Electronic Supplementary Material (ESI) for Polymer Chemistry.
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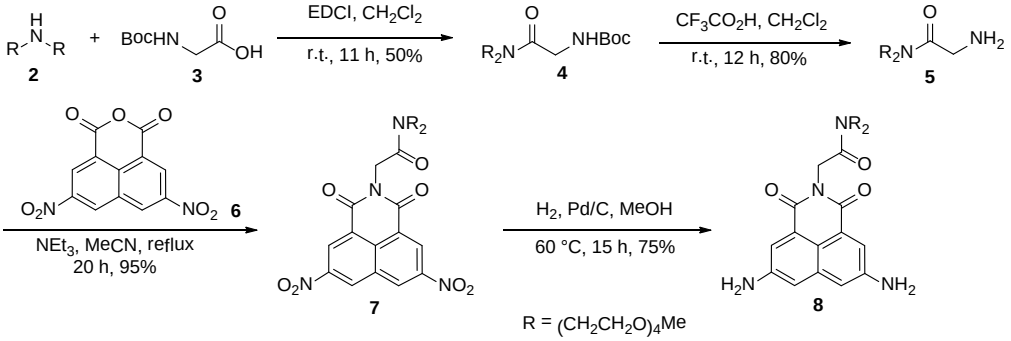
General methods. All reactions were performed under the protection of argon gas. ^{1}H and ^{13}C NMR spectra were obtained with Brucker ARX400 spectrometer with tetramethylsilane (TMS) as the internal standard; the chemical shifts (δ) are given in parts per million with respect to TMS. UV-vis spectra were recorded with a PerkinElmer Lambda 750 S spectrophotometer. Fluorescent spectra were obtained on a Cary-Eclipse Fluorescence Spectrometer. The 1-cm quartz cuvette was used for the UV and fluorescent measurement. Gel permeation chromatography (GPC) was performed on a Waters 1515 instrument with DMF containing lithium bromide (5 g/L) as the eluent with a flow rate of 1.0 mL/min at 60℃. The GPC was equipped with Waters 2414 Refractive Index detector, using TSK alpha-2500 and TSK alpha-3000 columns. The molecular weight was reported with the polymethylmethacrylate (PMMA) standard.



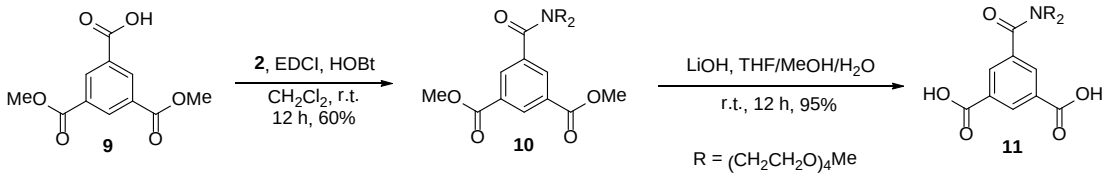
Compound 4. A mixture of compound **2**[1] (7.94 g, 20.0 mmol), Boc-glycin (**3**) (4.20 g, 24.0 mmol), 1-ethyl-3-(3-dimethyllaminopropyl)carbodiimide hydrochloride (EDCI, 5.74 g, 30.0 mmol) in dichloromethane (5 mL) was stirred at room temperature for 11 h. After removal of the solvent with a rotary evaporation, the resulting residue was purified by column chromatography on silica gel (CH_2Cl_2/MeOH 15/1) to give compound **4** as a pale yellow solid (6.40 g, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 5.48 (s, 1H), 4.03 (s, 2H), 3.62-3.47 (m, 32H), 3.35 (s, 6H), 1.41 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.1, 155.8, 79.3, 71.9, 70.8, 70.61, 70.6, 70.5, 70.4, 69.4, 68.6, 59.0, 47.9, 46.3, 42.2, 28.4. HRMS (ESI): Calcd for $C_{20}H_{42}N_2O_9$ $[M+H]^+$: 455.2969. Found 455.2961.

Compound 5. Compound **4** (3.20 g, 5.80 mmol) was dissolved in dichloromethane (40 mL), and trifluoroacetic acid (6.0 mL) was added. The mixture was stirred at room temperature for 12 h. After removal of the solvent with a rotavapor, the resulting residue was re-dissolved in dichloromethane (100 mL). The solution was washed with saturated sodium bicarbonate solution (50 mL) and brine (50 mL), and then dried over anhydrous sodium sulfate. The organic solvent was removed by a rotavapor, and the resulting residual was purified by column chromatography on silica gel (CH_2Cl_2/MeOH 30:1 to 10:1) to afford compound **5** as a colorless oil (2.10 g, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.60-3.50 (m, 34H), 3.37 (s, 3H), 3.35(s, 3H), 2.78(s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 172.5, 71.9, 71.8, 70.61, 70.58, 70.52, 70.49, 70.36, 69.5, 68.6, 59.0, 58.95, 48.0, 46.3, 42.5. HRMS (ESI): Calcd for $C_{25}H_{50}N_2O_{11}$ $[M+H]^+$: 555.3415. Found: 555.3469.

Compound 7. Compounds **5** (2.10 g, 4.60 mmol) and **6**[2] (1.33 g, 4.60 mmol) and triethylamine (0.65 mL, 4.60 mmol) were dissolved in acetonitrile (70 mL). The solution was stirred under

reflux for 20 h. After cooling to room temperature, the solvent was removed with rotavapor. The resulting residue was dissolved in chloroform (30 mL) and the solution was washed with water (10 mL) and brine (10 mL), and dried over sodium sulfate. Upon removal of the solvent, the resulting crude product was subjected to flash chromatography (CH_2Cl_2) to give compound **7** as a pale yellow oil (3.01 g, 95%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.38 (s, 2H), 9.33 (d, *J* = 4.0 Hz, 2H), 5.20 (s, 2H), 3.74-3.31 (m, 42H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.3, 161.2, 147.5, 132.1, 130.7, 130.4, 127.2, 125.1, 71.9, 71.0, 70.6, 70.5, 70.4, 69.4, 69.0, 59.0, 48.8, 46.8, 42.4. HRMS (ESI): Calcd for $C_{32}H_{44}N_4O_{15}$ $[M+Na]^+$: 747.2701. Found 747.2731.

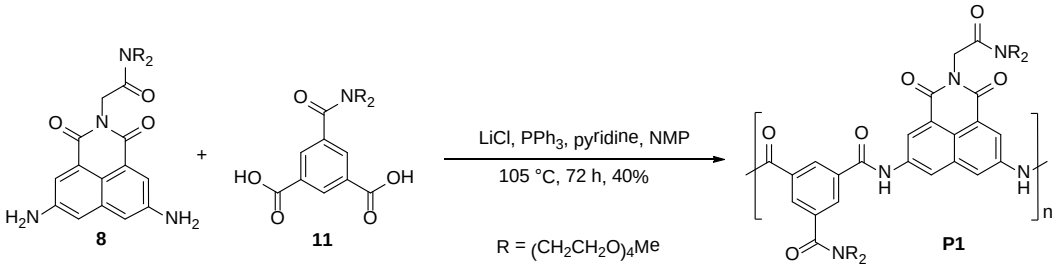
Compound 8. To a solution of compound **7** (3.00 g, 4.10 mmol) in methanol (25 mL) was added palladium on carbon (10 w%, 300 mg). The mixture was stirred at 60 °C for 15 h under an atmosphere of hydrogen gas (1 atm) and then cooled to room temperature. The solid was filtrated off and the solution was concentrated with a rotavapor. The resulting crude product was purified by flash chromatography on silica gel (CH_2Cl_2/MeOH 40:1 to 10:1) to give compound **8** (2.05 g, 75%) as a pale yellow solid. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 7.57 (d, *J* = 4.0 Hz, 2H), 6.95 (d, *J* = 4.0 Hz, 2H), 5.68 (s, 4H), 4.92 (s, 2H), 3.67-3.20 (m, 38H). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 167.2, 164.2, 148.1, 136.1, 122.7, 117.5, 115.0, 110.2, 71.7, 70.6, 70.3, 70.2, 70.1, 69.2, 68.6, 58.5, 47.9, 46.1, 41.6. HRMS (ESI): Calcd for $C_{32}H_{48}N_4O_{11}$ $[M+H]^+$: 664.3320. Found: 664.3360.



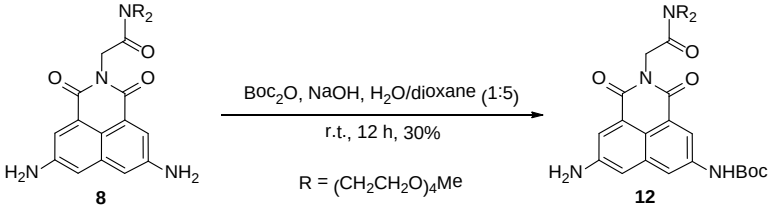
Compound 10. A mixture of compounds **2** (3.34 g, 8.40 mmol), **9** (2.00 g, 8.40 mmol) and EDCI (1.80 g, 9.20 mmol) and *N*-hydroxybenzotrizole (HOBt, 100 mg) in dichloromethane (30 mL) was stirred at room temperature for 12 h and then diluted with dichloromethane (100 mL). The solution was washed with water (2 × 30 mL) and brine (50 mL), and dried over sodium sulfate. After removal of the solvent under reduced pressure, the resulting residue was subjected to column chromatography (CH_2Cl_2/MeOH 15:1 to 10:1) to give compound **10** as a pale oil (2.00 g, 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.67 (d, *J* = 4.0 Hz, 1H), 8.27(s, 2H), 3.94 (s, 6H), 3.76-3.52 (m, 32H), 3.35 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.2, 165.5, 137.7, 132.3, 131.1, 130.8, 71.8, 70.5, 70.4, 69.1, 68.4, 58.9, 52.4, 49.8, 45.1. HRMS (ESI): Calcd for $C_{29}H_{47}NO_{13}$ $[M+H]^+$: 618.3126. Found: 618.3132.

Compound 11. To a stirred solution of compound **10** (1.00 g, 1.60 mmol) in THF (3 mL), methanol (3 mL), and water (10 mL) was added lithium hydroxide monohydrate (0.16 g, 3.20 mmol). The mixture was stirred at room temperature for 12 h and then water (20 mL) was added. The aqueous phase was extracted with ethyl acetate (10 mL) to remove the un-reacted starting material **10** and then acidified with 5% hydrochloric acid to pH = 3. The acidic aqueous solution was extracted with dichloromethane (3 × 100 mL). The combined organic phase was washed with water (60 mL) and brine (60 mL), and dried over sodium sulfate. The solvent was then removed under reduced pressure to give compound **11** as colorless oil (0.95 g, 95 %). ^{1}H NMR (400 MHz, DMSO-d_6) δ: 13.46 (s, 2H), 8.47 (s, 1H), 8.10 (s, 2H), 3.65-3.38 (m, 32H), 3.21 (s,

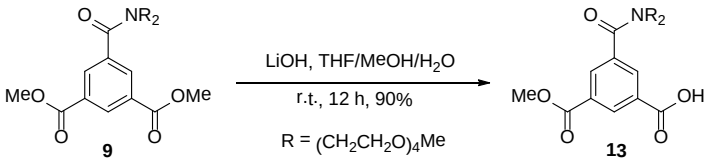
6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 169.9, 166.6, 138.4, 132.0, 130.6, 71.7, 70.3, 70.0, 68.3, 68.0, 58.5, 49.5, 44.4. HRMS (ESI): Calcd for $C_{27}H_{42}NO_{13}$ $[M–H]^-$: 588.2652: Found: 588.2973.



Polymer P1. A mixture of compounds **8** (0.44 g, 6.60 mmol), **11** (0.39 g, 6.60 mmol), lithium chloride (0.12 g), triphenylphosphine (0.41 g, 13.2 mmol), pyridine (0.3 mL), and NMP (1.2 mL) was stirred at 105 °C for 72 h. After cooling to room temperature, the solution was poured slowly into a stirred mixture of ethyl acetate (10 mL) and petroleum ether (10 mL). The yellow precipitate formed was filtrated, washed thoroughly with ethyl acetate and petroleum ether (1:1). The solid was re-dissolved in dichloromethane (20 mL) and washed with saturated sodium carbonate (5.0 mL), saturated ammonium chloride (5.0 mL) and brine (5.0 mL), and dried over sodium sulfate. The solvent was then removed under reduced pressure to afford polymer **P1** as a yellow solid (40%). M_w = 57903 g/mol, M_w/M_n =1.82. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 11.24 (s, 2H), 8.94 (br, 5H), 831 (br, 2H), 5.05 (s, 2H), 3.73-3.13 (m, 76H). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 170.2, 169.0, 167.7, 167.0, 165.3, 163.4, 138.9, 138.1, 135.2, 133.2, 129.9, 128.2, 124.1, 123.2, 122.7, 122.4, 121.8, 71.7, 70.7, 70.3, 70.2, 70.0, 69.2, 68.6, 68.5, 58.4, 49.6, 48.0, 46.1.

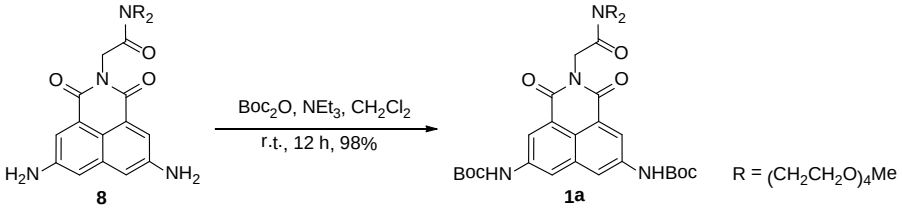


Compound 12. To a solution of compound **8** (1.00 g, 1.50 mmol) and di(tert-butyl) carbonate (0.33 g, 1.50 mmol) in 1,4-dioxane (25 mL) was added dropwise aqueous sodium hydroxide (60 mg, 1.50 mmol) (5 mL) in 2 h. The mixture was stirred at room temperature for another 12 h. The solution was concentrated with a rotavapor and the resulting residue was triturated with ethyl acetate (25 mL). The organic layer was washed with water (10 mL) and brine (10 mL), and dried over sodium sulfate. Upon removal of the solvent with a rotavapor, the resulting residue was subjected to column chromatography on silica gel (CH_2Cl_2/MeOH 40:1 to 20:1) to give compound **12** as a pale yellow solid (0.35 g, 30%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.18 (s, 1H), 7.93 (s, 1H), 7.78 (s, 1H), 7.16 (s, 2H), 5.02 (s, 2H), 3.72-3.61 (m, 32H), 3.38 (s, 3H), 3.35 (s, 3H), 1.56 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.7, 163.9, 163.8, 152.9, 146.0, 137.4, 134.1, 122.5,122.3, 120.2, 119.9, 118.8, 118.3, 113.4, 4, 71.9, 71.0, 70.6, 70.5, 70.4, 59.0, 48.7, 47.0, 41.5, 28.4. HRMS (ESI): Calcd for $C_{37}H_{56}N_4O_{13}$ $[M+H]^+$: 765.3844. Found: 765.3924. Calcd for $C_{37}H_{56}N_4O_{13}Na$: $[M+ Na]^+$: 787.3742. Found: 787.3750.



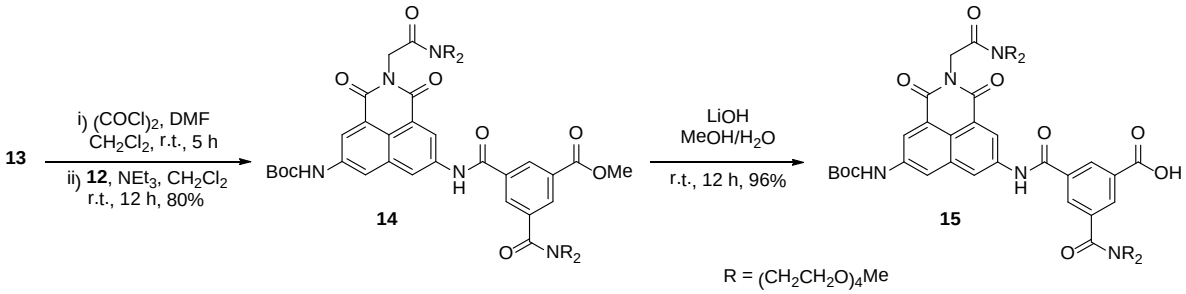
9 **13**

Compound 13. To a stirred solution of compound **9** (1.00 g, 1.60 mmol) in THF (10 mL), methanol (10 ml), and water (30 ml) was added lithium hydroxide monohydrate (78 mg, 1.60 mmol). The mixture was stirred at room temperature for 12 h and then concentrated with a rotavapor. The residue was triturated with water (10 mL) and the mixture was extracted with ethyl acetate (10 mL) to remove the un-reacted starting material **9**. The aqueous solution was then acidified with hydrochloric acid (1 M) to pH = 3. The acidic aqueous solution was extracted with dichloromethane (3 × 30 mL). The combined organic phase was washed with water (30 mL) and brine (30 mL), and dried over sodium sulfate. The solution was then concentrated under reduced pressure to give compound **13** as colorless oil (0.90 g, 90%). ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.48 (s, 1H), 8.13 (s, 2H), 3.90 (s, 3H), 3.65-3.23 (m, 32H), 3.21 (s, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 169.8, 166.4, 165.5, 138.6, 132.4, 132.1, 131.9, 130.7, 130.4, 71.7, 70.3, 70.0, 68.3, 68.0, 58.5, 53.1, 49.5, 44.4. HRMS (ESI): Calcd for $C_{28}H_{45}NO_{13}$ $[M+H]^+$: 604.2969. Found: 604.2975.



