

Supplementary Information

Triblock Near-Infrared Fluorescent Polymer Semiconductor Nanoparticles for Targeted Imaging

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Synthesis of BODIPY 2. ¹N-iodosuccinimide (1.09 g, 4.83 mmol) was added to a solution of compound **1** (623 mg, 1.38 mmol) in 25 mL DCM and the reaction mixture was stirred at room temperature for 4 h before washed with saturated Na₂S₂O₃ aqueous solution. The organic layer was dried over Na₂SO₄, concentrated and purified by column chromatography (SiO₂, 95:5 hexanes-EtOAc) to give the product as a red powder (782 mg, 81%). ¹H-NMR (400 MHz, CDCl₃): δ 7.08 (d, *J* = 8 Hz, 2H), 6.99 (d, *J* = 8 Hz, 2H), 4.00 (t, *J* = 7 Hz, 2H), 2.62 (s, 6H), 1.82 (p, *J* = 7 Hz, 2H), 1.43 (s, 6H), 1.36-1.22 (m, 10H), 0.88 (t, *J* = 7 Hz, 3H).

Synthesis of BODIPY 4a. A mixture of compound **2** (700 mg, 0.995 mmol), 4-octyloxybenzaldehyde (**3a**, 0.96 mL, 4.0 mmol), 0.6 mL acetic acid, 0.6 mL piperidine and 3 Å molecular sieves in 50 mL benzene was refluxed overnight. The reaction mixture was washed several times with water, dried over Na₂SO₄, concentrated and purified by column chromatography (SiO₂, DCM) to give the product as dark green powder (422 mg, 37%). ¹H-NMR (400 MHz, CDCl₃): δ 8.09 (d, *J* = 16 Hz, 2H), 7.57 (d, *J* = 9 Hz, 4H), 7.54 (d, *J* = 16 Hz, 2H), 7.12 (d, *J* = 9 Hz, 2H), 7.02 (d, *J* = 9 Hz, 2H), 6.93 (d, *J* = 9 Hz, 4H), 3.99 (m, 6H), 1.81 (m, 6H), 1.45 (m, 6H), 1.38-1.19 (m, 24H), 0.88 (m, 9H).

Synthesis of BODIPY 4b. Following the procedure for **4a**, compound **2** (100 mg, 0.142 mmol), 5-(4-methoxyphenyl)thiophene-2-carboxaldehyde (**3b**, 124 mg, 0.568 mmol), 0.1 mL acetic acid and 0.1 mL piperidine in 3 mL benzene gave the product as dark green powder (95 mg, 61%). ¹H-NMR (400 MHz, CDCl₃): δ 8.28 (d, *J* = 16 Hz, 2H), 7.59 (d, *J* = 9 Hz, 4H), 7.46 (d, *J* = 16 Hz, 2H), 7.23 (m, 2H), 7.18 (d, *J* = 4 Hz, 2H), 7.13 (d, *J* = 8 Hz, 2H), 7.01 (d, *J* = 8 Hz, 2H), 6.90 (d, *J* = 9 Hz, 4H), 4.01 (t, *J* = 7 Hz, 2H), 3.83 (s, 6H), 1.82 (p, *J* = 7 Hz, 2H), 1.48 (s, 6H), 1.36-1.22 (m, 10H), 0.88 (t, *J* = 7 Hz, 3H).

Synthesis of BODIPY 4c. Following the procedure for **4a**, compound **2** (160 mg, 0.213 mmol), 4-(dibutylamino)benzaldehyde (**3c**, 199 mg, 0.852 mmol), 0.15 mL acetic acid and 0.15 mL piperidine in 4.5 mL benzene gave the product as dark purple powder (241 mg, 93%). ¹H-NMR (400 MHz, CDCl₃): δ 8.12 (d, *J* = 16 Hz, 2H), 7.52 (m, 6H), 7.12 (d, *J* = 8 Hz, 2H), 6.99 (d, *J* = 8 Hz, 2H), 6.65 (d, *J* = 9 Hz, 4H), 4.01 (t, *J* = 7 Hz, 2H), 3.31 (t, *J* = 8 Hz, 8H), 1.82 (p, *J* = 7 Hz, 2H), 1.59 (p, *J* = 7 Hz, 8H), 1.46 (s, 6H), 1.36-1.22 (m, 18H), 0.95 (t, *J* = 7 Hz, 12H), 0.88 (t, *J* = 7 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 159.73, 152.62, 149.03, 145.50, 142.24, 140.03, 137.94, 136.95, 132.55, 129.90, 129.78, 129.61, 129.34, 127.05, 124.36, 115.01, 113.80, 111.79, 111.46, 85.61, 78.67, 68.19, 50.82, 31.81, 29.50, 29.41, 29.23, 26.06, 22.66, 20.32, 14.11, 13.97, 13.16.

Synthesis of BODIPY Monomer 5a. A mixture of compound **4a** (306 mg, 0.269 mmol), Pd(PPh₃)₄ (15 mg, 0.013 mmol), CuI (6 mg, 0.03 mmol), 9 mL PhMe and 3 mL diisopropylamine (DIPA) was degassed and backfilled with argon for three times before trimethylsilylacetylene (TMSA, 0.30 mL, 1.6 mmol) was added to the reaction flask through syringe. The mixture was stirred at 70 °C for 18 h before cooled to room temperature, washed with 50 mL saturated NH₄Cl aqueous solution, and extracted with DCM. The organic phase was dried over Na₂SO₄ and purified by column chromatography (SiO₂, 95:5 hexanes-EtOAc) to give the TMS-protected BODIPY-dialkyne intermediate as dark green powder (242 mg, 81%). ¹H-NMR (400 MHz, CDCl₃): δ 8.35 (d, *J* = 16 Hz, 2H), 7.65 (d, *J* = 16 Hz, 2H), 7.55 (d, *J* = 9 Hz, 2H), 7.13 (d, *J* = 9 Hz, 2H), 6.98 (d, *J* = 9 Hz, 2H), 6.93 (d, *J* = 9 Hz, 4H), 3.99 (m, 6H), 1.81 (m, 6H), 1.46 (m, 6H), 1.29 (m, 24H), 0.88 (m, 9H), 0.24 (s, 18H).

To deprotect the TMS group after the Sonogashira coupling, the aforementioned intermediate (242 mg, 0.224 mmol) was dissolved in a mixture of 7 mL THF and 5 mL MeOH, to which 1.6 mL 2.5 M K₂CO₃ aqueous solution was added. The reaction mixture was stirred at room temperature for 4 h before washed with water and extracted with DCM. The organic phase was dried over Na₂SO₄ and purified by column chromatography (SiO₂, DCM) to give the **5a** as dark green powder (82 mg, 39%). ¹H-NMR (400 MHz, CDCl₃): δ 8.29 (d, *J* = 16 Hz, 2H), 7.57 (m, 6H), 7.13 (d, *J* = 9 Hz, 2H), 7.02 (d, *J* = 9 Hz, 2H), 6.92 (d, *J* = 9 Hz, 4H), 3.99 (m, 6H), 3.54 (s, 2H), 1.81 (m, 6H), 1.45 (m, 6H), 1.38-1.19 (m, 24H), 0.88 (m, 9H). ¹³C-NMR (100 MHz, CDCl₃): δ 160.33, 159.95, 152.59, 146.46, 139.47, 138.79, 132.71, 129.53, 129.48, 129.31, 126.53, 116.44, 115.18, 114.76, 112.32, 86.02, 78.07, 68.13, 31.80, 29.68, 29.39, 29.35, 29.22, 26.01, 22.65, 14.09, 13.23.

Synthesis of BODIPY Monomer 5b. Following the procedure for **5a**, a mixture of compound **4b** (91 mg, 0.086 mmol), Pd(PPh₃)₄ (5 mg, 0.004 mmol), CuI (2 mg, 0.01 mmol), 3 mL PhMe, 1 mL DIPA and TMSA (0.10 mL, 0.53 mmol) yielded the TMS-protected intermediate as dark green powder (66 mg, 77%). ¹H-NMR (400 MHz, CDCl₃): δ 8.50 (d, *J* = 16 Hz, 2H), 7.60 (d, *J* = 9 Hz, 4H), 7.53 (d, *J* = 16 Hz, 2H), 7.19 (m, 4H), 7.12 (d, *J* = 8 Hz, 2H), 7.00 (d, *J* = 8 Hz, 2H), 6.94 (d, *J* = 9 Hz, 4H), 4.00 (t, *J* = 7 Hz, 2H), 3.84 (s, 6H), 1.82 (m, 2H), 1.48 (m, 6H), 1.36-1.22 (m, 10H), 0.88 (m, 3H), 0.26 (s, 18H). The intermediate was subsequently deprotected with K₂CO₃ (43 mg, 0.31 mmol) in 4 mL THF and 1 mL MeOH, and yielded **5b** as dark green powder (30 mg, 60%). ¹H-NMR (400 MHz, CDCl₃): δ 8.41 (d, *J* = 16 Hz, 2H), 7.59 (d, *J* = 9 Hz, 4H), 7.55 (d, *J* = 16 Hz, 2H), 7.22 (d, *J* = 4 Hz, 2H), 7.19 (d, *J* = 4 Hz, 2H), 7.13 (d, *J* = 8 Hz, 2H), 7.02 (d, *J* = 8 Hz, 2H), 6.93 (d, *J* = 9 Hz, 4H), 4.01 (t, *J* = 7 Hz, 2H), 3.83 (s, 6H), 3.57 (s, 2H), 1.82 (p, *J* = 7 Hz, 2H), 1.54 (s, 6H), 1.29 (m, 10H), 0.88 (m, 3H). ¹³C-NMR (100 MHz, CDCl₃): δ 159.94, 159.64, 151.70, 146.74, 146.36, 141.13, 138.87, 133.21, 131.75, 130.62, 129.53, 127.18, 126.83, 126.45, 123.09, 117.15, 115.16, 114.36, 112.70, 86.48, 77.94, 68.21, 55.37, 31.82, 29.42, 29.24, 26.06, 22.67, 14.14, 13.28.

