Supporting Information

for

Self-assembly and phase separation of amphiphilic dyads based on 4,7-bis(2-thienyl)benzothiodiazole and perylene diimide

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Contents **Page**

I. Optical Spectra of the Dyads

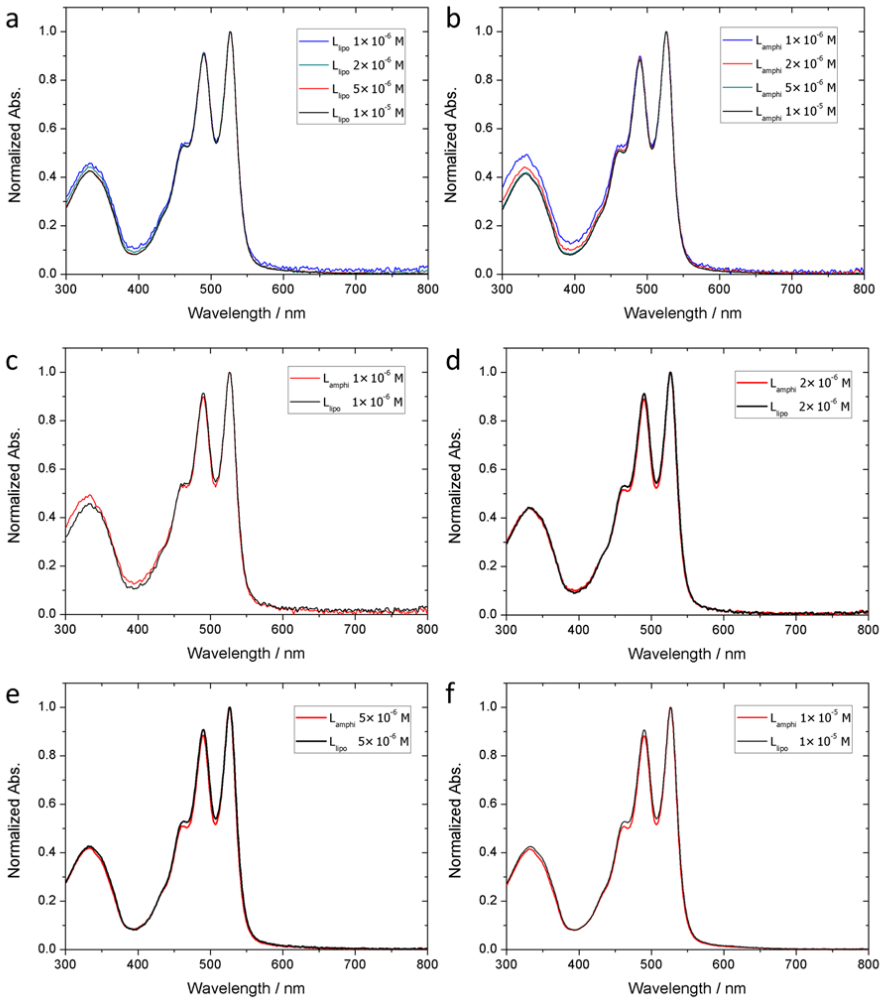


Fig. S1 Concentration-dependent UV-vis absorption spectra of $\mathbf{L_{lipo}}$ (a) and $\mathbf{L_{amphi}}$ (b) and comparison of normalized absorption spectra of $\mathbf{L_{lipo}}$ and $\mathbf{L_{amphi}}$ in selected concentrations of 1×10^{-6} M (c), 2×10^{-6} M (d), 5×10^{-6} M (e), 1×10^{-5} M (f). The peak shapes in (a) and (b) did not change with concentration except the fluctuation in short wavelength region, because the background noise was slightly enhanced when the concentration is lowered to 1×10^{-6} M (blue line, solvent: chloroform, 25 ^{o}C).

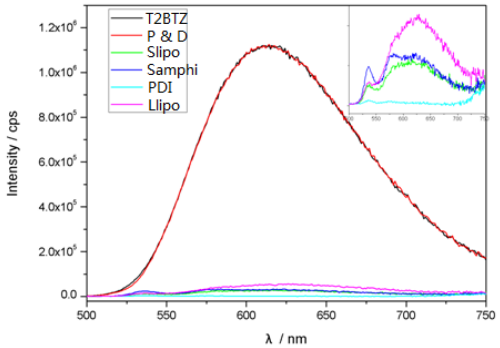


Fig. S2 Fluorescence spectra for dyads and reference compounds in chloroform at 2.5×10^{-6} M excited at 331 nm. The red line represents the mixture of **PDI** and **T2BTZ** both at 2.5×10^{-6} M.

II. Electrochemical Characterization

Ferrocene/ferrocenium was used as an external reference. The energy level of ferrocene (Fc)/ Fc^+ is assumed to be -4.8 eV under the vacuum level.[1] The half-wave potential of oxidation peak of Fc was measured to be 0.427 V against Ag/AgCl. The HOMO levels of **S_{lipo}, S_{amphi}, T2BTZ** were estimated from the half-wave potentials of the oxidation peaks. The LUMO levels of **S_{lipo}, S_{amphi}, PDI** were estimated from the half-wave potentials of the oxidation peaks.

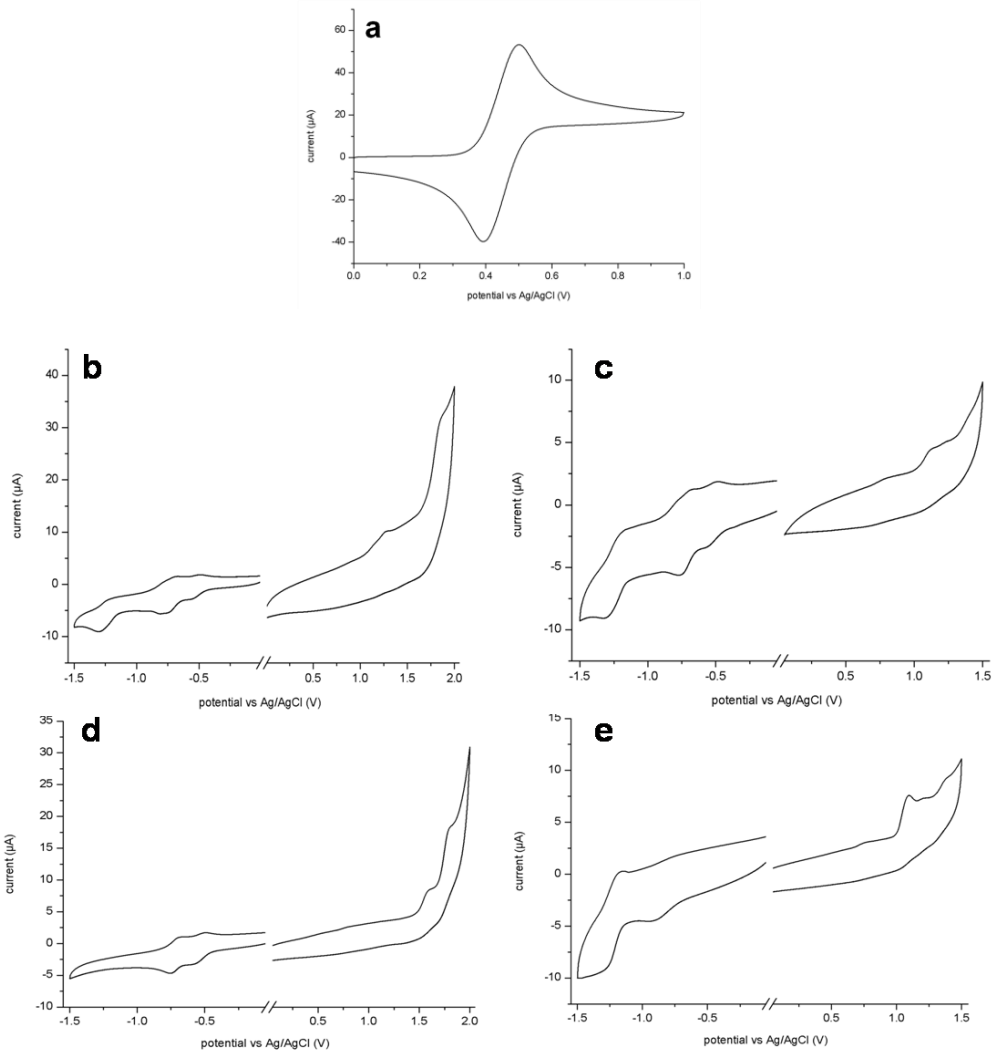


Fig. S3 Cyclic voltammetry curves of Ferrocene (a), **S_{lipo}** (b)**, S_{amphi}** (c), **PDI** (d) and **T2BTZ** (e).

1. B. W. D'Andrade, S. Datta, S. R. Forrest, P. Djurovich, E. Polikarpov and M. E. Thompson, *Organic Electronics*, 2005, **6**, 11.

III. Differential Scanning Calorimetry (DSC)

2.0 mg of the sample was encapsulated in a sealed aluminum pan, and was subjected to heating and cooling scans with temperature ranging from -50 ^{o}C to 200 ^{o}C at a rate of 10 ^{o}C/min.

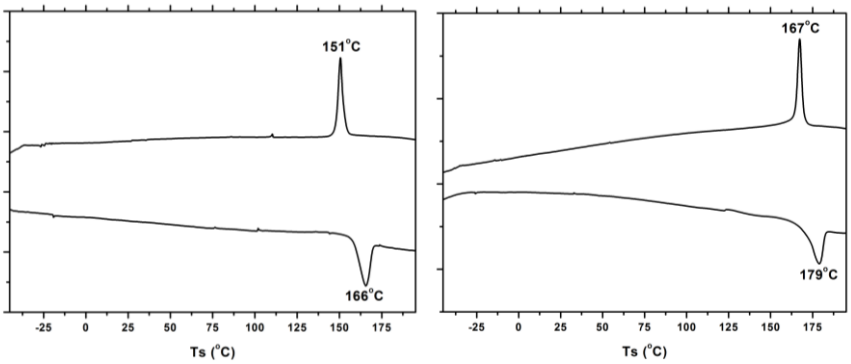


Fig. S4 Differential scanning calorimetry of **L$_{lipo}$** (left) and **L$_{amphi}$** (right).