8 **1a**

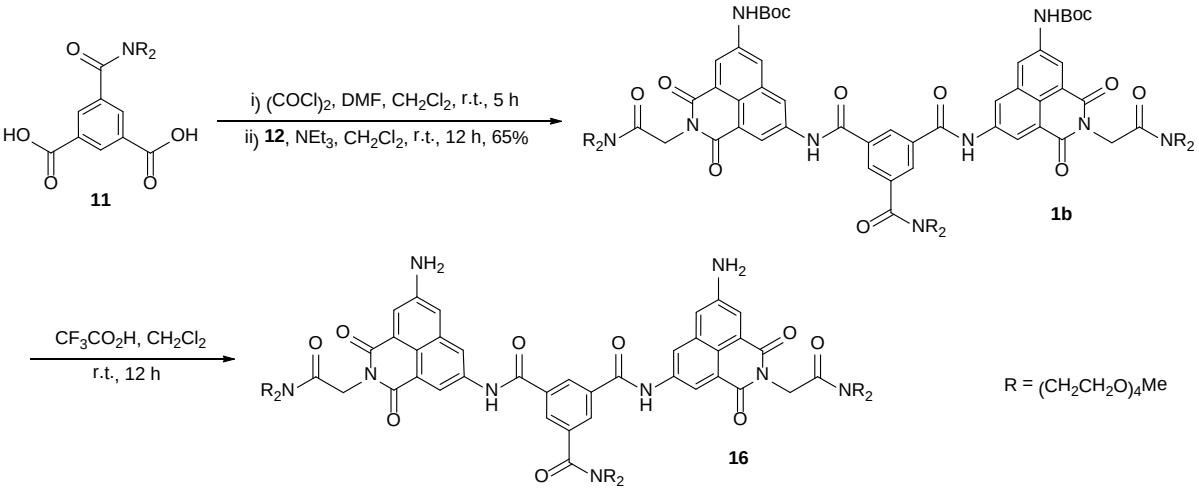
Compound 1a: To a solution of compounds **8** (66.0 mg, 0.10 mmol) and di(tert-butyl) carbonate (55 mg, 0.25 mmol) in dichloromethane (5 mL) was added triethylamine (35 μL, 0.25 mmol). The solution was stirred at room temperature for 12 h and another part of dichloromethane (25 mL) was added. The solution was washed with water (2 × 20 mL) and brine (20 mL), and dried over sodium sulfate. The solvent was then removed with a rotavapor. The resulting residue was purified by column chromatography on silica gel (CH_2Cl_2/MeOH 20:1) (85 mg, 98%) to give compound **1a** as a pale yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.01 (d, J = 4.0 Hz, 2H), 7.85 (s, 2H), 7.80 (s, 2H), 4.93 (s, 2H), 3.72-3.58 (m, 33H), 3.3 (s, 3H), 3.30 (s, 3H), 1.53 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 167.7, 163.3, 152.7, 137.7, 133.2, 122.3, 121.9, 120.7, 119.5, 80.7, 71.9, 71.0, 70.6, 70.5, 70.4, 69.5, 69.2, 59.0, 48.8, 47.2, 41,4, 28.4. HRMS (ESI): Calcd for $C_{42}H_{64}N_4O_{15}$ $[M+H]^+$: 865.4446. Found: 865.4469.



14 **15**

Compound 14. Oxalyl chloride (63 μL, 0.70 mmol) was added to the solution of compound **13** (86 mg, 0.14 mmol) in dichloromethane (3 mL) in the presence of catalytic amount of DMF (5 μL). The mixture was stirred at room temperature for 5 h and then concentrated under reduced pressure. The resulting residue was re-dissolved in dichloromethane (3 mL) and the solution was added dropwise to the solution of compound **12** (0.11 g, 0.14 mmol) and triethylamine (71 mg, 0.70 mmol) in dichloromethane (3 mL). The solution was stirred at room temperature for 12 h and then diluted with another part of dichloromethane (25 mL). The solution was washed with water (2 × 25 mL) and brine (25 m), and dried over sodium sulfate. The solvent was then removed with a rotavapor and the resulting residue was subjected to column chromatography on silica gel (CH_2Cl_2/MeOH 40:1 to 20:1) to give **14** as a colorless oil (76 mg, 40%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.98 (s, 1H), 8.68 (s, 2H), 8.45 (s, 1H), 8.40 (s, 1H), 8.33 (s, 1H), 8.18 (s, 1H), 8.13 (s, 1H), 8.12 (s, 1H), 7.49 (s, 1H), 5.05 (s, 2H), 3.96 (s, 2H), 3.66-3.33 (m, 79H), 1.56 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 170.8, 167.7, 165.9, 165.2, 163.5, 152.7, 137.8, 135.7, 133.4, 131.2, 130.5, 129.8, 123.8, 122.5, 121.6, 120.1, 81.0, 71.9, 71.7, 71.0, 70.6, 70.5, 70.4, 70.1, 69.4, 69.2, 67.9, 59.0, 58.6, 52.4, 50.6, 48.8, 47.7, 47.1, 45.0, 41.5, 37.8, 28.4. HRMS (ESI): Calcd for $C_{65}H_{99}N_5O_{25}$ $[M+Na]^+$: 1372.6527. Found: 1372.6535.

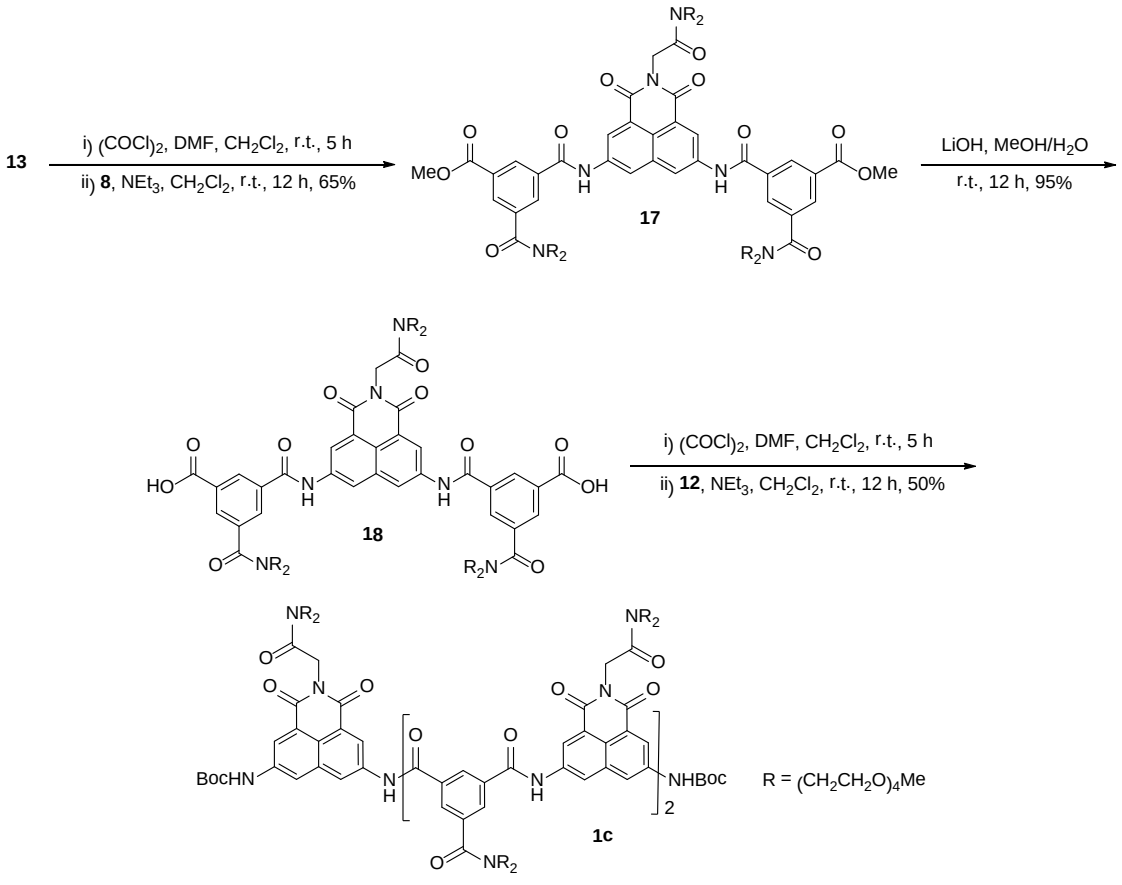
Compound 15. To a stirred solution of compound **14** (50 mg, 0.037 mmol) in methanol (3 ml) and water (3 ml) was added lithium hydroxide monohydrate (1.6 mg, 0.040 mmol). The mixture was stirred at room temperature for 12 h and then diluted with water (10 mL). The solution was acidified with 5% hydrochloric acid to pH = 3. The acidic aqueous solution was extracted with dichloromethane (3 × 10 mL). The combined organic phase was washed with water (15 mL) and brine (15 mL), and dried over sodium sulfate. The solvent was then removed under reduced pressure to give compound **15** as a yellow oil (48 mg, 96%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.3 (s, 1H), 8.47-8.20 (m, 7H), 8.16 (s, 1H), 4.96 (s, 2H), 3.78-3.25 (m, 78H), 1.59 (s, 9H). HRMS (ESI): Calcd for $C_{64}H_{97}N_5O_{25}$ $[M+Na]^+$: 1358.6370. Found: 1358.6392.



Compound 1b. Oxalyl chloride (0.12 mL, 1.35 mmol) was added to the solution of compound **11** (0.16 g, 0.27 mmol) in dichloromethane (3 mL) in the presence of catalytic amount of DMF (5 μL). The mixture was stirred at room temperature for 5 h and then concentrated under reduced pressure. The resulting residue was re-dissolved in dichloromethane (3 mL). The solution was added slowly to the solution of compound **12** (71 mg, 0.11 mmol) and triethylamine (0.11 mL, 1.35 mmol) in dichloromethane (3 mL). The solution was stirred at

room temperature for 12 h and then diluted with dichloromethane (25 mL). The solution was washed with water (15 mL) and brine (15 mL), and dried over sodium sulfate. The solvent was then removed under reduced pressure and the resulting residue was subjected to column chromatography on silica gel (CH_2Cl_2/MeOH 40:1 to 20:1) to give compound **1b** (0.15 g, 65%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.29 (s, 1H), 8.47-8.19 (m, 13H), 5.01 (s, 2H), 3.80-3.25 (m, 123H), 1.56 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 171.2, 168.4, 166.8, 164.9, 163.2, 153.0, 137.9, 137.4, 137.0, 135.4, 132.9, 131.1, 123.6, 123.0, 122.4, 121.9, 121.2, 71.9, 70.7, 70.5, 70.4, 69.3, 58.9, 55.0, 48.9, 47.5, 41.3, 28.4. HRMS (ESI): Calcd for $C_{101}H_{151}N_9O_{37}$ $[M+Na]^+$: 2106.0142. Found: 2106.0149.

Compound 16. To the solution of compound **1b** (75 mg, 0.036 mmol) in dichloromethane (5.0 mL) was added trifluoroacetic acid (1 mL). The solution was stirred at room temperature for 12 h and then concentrated under reduced pressure. The resulting residue was re-dissolved in dichloromethane (20 mL) and the solution was washed with saturated sodium bicarbonate solution (5 mL), water (5 mL) and brine (5 mL), and then dried over sodium sulfate. The solvent was then removed with a rotavapor to give compound **16** as a pale yellow solid. The resulting yellow solid was used in the next step without further purification.

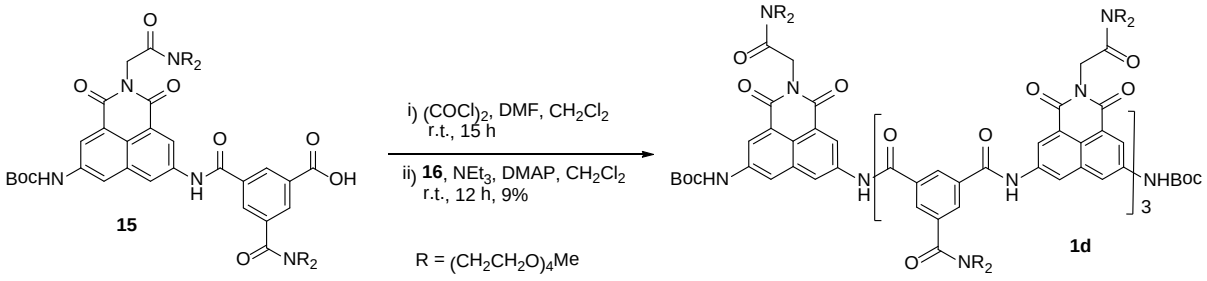


Compound 17. Oxalyl chloride (12 μL, 0.14 mmol) was added to the solution of compound **13** (42 mg, 0.028 mmol) in dichloromethane (3 mL) in the presence of catalytic amount of DMF (5 μL). The mixture was stirred at room temperature for 5 h and then concentrated under reduced pressure. The resulting residue was re-dissolved in dichloromethane (3 mL). The solution was added slowly to the solution of compound **8** (44 mg, 0.061 mmol) and triethylamine (20 μL,

0.14 mmol) in dichloromethane (3 mL). The solution was stirred at room temperature for 12 h and then diluted with dichloromethane (25 mL). The solution was washed with water (15 mL) and brine (15 mL), and dried over sodium sulfate. After the solvent was removed with a rotavapor, the resulting crude product was purified by column chromatography on silica gel (CH_2Cl_2/MeOH 20:1 to 10:1) to give compound **17** as a colorless oil (40 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ:10.0 (s, 2H), 8.97 (s, 2H), 8.71 (s, 2H), 8.56 (s, 2H), 8.51 (s, 2H), 8.16 (s, 2H), 5.09 (s, 2H), 3.95-3.30 (m, 135H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 169.9, 166.3, 164.9, 164.4, 162.7, 136.9, 134.6, 132.4, 130.1, 129.6, 129.5, 129.0, 123.3, 122.0, 121.7, 121.5, 70.9, 70.6, 69.6, 69.5, 69.0, 68.4, 56.0, 57.5, 51.5, 49.1, 47.7, 46.0, 43.8, 40.6. HRMS (ESI): Calcd for $C_{88}H_{134}N_6O_{35}$ $[M+H]^+$: 1835.8968. Found: 1835.8970.

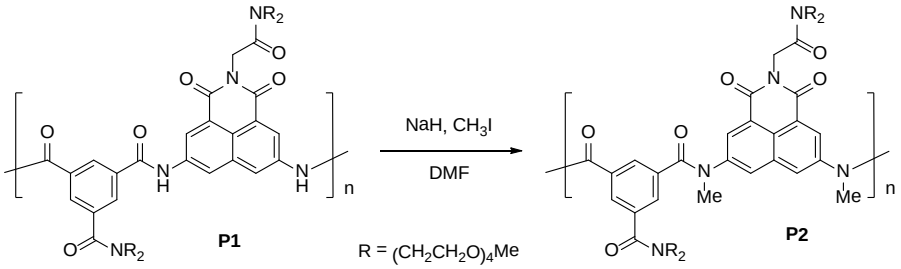
Compound 18. To a stirred solution of compound **17** (40 mg, 0.022 mmol) in methanol (3 mL) and water (3 mL) was added lithium hydroxide monohydrate (1.9 mg, 0.044 mmol). The mixture was stirred at room temperature for 12 h and then diluted with water (10 mL). The aqueous phase was acidified with 5% hydrochloric acid to pH = 3. The acidic aqueous solution was extracted with DCM (3 × 20 mL). The combined organic phase was washed with water (20 mL) and brine (20 mL), and dried over sodium sulfate. The solvent was then removed under reduced pressure to give compound **18** as colorless oil (38 mg, 95%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.3 (s, 1H), 8.62 (s, 1H), 8.58-8.23 (br, 10H), 5.02 (s, 2H), 3.78-3.30 (m, 146H). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 171.0, 168.5, 166.4, 165.0, 163.1, 137.2, 135.3, 132.7, 131.5, 131.2, 129.2, 124.7, 123.5, 122.0, 71.9, 70.6, 70.4, 69.4, 69.1, 68.7, 59.0, 50.0, 49.0, 47.7, 45.3, 41.4. HRMS (ESI): Calcd for $C_{86}H_{130}N_6O_{35}$ $[M+Na]^+$: 1829.8475. Found: 1829.8468.

Compound 1c. Oxalyl chloride (12 μL, 0.10 mmol) was added to the solution of compound **18** (38 mg, 0.021 mmol) in dichloromethane (3 mL) in the presence of catalytic amount of DMF (5 μL). The mixture was stirred at room temperature for 5 h and then concentrated under reduced pressure. The resulting residue was re-dissolved in dichloromethane (3 mL) and the solution was added slowly to a solution of compound **12** (41 mg, 0.053mmol) and triethylamine (14 μL, 0.10 mmol) in dichloromethane (3 mL). The solution was stirred at room temperature for 12 h and then diluted with dichloromethane (25 mL). The solution was washed with water (15 mL) and brine (15 mL), and dried over sodium sulfate. The solvent was then removed with a rotavapor and the resulting residue was subjected to column chromatography on silica gel (CH_2Cl_2/MeOH 20:1 to 7:1) to give compound **1c** as a colorless oil (34 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.22 (s, 4H), 8.79-7.99 (m, 18H), 5.34 (s, 2H), 3.81-3.24 (m, 176H), 1.57 (s, 9H). HRMS (ESI): Calcd for $C_{160}H_{238}N_{14}O_{59}$ $[M+2Na]^{2+}$: 1673.2941. Found: 1673.2970.



Compound 1d. Oxalyl chloride (4 μL, 0.045 mmol) was added to a solution of compound **15** (40 mg, 0.03 mmol) in dichloromethane (3 mL) in the presence of catalytic amount of DMF (5

μM). The mixture was stirred at room temperature for 15 h and then concentrated under reduced pressure. The resulting residue was re-dissolved in dichloromethane (3 mL). The solution was slowly added to a solution of compound **16** (23 mg, 0.012 mmol), 4-dimethylaminopyridine (DMAP, 0.73 mg, 0.006 mmol), and triethylamine (10 μL, 0.06 mmol) in dichloromethane (3 mL). The solution was stirred at room temperature for 12 h and then diluted with dichloromethane (15 mL). The solution was then washed with water (10 mL) and brine (10 mL), and dried over sodium sulfate. The solvent was then removed under reduced pressure and the resulting residue was subjected to column chromatography on silica gel (CH_2Cl_2/MeOH 20:1 to 7:1) to give compound **1d** as a colorless oil (13 mg, 9.0%). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 10.42 (s, 8H), 8.66-8.31 (m, 25H), 5.04 (s, 8H), 3.81-3.24 (m, 244H), 1.56 (s, 18H). HRMS (ESI): Calcd for $C_{219}H_{325}N_{19}O_{81}$ $[M+3NH_4]^{3+}$: 1524.4332. Found: 1524.4345.



Polymer P2. To a solution of **P1** (52 mg) in dry dimethylformamide (4.0 mL) at 0 °C was added sodium hydride (60wt%, 4.8 mg, 0.12 mmol) was added. The mixture was stirred for 1 h at room temperature and then methyl iodide (12.0 μL, 0.20 mmol) was added. The mixture was stirred for another 12 h at room temperature and then aqueous ammonium chloride solution (0.2 M, 4 mL) was added. The mixture was extracted with dichloromethane (3×10 mL). The combined organic layer was washed with water (2×10 mL), saturated aqueous ammonium chloride solution (10 mL) and brine (10 mL), and dried over anhydrous sodium sulfate. The solution was then concentrated under reduced pressure to about 0.5 mL. To the resulting solution was added slowly n-hexane (5 mL). The precipitate formed was collected by filtration and dried in vacuo to give polymer **P2** (24 mg, 45%) as a yellow solid. On the basis of the integration intensity of the NCH_2 signal and the unreacted NH signal, we calculated that the conversion yield from NH to NMe was about 90%.