Synthesis of BODIPY Monomer 5c. Following the procedure for **5a**, a mixture of compound **4c** (241 mg, 0.212 mmol), Pd(PPh₃)₄ (12 mg, 0.010 mmol), CuI (5 mg, 0.003 mmol), 7.2 mL PhMe, 2.4 mL DIPA and TMSA (0.25 mL, 0.53 mmol) yielded the TMS-protected intermediate as dark red powder (137 mg, 60%). ¹H-NMR (400 MHz, CDCl₃): δ 8.34 (d, *J* = 16 Hz, 2H), 7.52 (m, 6H), 7.10 (m, 2H), 6.98 (m, 2H), 6.64 (d, *J* = 9 Hz, 4H), 3.99 (t, *J* = 7 Hz, 2H), 3.32 (t, *J* = 8 Hz, 8H), 1.81 (m, 2H), 1.60 (m, 8H), 1.35 (m, 18H), 0.96 (t, *J* = 7 Hz, 12H), 0.89 (t, *J* = 7 Hz, 3H), 0.24 (s, 18H). The intermediate was subsequently deprotected with K₂CO₃ (43 mg, 0.31 mmol) in 4 mL THF and 1 mL MeOH, and yielded **5c** as dark red powder (82 mg, 69%). ¹H-NMR (400 MHz, CDCl₃): δ 8.27 (d, *J* = 16 Hz, 2H), 7.54 (m, 6H), 7.12 (d, *J* = 8 Hz, 2H), 7.00 (d, *J* = 8 Hz, 2H), 6.65 (d, *J* = 9 Hz, 4H), 4.01 (t, *J* = 7 Hz, 2H), 3.52 (s, 2H), 3.31 (t, *J* = 8 Hz, 8H), 1.82 (m, 2H), 1.59 (m, 8H), 1.35 (m, 18H), 0.96 (t, *J* = 7 Hz, 12H), 0.88 (t, *J* = 7 Hz, 3H). ¹³C-NMR (100 MHz, CDCl₃): δ 159.73, 152.62, 149.03, 145.50, 139.21, 132.55, 129.78, 129.61, 129.34, 127.05, 124.36, 115.01, 113.80, 111.46, 85.61, 78.67, 68.19, 50.82, 31.81, 29.50, 29.41, 29.23, 26.06, 22.66, 20.32, 14.11, 13.97, 13.16.

Synthesis of conjugated polymer P1. The mixture of compound **5a** (8.0 mg, 0.0086 mmol) **FL** (273 mg, 0.314 mmol), **BT** (51 mg, 0.28 mmol), Pd(PPh₃)₄ (33 mg, 0.029 mmol), and CuI (5.0 mg, 0.029 mmol) was degassed and backfilled with argon three times in a round bottom flask. 42 mL toluene and 14 mL diisopropylamine were added to the flask by syringe, and the reaction mixture was stirred at 70°C. After 5 h, 5 mL norbornadiene was added to the reaction mixture, followed by the addition of 2.5 mL formic acid in 1 h. The mixture was stirred for another 18 h before cooled to room temperature, washed with saturated NH₄Cl solution, and precipitated twice from acetone. The precipitate was collected by filtration, washed with acetone and dried under vacuum to give **P1** as a dark green powder (145 mg, 56%). ¹H-NMR (400 MHz, CDCl₃): δ 7.85 (br, Ar-H), 7.67 (br, Ar-H), 6.19 (br, norbornene 4H), 4.01 (br, BODIPY 6H), 2.92 (br, norbornene 4H), 2.00 (br, fluorene 4H), 1.20 (br, methylene groups), 0.85 (br, methyl groups), 0.59 (br, 4H). GPC: *M*_n = 17.6 kDa, PD = 1.35. (Note: due to the large difference between BODIPY and fluorene content in the conjugated polymers, not all the peaks could be visualized and quantified.)

Synthesis of conjugated polymer P2. Following the procedure for **P1**, **5b** (0.80 mg, 0.0089 mmol) **FL** (27 mg, 0.031 mmol), **BT** (5.0 mg, 0.027 mmol), Pd(PPh₃)₄ (1.6 mg, 0.0014 mmol), and CuI (0.25 mg, 0.0014 mmol) gave **P2** as a brown powder (12 mg, 47%). ¹H-NMR (400 MHz, CDCl₃): δ 7.84 (br, Ar-H), 7.65 (br, Ar-H), 6.19 (br, norbornene 4H), 3.86 (br, BODIPY 6H), 2.92 (br, norbornene 4H), 2.02 (br, fluorene 4H), 1.21 (br, methylene groups) 0.84 (br, methyl groups), 0.58 (br, 4H). GPC: *M*_n = 15.1 kDa, PD = 1.38.

Synthesis of conjugated polymer P3. Following the procedure for **P1**, **5c** (0.9 mg, 0.001 mmol) **FL** (32 mg, 0.037 mmol), **BT** (6.0 mg, 0.033 mmol), Pd(PPh₃)₄ (1.9 mg, 0.0017 mmol), and CuI (0.32 mg, 0.0017 mmol) gave **P3** as a brown powder (24 mg, 79%). ¹H-NMR (400 MHz, CDCl₃): 7.85 (br, Ar-H), 7.65 (br, Ar-H), 6.70 (br,

Ar-H), 6.18 (br, norbornene 4H), 3.34 (br, BODIPY 8H), 2.92 (br, norbornene 4H), 2.00 (br, fluorene 4H), 1.17 (br, methylene groups), 0.84 (br, methyl groups), 0.60 (br, 4H). GPC: $M_n = 29.9$ kDa, PD = 1.64.

Synthesis of conjugated polymer P4. Following the procedure for **P1**, **5a** (20 mg, 0.021 mmol) **FL** (67 mg, 0.077 mmol), **BT** (9.0 mg, 0.05 mmol), Pd(PPh₃)₄ (8.1 mg, 0.0070 mmol), and CuI (1.3 mg, 0.0070 mmol) gave **P4** as a brown powder (27 mg, 35%). ¹H-NMR (400 MHz, CDCl₃): δ 8.48 (br, 2H), 7.65 (br, 25H), 7.47 (br, 7H), 7.06 (br, 2H), 6.93 (br, 5H), 6.09 (br, norbornene 4H), 4.01 (br, BODIPY 6H), 2.81 (br, norbornene 4H), 2.00 (br, fluorene 4H), 1.81 (br, 12H) 1.17 (br, methylene groups), 0.85 (br, methyl groups), 0.58 (br, 4H). GPC: $M_n = 20.8$ kDa, PD = 1.59.

Synthesis of conjugated polymer P5. Following the procedure for **P1**, **5b** (10.5 mg, 0.0116 mmol) **FL** (37 mg, 0.043 mmol), **BT** (5.0 mg, 0.027 mmol), Pd(PPh₃)₄ (2.2 mg, 0.0019 mmol), and CuI (0.37 mg, 0.0019 mmol) gave **P5** as a brown powder (22 mg, 51%). ¹H-NMR (400 MHz, CDCl₃): δ 8.64 (br, 2H), 7.84 (br, 6H), 7.65 (br, 32H), 7.48 (br, 7H), 7.05 (br, 3H), 6.94 (br, 5H), 6.19 (br, norbornene 4H), 4.03 (br, BODIPY 2H), 3.86 (br, BODIPY 6H), 2.99 (br, norbornene 2H), 2.81 (br, norbornene 2H), 2.02 (br, fluorene 4H), 1.20 (br, methylene groups) 0.84 (br, methyl groups), 0.60 (br, 4H). GPC: $M_n = 13.2$ kDa, PD = 1.37.

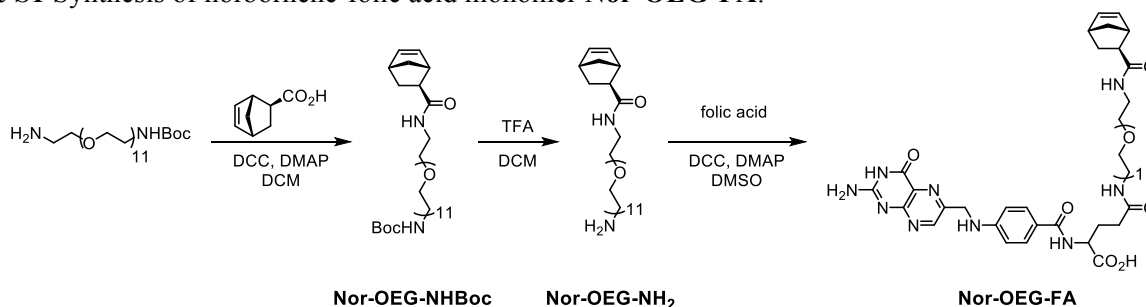
Synthesis of conjugated polymer P6. Following the procedure for **P1**, **5c** (10.8 mg, 0.0116 mmol) **FL** (37 mg, 0.043 mmol), **BT** (5.0 mg, 0.027 mmol), Pd(PPh₃)₄ (2.2 mg, 0.0019 mmol), and CuI (0.37 mg, 0.0019 mmol) gave **P6** as a brown powder (23 mg, 54%). ¹H-NMR (400 MHz, CDCl₃): δ 8.47 (br, 2H), 7.85 (br, 5H), 7.65 (br, 25H), 7.47 (br, 7H), 7.04 (br, 2H), 6.68 (br, 5H), 6.19 (br, norbornene 4H), 4.05 (br, BODIPY 2H), 3.33 (br, BODIPY 8H), 2.76 (br, norbornene 4H), 1.99 (br, fluorene 4H), 1.17 (br, methylene groups), 0.84 (br, methyl groups), 0.60 (br, 4H). GPC: $M_n = 37.6$ kDa, PD = 1.89.

Determination of BODIPY composition in P1-P6 by ¹H-NMR. The two methylene groups (around 2.0 ppm) adjacent to the fluorene 9-position quaternary carbon was selected to determine the quantity of fluorene composition. For polymers made from BODIPY **5a**, three methylene groups (including the one on BODIPY *meso*-phenyl position, all around 4.0 ppm) adjacent to O were selected for BODIPY composition. For polymers made from **5b**, two methoxy groups (3.9 ppm) were selected. For polymers made from **5c**, four methylene groups adjacent to N on the dibutyl amino groups (3.3 ppm) were selected. BODIPY composition was calculated from the proton integration of each BODIPY of interest, divided by the integration of aforementioned fluorene protons (Table S1).

Table S1 BODIPY composition in **P1-P6** by ¹H-NMR.

Polymer	BODIPY monomer	Initial loading	Calculated composition
P1	5a	3.0%	2.7%
P2	5b	3.0%	2.2%
P3	5c	3.0%	3.0%
P4	5a	30%	27%
P5	5b	30%	21%
P6	5c	30%	28%

Synthesis of Nor-OEG. To a mixture of *exo*-5-norbornenecarboxylic acid (275 mg, 1.99 mmol), methoxyundecaethylene glycol (858 mg, 1.66 mmol), 4-(dimethylamino)pyridine (DMAP, 304 mg, 2.49 mmol) and 5 mL DCM, dicyclohexylcarbodiimide (DCC, 573 mg, 2.78 mmol) was added in one portion. The reaction mixture was stirred at room temperature for 24 h before the precipitate was filtered. The filtrate was purified by column chromatography (SiO₂, 1:1 CHCl₃-acetone) to give the product as pale yellow oil (829 mg, 78%). ¹H-NMR (400 MHz, CDCl₃): δ 6.11 (m, 2H), 4.23 (m, 2H), 3.69 (m, 2H), 3.69 (br m, 44H), 3.54 (m, 2H), 3.36 (s, 3H), 3.03 (m, 1H), 2.90 (m, 1H), 2.25 (m, 1H), 1.92 (m, 1H), 1.50 (m, 1H), 1.35 (m, 2H).

Scheme S1 Synthesis of norbornene-folic acid monomer **Nor-OEG-FA**.