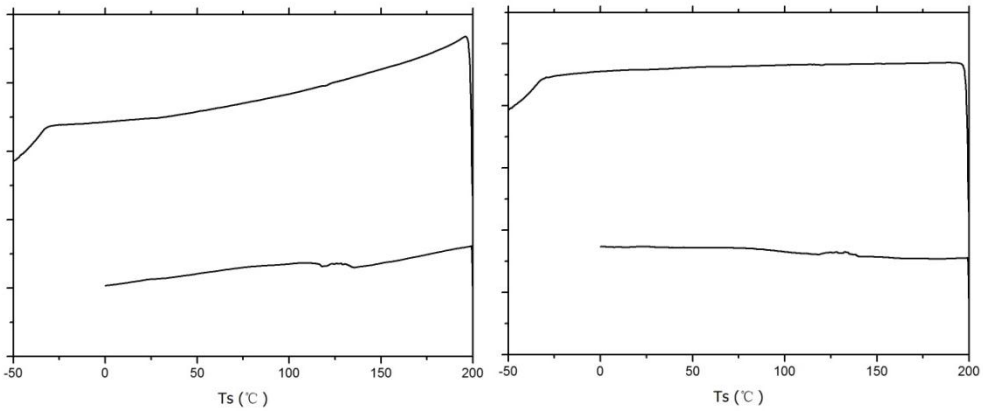


Fig. S5 Differential scanning calorimetry of **S$_{lipo}$** (left) and **S$_{amphi}$** (right).

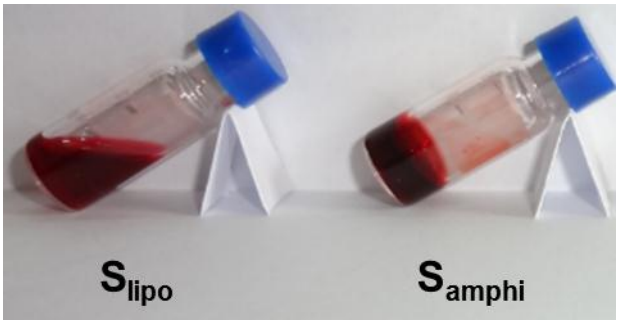
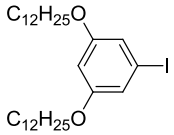


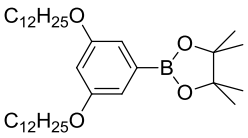
Fig. S6 Photograph of short-linker dyads in DCM/n-hexane.

IV. Synthetic Procedures and Characterization Data

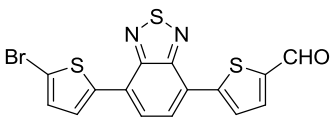
a. Synthesis of p-type fragment



3,5-Bis(dodecyloxy)iodobenzene (p-3). To an acetone solution (75 mL) of **p-1** (472 mg, 2.0 mmol) was added anhydrous K_2CO_3 (1.10 g, 8 mmol) and dodecyl bromide (1.5 mL). The mixture was stirred at reflux under air. After removal of volatiles *in vacuo*, the residue was dissolved by water. The aqueous phase was extracted by EtOAc. The organic extract was washed with water and brine, dried over anhydrous $MgSO_4$, filtered and concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, PE) to afford **p-3** (955 mg, 83%) as a white crystal. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 6.83 (d, 2*H*, *J* = 2.1 Hz), 6.39 (t, 1*H*, *J* = 2.1 Hz), 3.88 (t, 4*H*, *J* = 6.6 Hz), 1.74 (quint, 4*H*, *J* = 6.6 Hz), 1.53-1.11 (m, 36*H*), 0.88 (t, 6*H*, *J* = 6.6 Hz). MS (ESI): Calcd. for $C_{30}H_{53}IO_2$: 572.31. Found: 573.32 (m/z+H^+).

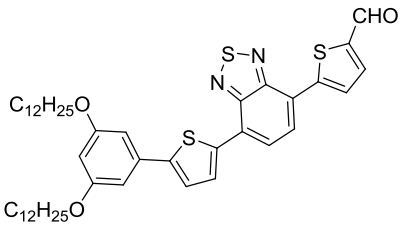


2-(3,5-Bis(dodecyloxy)phenyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (p-4). A 50 mL Schlenk tube was charged with **p-3** (2.10 g, 3.67 mmol), bis(pinacolato)diboron (1.40 g, 5.51 mmol), Pd(dppf)Cl_2 (134 mg, 0.18 mmol) and anhydrous potassium acetate (1.08 g, 11 mmol). The tube was evacuated and refilled with nitrogen for 3 times. To the mixture was added degassed DMF (20 mL) under nitrogen. The mixture was stirred at 60 ^{o}C for 40 h. The mixture was diluted with EtOAc and washed with water repeatedly to remove DMF. The organic extract was dried over anhydrous $MgSO_4$, filtered and concentrated *in vacuo*. The solid residue was subjected to flash column chromatography (silica gel, PE/DCM, 2/1 to 1/1 v/v) to afford **p-4** (1.56 g, 74%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 6.93 (d, 2*H*, *J* = 2.1 Hz), 6.57 (t, 1*H*, *J* = 2.1 Hz), 3.96 (t, 4*H*, *J* = 6.6 Hz), 1.76 (quint, 4*H*, *J* = 6.6 Hz), 1.56-1.06 (m, 48*H*), 0.89 (t, 6*H*, *J* = 6.6 Hz).^{13}C NMR (100 MHz, $CDCl_3$, 40 ^{o}C): δ (ppm) 160.0, 112.3, 105.2, 83.9, 68.0, 32.0, 29.7, 29.6, 29.4, 26.1, 24.8, 22.7, 14.2. MS (EI): Calcd. for $C_{36}H_{65}BO_4$: 572. Found: 572 (m/z). Elem. Anal.: Calcd. for $C_{36}H_{65}BO_4$: C, 75.50; H, 11.44. Found: C, 75.55; H, 11.43.

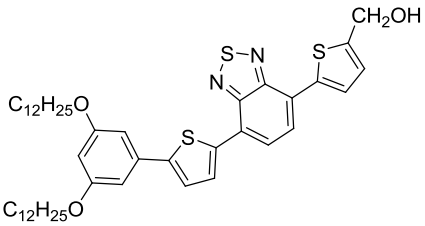


Compound p-5. Protected from light, NBS (413 mg, 1.83 mmol) was added by portions to a solution of aldehyde **p-2** (500 mg, 1.52 mmol) in $CHCl_3$/AcOH (25 mL/25 mL) at 0 ^{o}C under air. The mixture was stirred at room temperature for 18 h. The mixture was filtered and the solid residue was washed with water, methanol and $CHCl_3$. The solid was purified by washing with hot $CHCl_3$ to afford **p-5** (449 mg, 72%) as a brown powder. NMR data is absent due to the extremely

poor solubility of **p-5** in most solvents. MS (ESI): Calcd. for $C_{15}H_7BrN_2OS_3$: 407.89. Found: 407.89 (m/z), 408.90 (m/z+H^+).



Compound p-6. A 50 mL Schlenk tube was charged with **p-5** (55.3 mg, 0.136 mmol), **p-4** (117 mg, 0.204 mmol), $Pd(PPh_3)_4$ (8 mg, 0.07 mmol). The tube was evacuated and refilled with nitrogen for 3 times. To the mixture was added degassed 2 M K_2CO_3 (0.5 mL) and THF (2 mL) under nitrogen. The mixture was stirred at reflux for 22 h, then quenched with aqueous NH_4Cl. The aqueous layer was extracted by DCM. The combined organic extract was washed with water and brine, dried over anhydrous $MgSO_4$, filtered and concentrated *in vacuo*. The solid residue was subjected to flash column chromatography (silica gel, PE/DCM, 1/1 v/v) to get the crude product. Recrystallization from DCM/MeOH afforded **p-6** (79 mg, 76%) as a magenta powder. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 9.97 (s, 1*H*), 8.20 (d, 1*H*, *J* = 3.9 Hz), 8.14 (d, 1*H*, *J* = 3.9 Hz), 7.98 (d, 1*H*, *J* = 7.6 Hz), 7.89 (d, 1*H*, *J* = 7.6 Hz), 7.83 (d, 1*H*, *J* = 3.9 Hz), 7.39 (d, 1*H*, *J* = 3.9 Hz), 6.83 (d, 2*H*, *J* = 2.1 Hz), 6.44 (t, 1*H*, *J* = 2.1 Hz), 4.00 (t, 4*H*, *J* = 6.6 Hz), 1.81 (quint, 4*H*, *J* = 6.6 Hz), 1.54-1.13 (m, 36*H*), 0.88 (t, 6*H*, *J* = 6.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 182.8, 160.7, 152.3, 152.2, 148.5, 146.7, 143.3, 137.9, 136.6, 135.4, 129.3, 127.8, 127.2, 124.5, 124.3, 124.0, 68.2, 32.0, 29.7, 29.5, 29.4, 26.1, 22.7, 14.1. MS (EI): Calcd. for $C_{45}H_{60}N_2O_3S_3$: 772. Found: 772 (m/z). Elem. Anal.: Calcd. for $C_{45}H_{60}N_2O_3S_3$: C, 69.91; H, 7.82; N, 3.62. Found: C, 70.04; H, 7.85; N, 3.50.