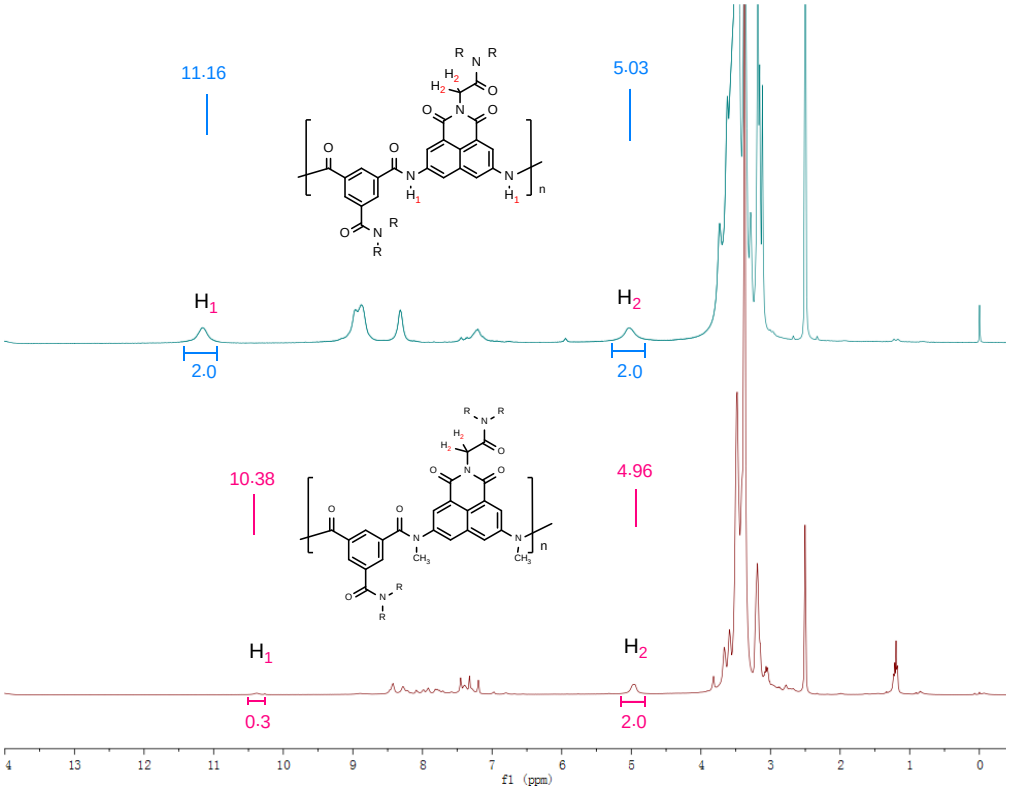


Fig. S1 ^{1}H NMR spectrum of **P1** and **P2** in DMSO-d_6 at 25 °C.

Molecular modeling and computational method: Polymer **P1** (54 aromatic subunits) was constructed using Polymer Builder module in the MS Modeling software (version 7.0, Accelrys, Inc., San Diego, CA)[3] to satisfy the helical parameters (3.5 Å for the aromatic stacking and about subunits per turn). The structure was then optimized with the semiempirical PM6 calculation method using the GAUSSIAN 09 program.[4]

References:

(1) C. Selve, J. C. Ravey, M. J. Stebe, C. El Moudjahid, E. M. Moumni and J. I. Delpuech, *Tetrahedron*, 1991, **47**, 411.
(2) S. Girouard, M.-H. Houle, A. Grandbois, J. W. Keillor and S. W. Michnick, *J. Am. Chem. Soc.*, 2005, **127**, 559.
(3) M. Banno, Y. Yamaguchi, Y. Nagai, C. Kaiser, S. Hecht and E. Yashima, *J. Am. Chem. Soc.*, 2012, **134**, 8718.
(4) J. X. Cao, M. Kline, Z. Z. Chen, B. Luan, M. L. Lü, W. R. Zhang, C. X. Lian, Q. W. Wang, Q. F. Huang, X. X. Wei, J. E. Deng, J. Zhu and B. Gong, *Chem. Commun.*, 2012, **48**, 11112.

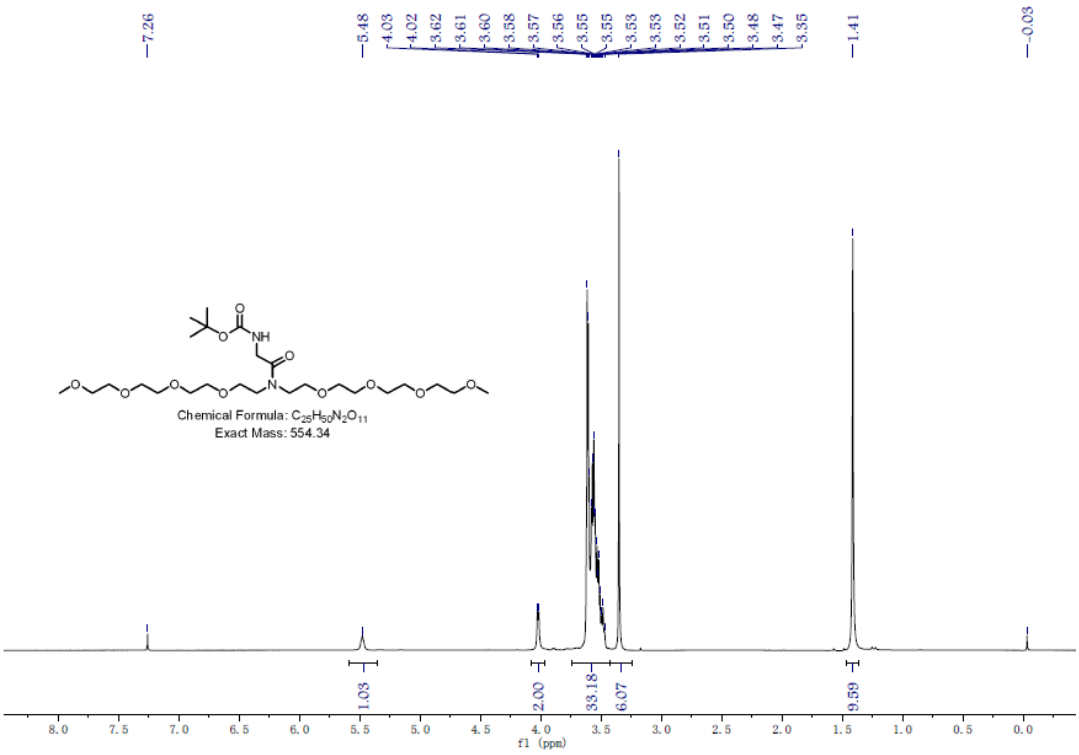


Fig. S2 ^{1}H NMR spectrum (400 MHz) of compound **4** in $CDCl_3$ at 25 °C.

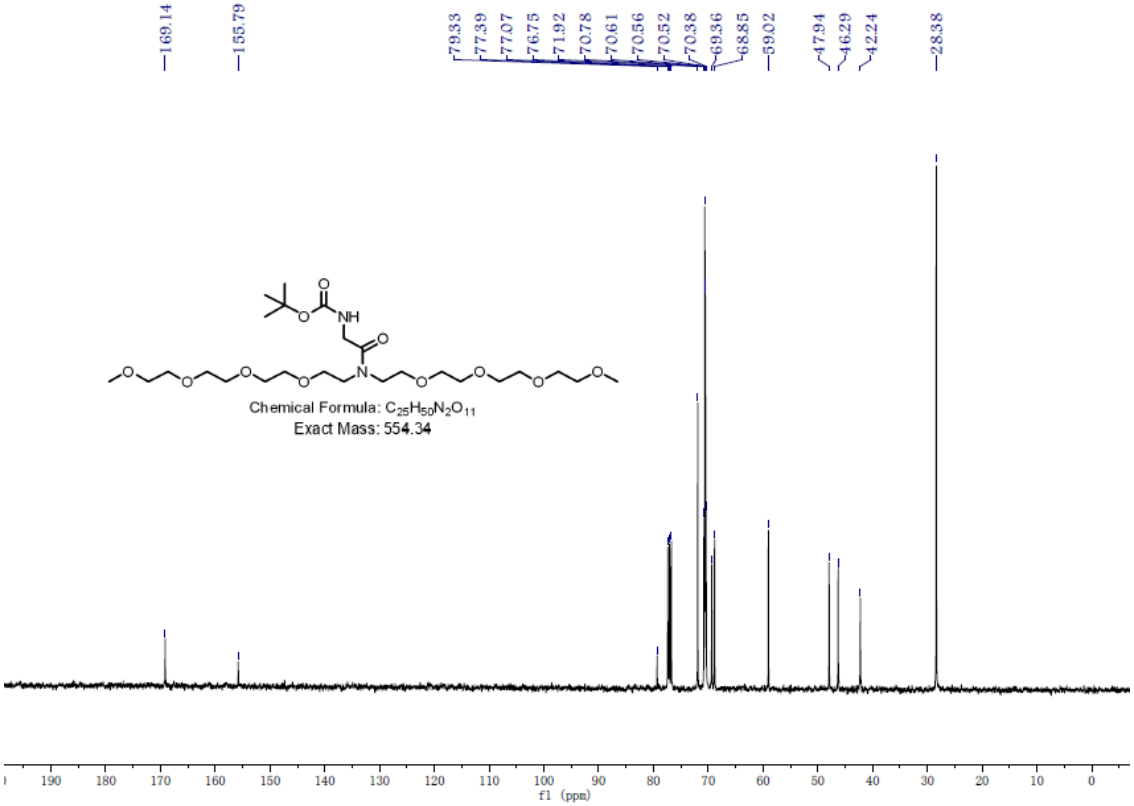


Fig. S3 ^{13}C NMR spectrum (100 MHz) of compound **4** in $CDCl_3$ at 25 °C.

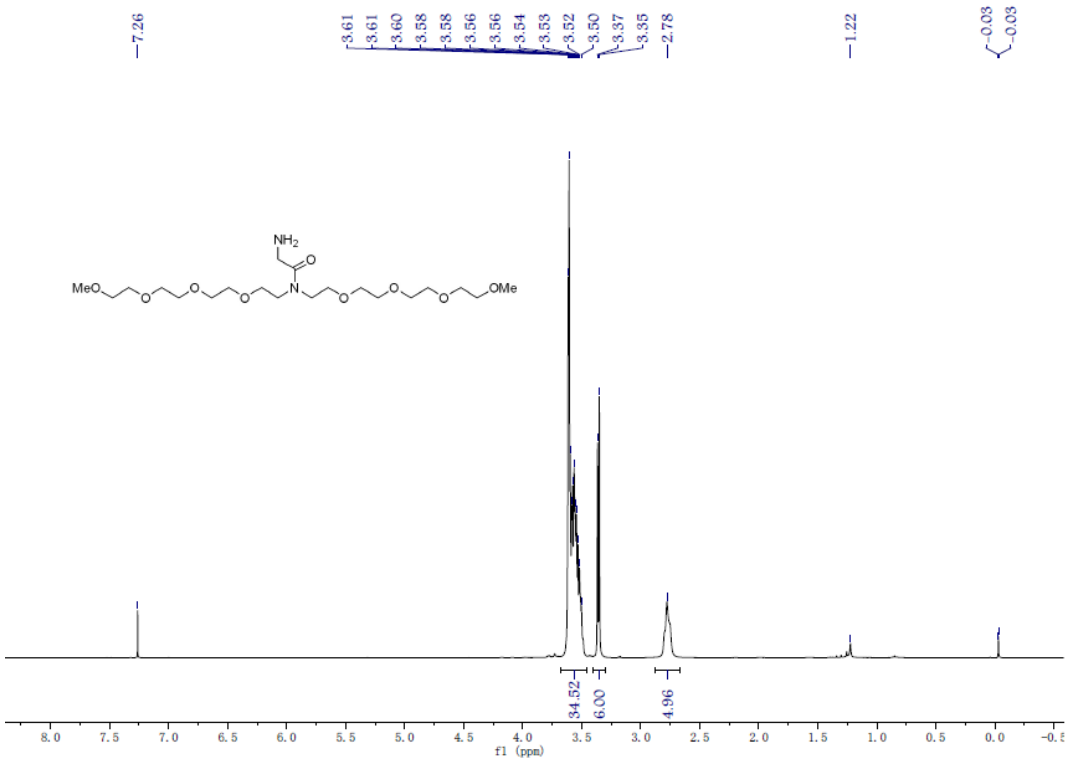


Fig. S4 ^{1}H NMR spectrum (400 MHz) of compound **5** in $CDCl_3$ at 25 °C.

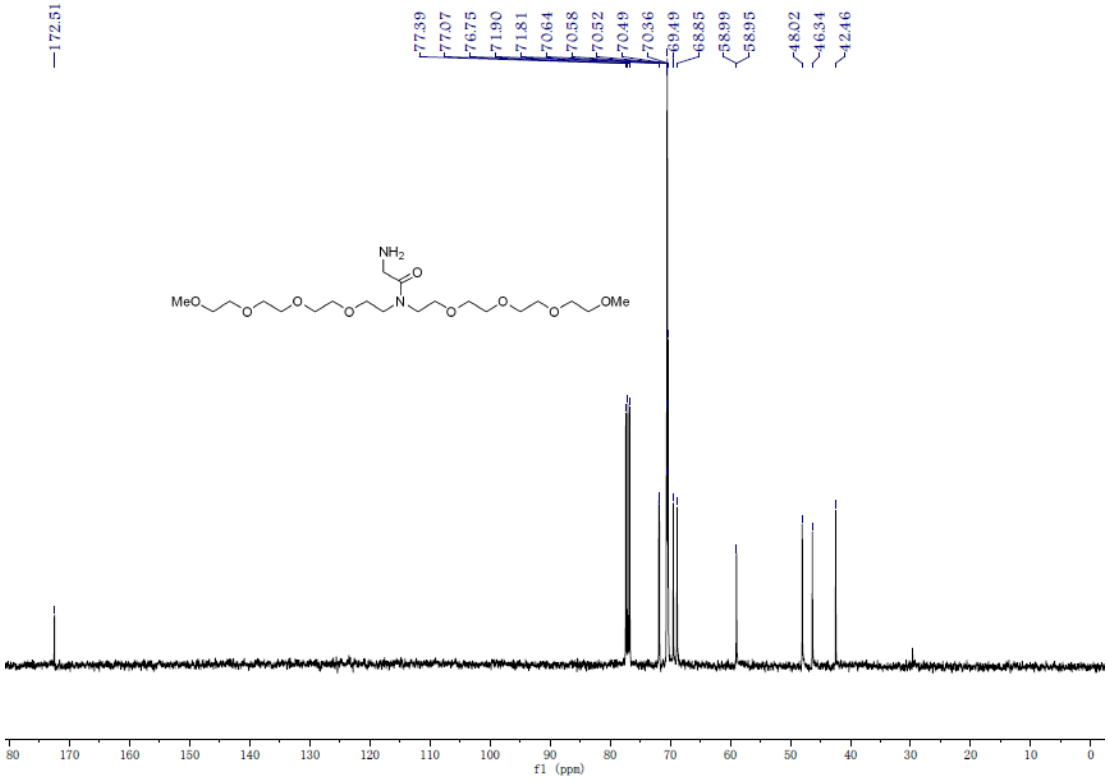


Fig. S5 ^{13}C NMR spectrum (100 MHz) of compound **5** in $CDCl_3$ at 25 °C.

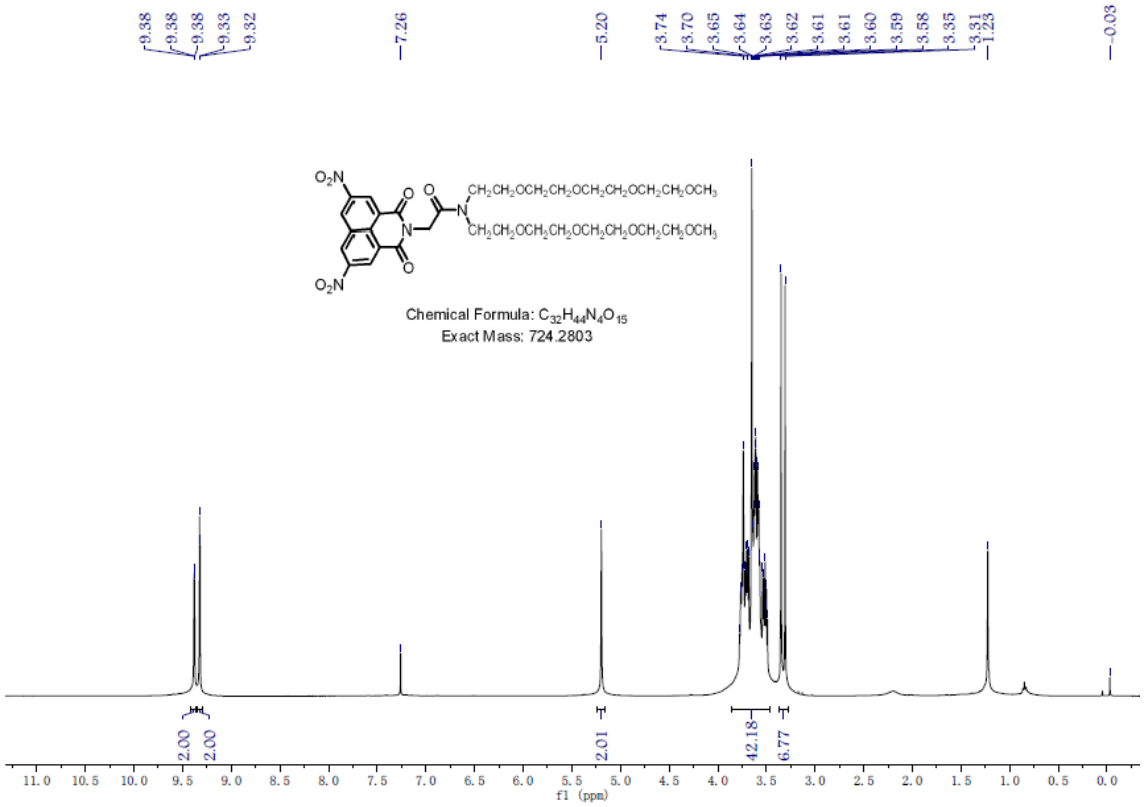


Fig. S6 ^{1}H NMR spectrum (400 MHz) of compound **7** in $CDCl_3$ at 25 °C.