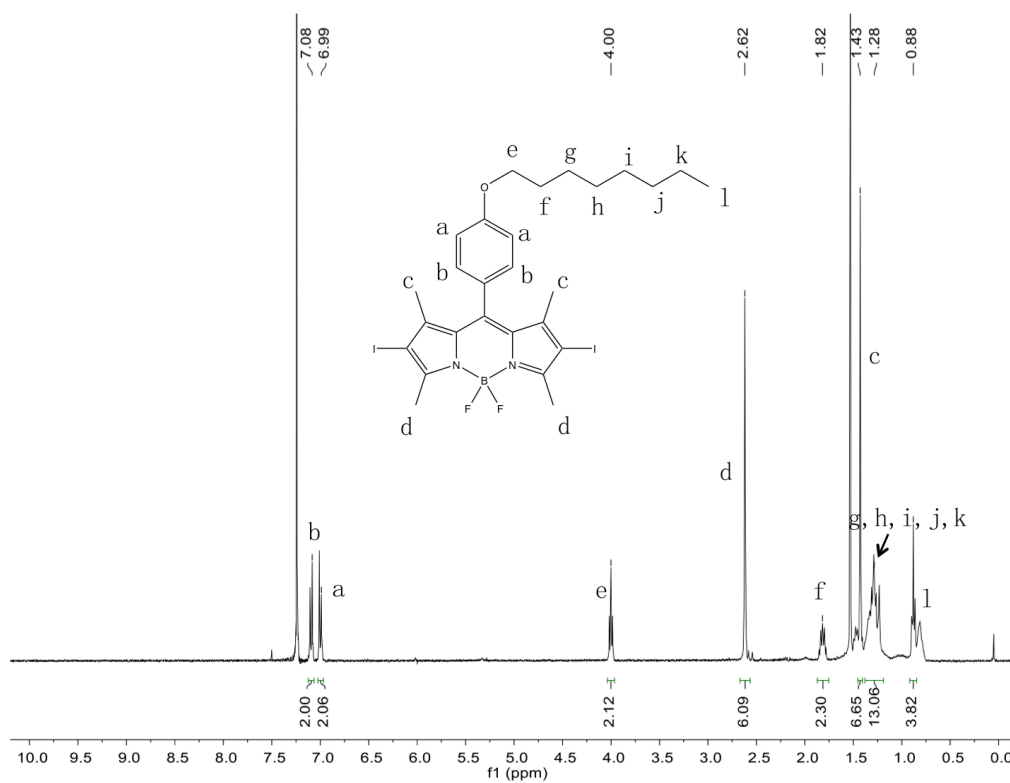
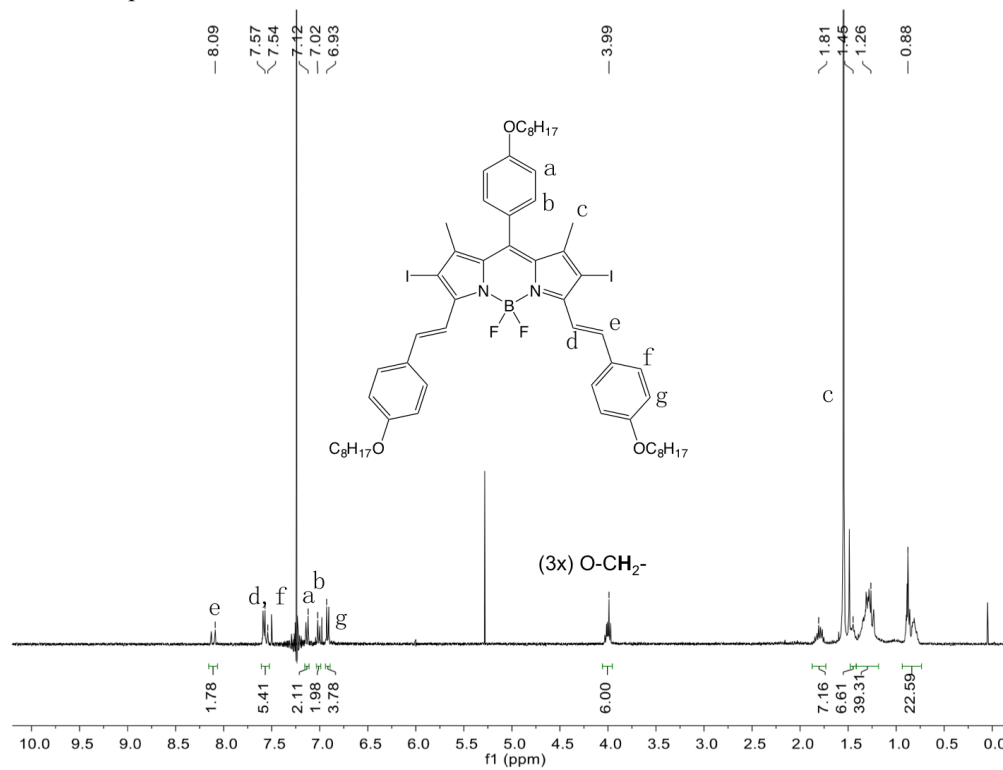
Synthesis of Nor-OEG-NHBoc. *O*-(2-Aminoethyl)-*O'*-[2-(Boc-amino)ethyl]decaethylene glycol (271 mg, 0.420 mmol) and *exo*-5-norbornenecarboxylic acid (62 mg, 0.45 mmol) were dissolved in 8.8 mL anhydrous DCM followed by addition of DCC (108 mg, 0.520 mmol) and DMAP (7.0 mg, 0.057 mmol). The reaction mixture was stirred at room temperature for 15 h, then filtered to remove white precipitate. The filtrate was evaporated under reduced pressure and purified by column chromatography (SiO₂, 1:2 CHCl₃-acetone, *R_f* = 0.18) to give the **Nor-OEG-NHBoc** as a colorless sticky oil (164 mg, 51%). ¹H NMR (400 MHz, CDCl₃): δ = 6.14 – 6.06 (m, 2H), 5.04 (s, 1H), 3.68 – 3.58 (m, 40H), 3.53 (dt, *J* = 12, 5 Hz, 4H), 3.45 (t, *J* = 5.2 Hz, 2H), 3.33 – 3.24 (m, 2H), 2.90 (s, 2H), 2.05 – 1.95 (m, 1H), 1.89 (dt, *J* = 11.3, 3.9 Hz, 1H), 1.42 (s, 9H), 1.36 – 1.28 (m, 2H), 0.85 (dd, *J* = 15.2, 8.4 Hz, 1H).

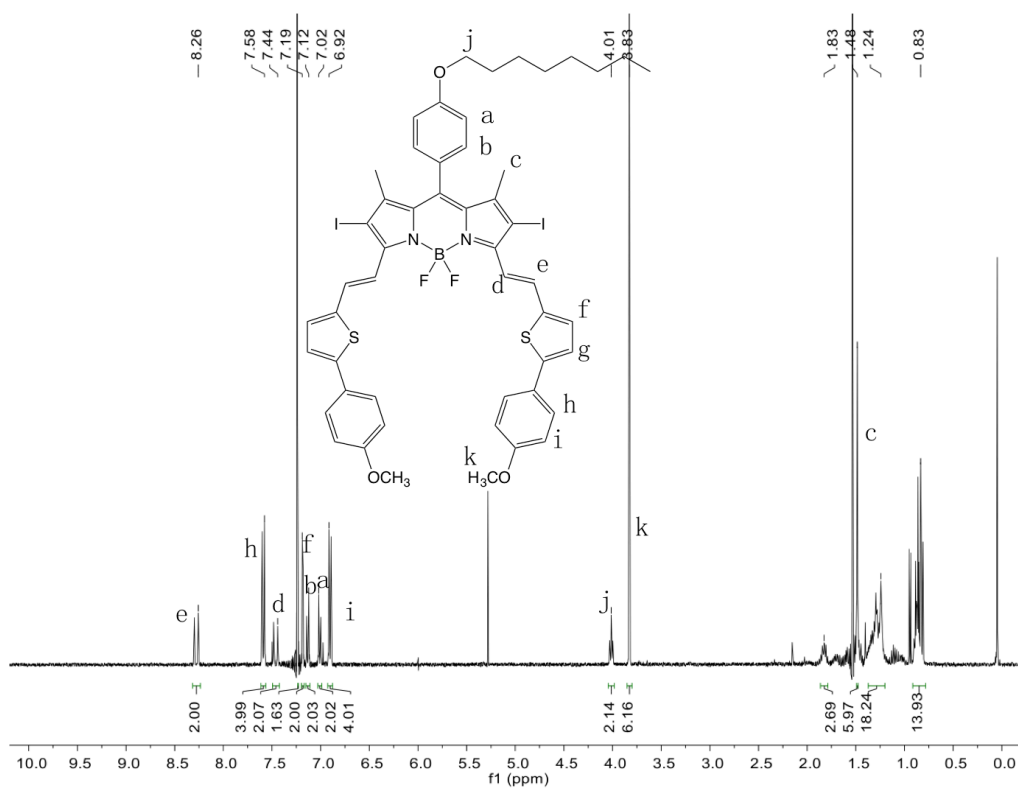
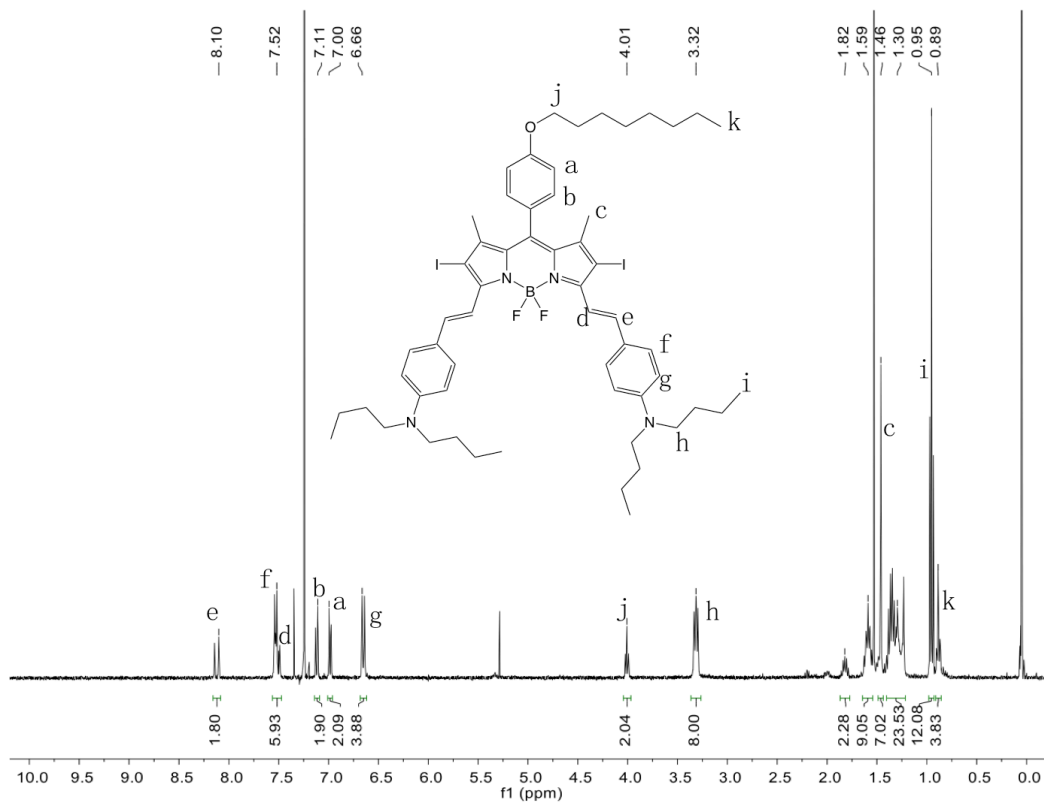
Synthesis of Nor-OEG-NH₂. **Nor-OEG-NHBoc** (164 mg, 0.210 mmol) was dissolved in 3.5 mL anhydrous DCM. TFA (0.40 mL, 1/40×) was added dropwise and the mixture was stirred at room temperature for 50 min. The reaction mixture was washed with saturated NaHCO₃ solution followed by saturated NaCl solution. The DCM layer was dried over Na₂SO₄ and concentrated to give the **Nor-OEG-NH₂** as a colorless sticky oil (67 mg, 47%). ¹H NMR (400 MHz, CDCl₃): δ = 6.20 – 6.01 (m, 2H), 3.90 (t, *J* = 4.9 Hz, 2H), 3.77 – 3.54 (m, 40H), 3.56 (t, *J* = 5.0 Hz, 2H), 3.45 (t, *J* = 5.2 Hz, 2H), 3.16 (t, *J* = 4.9 Hz, 2H), 2.90 (d, *J* = 9.5 Hz, 2H), 2.02 (dd, *J* = 9.1, 4.4 Hz, 1H), 1.91 (t, *J* = 4.0 Hz, 1H), 1.36 – 1.28 (m, 2H), 0.98 – 0.78 (m, 1H).