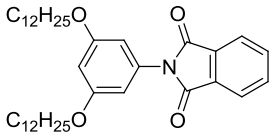


Compound p-7. To a suspension of $LiAlH_4$ (28 mg, 0.74 mmol) in THF (5 mL) at 0 ^{o}C was added dropwise a solution of **p-6** (570 mg, 0.74 mmol) in THF (20 mL). The reaction mixture was stirred at room temperature for 30 min and then quenched with saturated aqueous Na_2SO_4. The insoluble solid was removed by filtration and the filtrate was concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, PE/DCM, 1/1 to 0/1 v/v) to afford **p-7** (508 mg, 89%) as a reddish solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.08 (d, 1*H*, *J* = 3.9 Hz), 7.98

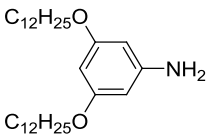
(d, 1*H*, *J* = 3.9 Hz), 7.86 (d, 1*H*, *J* = 7.6 Hz), 7.82 (d, 1*H*, *J* = 7.6 Hz), 7.38 (d, 1*H*, *J* = 3.9 Hz), 7.10 (d, 1*H*, *J* = 3.9 Hz), 6.83 (d, 2*H*, *J* = 2.2 Hz), 6.43 (t, 1*H*, *J* = 2.2 Hz), 4.91 (d, 2*H*, *J* = 6.0 Hz), 4.01 (t, 4*H*, *J* = 6.6 Hz), 1.90 (t, 1*H*, *J* = 6.0 Hz), 1.81 (quint, 4*H*, *J* = 6.6 Hz), 1.54-1.13 (m, 36H), 0.88 (t, 6*H*, *J* = 6.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 160.7, 152.5, 152.4, 145.6, 145.4, 139.4, 138.4, 135.7, 128.4, 127.3, 126.2, 125.7, 125.6, 125.4, 125.1, 124.2, 104.7, 100.9, 68.3, 60.3, 32.0, 29.7, 29.5, 29.4, 26.1, 22.7, 14.1. MS (EI): Calcd. for $C_{45}H_{62}N_2O_3S_3$: 774. Found: 774 (m/z). Elem. Anal.: Calcd. for $C_{45}H_{62}N_2O_3S_3$: C, 69.72; H, 8.06; N, 3.61. Found: C, 69.53; H, 8.08; N, 3.66.

b. Synthesis of dyads L_{lipo} and L_{amphi}

When R=R_1:

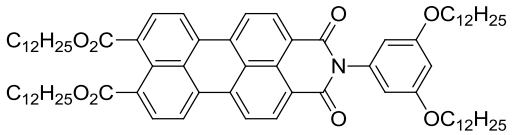


2-(3,5-Bis(dodecyloxy)phenyl)isoindoline-1,3-dione (n-3). To a DMF solution (5 mL) of 2-(3,5-dihydroxyphenyl)isoindoline-1,3-dione (**n-1**) (80 mg, 0.31 mmol) was added anhydrous K_2CO_3 (173 mg, 1.25 mmol) and dodecyl bromide (0.5 mL). The mixture was stirred at 80 ^{o}C under air. After removal of DMF *in vacuo*, the residue was extracted by DCM. The organic extract was washed with water and brine, dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, PE/EtOAc, 20/1 v/v) to afford **n-3** (169 mg, 91%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.95 (m, 2*H*), 7.79 (m, 2*H*), 6.55 (d, 2*H*, *J* = 2.2 Hz), 6.49 (t, 1*H*, *J* = 2.2 Hz), 3.94 (t, 4*H*, *J* = 6.4 Hz), 1.77 (quint, 4*H*, *J* = 6.9 Hz), 1.52-1.03 (m, 36*H*), 0.88 (t, 6*H*, *J* = 6.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 167.2, 160.5, 134.4, 132.9, 131.8, 123.7, 105.5, 101.6, 68.3, 32.0, 29.7, 29.6, 29.4, 29.2, 26.1, 22.7, 14.2. MS (EI): Calcd. for $C_{38}H_{57}NO_4$: 591. Found: 591 (m/z). Elem. Anal.: Calcd. for $C_{38}H_{57}NO_4$: C, 77.11; H, 9.71; N, 2.37. Found: C, 77.07; H, 10.06; N, 2.30.

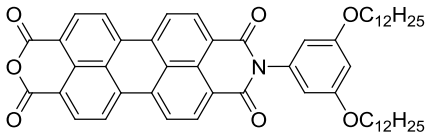


3,5-Bis(dodecyloxy)aniline (n-4). Phthalimide **n-3** (919 mg, 1.55 mmol) was dissolved in absolute ethanol/THF (40 mL/10 mL) at 60 ^{o}C. Hydrazine monohydrate (85%, 1 mL) was added to the mixture and the mixture was stirred at reflux for 2 h. After removal of insoluble solid by

filtration, the solution was concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, PE/EtOAc/Et_3N, 10/1/0.2 v/v) to afford **n-4** (586 mg, 82%) as a pale yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 5.91 (t, 1*H*, *J* = 2.1 Hz), 5.85 (d, 2*H*, *J* = 2.1 Hz), 3.88 (t, 4*H*, *J* = 6.6 Hz), 3.63 (br, 2*H*), 1.74 (quint, 4*H*, *J* = 6.9 Hz), 1.50-1.14 (m, 36*H*), 0.88 (t, 6*H*, *J* = 6.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 161.2, 148.2, 94.3, 92.0, 67.8, 31.9, 29.7, 29.6, 29.4, 29.3, 26.1, 22.7, 14.1. MS (EI): Calcd. for $C_{30}H_{55}NO_2$: 461. Found: 461 (m/z). Elem. Anal.: Calcd. for $C_{30}H_{55}NO_2$: C, 78.03; H, 12.01; N, 3.03. Found: C, 77.95; H, 12.24; N, 2.79.

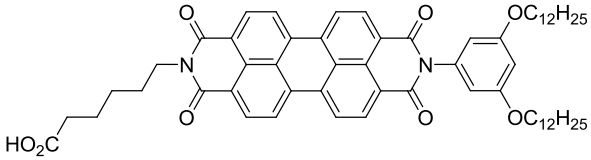


***N*-(3,5-Bis(dodecyloxy)phenyl)-9,10-bis(dodecyloxycarbonyl)-perylene-3,4-dicarboximide (n-5).** A dry 50 mL Schlenk tube was charged with anhydride **n-2** (243 mg, 0.325 mmol), **n-4** (166 mg, 0.358 mmol), DMAP (40 mg, 0.325 mmol) and imidazole (3 g). The reaction mixture was stirred at 130 ^{o}C under nitrogen for 24 h, quenched with 3 M HCl. The aqueous layer was extracted by chloroform. The combined organic extract was washed with brine, dried over anhydrous Na_2SO_4. After removal of solvents *in vacuo*, the residue was subjected to flash column chromatography (silica gel, PE/DCM, 1/1 v/v) to afford **n-5** (307 mg, 79%) as a red solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.32 (br, 2*H*), 7.89 (br, 6*H*), 6.56 (br, 3*H*), 4.37 (t, 4*H*, *J* = 7.0 Hz), 3.96 (t, 4*H*, *J* = 6.8 Hz), 1.86 (quint, 4*H*, *J* = 7.0 Hz), 1.75 (quint, 4*H*, *J* = 6.8 Hz), 1.55-1.04 (m, 72*H*), 0.97-0.76 (m, 6*H*). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.1, 163.2, 160.8, 136.7, 135.0, 131.8, 131.5, 131.1, 130.1, 128.8, 128.7, 128.5, 125.4, 122.3, 121.9, 121.6, 107.1, 102.2, 68.3, 65.9, 32.0, 29.7, 29.5, 29.4, 29.3, 28.7, 26.1, 22.7, 14.1. MS (ESI): Calcd. for $C_{78}H_{111}NO_8$: 1189.8. Found: 1190.8 (m/z + H^+). Elem. Anal.: Calcd. for $C_{78}H_{111}NO_8$: C, 78.68; H, 9.40; N, 1.18. Found: C, 78.76; H, 9.50; N, 1.19.

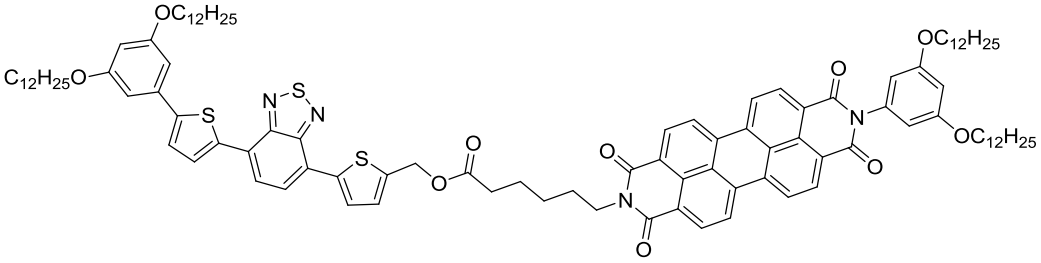


***N*-(3,5-Bis(dodecyloxy)phenyl)-perylene-9,10-dicarboximide-3,4-dicarboxylic anhydride (n-6).** To a solution of **n-5** (501 mg, 0.42 mmol) in toluene (50 mL) was added 4-toluenesulfonic acid hydrate (684 mg, 3.6 mmol). The mixture was stirred at 100 ^{o}C under air for 15 h. After removal of volatiles *in vacuo*, the residue was washed with methanol and then purified by flash

column chromatography (silica gel, $CHCl_3$) to afford **n-6** (310 mg, 88%) as a red solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm). 8.72 (m, 8*H*), 6.59 (t, 1*H*, *J* = 2.1 Hz), 6.46 (d, 2*H*, *J* = 2.1 Hz), 3.95 (t, 4*H*, *J* = 6.3 Hz), 1.76 (quint, 4*H*, *J* = 6.3 Hz), 1.48-1.15 (m, 36*H*), 0.87 (t, 6*H*, *J* = 6.6 Hz). MS (ESI): Calcd. for $C_{54}H_{61}NO_7$: 835.4. Found: 835.4 (m/z). Elem. Anal.: Calcd. for $C_{54}H_{61}NO_7$: C, 77.58; H, 7.35; N, 1.68. Found: C, 77.65; H, 7.37; N, 1.68.