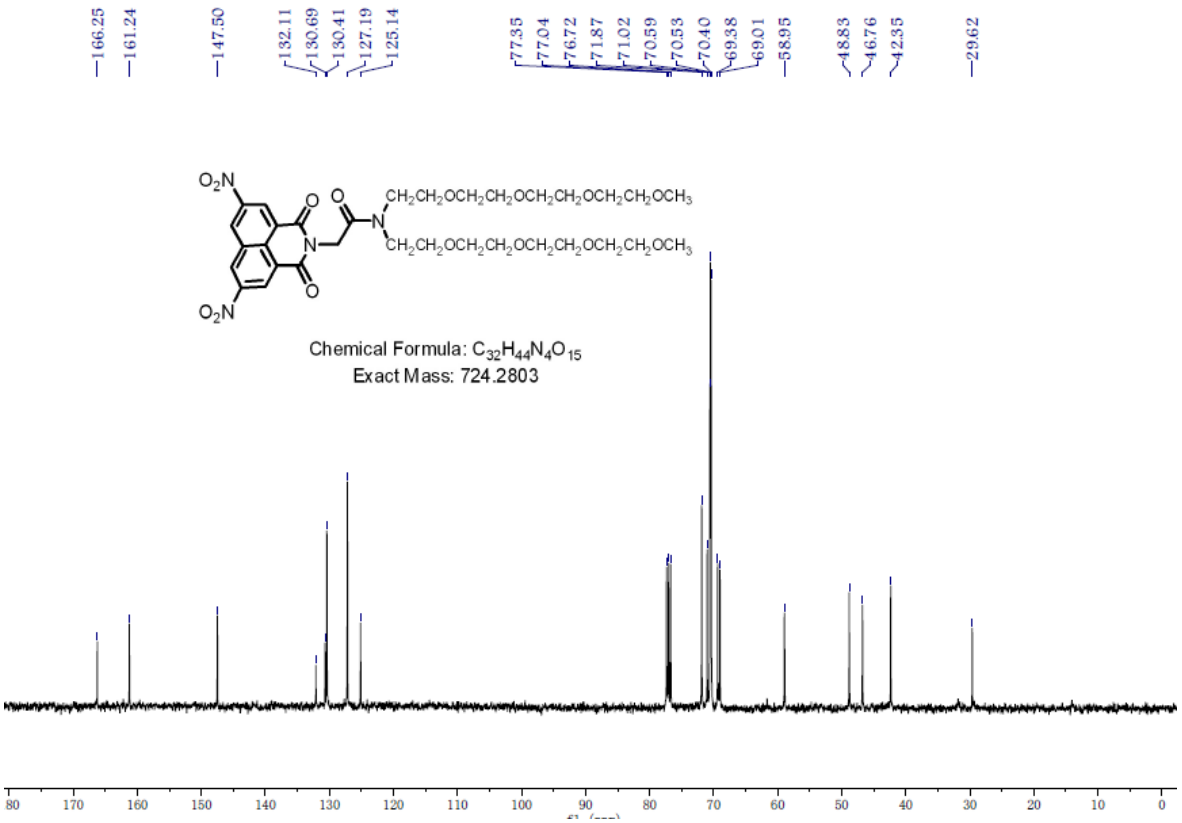


Fig. S7 ^{13}C NMR spectrum (100 MHz) of compound **7** in $CDCl_3$ at 25 °C.

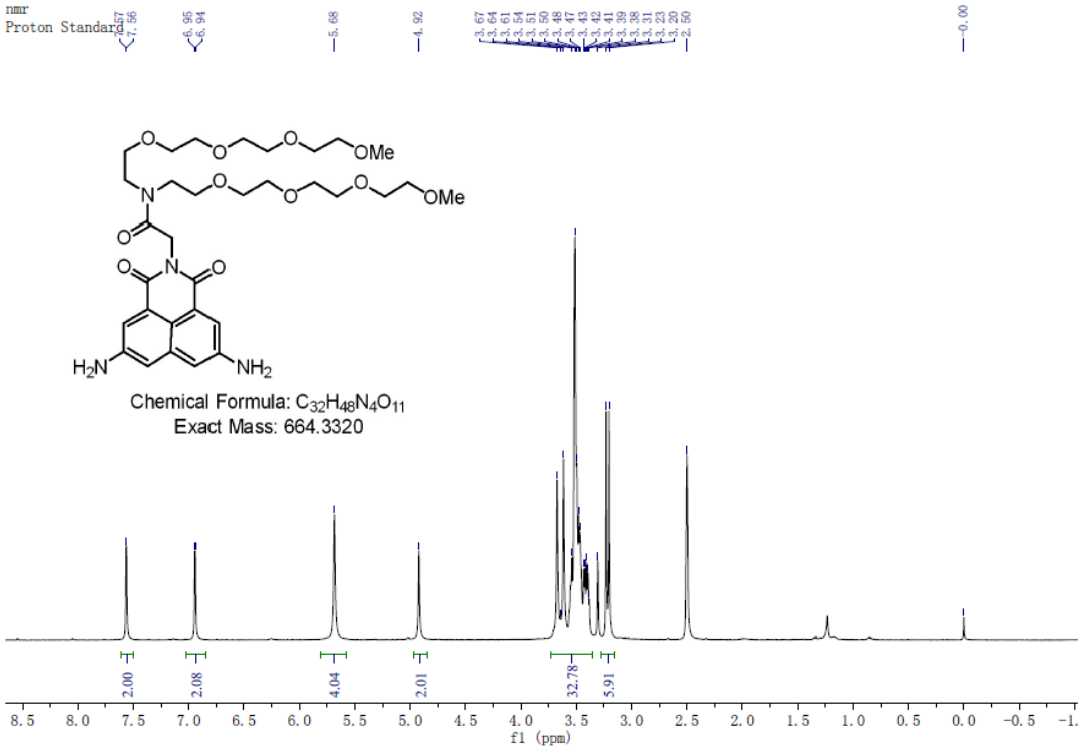


Fig. S8 ^{1}H NMR spectrum (400 MHz) of compound **8** in DMSO-d_6 at 25 °C.

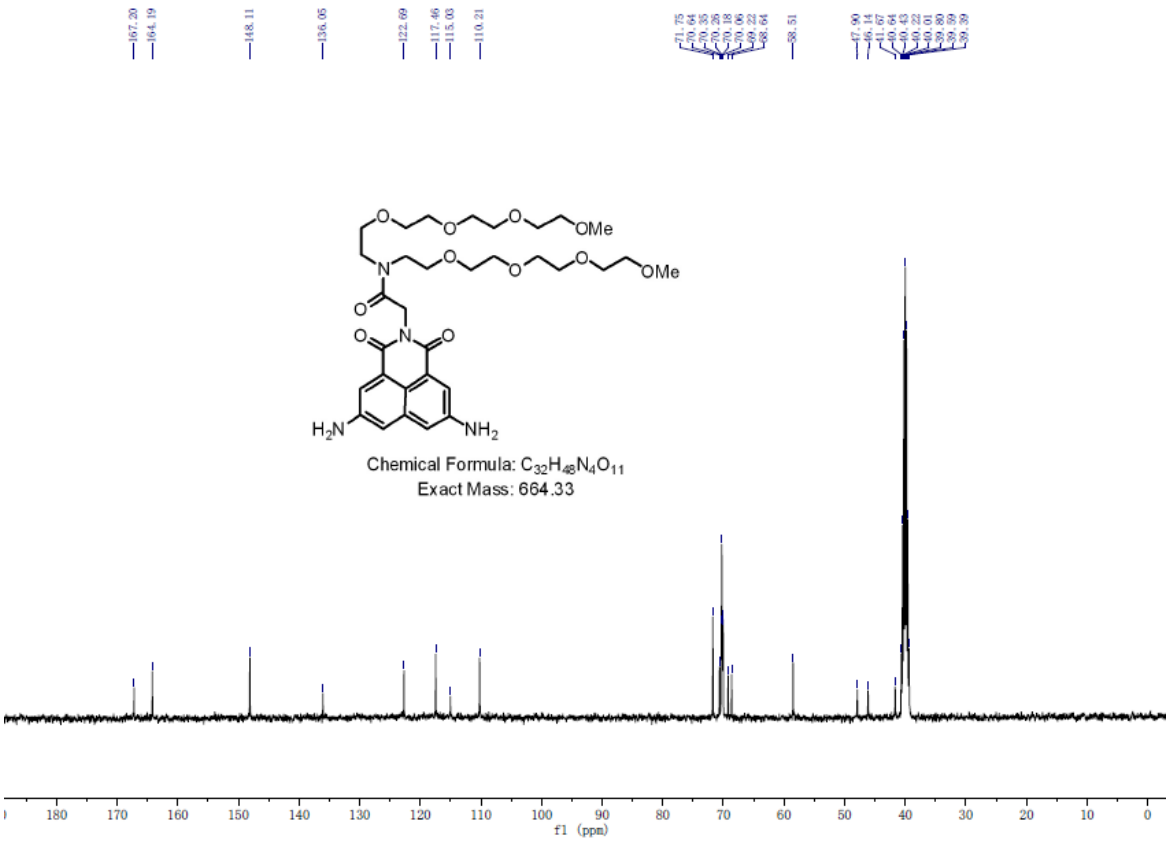


Fig. S9 ^{13}C NMR spectrum (100 MHz) of compound **8** in DMSO-d_6 at 25 °C.

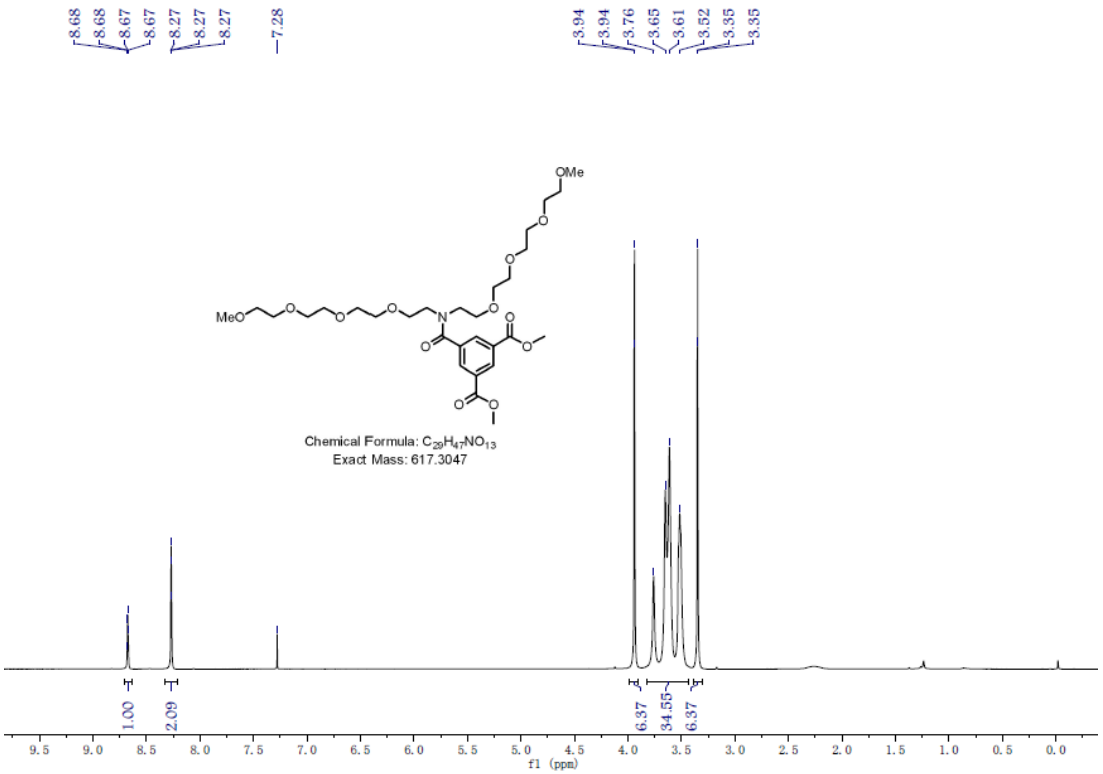


Fig. S10 ^{1}H NMR spectrum (400 MHz) of compound **10** in $CDCl_3$ at 25 °C.

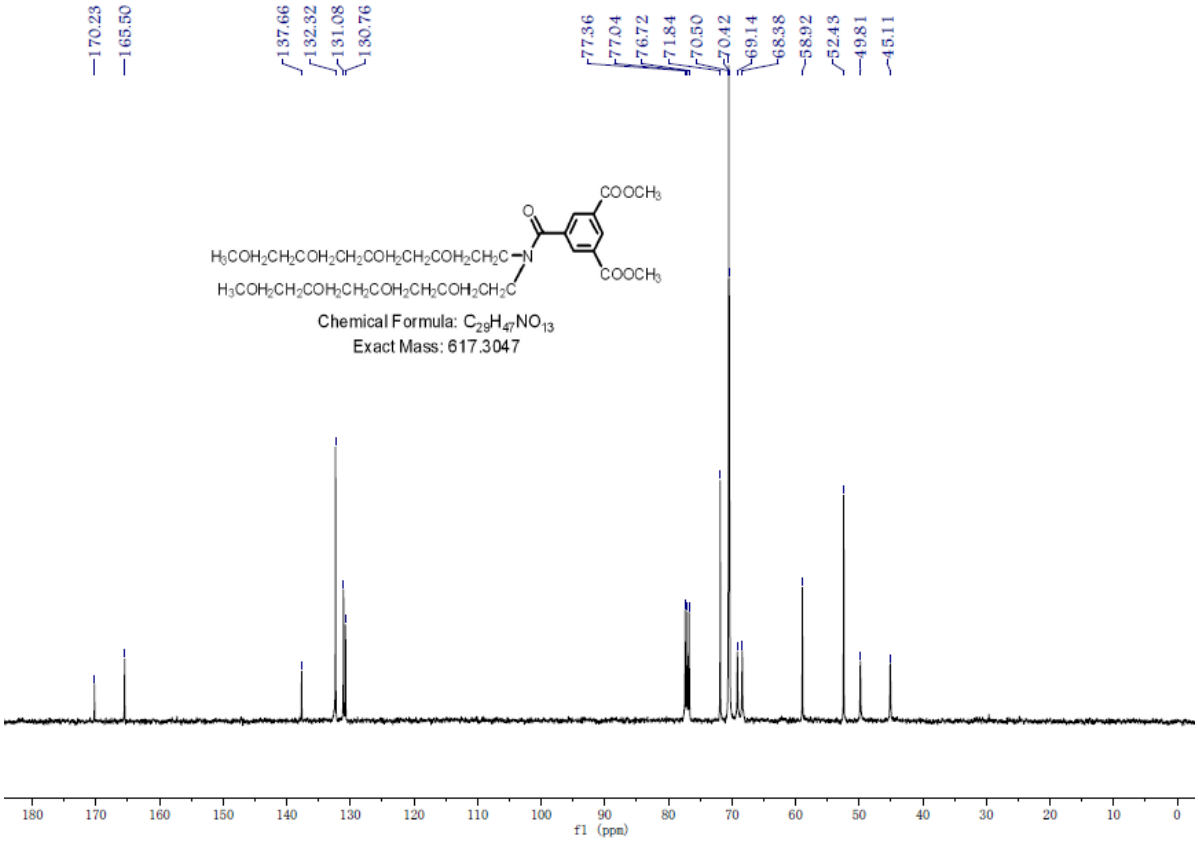


Fig. S11 ^{13}C NMR spectrum (100 MHz) of compound **10** in $CDCl_3$ at 25 °C.

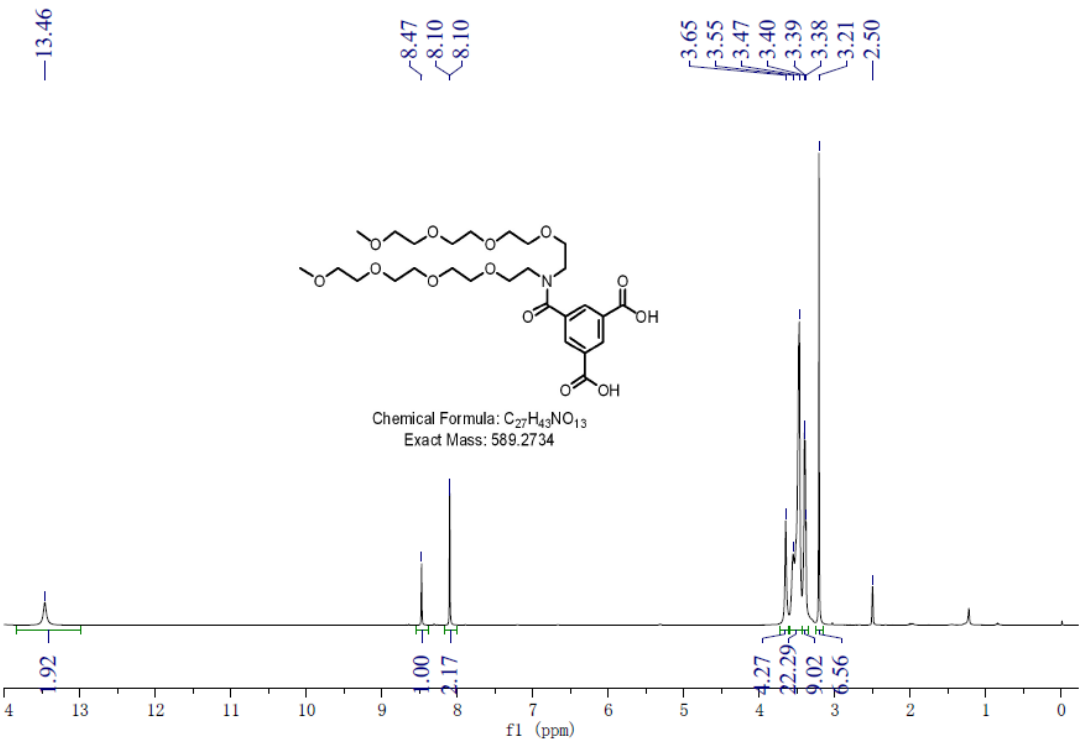


Fig. S12 ^{1}H NMR spectrum (400 MHz) of compound **11** in DMSO-d_6 at 25 °C.