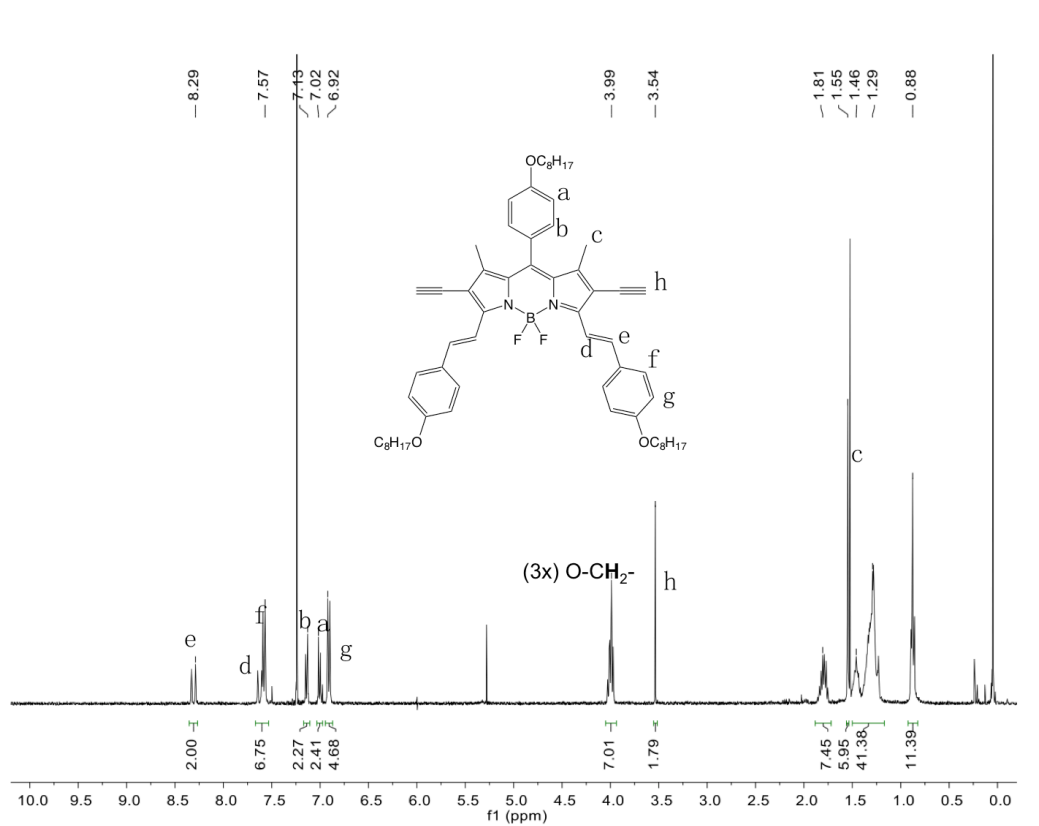
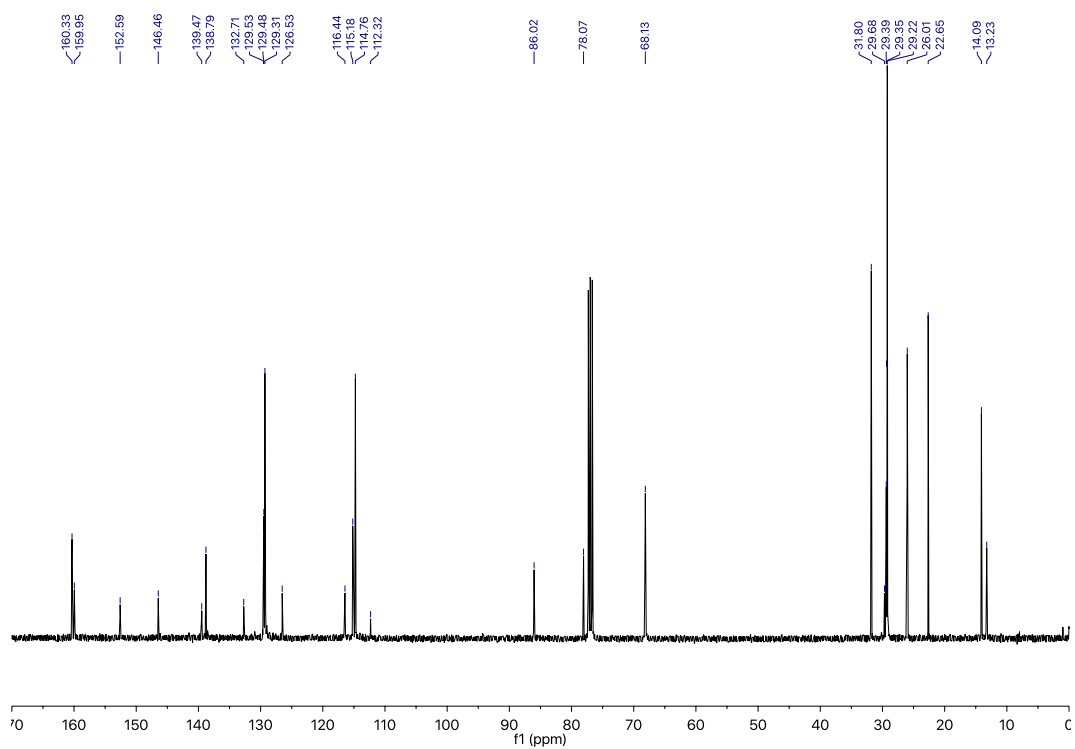
Synthesis of Nor-OEG-FA. **Nor-OEG-NH₂** (67 mg, 0.10 mmol) and folic acid (42 mg, 0.095 mmol) was dissolved into 3.5 mL anhydrous DMSO followed by addition of DCC (27 mg, 0.13 mmol) and DMAP (2.0 mg, 0.016 mmol). The reaction mixture was stirred at room temperature for 24 h. The mixture was filtered and precipitate in diethyl ether. The precipitate was washed with diethyl ether and dried under vacuum to give the **Nor-OEG-FA** as a yellow solid (90 mg, 85%). ¹H NMR (400 MHz, DMSO-*d*₆): δ = 12.27 (br, 1H), 11.57 (s, 1H), 8.64 (d, *J* = 2.4 Hz, 1H), 8.28 – 8.19 (m, 1H), 7.90 (d, *J* = 10.9 Hz, 1H), 7.65 (dd, *J* = 9.1, 3.2 Hz, 2H), 6.97 (dt, *J* = 8.5, 2.6 Hz, 1H), 6.63 (dd, *J* = 8.9, 2.6 Hz, 2H), 6.11 (q, *J* = 2.6 Hz, 2H), 4.48 (d, *J* = 5.3 Hz, 2H), 4.38 – 4.23 (m, 1H), 3.53 – 3.44 (m, 40H), 3.38 (dt, *J* = 13.6, 5.3 Hz, 2H), 3.44 – 3.33 (m, 2H), 3.24 – 3.15 (m, 2H), 2.82 (s, 1H), 2.75 (d, *J* = 2.6 Hz, 1H), 2.31 (t, *J* = 7.4 Hz, 2H), 2.03 (dd, *J* = 9.3, 3.9 Hz, 1H), 1.79 – 1.69 (m, 1H), 1.63 (d, *J* = 7.8 Hz, 1H), 1.27 – 1.08 (m, 2H).