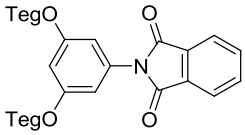
Carboxylic acid n-7. A mixture of **n-6** (416 mg, 0.50 mmol), 6-aminohexanoic acid (131 mg, 1.00 mmol), imidazole (680 mg, 10 mmol) and DMF (10 mL) was stirred at 95 ^{o}C under nitrogen for 22 h. The product was precipitated by addition of ethanol/3M aqueous HCl (1/1, v/v, 40 mL), and then filtered. The solid residue was washed with ethanol and dried at 100 ^{o}C to afford **n-7** (419 mg, 89%) as a brown solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.56 (d, 2*H*, *J* = 8.1 Hz), 8.45 (d, 2*H*, *J* = 8.1 Hz), 8.39-8.18 (m, 4*H*), 6.57 (br, 1*H*), 6.56 (br, 2*H*), 4.19 (t, 2*H*, *J* = 7.5 Hz), 3.90 (t, 4*H*, *J* = 6.3 Hz), 2.43 (t, 2*H*, *J* = 6.9 Hz), 2.00-1.62 (m, 8*H*), 1.62-1.00 (m, 38*H*), 0.86 (t, 6*H*, *J* = 6.6 Hz). MS (ESI): Calcd. for $C_{60}H_{72}N_2O_8$: 948.5. Found: 949.5 (m/z + H^+). Elem. Anal.: Calcd. for $C_{30}H_{36}NO_4$: C, 75.92; H, 7.65; N, 2.95. Found: C, 76.04; H, 7.62; N, 2.97.



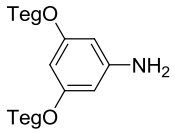
Dyad L_{lipo}. p-7 (150 mg, 0.194 mmol), **n-7** (184 mg, 0.194 mmol) and PPh_3 (206 mg, 0.97 mmol) was dissolved in dry THF (40 mL) in a dry 100 mL Schlenk tube under nitrogen. After cooling the mixture to 0 ^{o}C, DIAD (196 mg, 0.97 mmol) was added dropwise to the mixture under nitrogen. The mixture was stirred at room temperature for 30 min. After removal of volatiles *in vacuo*, the solid residue was subjected to flash column chromatography (silica gel, DCM) to get the crude product. Recrystallization from DCM/EtOH afforded dyad **L_{lipo}** (171 mg, 52%) as a brown solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.33 (d, 2*H*, *J* = 7.6 Hz), 8.07 (d, 2*H*, *J* = 7.6 Hz), 7.98 (d, 2*H*, *J* = 8.0 Hz), 7.89 (d, 2*H*, *J* = 8.0 Hz), 7.84 (d, 2*H*, *J* = 3.6 Hz), 7.68 (d, 2*H*, *J* = 3.6 Hz), 7.49 (d, 2*H*, *J* = 7.6 Hz), 7.33 (d, 2*H*, *J* = 7.6 Hz), 7.12 (d, 2*H*, *J* = 3.6 Hz), 7.04 (d, 2*H*, *J* = 3.6 Hz), 6.60 (d, 2*H*, *J* = 2.0 Hz), 6.59 (t, 1*H*, *J* = 2.0 Hz), 6.47 (d, 2*H*, *J* = 2.0 Hz), 6.31 (t, 1*H*, *J* = 2.0 Hz),

5.32 (s, 2*H*), 3.94 (t, 4*H*, *J* = 6.4 Hz), 3.90 (t, 4*H*, *J* = 6.4 Hz), 3.84 (t, 2*H*, *J* = 7.2 Hz), 2.44 (t, 2*H*, *J* = 6.8 Hz), 1.83-1.56 (m, 14*H*), 1.52-1.20 (m, 72*H*), 0.88 (t, 6*H*, *J* = 6.4 Hz), 0.87 (t, 6*H*, *J* = 6.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 173.2, 162.8, 162.4, 160.9, 160.5, 152.1, 151.8, 145.4, 140.3, 139.9, 137.8, 136.6, 135.0, 133.8, 133.5, 130.8, 130.3, 129.0, 128.2, 127.1, 125.5, 125.3, 125.2, 124.4, 123.8, 123.0, 122.5, 107.2, 104.0, 102.4, 100.8, 68.3, 68.2, 60.4, 40.3, 34.3, 32.0, 29.8, 29.7, 29.6, 29.5, 29.4, 27.3, 26.5, 26.2, 24.7, 22.7, 14.1. MS (MALDI-TOF): Calcd. for $C_{105}H_{132}N_4O_{10}S_3$: 1705.91. Found: 1705.76 (m/z), 1728.74 (m/z + Na^+), 1744.72 (m/z + K^+). Elem. Anal.: Calcd. for $C_{105}H_{132}N_4O_{10}S_3$: C, 73.91; H, 7.80; N, 3.28. Found: C, 73.85; H, 7.86; N, 3.32.

When R=R$_2$:

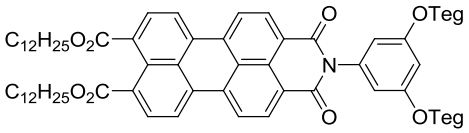


2-(3,5-Bis((2-(2-(2-methoxy)ethoxy)ethoxy)ethoxy)phenyl)isoindoline-1,3-dione (n-3). To a DMF solution (15 mL) of 2-(3,5-dihydroxyphenyl)isoindoline-1,3-dione (**n-1**) (361 mg, 1.42 mmol) was added anhydrous K_2CO_3 (781 mg, 5.66 mmol) and triethylene glycol monomethyl ether *p*-tosylate (TegOTs, 1.35 g, 4.24 mL). The mixture was stirred at 90 ^{o}C under nitrogen. After removal of DMF *in vacuo*, the residue was extracted by water. The aqueous phase was extracted repeatedly by diethyl ether. The organic extract was dried over anhydrous $MgSO_4$, filtered and concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, DCM/methanol, 70/1 v/v) to afford **n-3** (365 mg, 47%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.94 (m, 2*H*), 7.79 (m, 2*H*), 6.61 (d, 2*H*, *J* = 2.2 Hz), 6.55 (t, 1*H*, *J* = 2.2 Hz), 4.13 (t, 4*H*, *J* = 4.8 Hz), 3.95-3.79 (m, 4*H*), 3.78-3.59 (m, 12*H*), 3.59-3.46 (m, 4*H*), 3.37 (s, 6*H*). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 166.9, 159.9, 134.3, 132.9, 131.6, 123.6, 105.8, 101.6, 71.8, 70.7, 70.5, 70.4, 69.4, 67.6, 58.9. MS (EI): Calcd. for $C_{28}H_{37}NO_{10}$: 547. Found: 547 (m/z). Elem. Anal.: Calcd. for $C_{28}H_{37}NO_{10}$: C, 61.41; H, 6.81; N, 2.56. Found: C, 61.71; H, 6.79; N, 2.47.

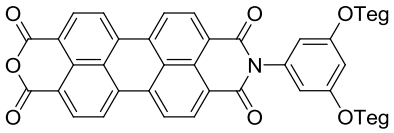


3,5-Bis((2-(2-(2-methoxy)ethoxy)ethoxy)ethoxy)aniline (n-4). Phthalimide **n-3** (456 mg, 0.83 mmol) was dissolved in absolute ethanol (20 mL) at 60 ^{o}C. Hydrazine monohydrate (85%, 0.25 mL, 4.2 mmol) was added to the mixture and the mixture was stirred at reflux for 2 h. After removal of insoluble solid by filtration, the solution was concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, DCM/methanol, 15/1 v/v) to afford **n-4** (317 mg, 91%) as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 5.90 (d, 2*H*, *J* = 2.1 Hz), 5.84 (t, 1*H*, *J* = 2.1 Hz), 4.02 (t, 4*H*, *J* = 4.8 Hz), 3.92-3.46 (m, 22*H*), 3.35 (s, 6*H*). ^{13}C NMR (100

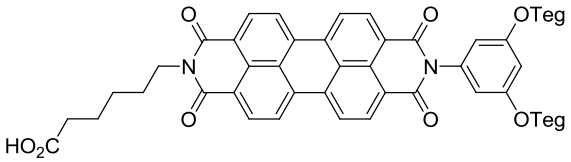
MHz, $CDCl_3$): δ (ppm) 160.8, 148.3, 94.8, 92.4, 72.0, 70.8, 70.6, 69.7, 67.3, 59.0. MS (ESI): Calcd. for $C_{20}H_{35}NO_8$: 417.24. Found: 418.24 (m/z+H^+).