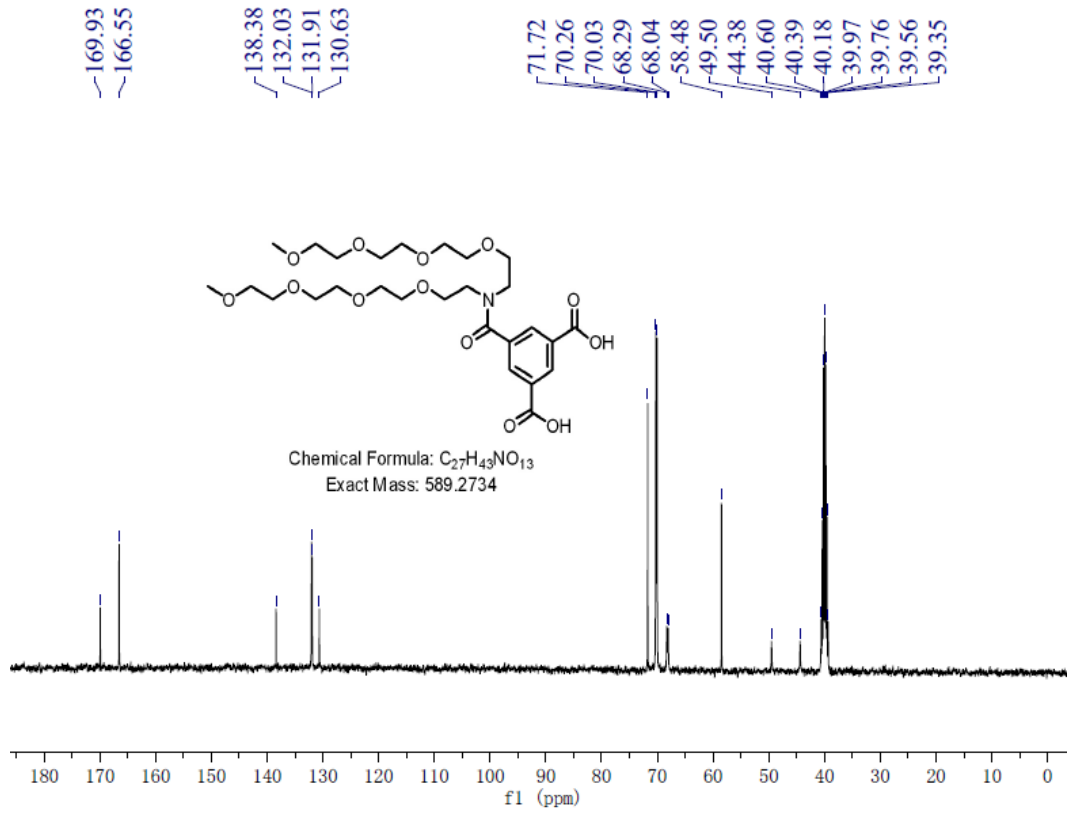


Fig. S13 ^{13}C NMR spectrum (100 MHz) of compound **11** in DMSO-d_6 at 25 °C.

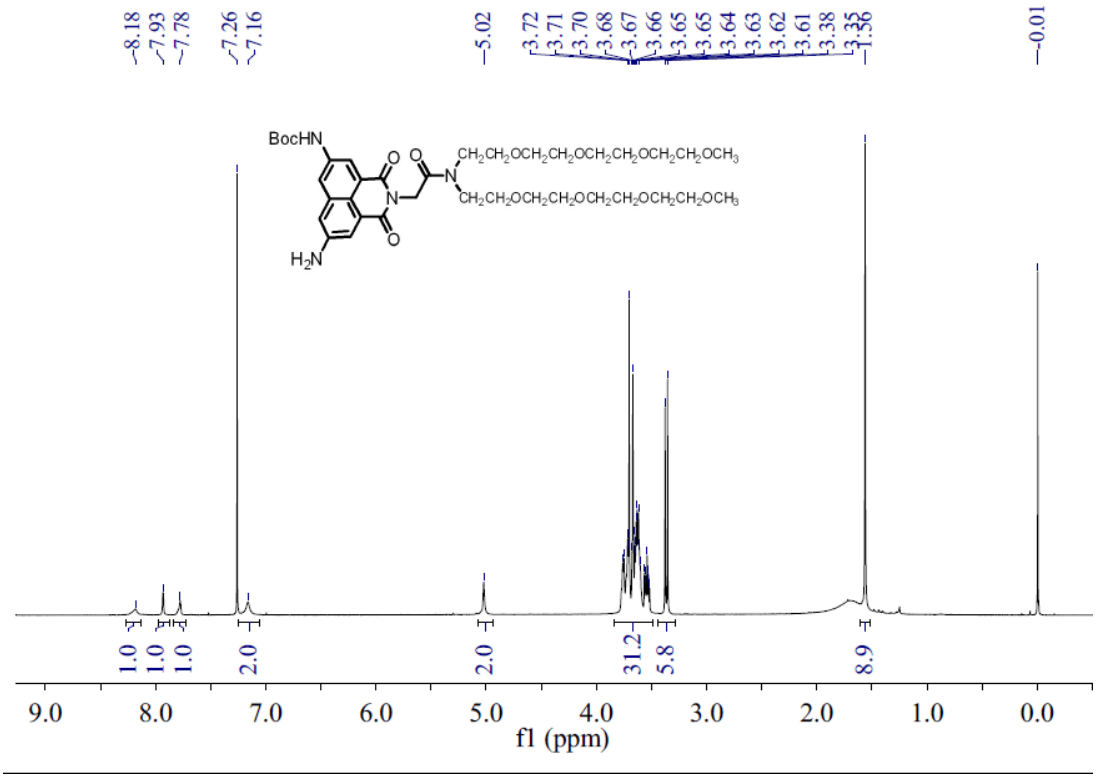


Fig. S14 ^{1}H NMR spectrum (400 MHz) of compound **12** in $CDCl_3$ at 25 °C.

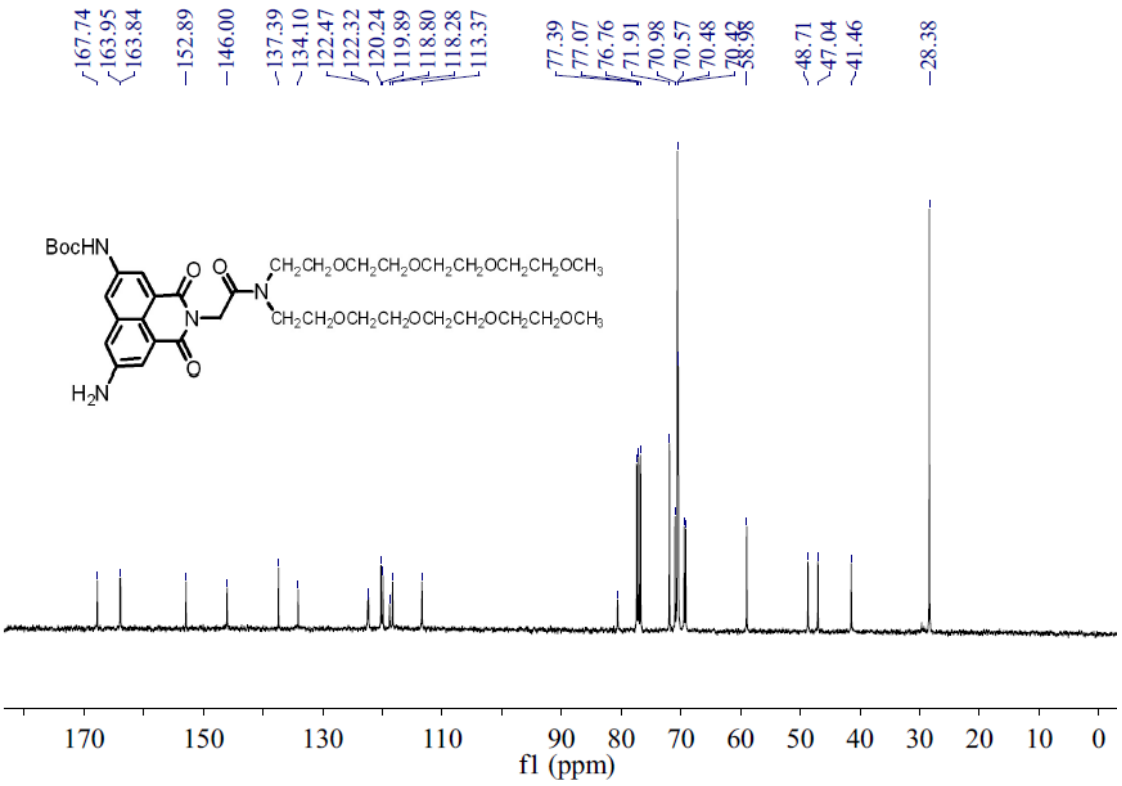


Fig. S15 ^{13}C NMR spectrum (100 MHz) of compound **12** in $CDCl_3$ at 25 °C.

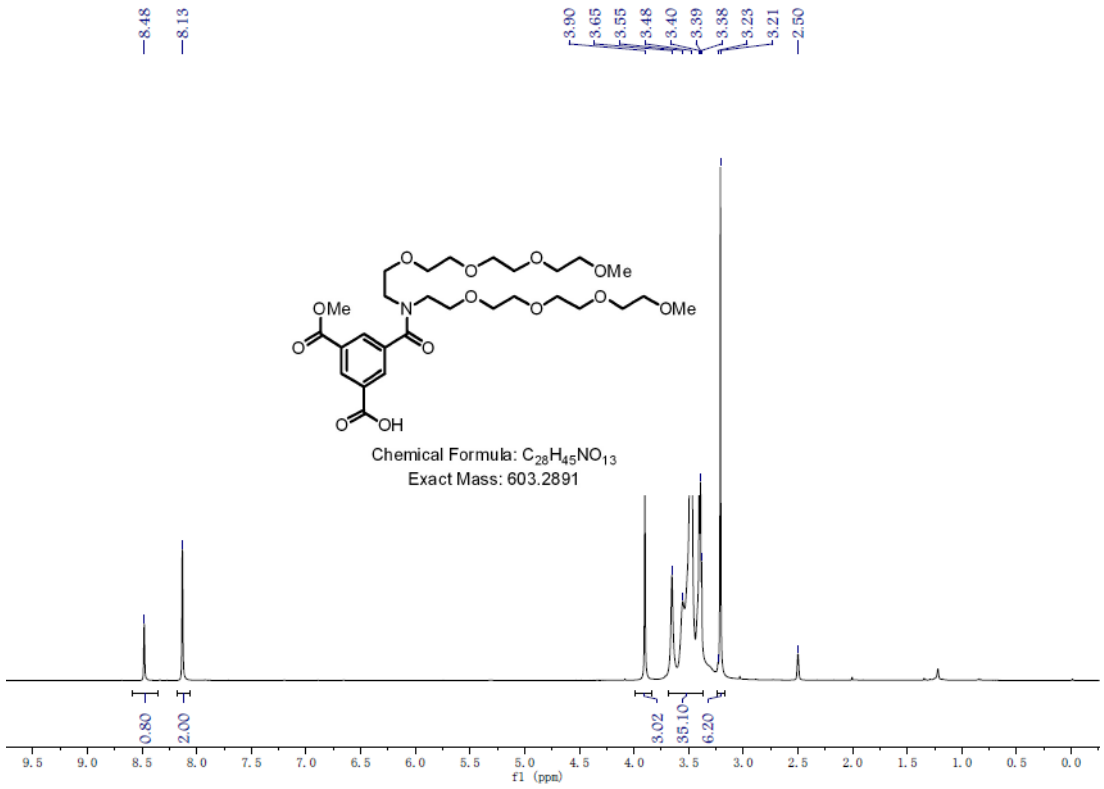


Fig. S16 ^{1}H NMR spectrum (400 MHz) of compound **13** in DMSO-d_6 at 25 °C.

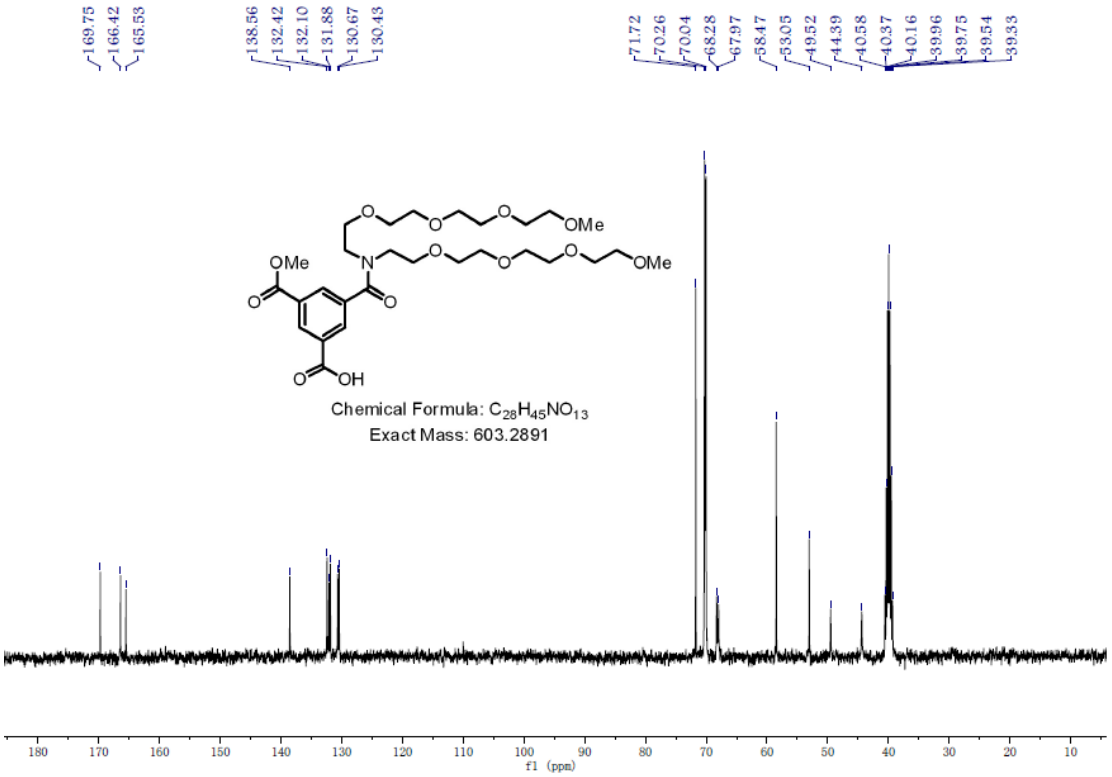


Fig. S17 ^{13}C NMR spectrum (100 MHz) of compound **13** in DMSO-d_6 at 25 °C.

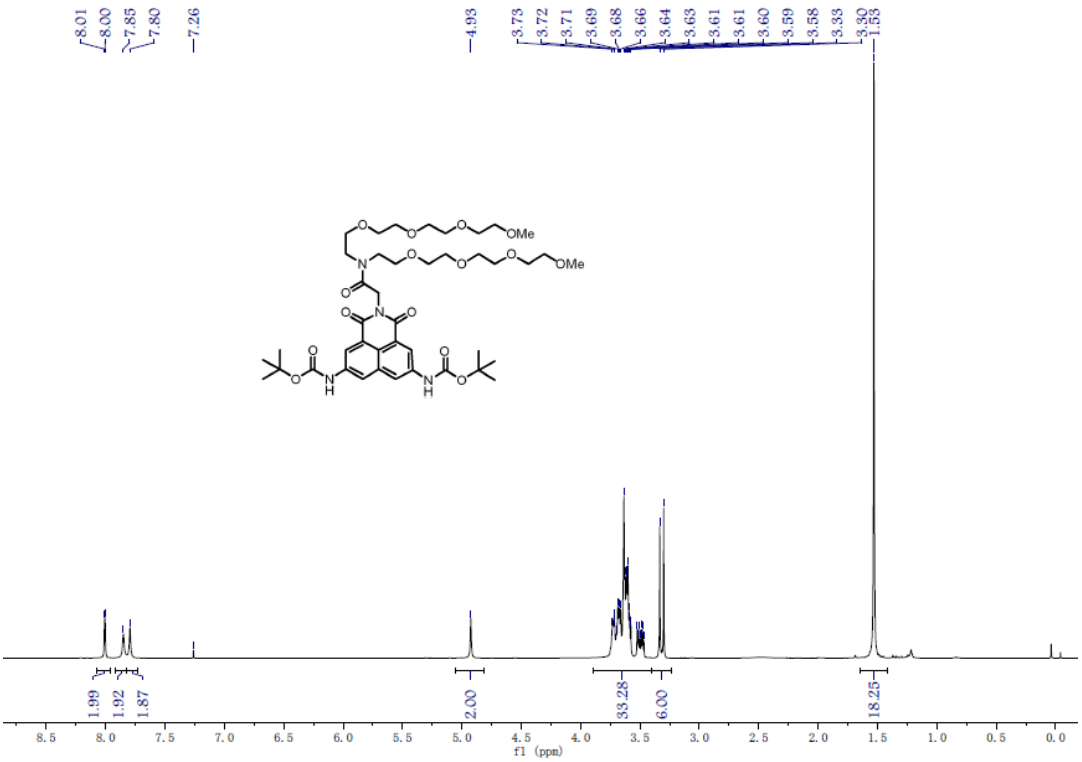


Fig. S18 ^{1}H NMR spectrum (400 MHz) of compound **1a** in $CDCl_3$ at 25 °C.

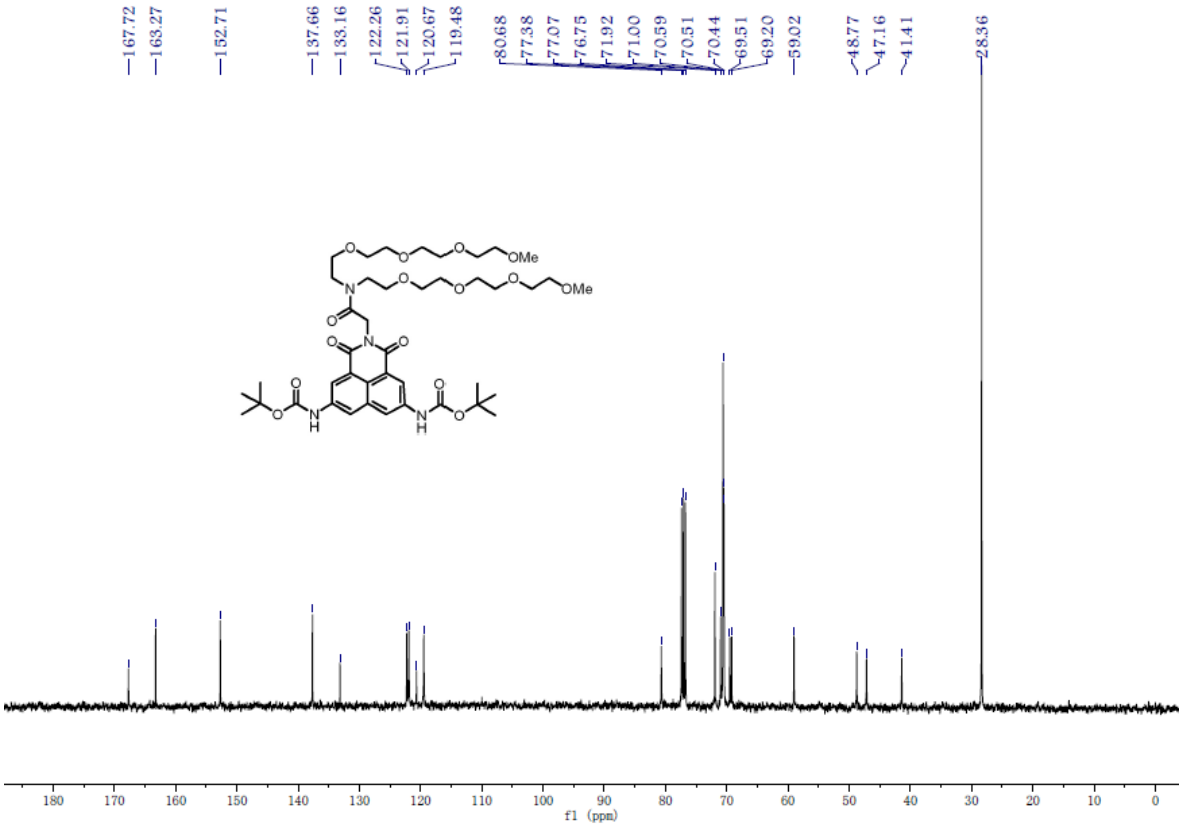


Fig. S19 ^{13}C NMR spectrum (100 MHz) of compound **1a** in $CDCl_3$ at 25 °C.

