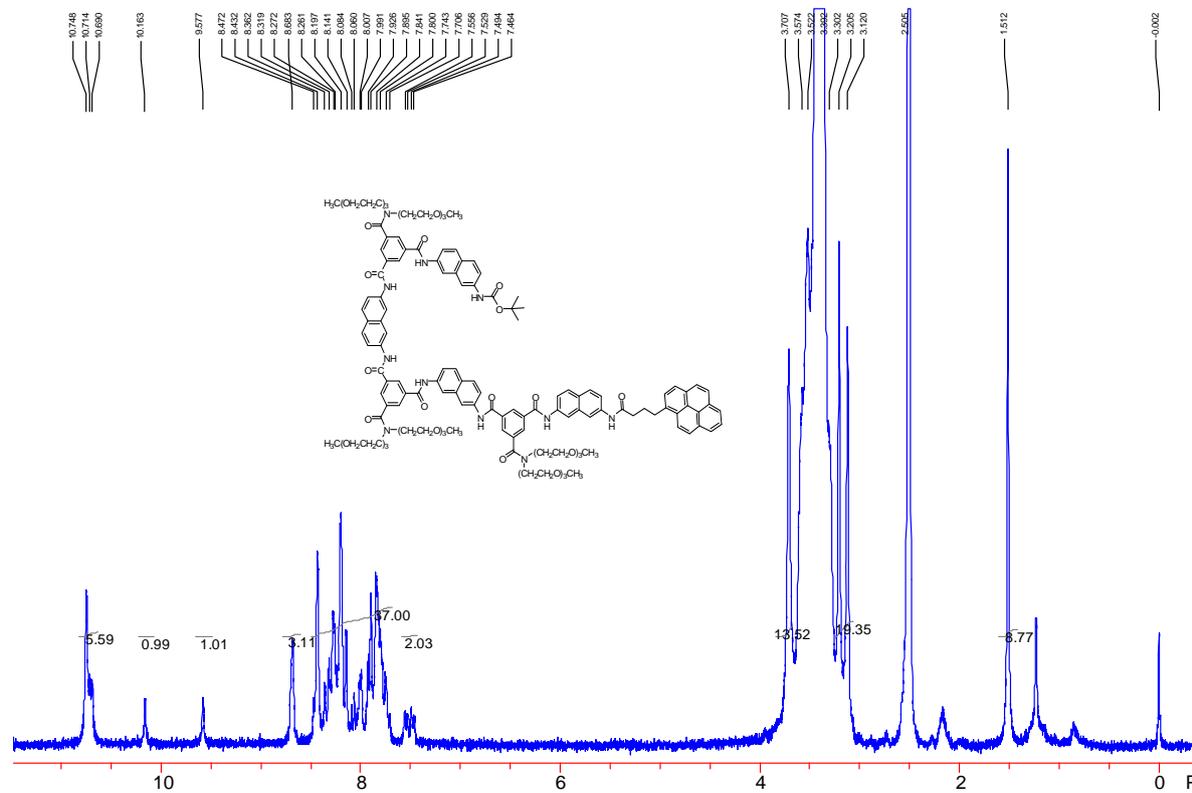


Figure S34. ^1H NMR spectrum (300 MHz) of **4** in DMSO-d_6 .

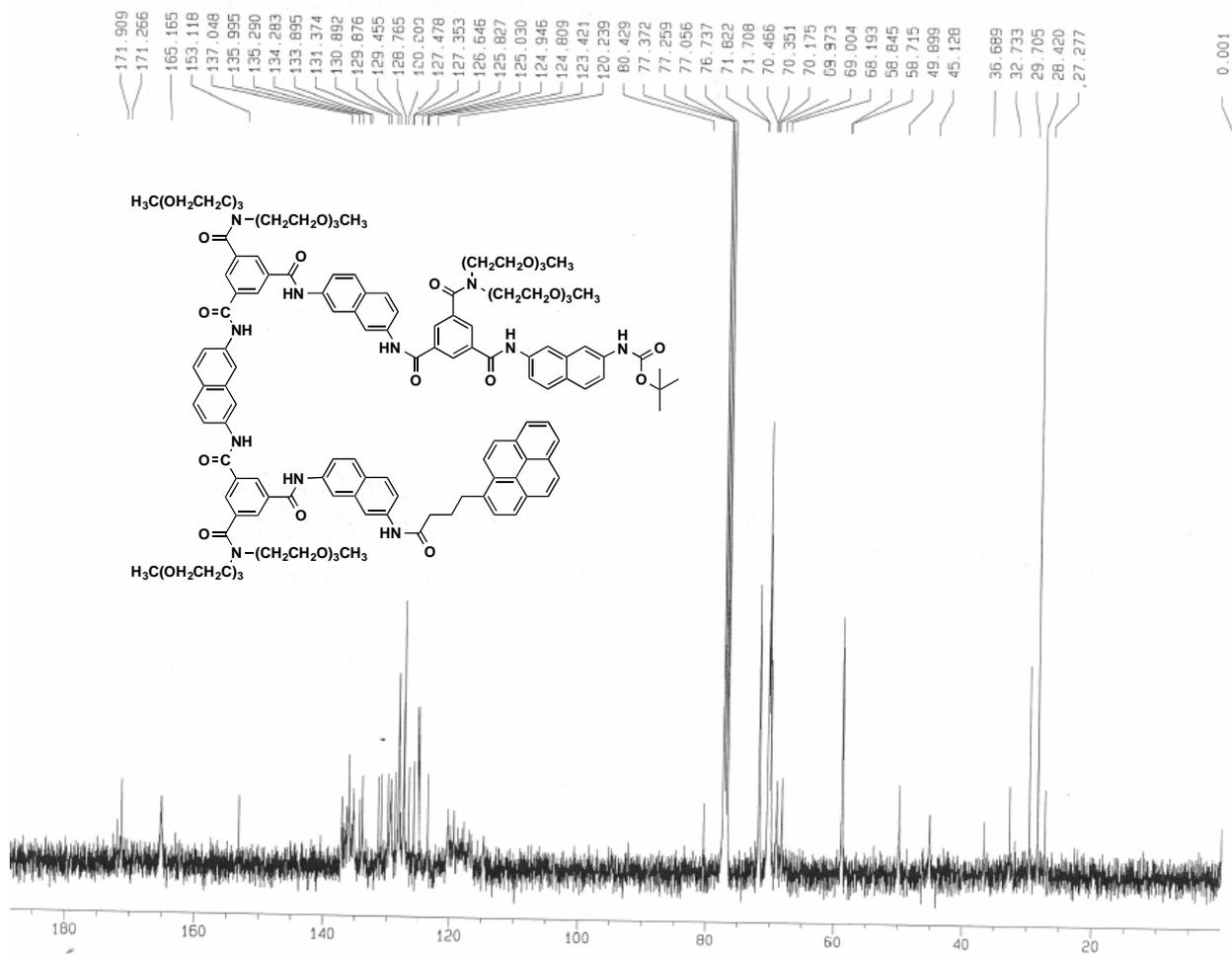


Figure S35. ^{13}C NMR spectrum (125 MHz) of 4 in CDCl_3 .