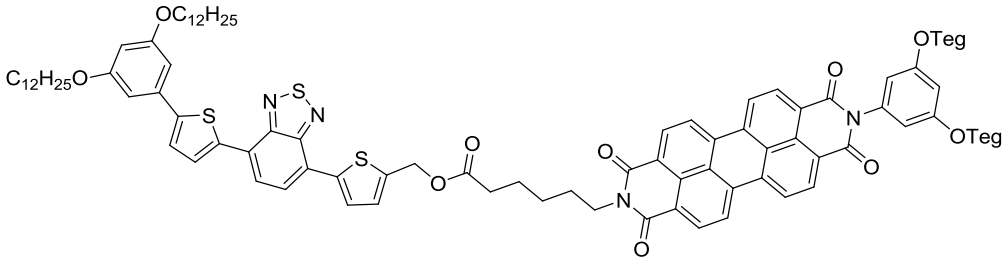
***N*-(3,5-Bis(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl)-9,10-bis(dodecyloxycarbonyl)-perylene-3,4-dicarboximide (n-5).** A dry 50 mL Schlenk tube was charged with anhydride **n-2** (50 mg, 0.067 mmol), **n-4** (34 mg, 0.080 mmol), DMAP (8 mg, 0.067 mmol) and imidazole (1 g). The reaction mixture was stirred at 130 ^oC under nitrogen for 12 h, quenched with 3M HCl. The aqueous layer was extracted by chloroform. The combined organic extract was washed with brine, dried over anhydrous Na_2SO_4, and filtered off from an insoluble fraction. After removal of solvents *in vacuo*, the residue was subjected to flash column chromatography (silica gel, DCM / MeOH, 100/1 v/v) to afford **n-5** (57 mg, 75%) as a red solid. ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.35 (m, 2*H*), 7.94 (m, 6*H*), 6.63 (t, 1*H*, *J* = 2.1 Hz), 6.55 (d, 2*H*, *J* = 2.1 Hz), 4.36 (t, 4*H*, *J* = 6.9 Hz), 4.15 (t, 4*H*, *J* = 4.8 Hz), 3.84 (t, 4*H*, *J* = 4.8 Hz), 3.74-3.64 (m, 12*H*), 3.55-3.53 (m, 4*H*), 3.37 (s, 6*H*), 1.83 (quint, 4*H*, *J* = 7.2 Hz), 1.52-1.14 (m, 36*H*), 0.87 (t, 6*H*, *J* = 6.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.1, 163.2, 160.4, 136.7, 135.3, 131.9, 131.7, 131.3, 130.3, 129.0, 128.7, 125.7, 122.5, 121.9, 121.7, 107.7, 102.5, 71.9, 70.8, 70.7, 70.6, 69.6, 67.7, 65.9, 59.0, 53.4, 31.9, 29.7, 29.6, 29.4, 28.6, 26.0, 22.7, 14.1. Elem. Anal.: Calcd. for $C_{68}H_{91}NO_{14}$: C, 71.24; H, 8.00; N, 1.22. Found: C, 71.20; H, 8.06; N, 1.18.



***N*-(3,5-Bis(2-(2-(2-methoxyethoxy)ethoxy)ethoxy)phenyl)-perylene-9,10-dicarboximide-3,4-dicarboxylic anhydride (n-6).** To a solution of **n-5** (279 mg, 0.243 mmol) in toluene (30 mL) was added 4-toluenesulfonic acid hydrate (300 mg, 1.5 mmol). The mixture was stirred at 95 ^oC under air for 16 h. After removal of volatiles *in vacuo*, the residue was subsequently washed with ethanol and PE. The red solid was dried at 80 ^oC to afford **n-6** (170 mg, 88%) as a red solid. ^1H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.72 (br, 8*H*), 6.65 (br, 1*H*), 6.51 (br, 2*H*), 4.15 (br, 4*H*), 3.86 (br, 4*H*), 3.80-3.46 (br, 16*H*), 3.37 (s, 6*H*). MS (ESI): Calcd. for $C_{44}H_{41}NO_{13}$: 791.26. Found: 792.27 (m/z+H^+), 814.25 (m/z+Na^+).

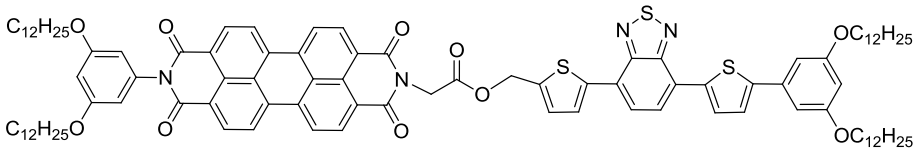


Carboxylic acid n-7. A mixture of **n-6** (416 mg, 0.50 mmol), 6-aminohexanoic acid (131 mg, 1 mmol), imidazole (700 mg) and DMF (10 mL) was stirred at 95 ^{o}C under nitrogen for 12 h. The product was precipitated by addition of ethanol/3M aqueous HCl (1/1, v/v, 40 mL), and then filtered. The solid residue was washed with ethanol and dried at 100 ^{o}C to afford **n-7** (419 mg, 89%) as a brown solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.45 (d, 2*H*, *J* = 8.1 Hz), 8.25 (d, 2*H*, *J* = 8.1 Hz), 8.11 (d, 2*H*, *J* = 8.1 Hz), 8.02 (d, 2*H*, *J* = 8.1 Hz), 6.68 (br, 3*H*), 4.18 (br, 4*H*), 4.12 (br, 2*H*), 4.04-3.49 (m, 20*H*), 3.38 (s, 6*H*), 2.37 (br, 2*H*), 1.75 (br, 4*H*), 1.51 (br, 2*H*). MS (ESI): Calcd. for $C_{50}H_{52}N_2O_{14}$: 904.34. Found: 905.35 (m/z+H^+).

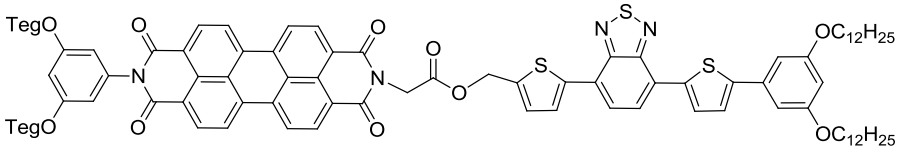


Dyad L_{amphi}. p-7 (160 mg, 0.206 mmol), **n-7** (186 mg, 0.206 mmol) was dissolved in dry DCM (50 mL) in a dry 100 mL Schlenk tube under nitrogen. To the mixture was added 2-chloro-1-methylpyridinium iodide (263 mg, 1.03 mmol) and Et_3N (0.3 mL). The mixture was stirred under nitrogen at room temperature for 24 h. After removal of volatiles *in vacuo*, the solid residue was subjected to flash column chromatography (silica gel, DCM/THF, 5/1 v/v) to afford pure **dyad L_{amphi}** (116 mg, 34%) as a brown solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.51 (d, 2*H*, *J* = 8.1 Hz), 8.43-8.22 (m, 6*H*), 7.93 (d, 2*H*, *J* = 3.6 Hz), 7.82 (d, 2*H*, *J* = 3.6 Hz), 7.65 (d, 2*H*, *J* = 7.6 Hz), 7.50 (d, 2*H*, *J* = 7.6 Hz), 7.17 (d, 2*H*, *J* = 3.6 Hz), 7.13 (d, 2*H*, *J* = 3.6 Hz), 6.66 (t, 1*H*, *J* = 2.1 Hz), 6.59 (d, 2*H*, *J* = 2.1 Hz), 6.53 (d, 2*H*, *J* = 2.1 Hz), 6.34 (t, 1*H*, *J* = 2.1 Hz), 5.35 (s, 2*H*), 4.15 (t, 4*H*, *J* = 4.2 Hz), 3.99-3.80 (m, 10*H*), 3.79-3.48 (m, 16*H*), 3.37 (s, 6*H*), 2.43 (t, 4*H*, *J* = 6.9 Hz), 1.88-1.15 (m, 46*H*), 0.88 (t, 6*H*, *J* = 6.3 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 173.2, 162.8, 162.5, 160.5, 160.4, 152.1, 151.9, 145.4, 140.3, 139.8, 137.9, 136.7, 135.0, 134.0, 133.6, 130.9, 130.4, 129.1, 128.4, 128.2, 127.1, 125.6, 125.4, 125.3, 124.5, 123.9, 123.0, 122.6, 122.5, 107.7, 104.0, 102.7, 100.7, 72.0, 70.8, 70.7, 70.6, 69.7, 68.2, 67.7, 60.4, 59.0, 40.3, 34.3, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 27.3, 26.4, 26.1, 24.6, 22.7, 14.1. MS (MALDI-TOF): Calcd. for $C_{95}H_{112}N_4O_{16}S_3$: 1661.73. Found: 1661.73 (m/z), 1684.74 (m/z + Na^+), 1700.72 (m/z + K^+).