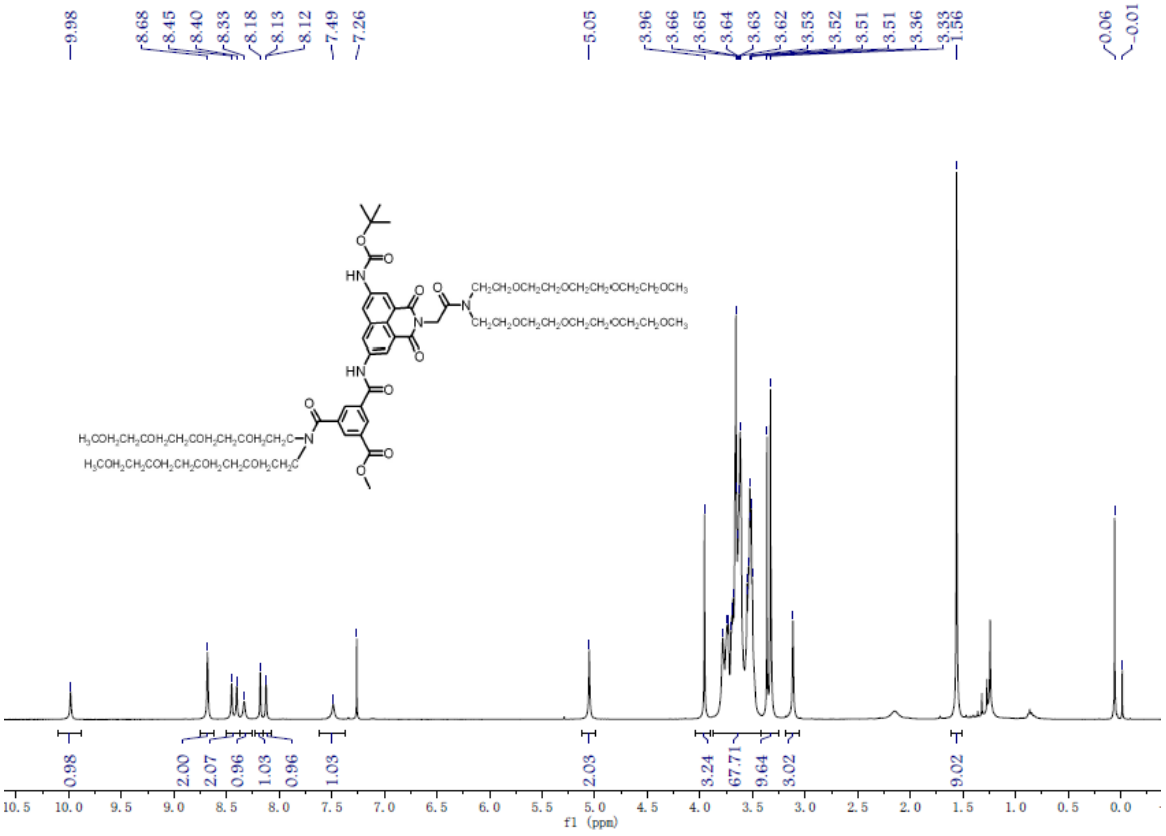


Fig. S20 ^{1}H NMR spectrum (400 MHz) of compound **14** in $CDCl_3$ at 25 °C.

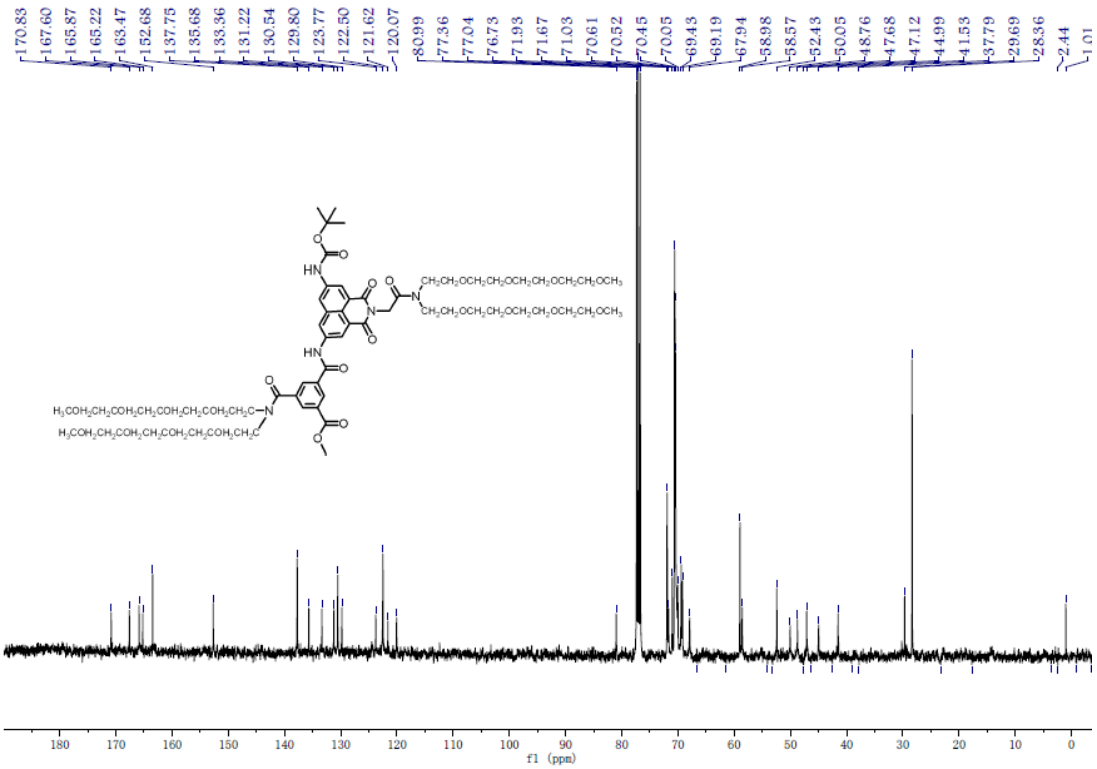


Fig. S21 ^{13}C NMR spectrum (100 MHz) of compound **14** in $CDCl_3$ at 25 °C.

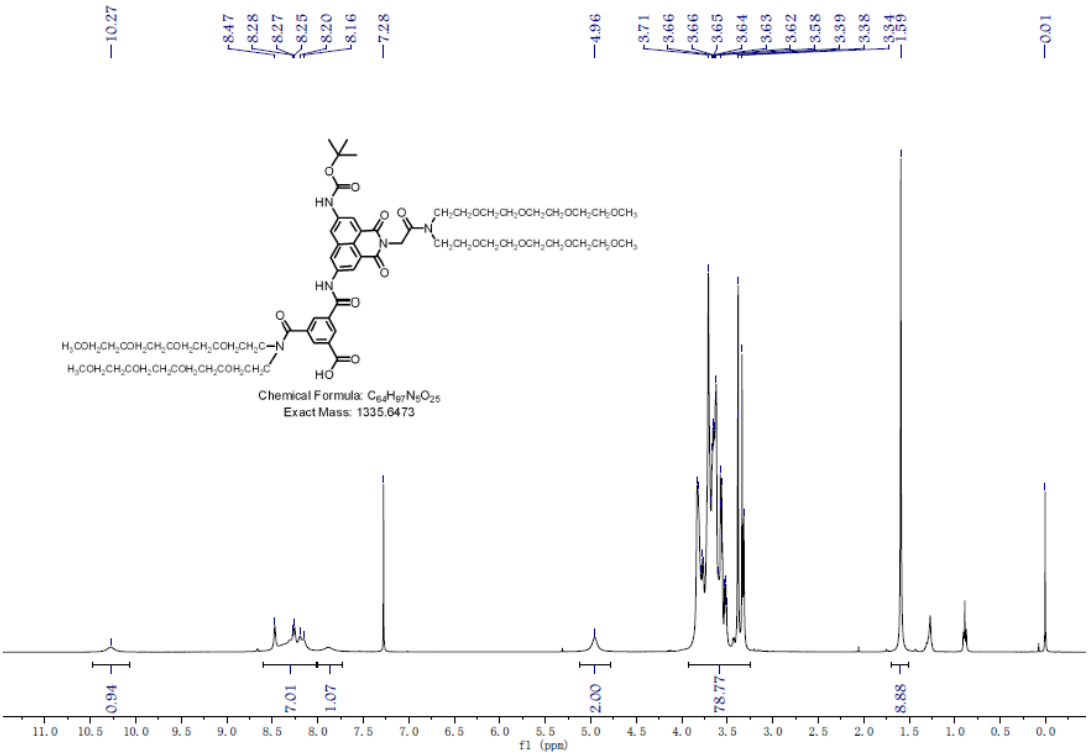


Fig. S22 ^{1}H NMR spectrum (400 MHz) of compound **15** in $CDCl_3$ at 25 °C.

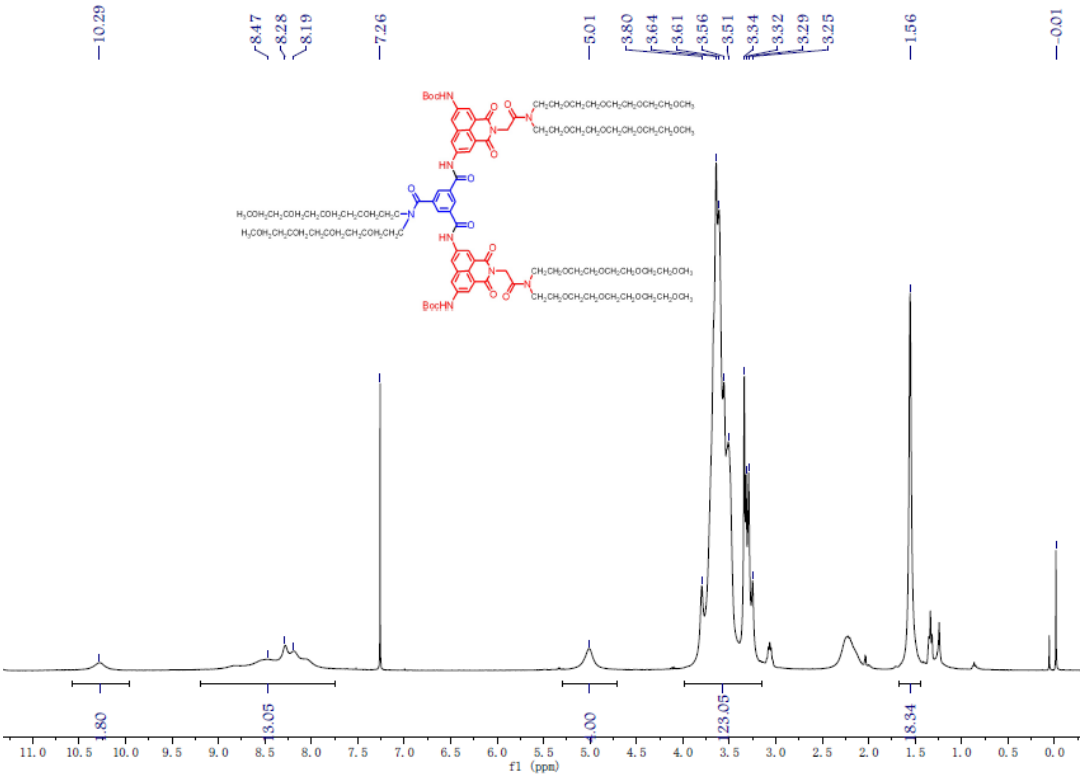


Fig. S23 ^{1}H NMR spectrum (400 MHz) of compound **1b** in $CDCl_3$ at 25 °C.

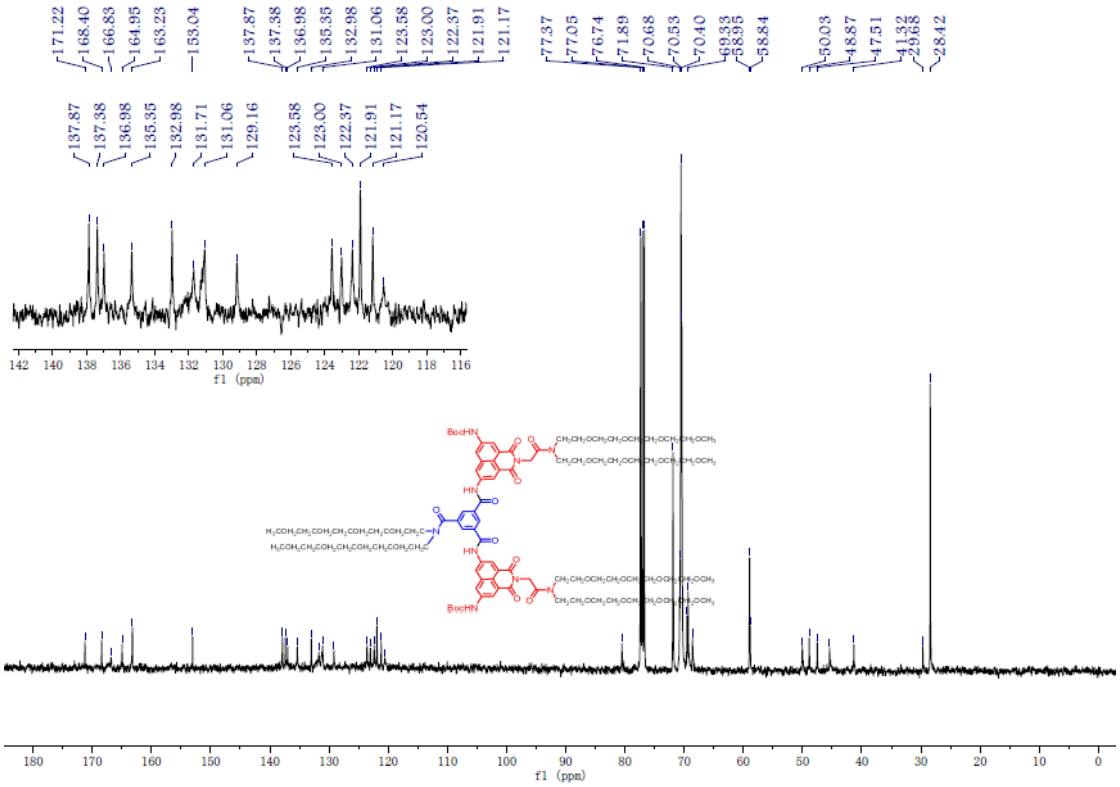


Fig. S24 ^{13}C NMR spectrum (100 MHz) of compound **1b** in $CDCl_3$ at 25 °C.

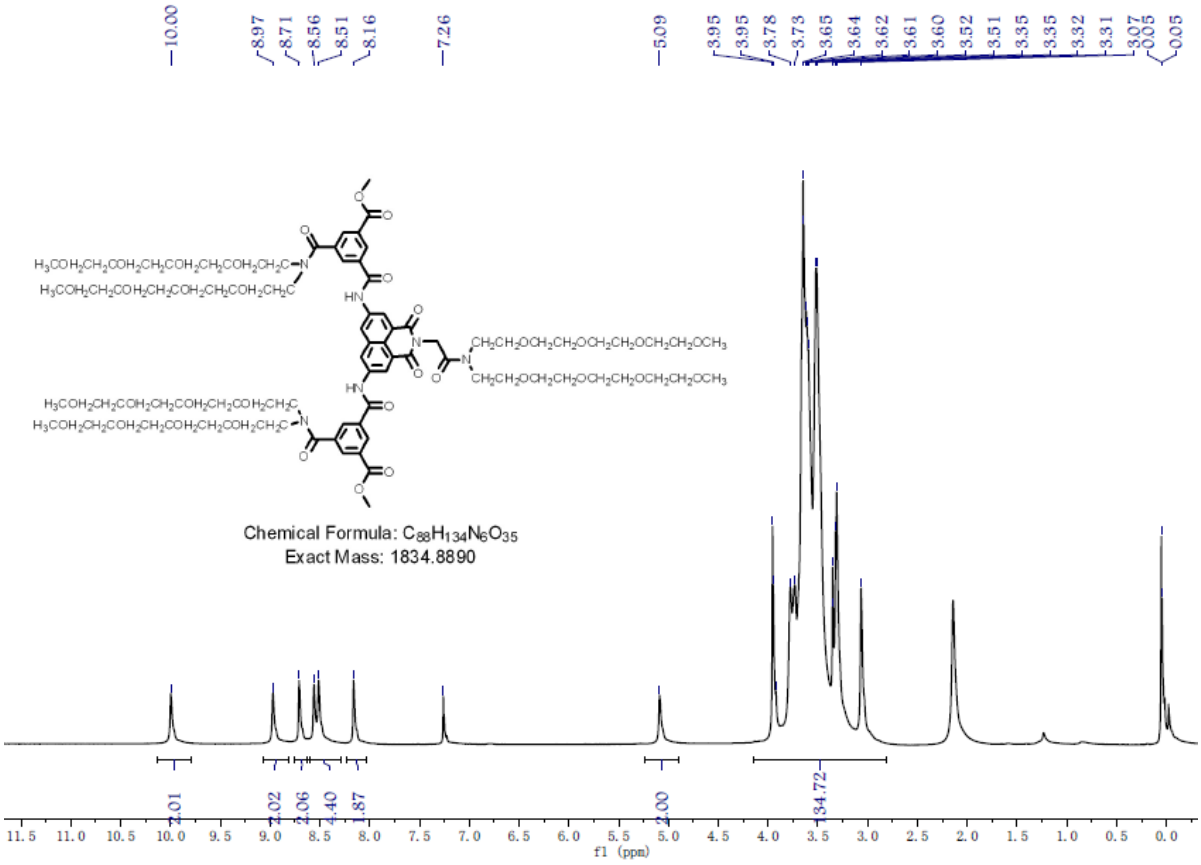


Fig. S25 ^{1}H NMR spectrum (400 MHz) of compound **17** in $CDCl_3$ at 25 °C.