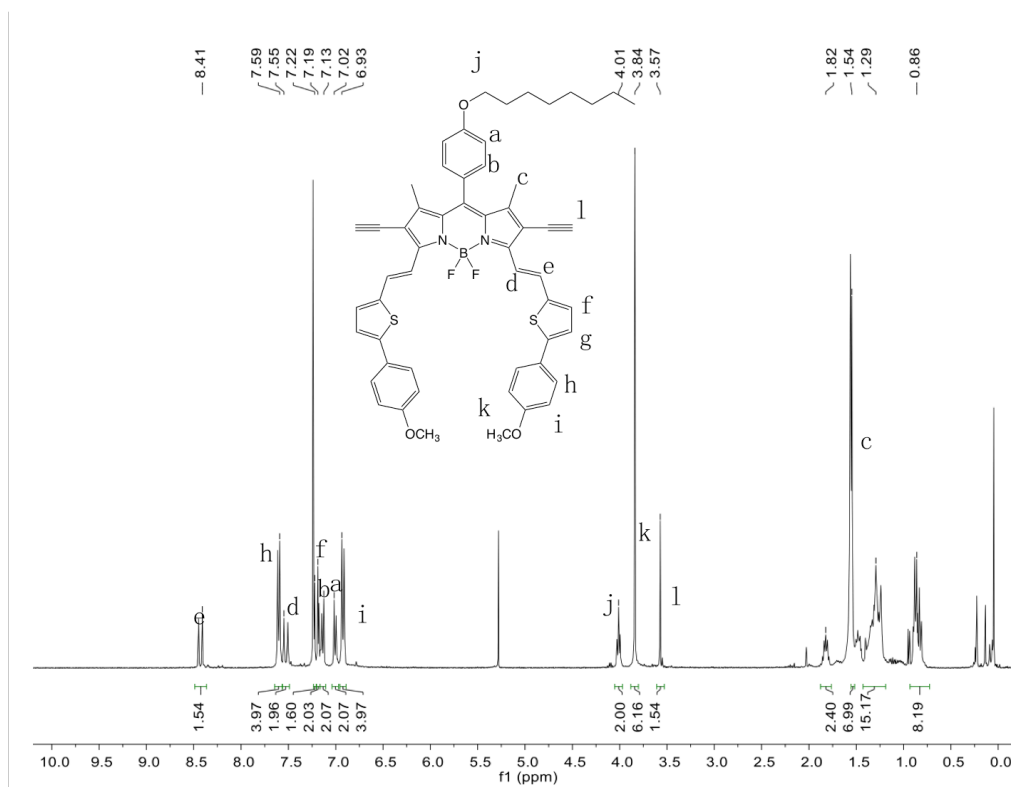
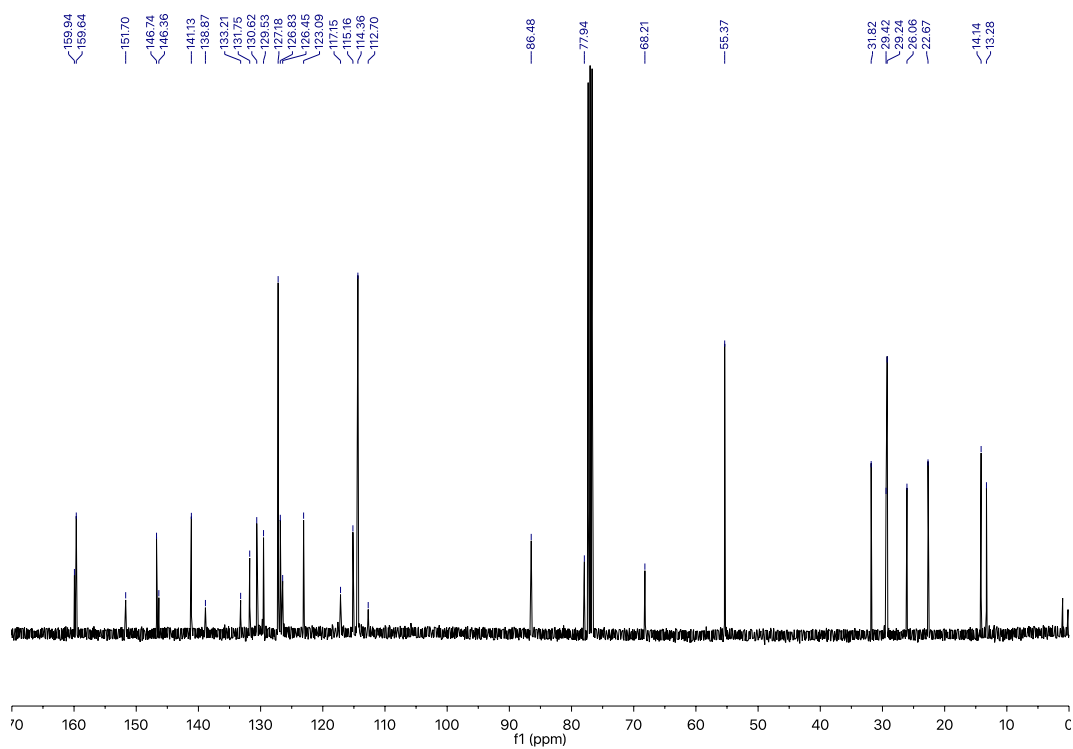
Table S2 Molecular weight and molecular weight distribution of **P1a-c**.

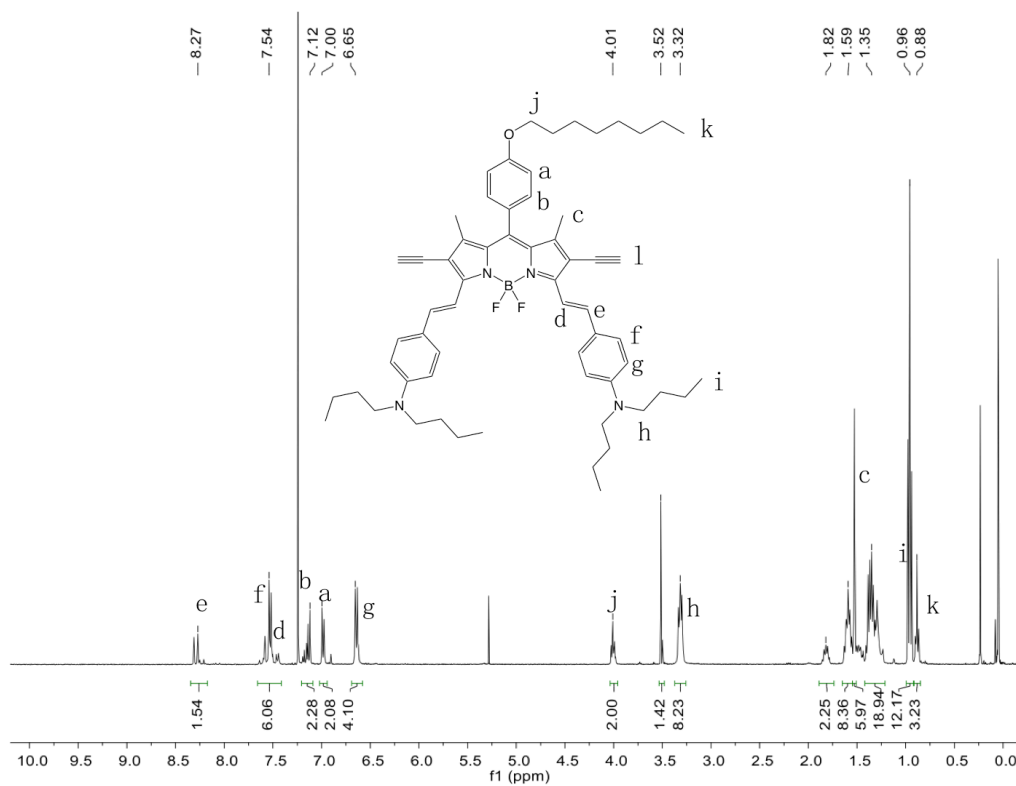
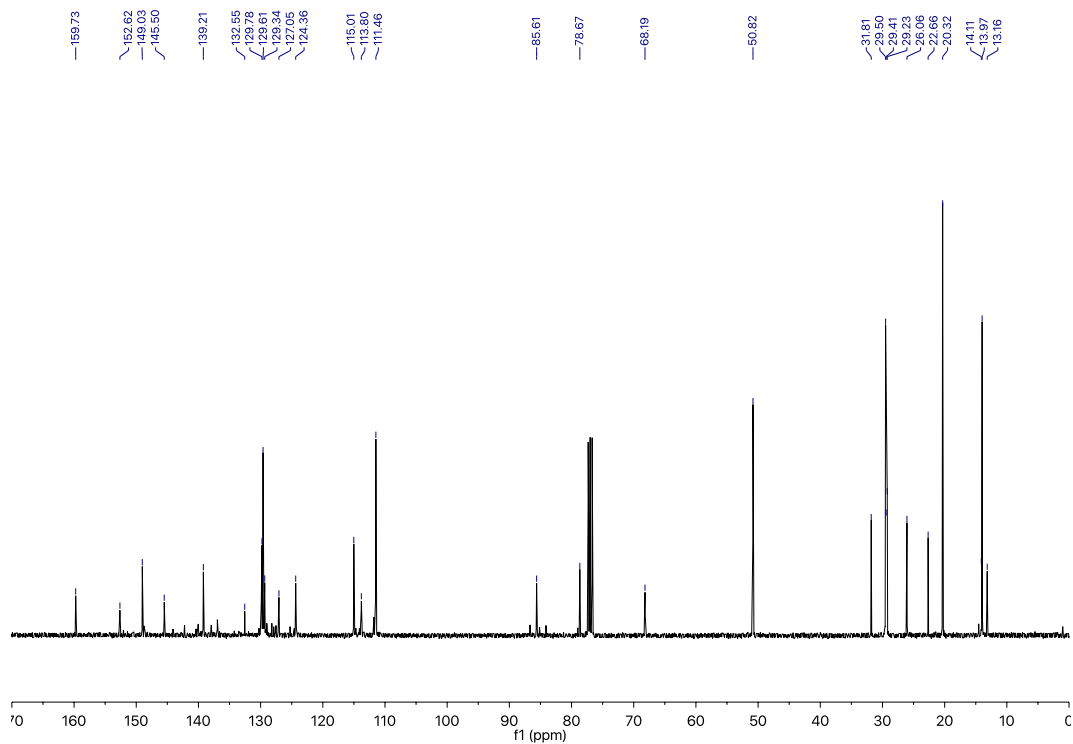
Entry	FA%	<i>M_n</i> of living block (kDa)	PDI of living block	<i>M_n</i> of block copolymer (kDa)	PDI of block copolymer
P1a	0%	16.3	1.20	60	2.86
P1b	10%	14.0	1.12	41	2.97
P1c	20%	15.2	1.08	28	1.82

Fig. S1 $^1\text{H-NMR}$ of compound 2.Fig. S2 $^1\text{H-NMR}$ of compound 4a.

Fig. S3 ¹H-NMR of compound 4b.Fig. S4 ¹H-NMR of compound 4c.

Fig. S5 $^1\text{H-NMR}$ of compound **5a**.Fig. S6 $^{13}\text{C-NMR}$ of compound **5a**.

Fig. S7 ¹H-NMR of compound **5b**.Fig. S8 ¹³C-NMR of compound **5b**.

Fig. S9 ¹H-NMR of compound 5c.Fig. S10 ¹³C-NMR of compound 5c.

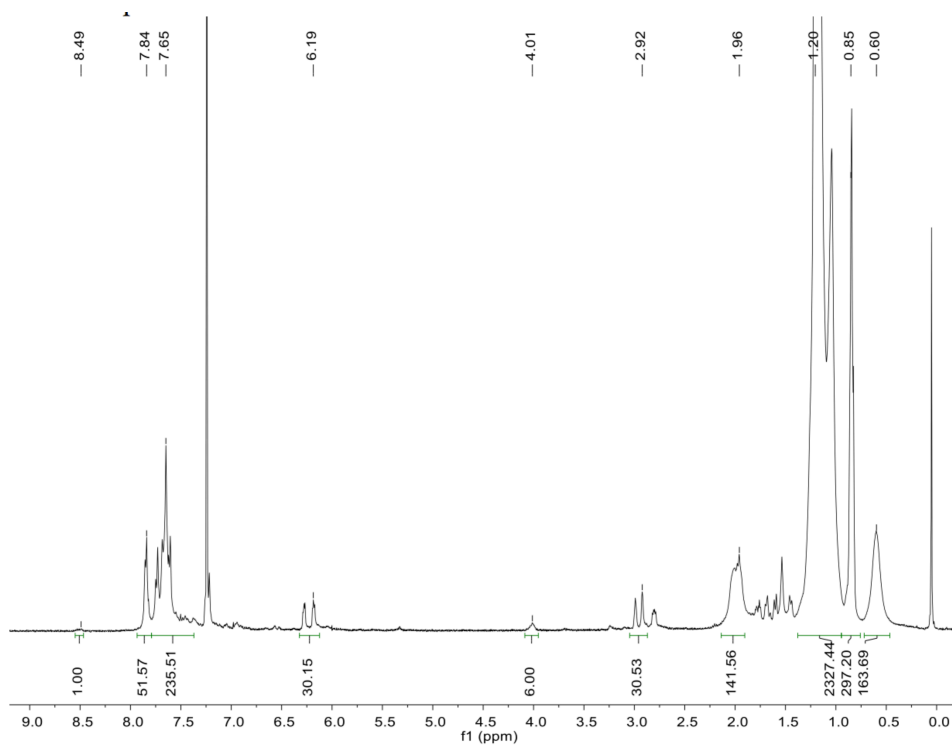


Fig. S11 $^1\text{H-NMR}$ of conjugated polymer **P1**.