c. Synthesis of dyads S_{lipo} and S_{amphi}

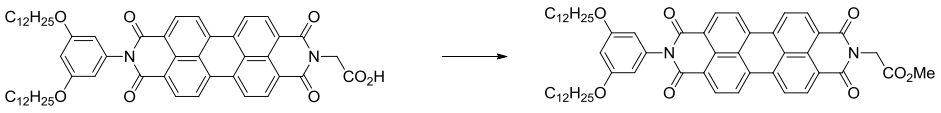


Dyad S_{lipo}. A mixture of **n-6** (520 mg, 0.62 mmol), glycine (93.4 mg, 1.24 mmol), imidazole (847 mg, 12.4 mmol) and DMF (10 mL) was stirred at 95 ^{o}C under nitrogen for 17 h. The product was precipitated by addition of 3M aqueous HCl (1/1, v/v, 100 mL), and then filtered. The solid residue was washed by water and dried at 110 ^{o}C to afford **n-8** (513 mg, 92%) as a dark red solid. The solid (450 mg, 0.50 mmol) was suspended in 50 mL DCM, oxalyl chloride (85 μL) was added into the suspension and then one drop of DMF was added. The suspension was stirred for 2 h at room temperature. Then DCM was removed *in vacuo*, the residue was redissolved in 20 mL DCM, a mixture of 4-nitrophenol (210 mg, 1.51mmol), 10 mL DCM and 0.5 mL Et_3N was added dropwise at 0 ^{o}C. After that, the solution was stirred overnight at room temperature. The reaction mixture was successively washed with aqueous $NaHCO_3$, water, and brine, then dried with sodium sulfate. After removal of solvent, the solid residue was subjected to flash column chromatography (silica gel, DCM/Methanol, 100/1 v/v) to afford pure active ester **n-9** (466 mg, 91%) as a dark red solid. The active ester **n-9** (120 mg, 0.13 mmol) and the alcohol **p-7** (110 mg, 0.14 mmol), DMAP (97 mg, 0.79 mmol) was dissolved in dry DMF, stirred at room temperature for 72 h. The reaction mixture was poured into water, extracted with DCM, washed with aqueous ammonium chloride, brine, dried with sodium sulfate. After removal of DCM, the solid residue was purified by column chromatography (silica gel, DCM) to afford pure dyad **S_{lipo}** (103 mg, 53%). ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.46 (d, 2*H*, *J* = 7.6 Hz), 8.26 (d, 2*H J*= 8.0 Hz), 8.14 (d, 2*H*, *J* = 8.0 Hz), 8.05 (d, 2*H*, *J* = 8.0 Hz), 7.75 (d, 1*H*, *J* = 3.2 Hz), 7.70 (d, 1*H*, *J* = 3.6 Hz), 7.40 (d, 1*H*, *J* = 7.6 Hz), 7.32 (d, 1*H*, *J* = 7.6 Hz), 7.14 (m, 2*H*), 6.64 (d, 2*H*, J = 1.6 Hz), 6.62 (t, 1*H*, *J* = 2.0 Hz), 6.58 (d, 2*H*, *J* = 1.6 Hz), 6.37 (t, 1*H*, *J* = 2.0 Hz), 5.44 (s, 2*H*), 5.00 (s, 2*H*), 3.95 (m, 8*H*), 1.78 (m, 8*H*), 1.45 (m, 8*H*), 1.31 (m, 64*H*), 0.88 (m, 12*H*). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.1, 163.0, 162.4, 160.9, 160.6, 151.8, 151.7, 145.7, 140.7, 138.3, 137.8, 136.6, 135.3, 134.0, 133.8, 131.1, 129.7, 128.9, 128.5, 128.3, 127.1, 125.6, 125.4, 125.1, 124.4, 124.0, 123.1, 122.9, 122.6, 122.1, 107.2, 104.3, 102.3, 100.9, 68.3, 68.2, 61.6, 41.5, 31.9, 29.7, 29.5, 29.4, 26.1, 22.7, 14.1. MS (MALDI-TOF): Calcd. for $C_{101}H_{124}N_4O_{10}S_3$: 1649.85. Found: 1649.69 (m/z). Elem. Anal.: Calcd. for $C_{101}H_{124}N_4O_{10}S_3$: C, 73.51; H, 7.57; N, 3.39. Found: C, 73.59; H, 7.63; N, 3.37.

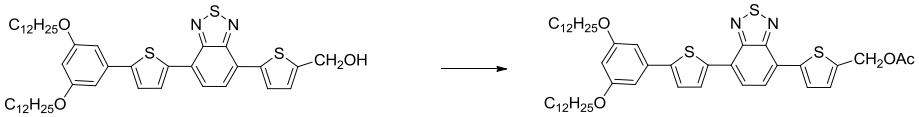


Dyad S_{amphi}. A mixture of **n-6** (290 mg, 0.37 mmol), glycine (55 mg, 0.73 mmol), imidazole (499 mg, 7.3 mmol) and DMF (7 mL) was stirred at 95 ^{o}C under nitrogen for 17 h. The product was precipitated by addition of 3M aqueous HCl (1/1, v/v, 200 mL), and centrifuged. The solid residue was dried at 100 ^{o}C to afford **n-8** (193 mg, 62%) as a dark red solid. The solid **n-8** (144 mg, 0.17 mmol) was suspended in 50 mL DCM, oxalyl chloride (21 μL) was added into the suspension and then one drop of DMF was added. The suspension was stirred for 2 h at room temperature. Then DCM was removed *in vacuo*, the residue was redissolved in 20 mL DCM, a mixture of 4-nitrophenol (47 mg, 0.34 mmol), 10 mL DCM and 0.5 mL Et_3N was added dropwise at 0 ^{o}C. After dropwise, the solution was stirred overnight at room temperature. The reaction mixture was successively washed with aqueous $NaHCO_3$, water, and brine, then dried with sodium sulfate. After removal of solvent, the solid residue was subjected to flash column chromatography (silica gel, DCM/Methanol, 40/1 v/v) to afford pure active ester **n-9** (36 mg, 47%) as a dark red solid. The active ester **n-9** (36 mg, 0.037 mmol) and the alcohol **p-7** (28.8 mg, 0.037 mmol), DMAP (12.7 mg, 0.1 mmol) was dissolved in dry DMF, stirred at room temperature for 72 h. The reaction mixture was poured into water, extracted with DCM, washed with aqueous ammonium chloride, brine, and dried with sodium sulfate. After removal of DCM, the solid residue was purified by column chromatography (silica gel, DCM/methanol, 60/1 v/v) to afford pure dyad $\mathbf{S_{amphi}}$ (41.2 mg, 69%). ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 8.65 (d, 2*H*, *J* = 8.1 Hz), 8.59 (d, 2*H* *J*= 7.8 Hz), 8.5 (d, 2*H*, *J* = 8.1 Hz), 8.46 (d, 2*H*, *J* = 8.4 Hz), 7.99 (d, 1*H*, *J* = 4.2 Hz), 7.94 (d, 1*H*, *J* = 3.6 Hz), 7.74 (d, 1*H*, *J* = 7.5 Hz), 7.71 (d, 1*H*, *J* = 7.5 Hz), 7.33 (d, 1*H*, *J* = 3.6 Hz), 7.21 (d, 1*H*, J = 3.6 Hz), 6.78 (d, 2*H*, *J* = 2.1 Hz), 6.65 (t, 1*H*, *J* = 2.1 Hz), 6.55 (d, 2*H*, *J* = 2.1 Hz), 6.43 (t, 1*H*, 2.1 Hz), 5.47 (s, 2*H*), 5.04 (s, 2*H*), 4.14 (t, 4*H*, *J* = 4.2 Hz), 4.00 (t, 4*H*, *J* = 6.3 Hz), 3.85 (t, 4*H*, *J* = 4.2 Hz), 3.73 (m, 4*H*), 3.67 (m, 8*H*), 3.55 (m, 4*H*), 3.37 (s, 6*H*), 1.81 (t, 4*H*, *J* = 6.6 Hz), 1.88-1.15 (m, 36*H*), 0.88 (t, 6*H*, *J* = 6.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.0, 162.9, 162.4, 160.6, 160.5, 151.8, 145.7, 140.7, 138.3, 137.9, 136.6, 135.3, 134.0, 133.8, 131.0, 129.7, 128.9, 128.5, 128.3, 127.0, 125.5, 125.4, 125.2, 124.7, 124.4, 124.0, 123.1, 122.9, 122.7, 122.1, 107.7, 104.3, 102.7, 100.9, 72.0, 70.8, 70.7, 70.6, 69.6, 68.2, 67.8, 59.0, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 26.1, 22.7, 14.1. MS (MALDI-TOF): Calcd. for $C_{91}H_{104}N_4O_{16}S_3$: 1605.7. Found: 1605.48 (m/z), 1627.48 (m/z + Na^+), 1644.45 (m/z + K^+). Elem. Anal.: Calcd. for $C_{101}H_{124}N_4O_{10}S_3$: C, 68.06; H, 6.53; N, 3.49. Found: C, 67.74; H, 6.86; N, 3.22.

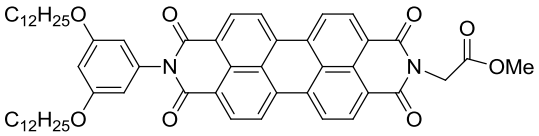
d. Synthesis of reference compounds



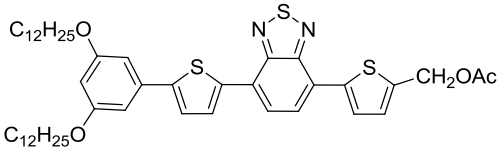
PDI



T2BTZ



Compound PDI. To a solution of **n-8** (80 mg, 0.09 mmol) in DCM (20 mL) was added DCC (37 mg, 0.18 mmol), DMAP (5.5 mg, 0.045 mmol) and methanol (14 mg, 0.44 mmol). The mixture was stirred at room temperature under air for 19 h. After removal of volatiles *in vacuo*, the residue was subjected to flash column chromatography (silica gel, DCM) to afford **PDI** (43 mg, 53%) as a red solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.45 (d, 2*H*, *J*= 7.8 Hz), 8.12 (d, 2*H*, *J*= 8.1 Hz), 8.02 (d, 2*H*, *J*= 8.1 Hz), 7.86 (d, 2*H*, *J*= 8.4 Hz), 6.62 (d, 2*H*, *J*= 2.1 Hz), 6.56 (t, 1*H*, *J*= 1.8 Hz), 4.92 (s, 2*H*), 3.90 (t, 4*H*, *J*= 6.0 Hz), 3.85 (s, 3*H*), 1.71 (m, 4*H*), 1.25 (m, 36*H*), 0.86 (m, 6*H*). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 168.5, 162.9, 162.4, 160.9, 136.5, 134.0, 133.8, 131.2, 131.0, 129.0, 128.6, 125.6, 125.5, 123.3, 123.0, 122.7, 122.3, 107.0, 102.4, 68.3, 52.6, 41.3, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 26.1, 22.7, 14.1. MS (ESI): Calcd. for $C_{57}H_{66}N_2O_8$: 906.48. Found: 907.37 (m/z + H+). Elem. Anal.: Calcd. for $C_{57}H_{66}N_2O_8$: C, 75.47; H, 7.33; N, 3.09. Found: C, 75.48; H, 7.49; N, 2.83.