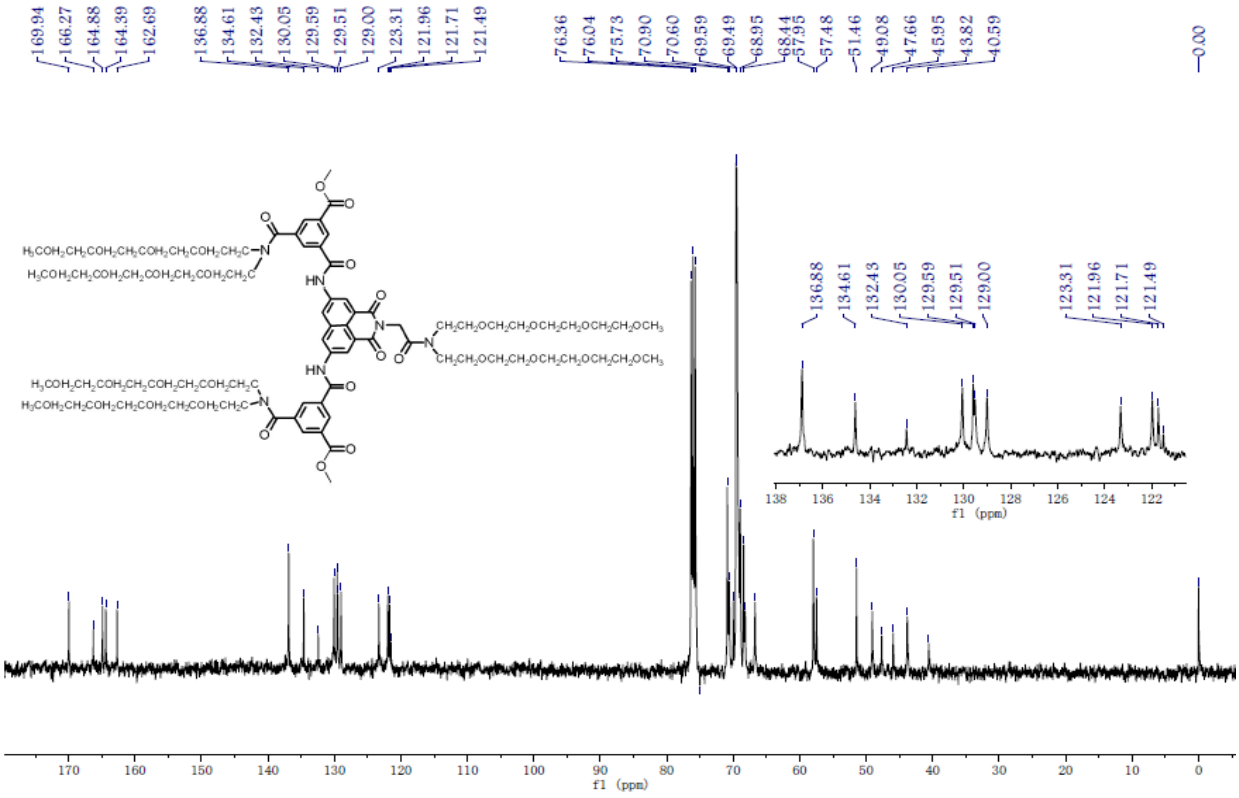


Fig. S26 ^{13}C NMR spectrum (100 MHz) of compound **17** in $CDCl_3$ at 25 °C.

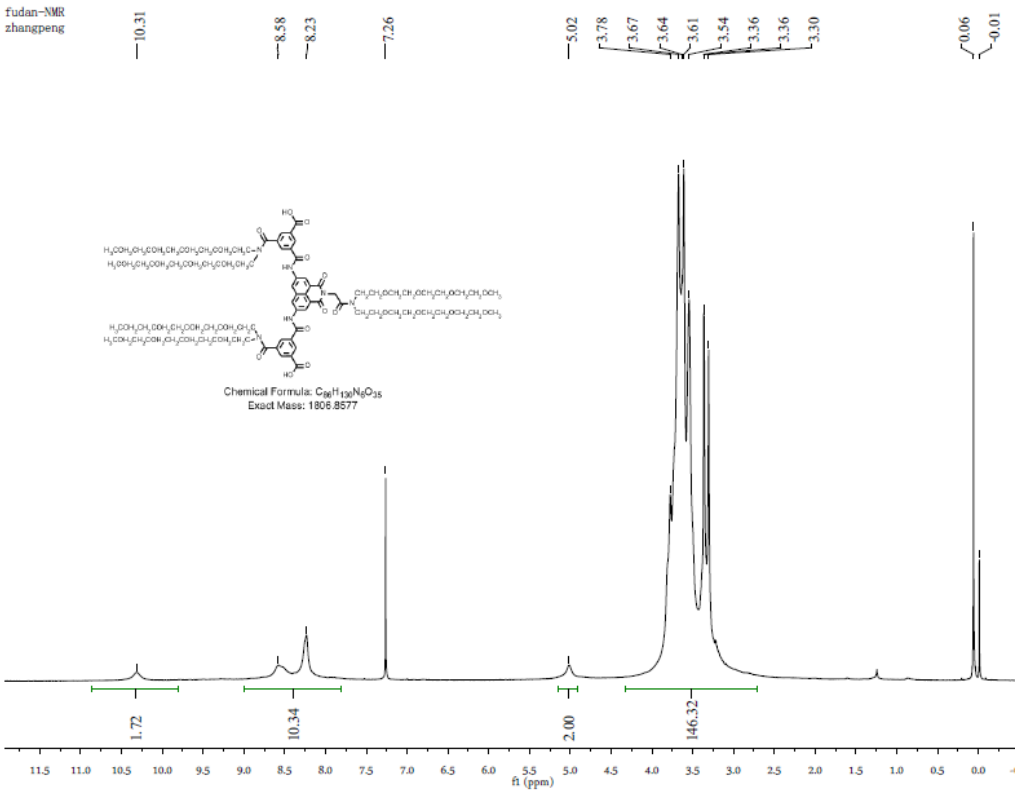


Fig. S27 ^{1}H NMR spectrum (400 MHz) of compound **18** in $CDCl_3$ at 25 °C.

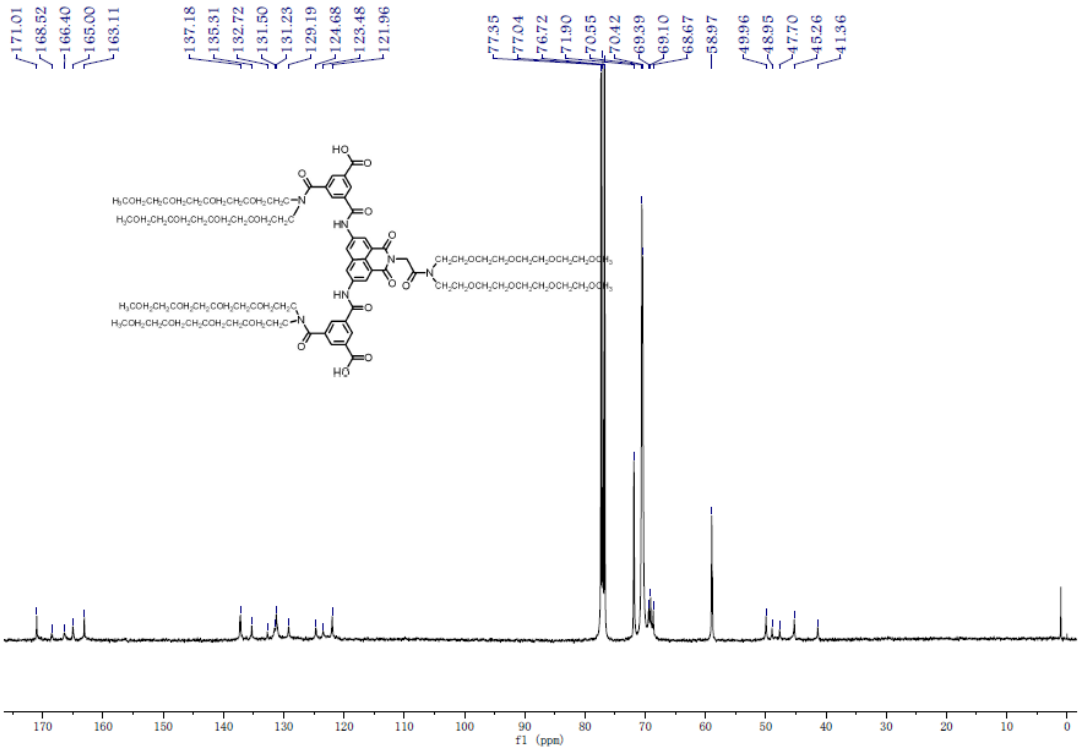


Fig. S28 ^{13}C NMR spectrum (100 MHz) of compound **18** in $CDCl_3$ at 25 °C.

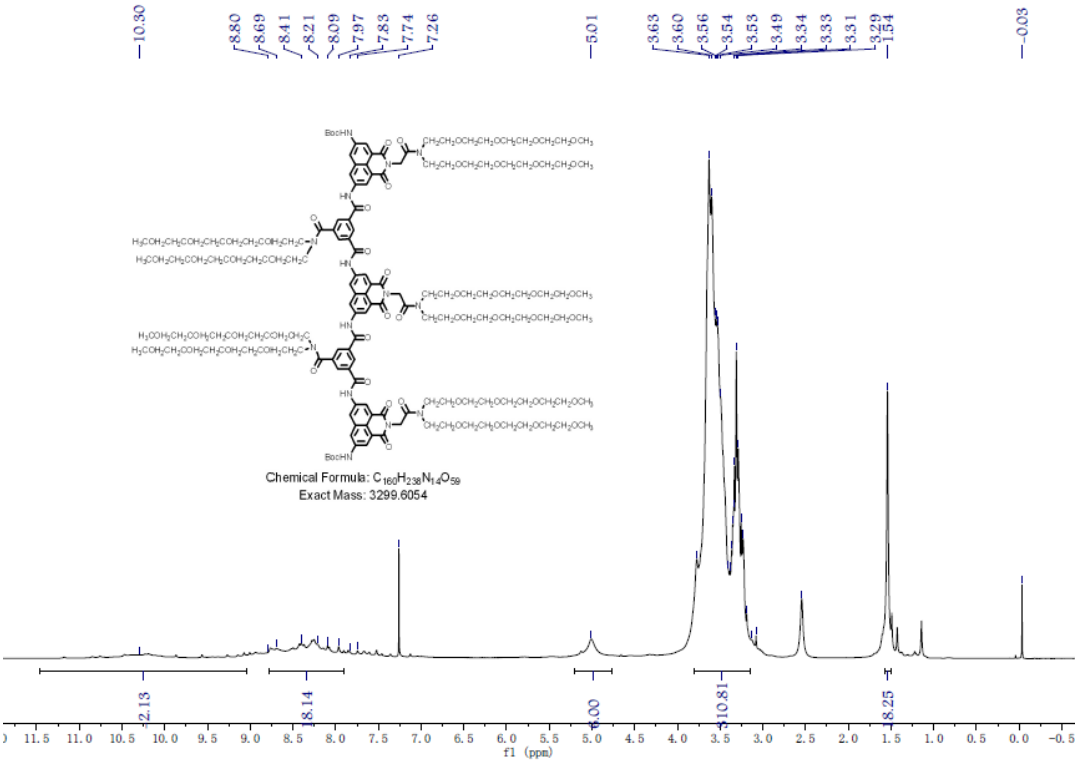


Fig. S29 ^{13}C NMR spectrum (100 MHz) of compound **1c** in $CDCl_3$ at 25 °C.

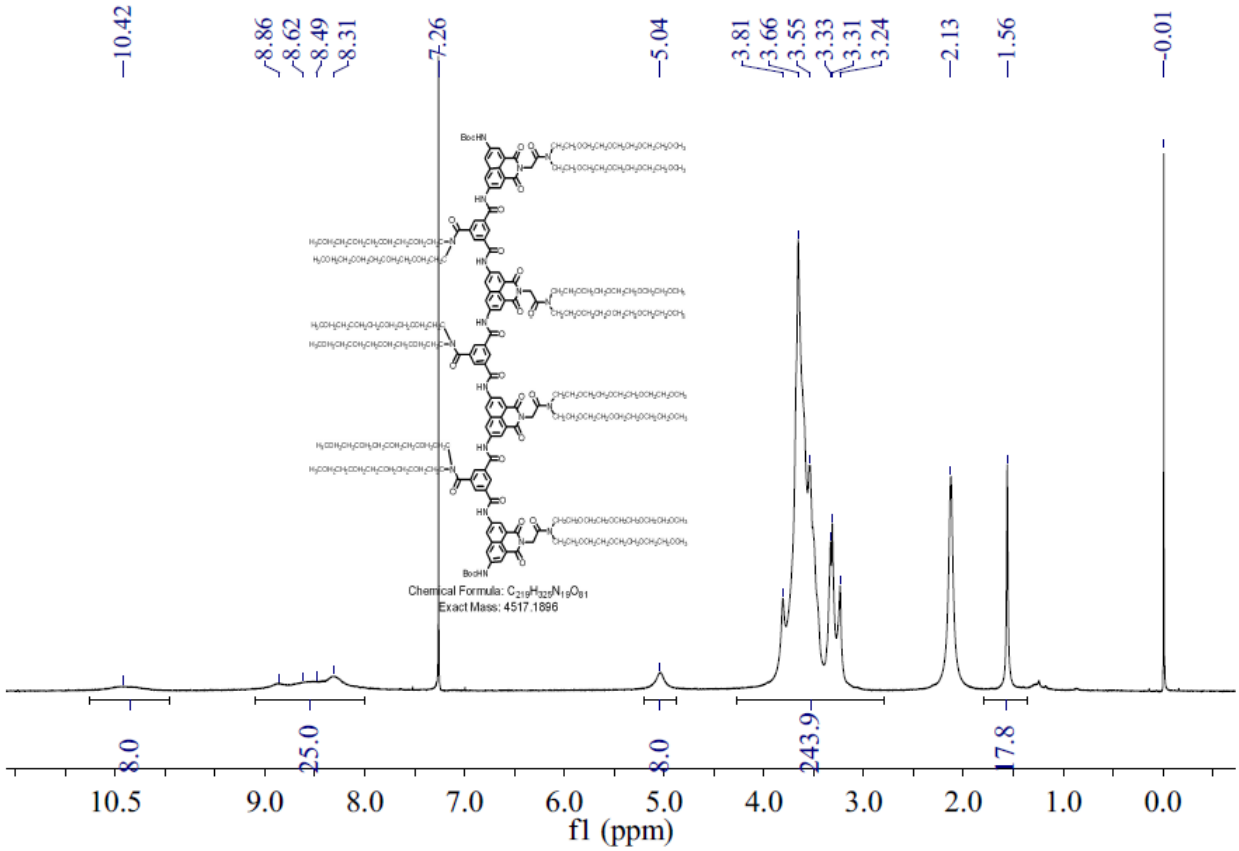


Fig. S30 ^{1}H NMR spectrum (400 MHz) of compound **1d** in $CDCl_3$ at 25 °C.

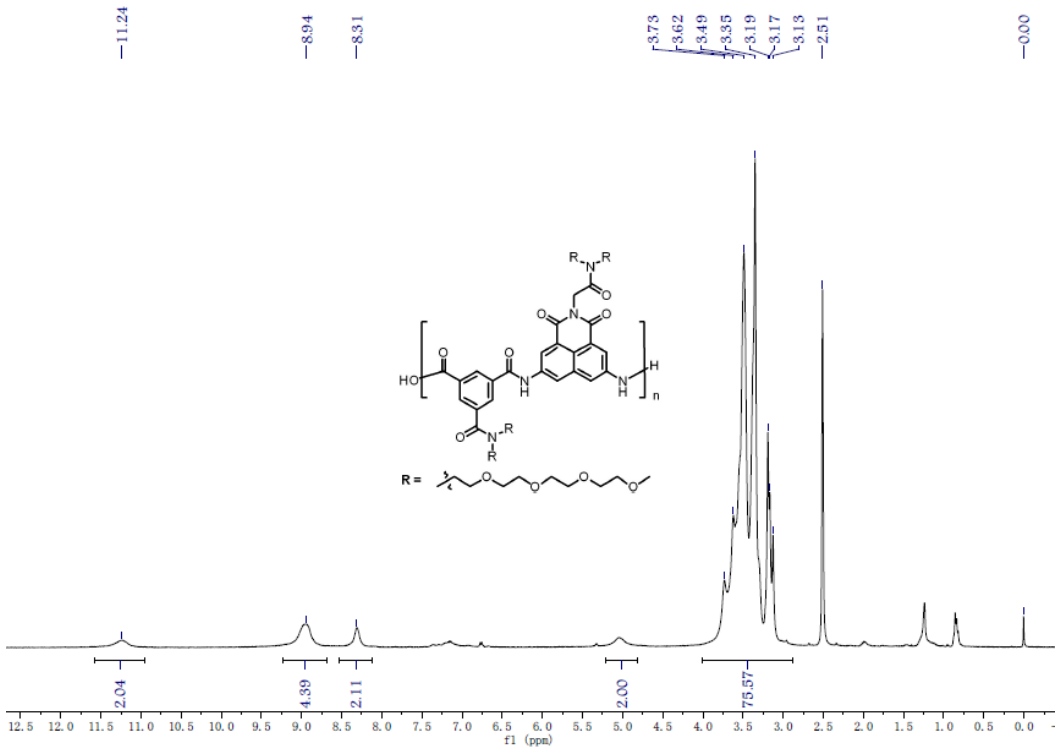


Fig. S31 ^{1}H NMR spectrum (100 MHz) of **Polymer P1** in DMSO-d_6 at 25 °C.

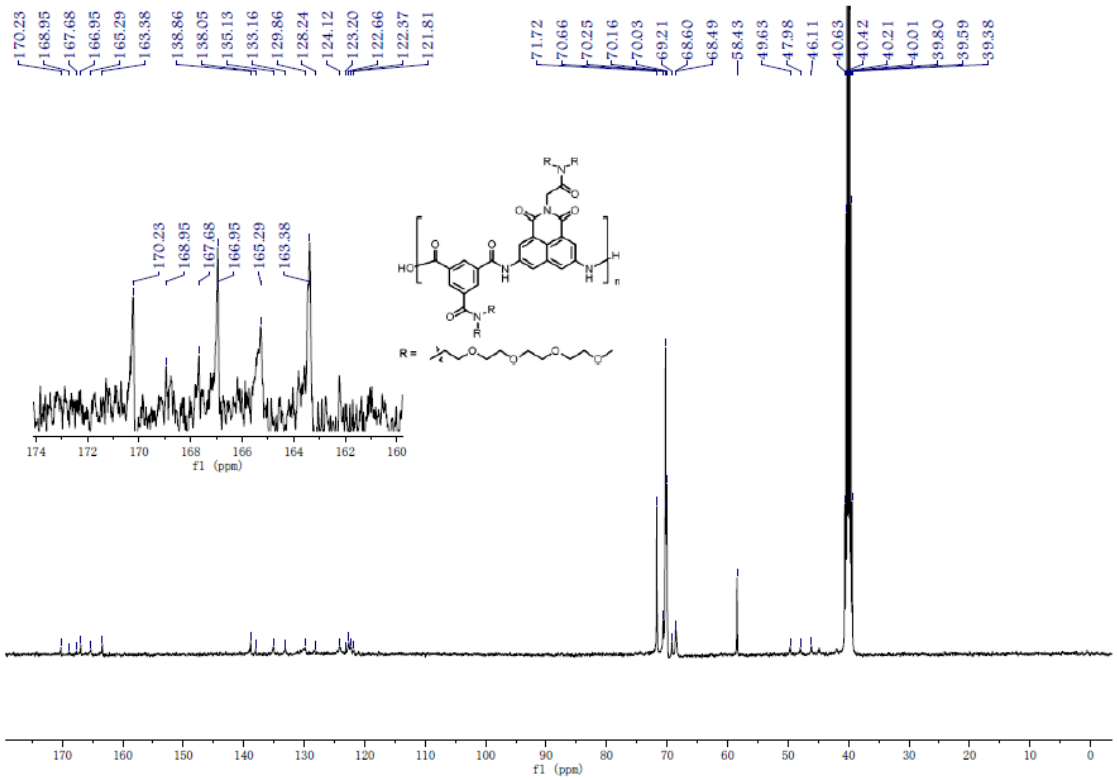


Fig. S32 ^{13}C NMR spectrum (100 MHz) of **Polymer P1** in DMSO-d_6 at 25 °C.