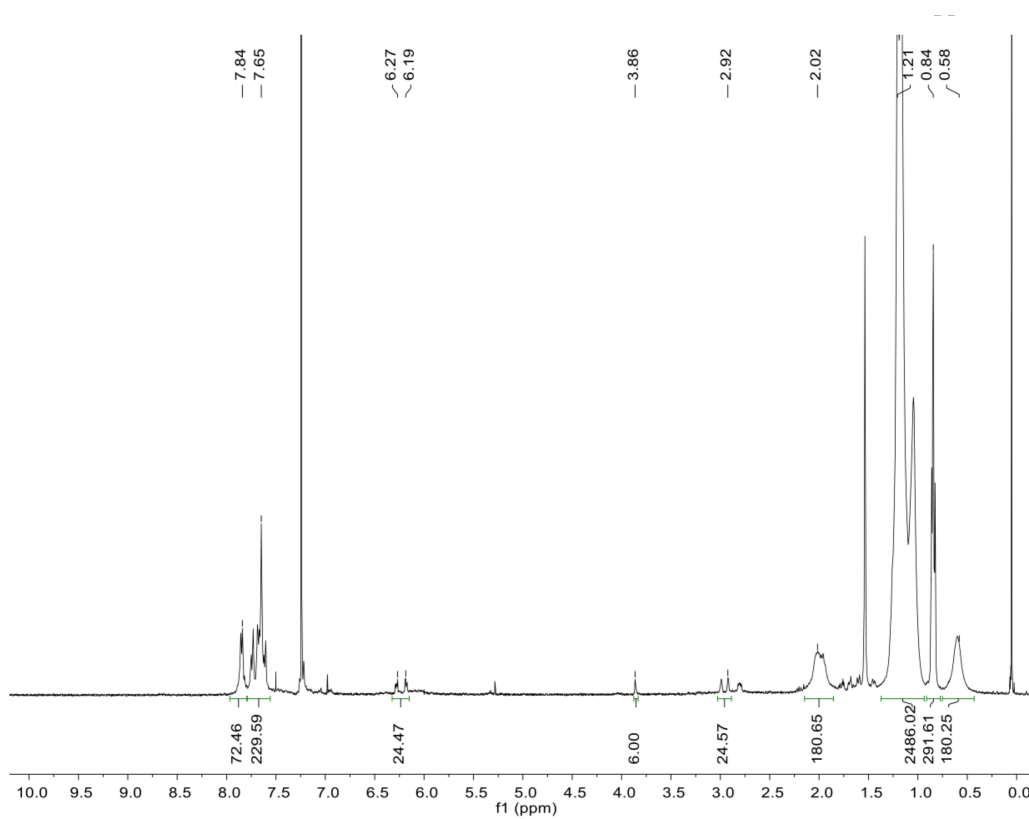


Fig. S12 $^1\text{H-NMR}$ of conjugated polymer **P2**.

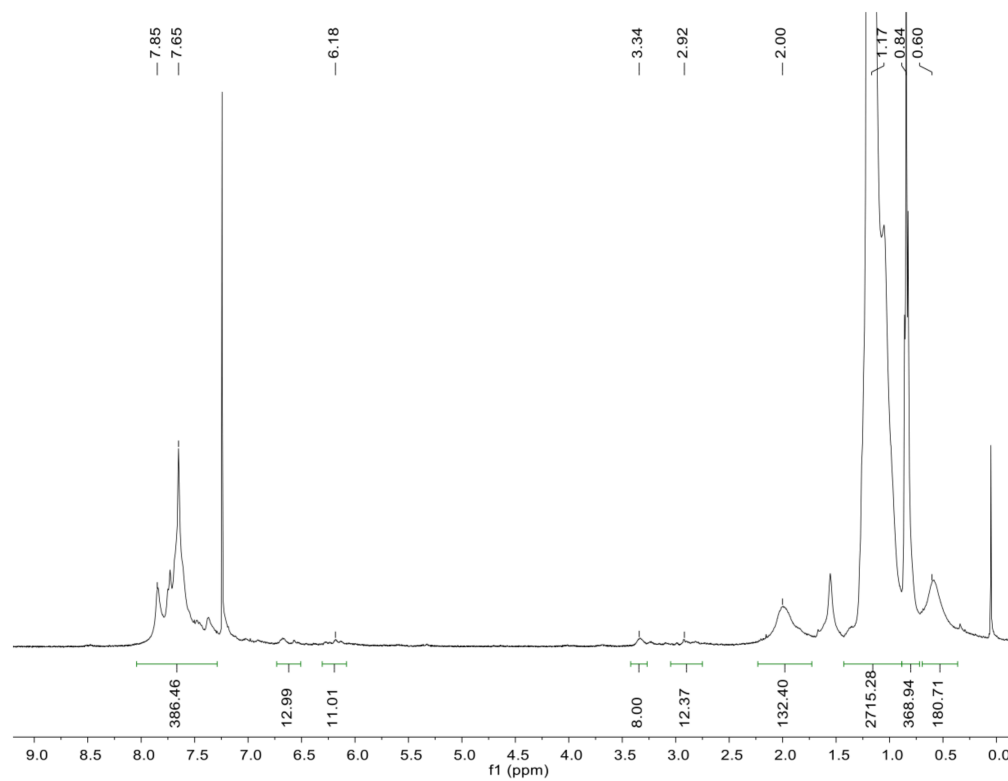


Fig. S13 $^1\text{H-NMR}$ of conjugated polymer **P3**.

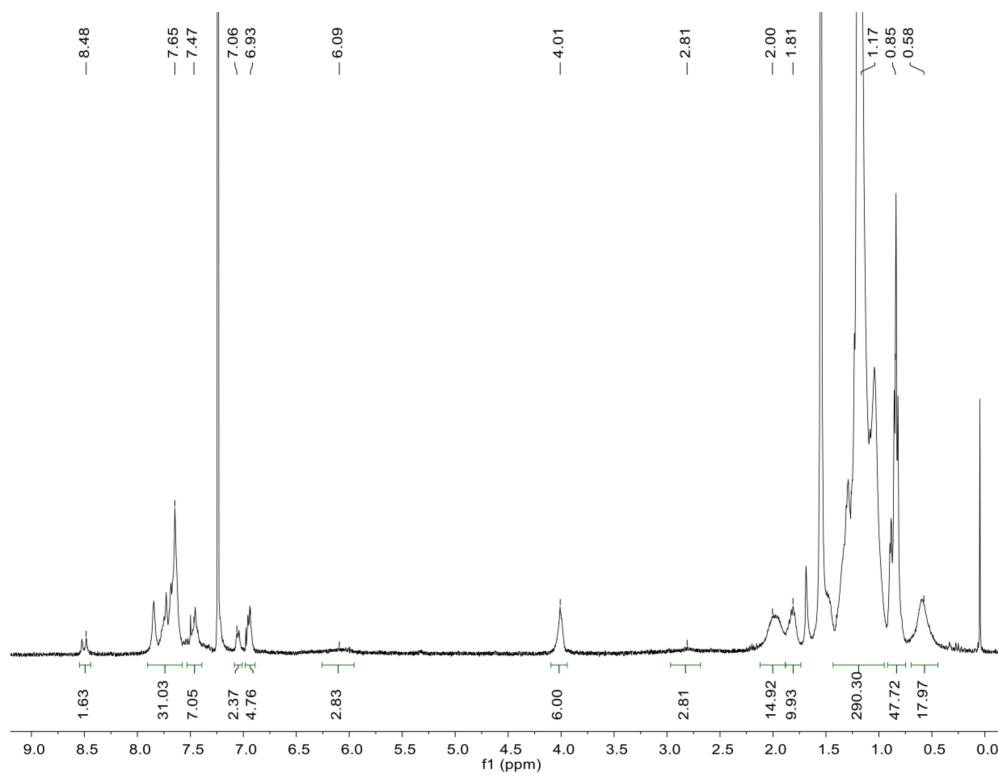


Fig. S14 $^1\text{H-NMR}$ of conjugated polymer **P4**.

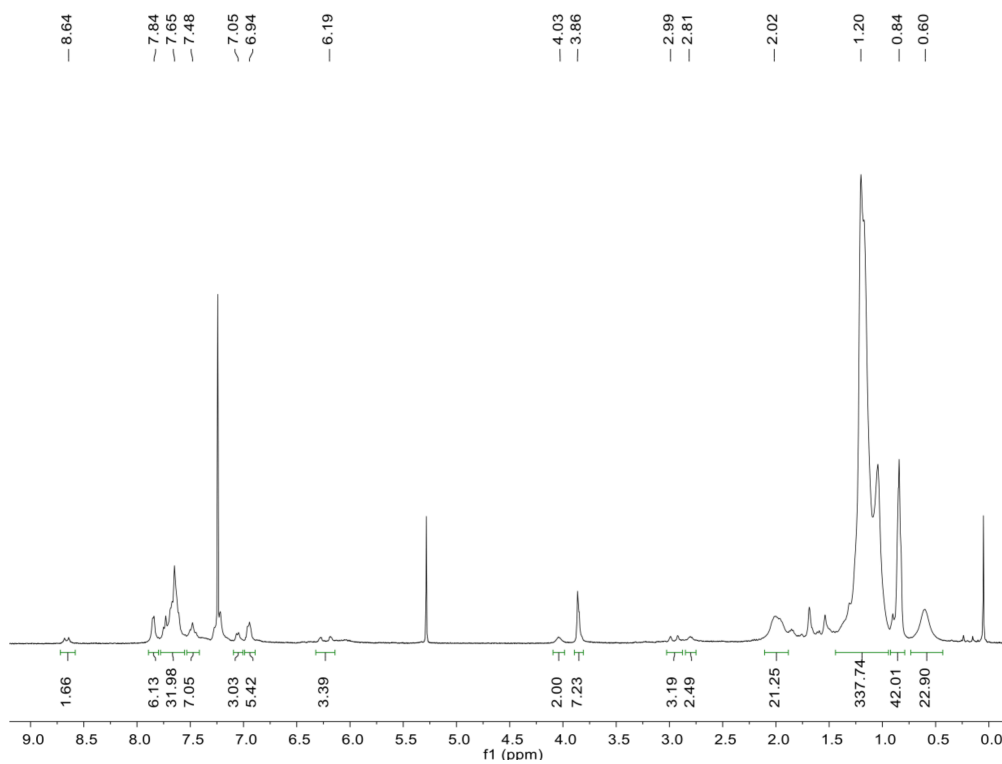


Fig. S15 $^1\text{H-NMR}$ of conjugated polymer P5.