Compound T2BTZ. p-11 (80 mg, 0.10 mmol) and DMAP (4 mg, 0.03 mmol) was dissolved in dry DCM (8 mL) in a dry 50 mL Schlenk tube under nitrogen. To the mixture was added Et_3N (44 μL, 0.318 mmol) and acetic anhydride (20 μL, 0.212 mmol). The mixture was stirred at room temperature for 4 h and then quenched with water. After removal of aqueous layer, the organic extract was dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The residue was subjected to flash column chromatography (silica gel, PE/DCM, 1/1 v/v) to afford **T2BTZ** (83 mg, 98%) as an orange solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 8.05 (d, 1*H*, *J* = 3.6 Hz), 7.94 (d, 1*H*, *J* = 3.6 Hz), 7.79 (br, 2*H*), 7.35 (d, 1*H*, *J* = 3.6 Hz), 7.15 (d, 1*H*, *J* = 3.6 Hz), 6.81 (d, 2*H*, *J* =

2.0 Hz), 6.42 (t, 1*H*, *J* = 2.0 Hz), 5.30 (s, 2*H*), 3.99 (t, 4*H*, *J* = 6.4 Hz), 2.12 (s, 3*H*), 1.80 (quint, 4*H*, *J* = 6.4 Hz), 1.53-1.16 (m, 36*H*), 0.88 (t, 6*H*, *J* = 6.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 170.7, 160.7, 152.6, 152.5, 145.9, 140.7, 139.2, 138.4, 135.7, 129.0, 128.6, 127.1, 126.2, 125.7, 125.5, 125.1, 124.3, 104.7, 101.0, 68.3, 60.7, 32.0, 29.7, 29.5, 29.4, 26.1, 22.7, 21.0, 14.2. MS (ESI): Calcd. for $C_{47}H_{64}N_2O_4S_3$: 816.4. Found: 817.4 (m/z + H+). Elem. Anal.: Calcd. for $C_{47}H_{64}N_2O_4S_3$: C, 69.08; H, 7.89; N, 3.43. Found: C, 69.11; H, 7.99; N, 3.40.

V. Concentration-dependent ^{1}H NMR Spectra

Concentration-dependent ^{1}H NMR spectra were recorded on Mercury plus 300 (300 MHz), using $CDCl_3$ as the solvent. The spectra were recorded at the concentration of 0.03 M (1), 0.02 M (2), 0.013 M (3), and 0.009 M (4), respectively. Δδ is the chemical shift changes of the most downfield proton attached on a) **PDI** and b) **T2BTZ**.

Table S1. Concentration-dependent ^{1}H NMR experiment of four dyads, reference compounds and corresponding mixtures. Δδ is the chemical shift changes of the proton attached on a) **PDI** and b) **T2BTZ**. ($CDCl_3$, 0.03 M to 0.009 M, 25 ℃)

	$\mathbf{S_{lipo}}$	$\mathbf{S_{amphi}}$	$\mathbf{L_{lipo}}$	$\mathbf{L_{amphi}}$	**PDI**	**T2BTZ**	**PDI&T2BTZ**
Δδ of **PDI**-H[a]	0.127	0.143	0.13	0.15	0.093	---	0.12
Δδ of **T2BTZ**-H[b]	0.138	0.17	0.068	0.07	---	0.013	0.087