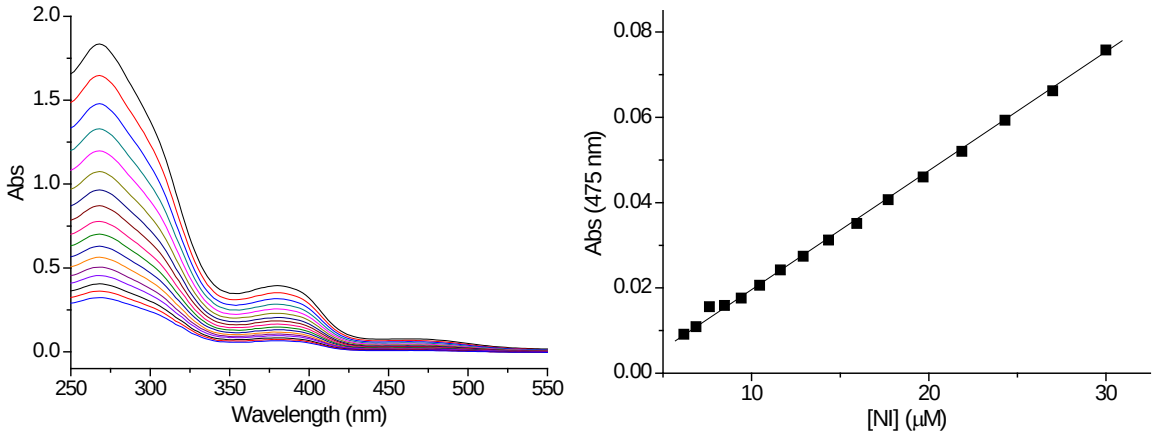


Fig. S33 UV-vis spectrum (right) of polymer **P1** in water at 25 °C and the plot of the absorbance (475 nm) versus [NI].

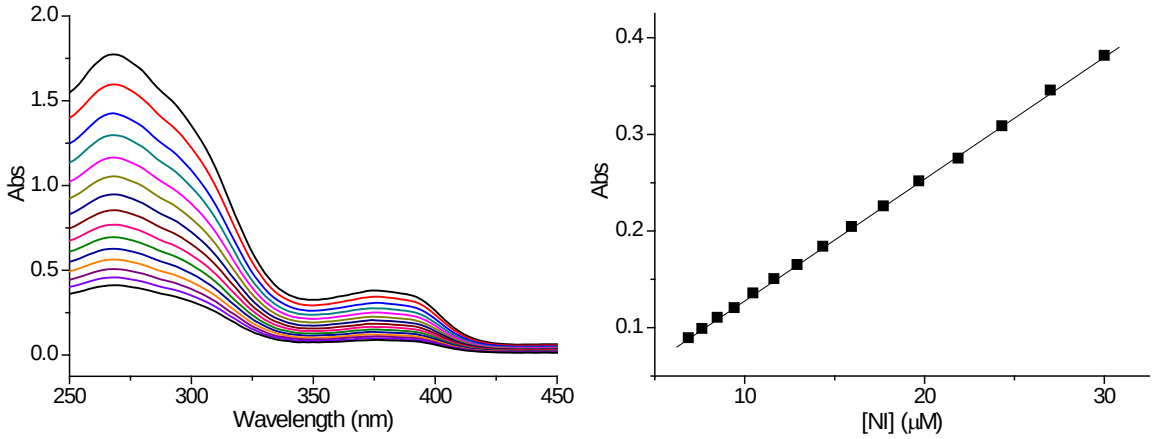


Fig. S34 UV-vis spectrum (right) of polymer **P1** in methanol at 25 °C and the plot of the absorbance (375 nm) versus [NI].

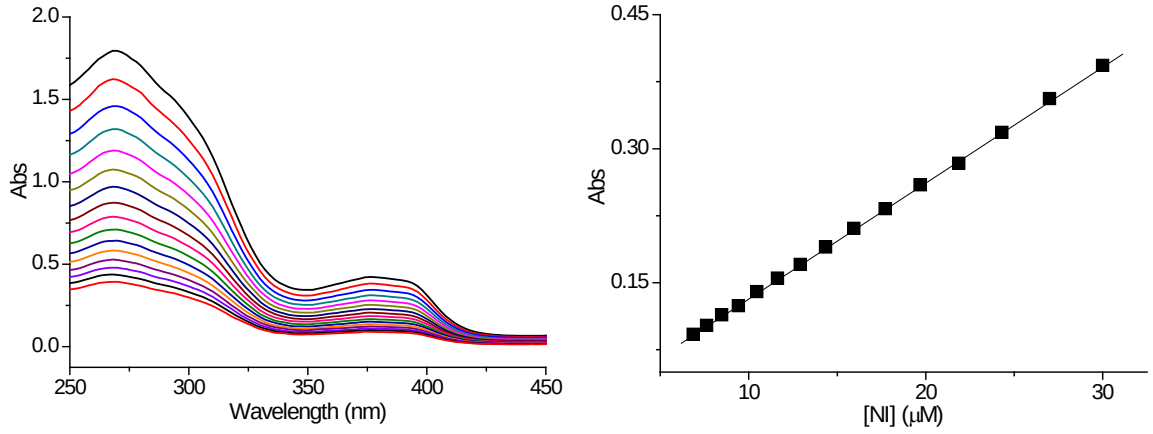


Fig. S35 UV-vis spectrum (right) of polymer **P1** in dichloromethane at 25 °C and the plot of the absorbance (376 nm) versus [NI].

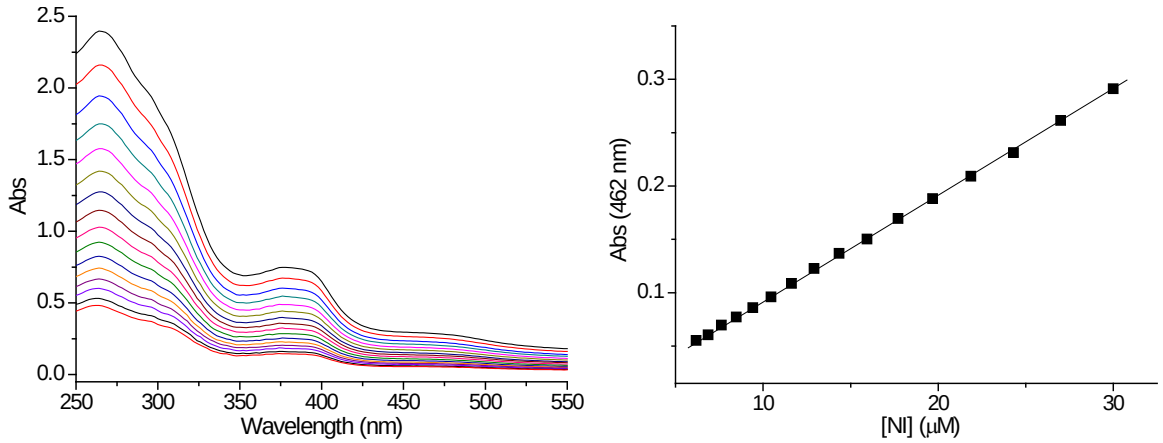


Fig. S36 UV-vis spectrum (right) of polymer **P1** in dichloromethane/n-hexane at 25 °C and the plot of the absorbance (462 nm) versus [NI].

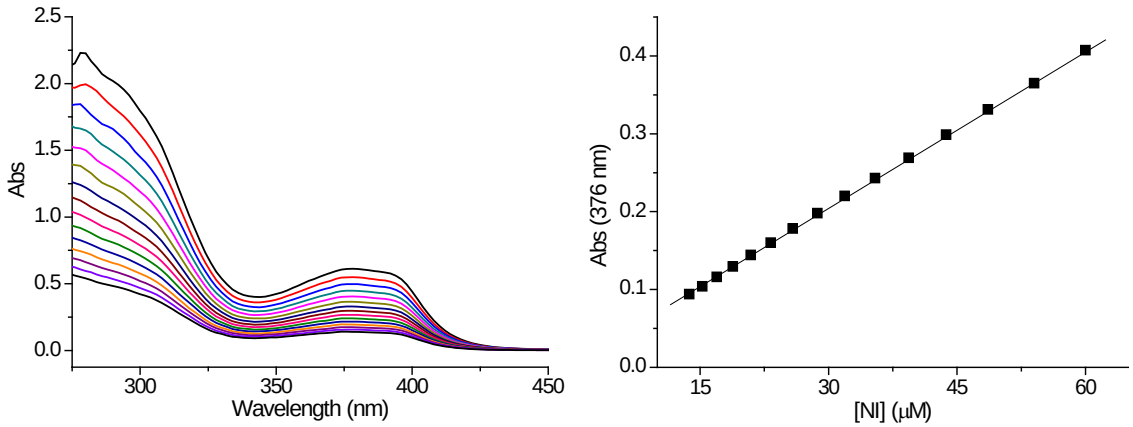


Fig. S37 UV-vis spectrum (right) of compound **1d** in dichloromethane at 25 °C and the plot of the absorbance (376 nm) versus [NI].

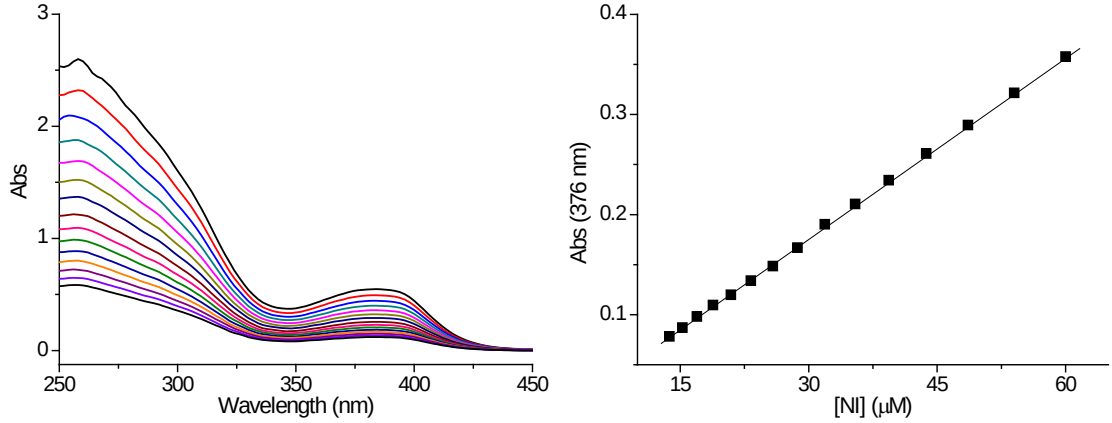


Fig. S38 UV-vis spectrum (right) of compound **1d** in dichloromethane/n-hexane (2:3 v/v) at 25 °C and the plot of the absorbance (376 nm) versus [NI].

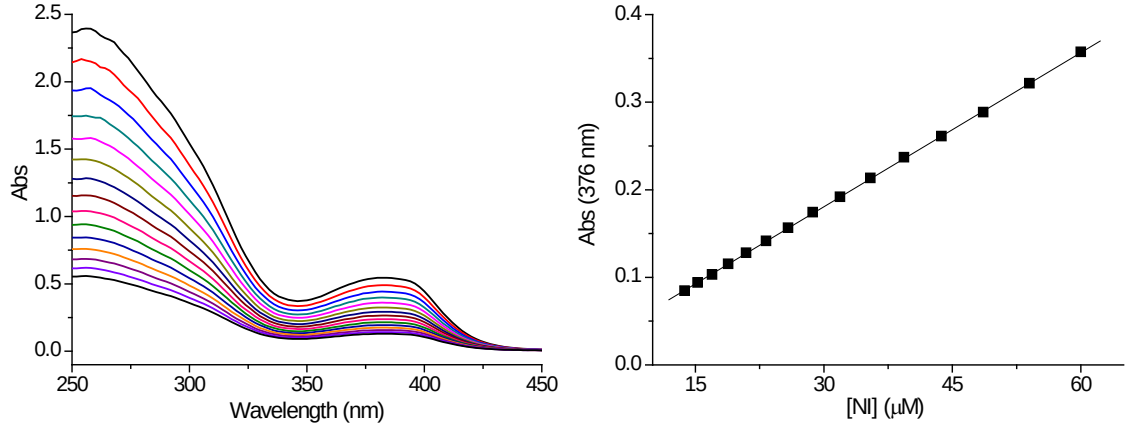


Fig. S39 UV-vis spectrum (right) of compound **1d** in dichloromethane/cyclohexane (2:3 v/v) at 25 °C and the plot of the absorbance (376 nm) versus [NI].

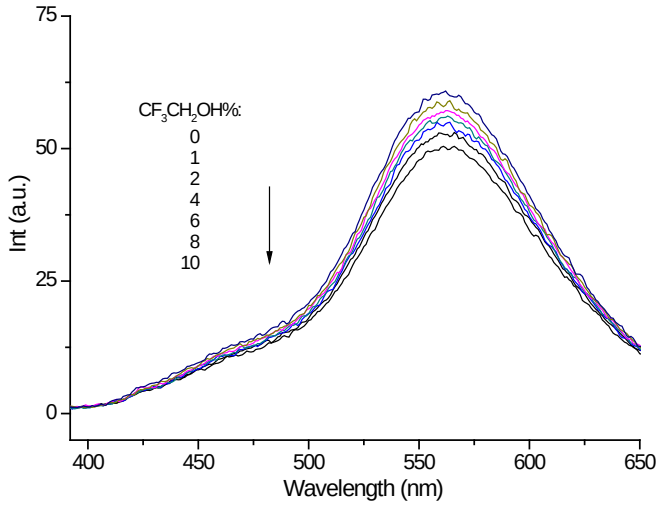


Fig. S40 Fluorescent spectra of Polymer **P1** at 25 °C in mixture of water and trifluoroethanol of increasing content. [NI] = 20 μM.

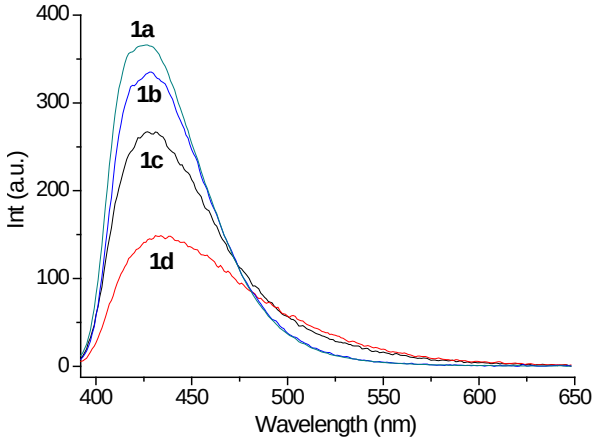


Fig. S41 Fluorescent spectra of **1a-d** at 25 °C in chloroform. [NI] = 20 μM.

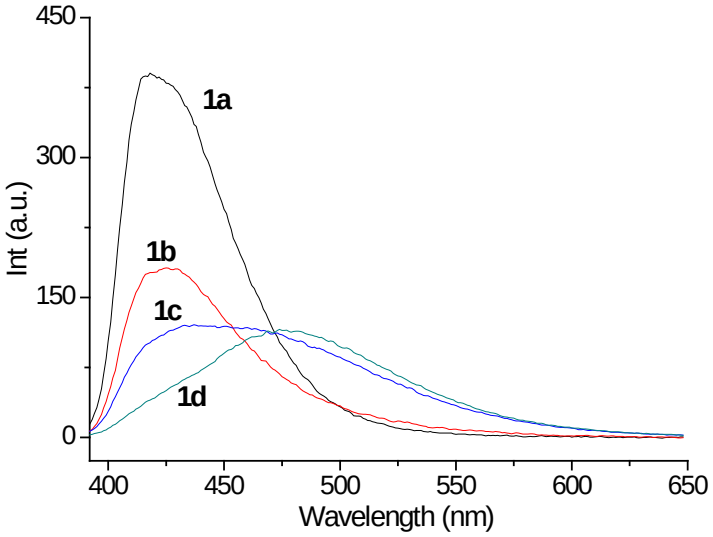


Fig. S42 Fluorescent spectra of **1a-d** at 25 °C in 1,2-dichloroethane. [NI] = 20 μM.

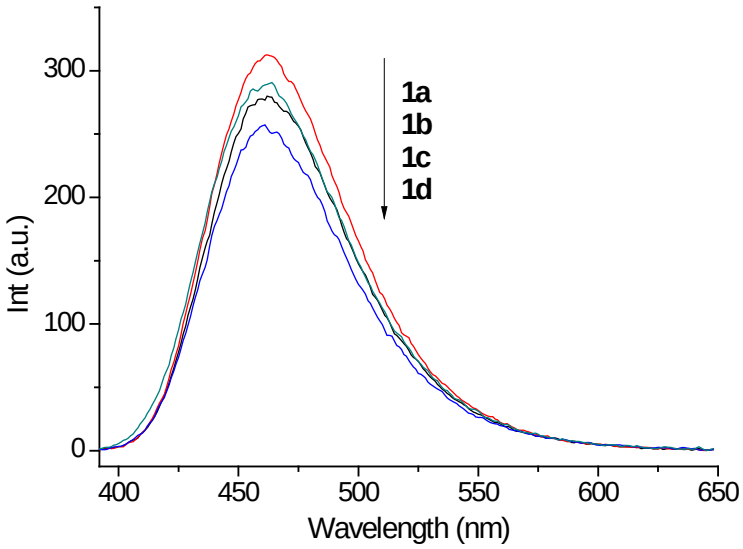


Fig. S43 Fluorescent spectra of **1a-d** at 25 °C in *N*-methylpyridone. [NI] = 20 μM.