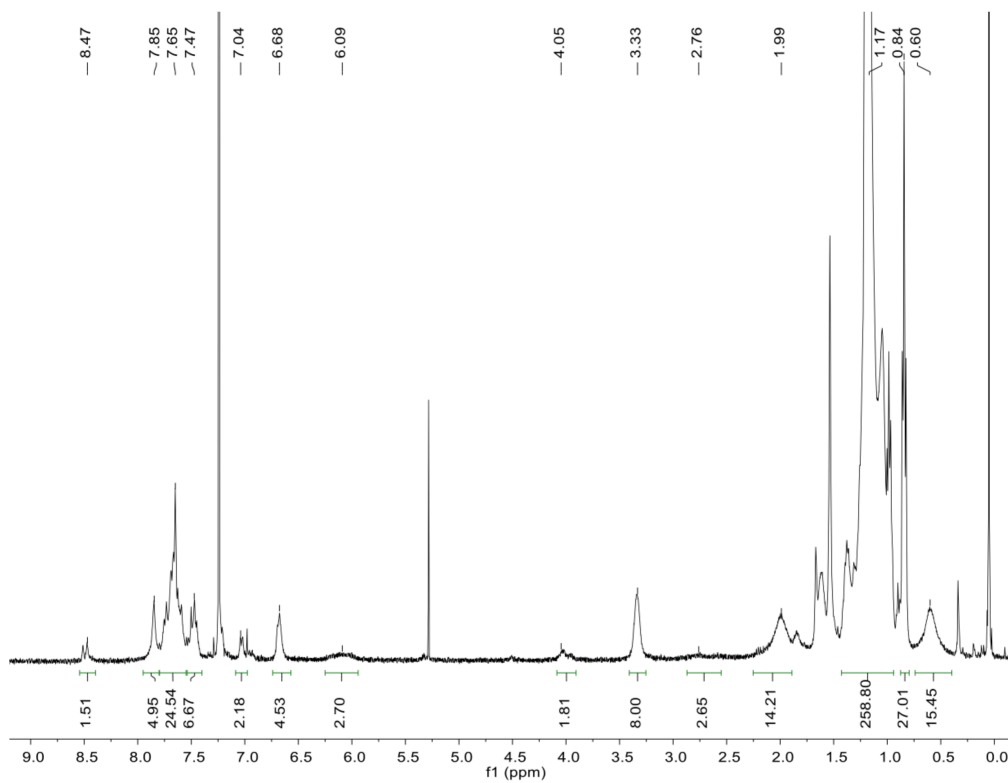
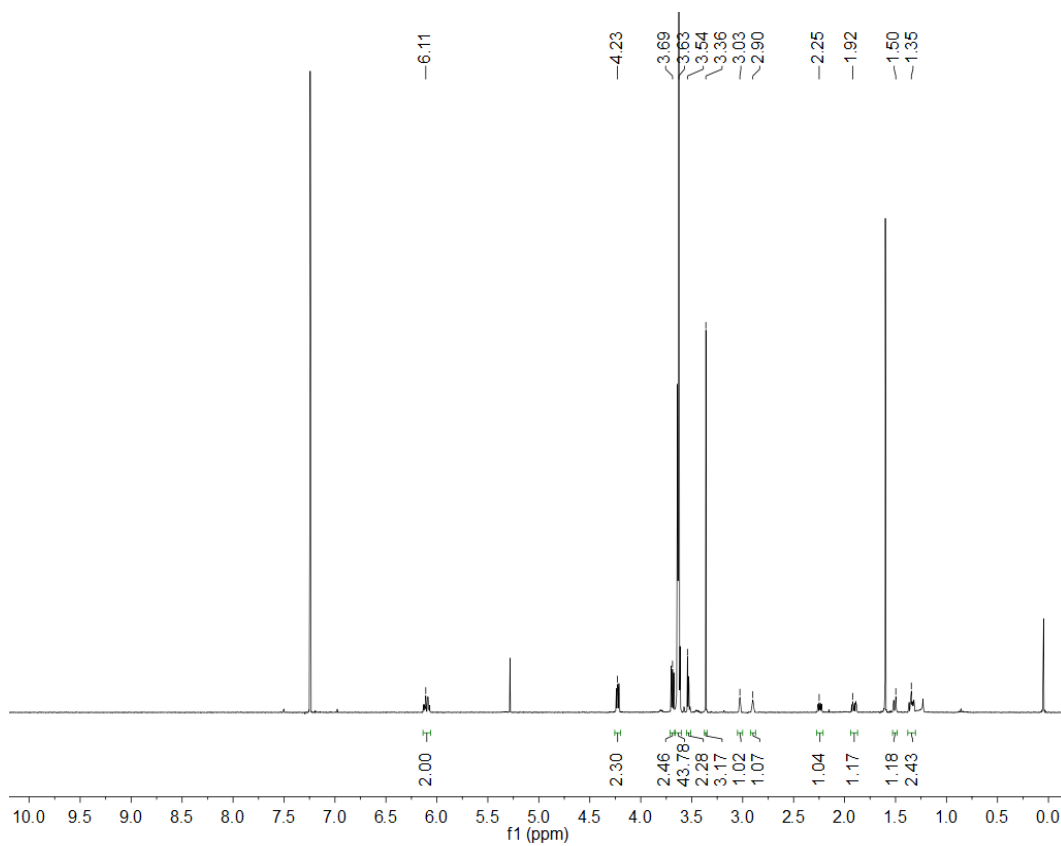
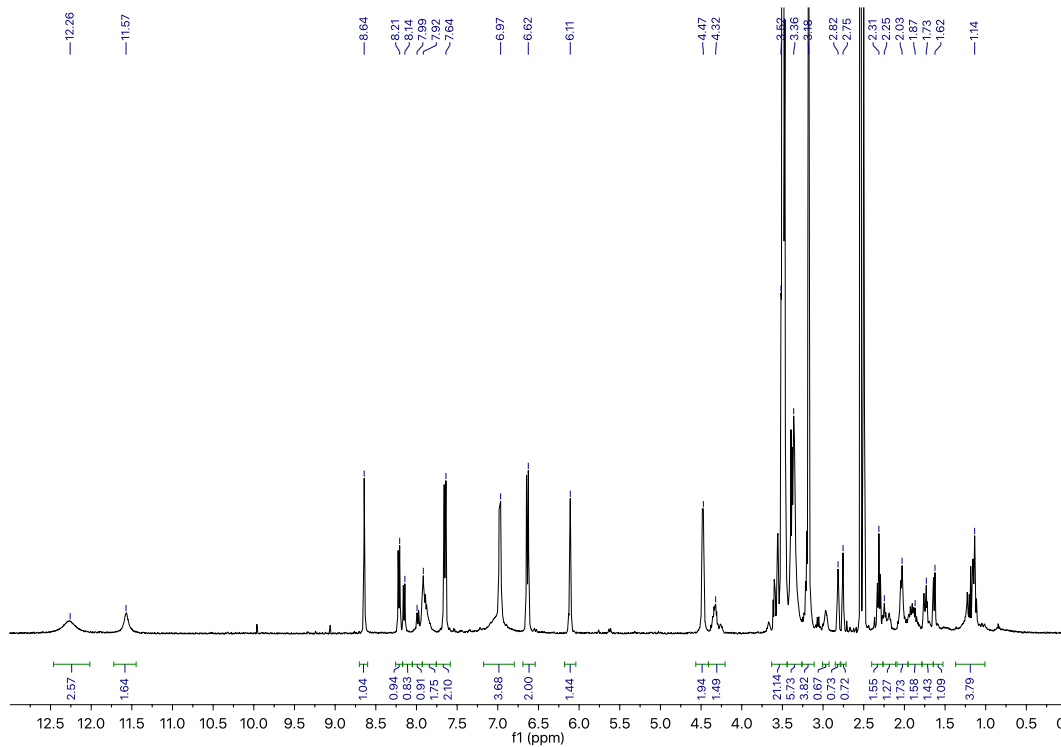
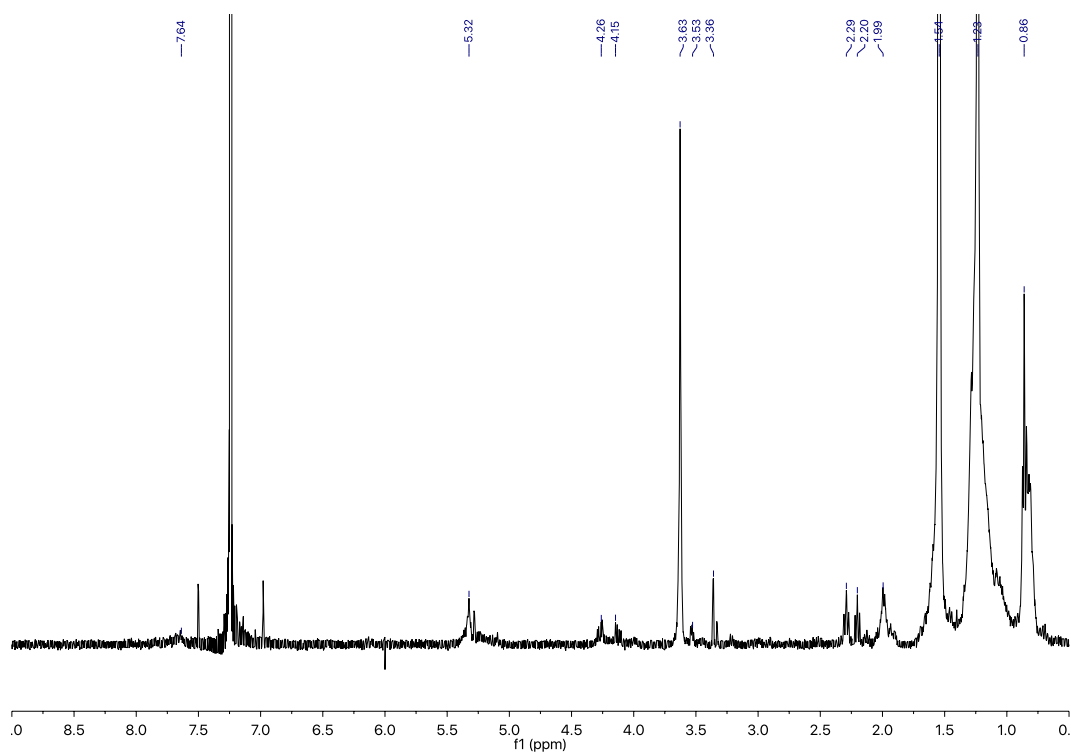
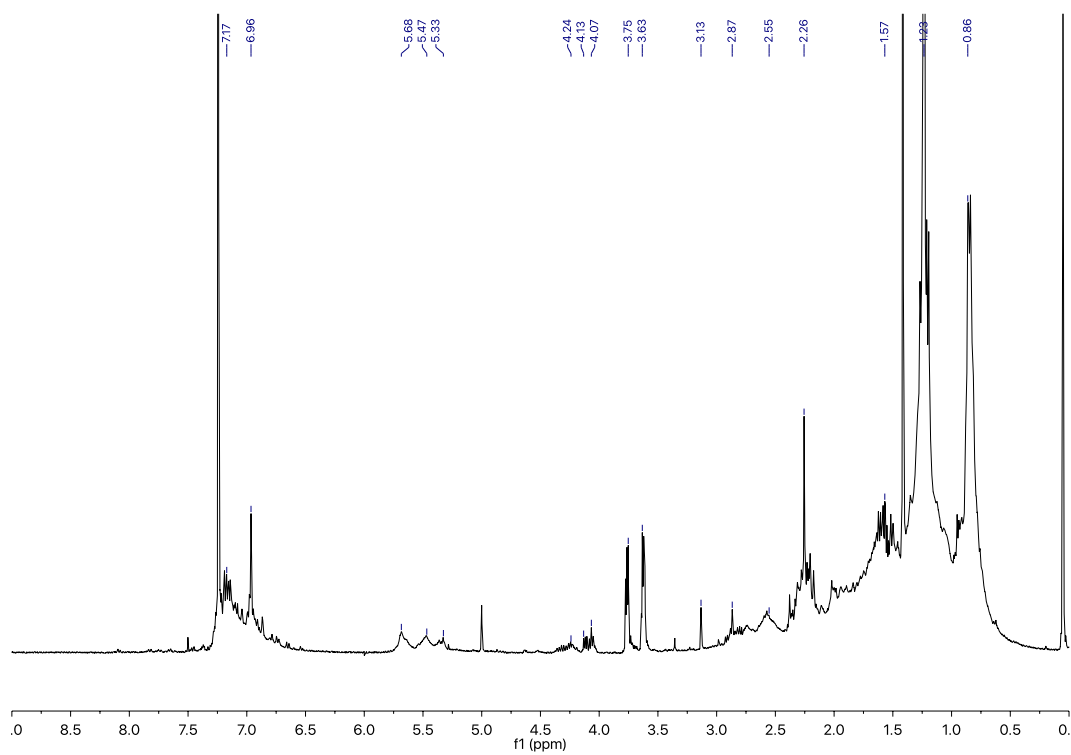


Fig. S16 $^1\text{H-NMR}$ of conjugated polymer P6.

Fig. S17 $^1\text{H-NMR}$ of Nor-OEG.Fig. S18 $^1\text{H-NMR}$ of Nor-OEG-FA in DMSO- d_6 .

Fig. S19 $^1\text{H-NMR}$ of P1a.Fig. S20 $^1\text{H-NMR}$ of P1b.

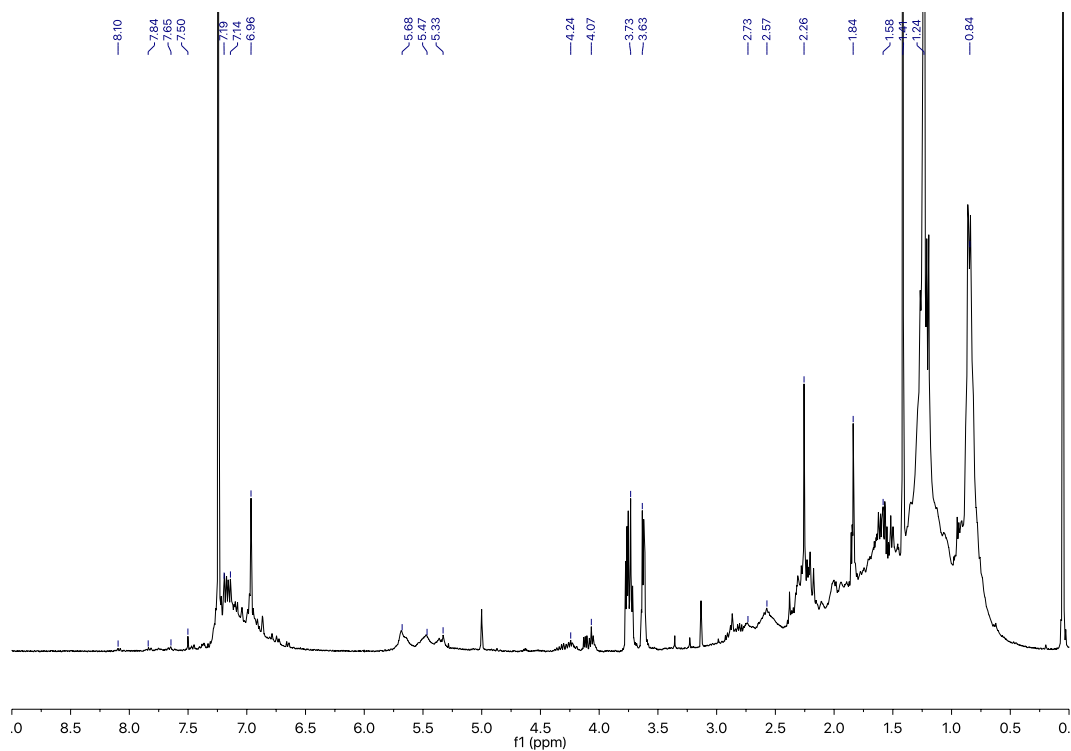


Fig. S21 $^1\text{H-NMR}$ of **P1c**.

Characterization of **PFBT**

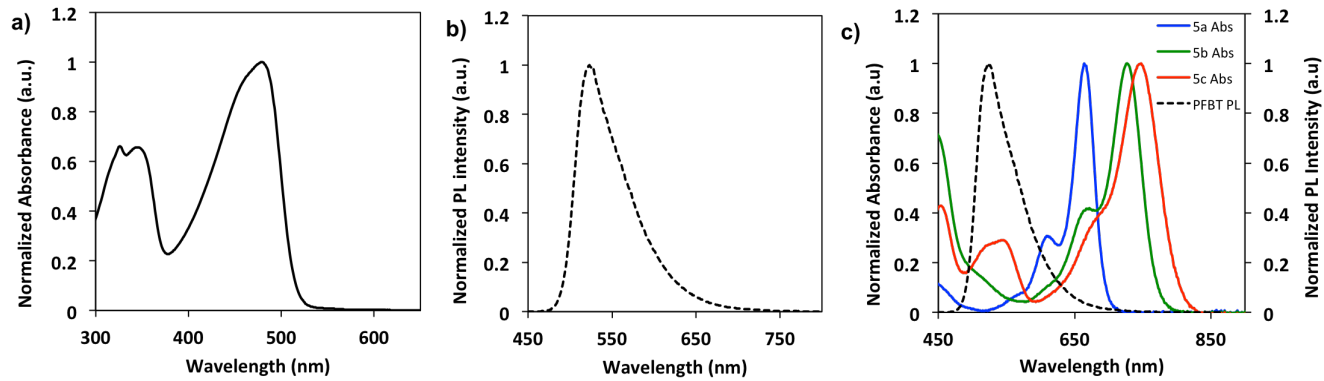


Fig. S22 Normalized a) absorption and b) emission spectra of **PFBT** in THF. c) Overlap of the absorption spectra of **5a-c** and the emission spectrum of **PFBT**.

Characterization of nanoparticles

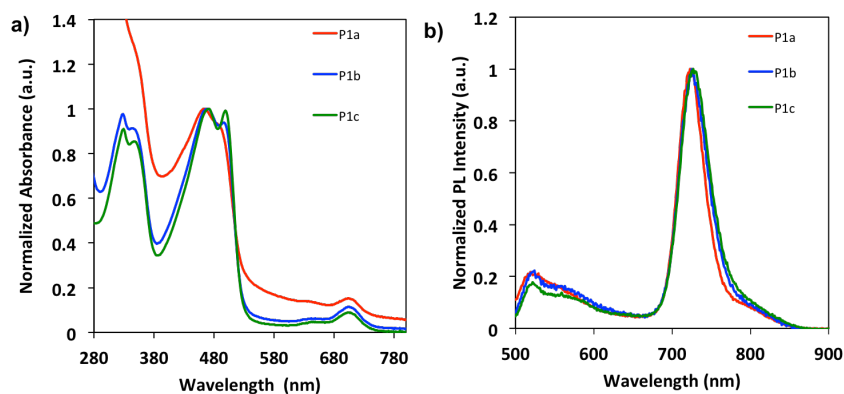


Fig. S23 Normalized a) absorption and b) emission spectra of **P1a-c** nanoparticles.

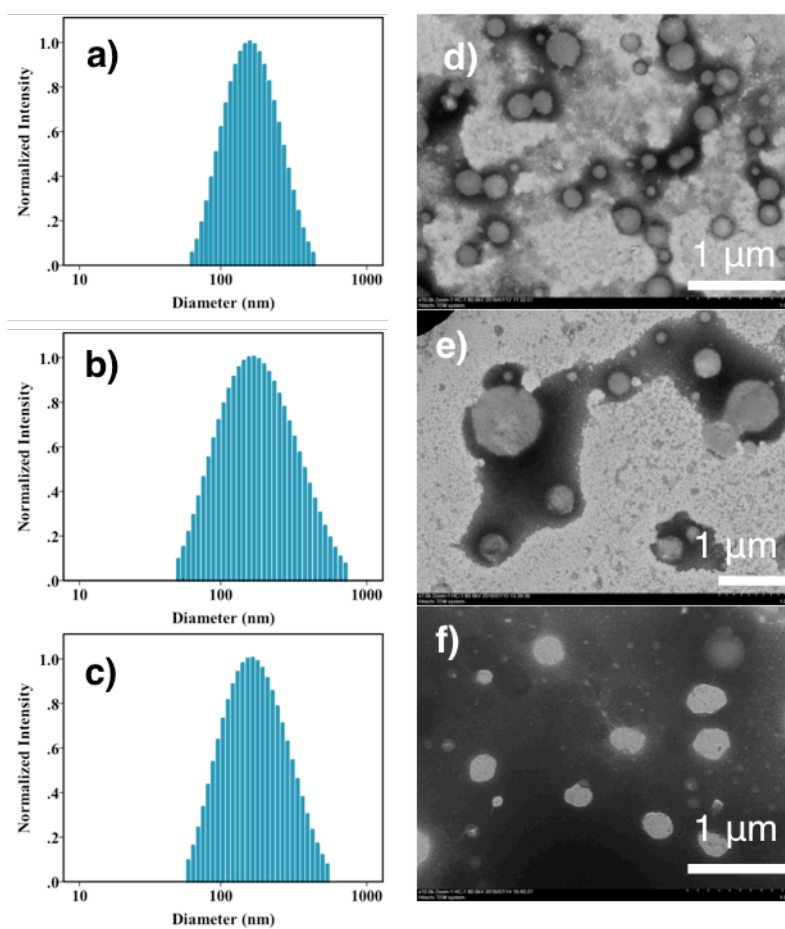


Fig. S24 a)-c) Histogram of the size distribution of **P1-c** NPs measured by DLS; d)-f) TEM image of **P1a-c** NPs.

References

1. Y. Rong, C. Wu, J. Yu, X. Zhang, F. Ye, M. Zeigler, M. E. Gallina, I.-C. Wu, Y. Zhang and Y.-H. Chan, *Acs Nano*, 2013, **7**, 376-384.