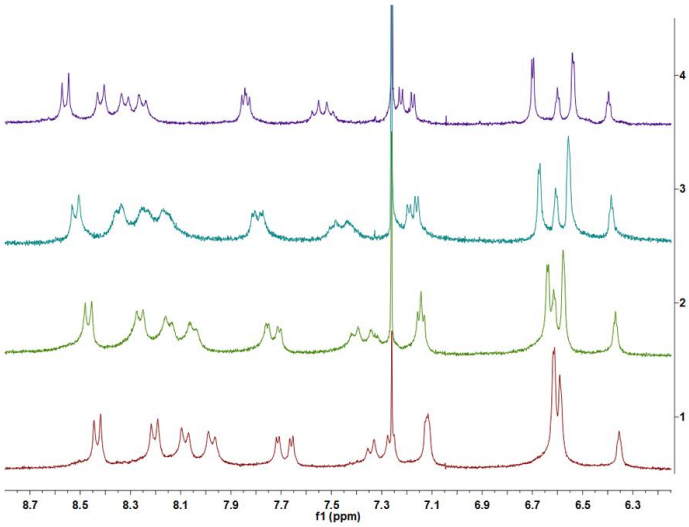


Fig. S7 Concentration-dependent ^{1}H NMR spectra of $\mathbf{S_{lipo}}$

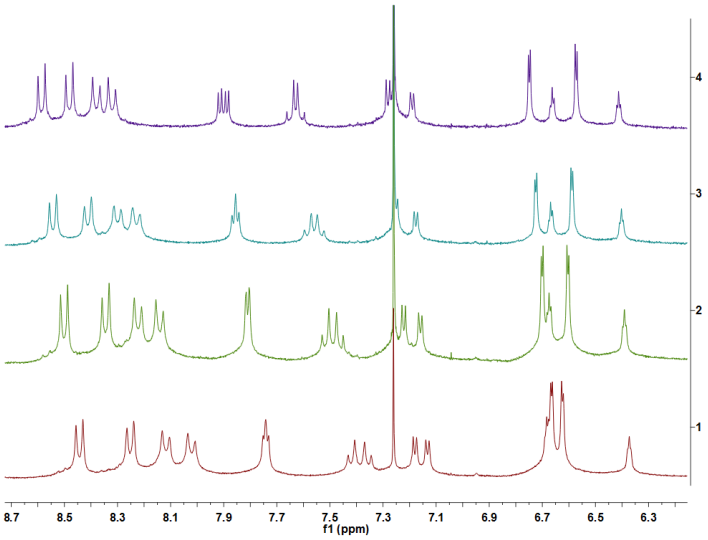


Fig. S8 Concentration-dependent ^{1}H NMR spectra of $\mathbf{S_{amphi}}$

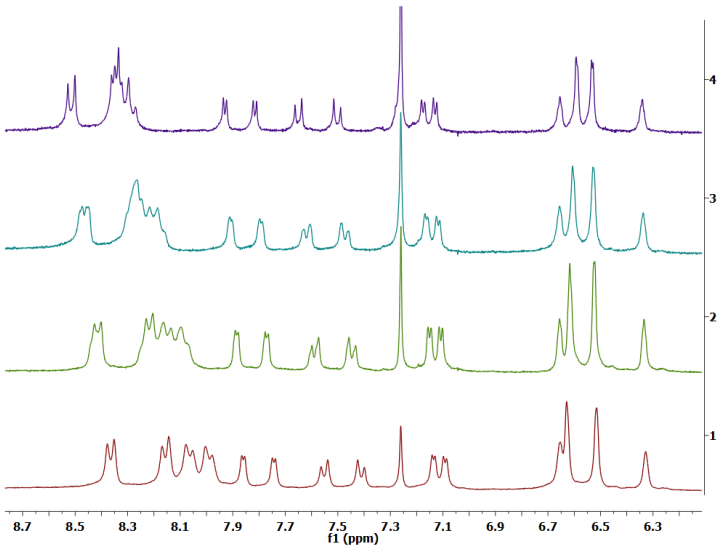


Fig. S9 Concentration-dependent ^{1}H NMR spectra of $\mathbf{L_{amphi}}$

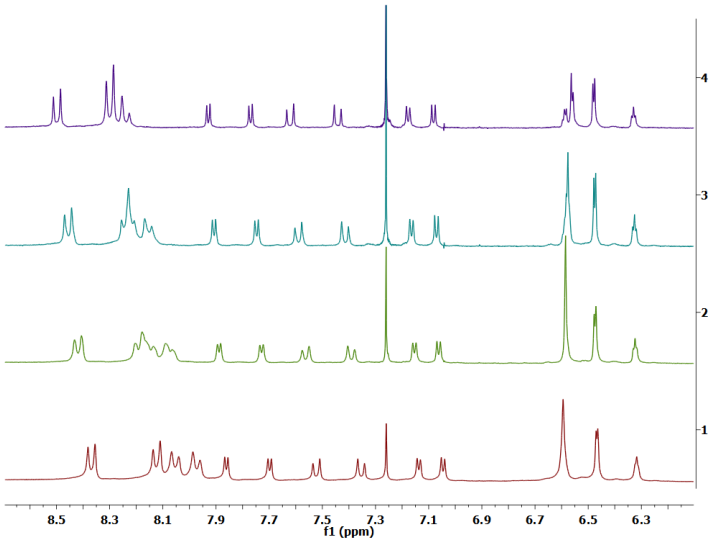


Fig. S10 Concentration-dependent ^{1}H NMR spectra of $\mathbf{L_{lipo}}$

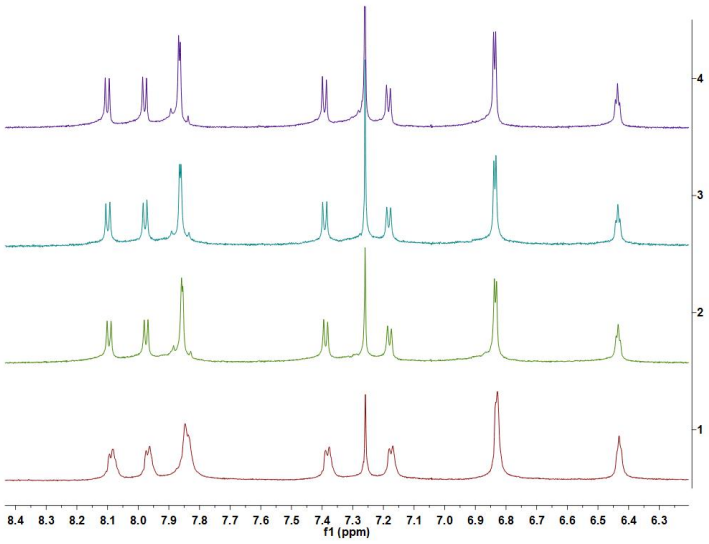


Fig. S11 Concentration-dependent ^{1}H NMR spectra of **T2BTZ**

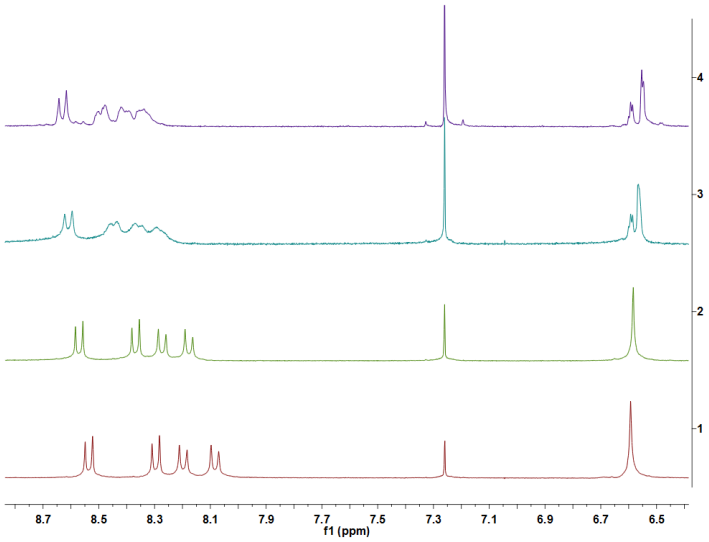


Fig. S12 Concentration-dependent ^{1}H NMR spectra of **PDI**

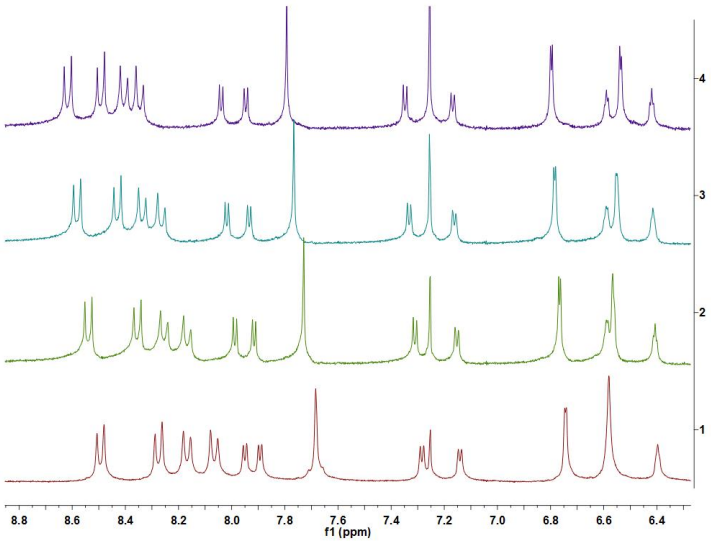


Fig. S13 Concentration-dependent ^{1}H NMR spectra of Mixture of **PDI** and **T2BTZ**

VI. Scanning electron microscopy

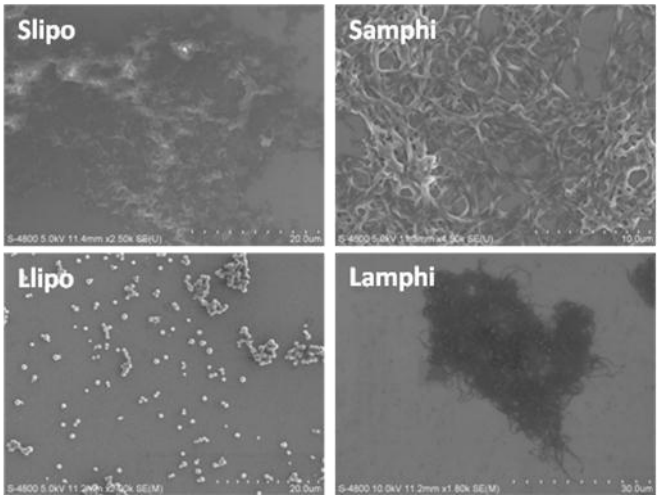
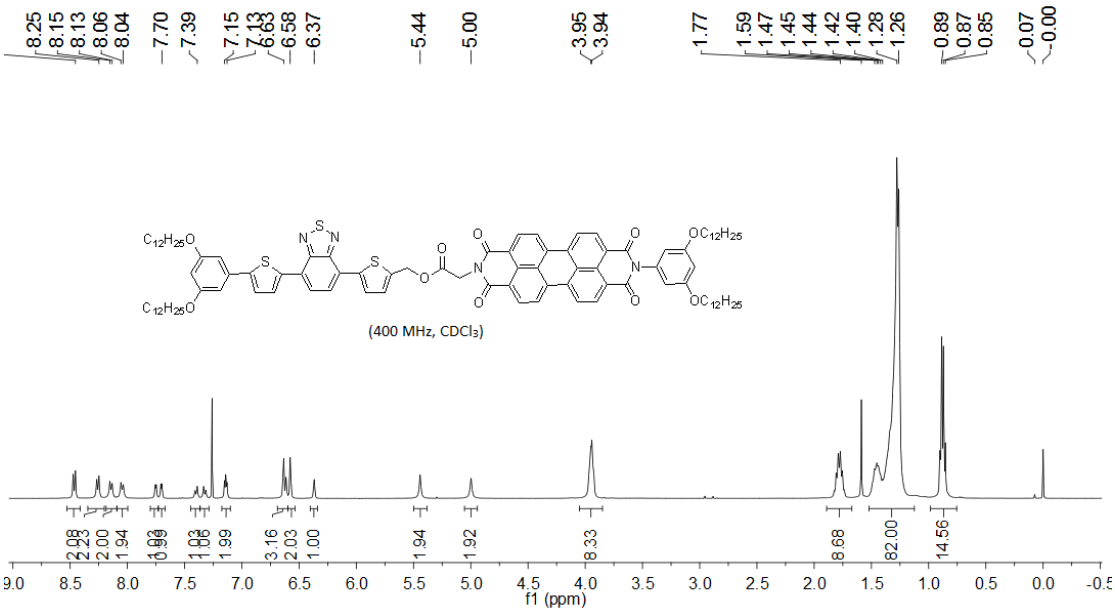
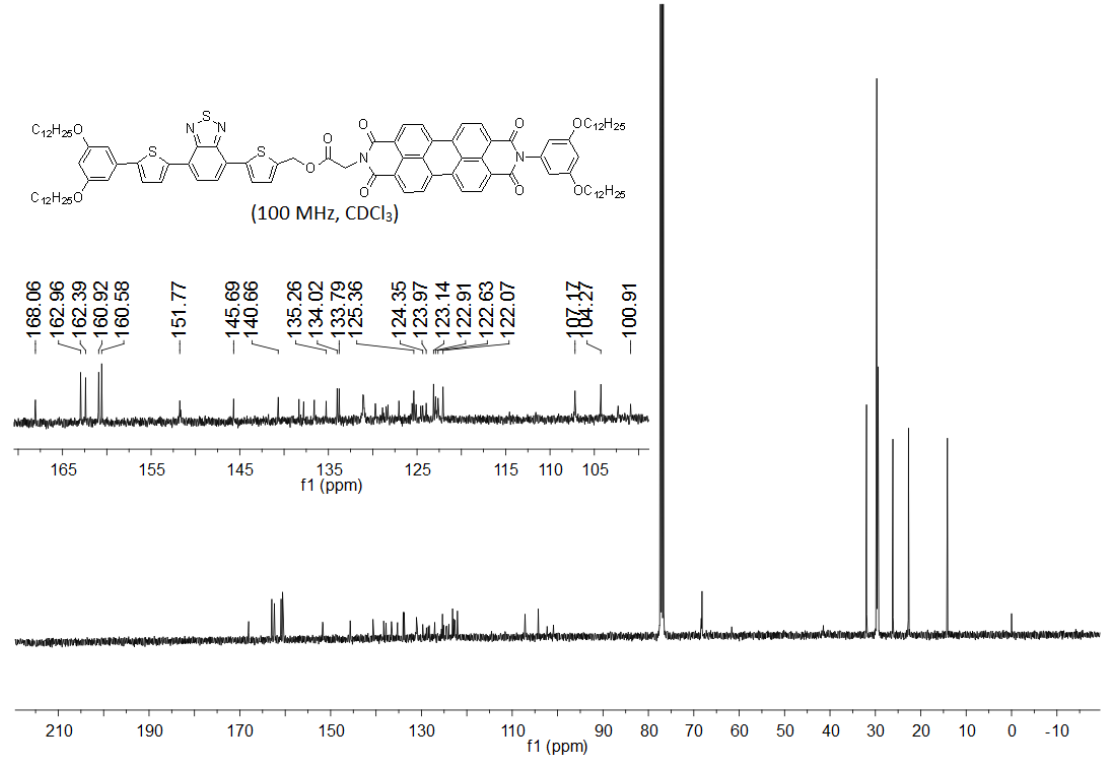
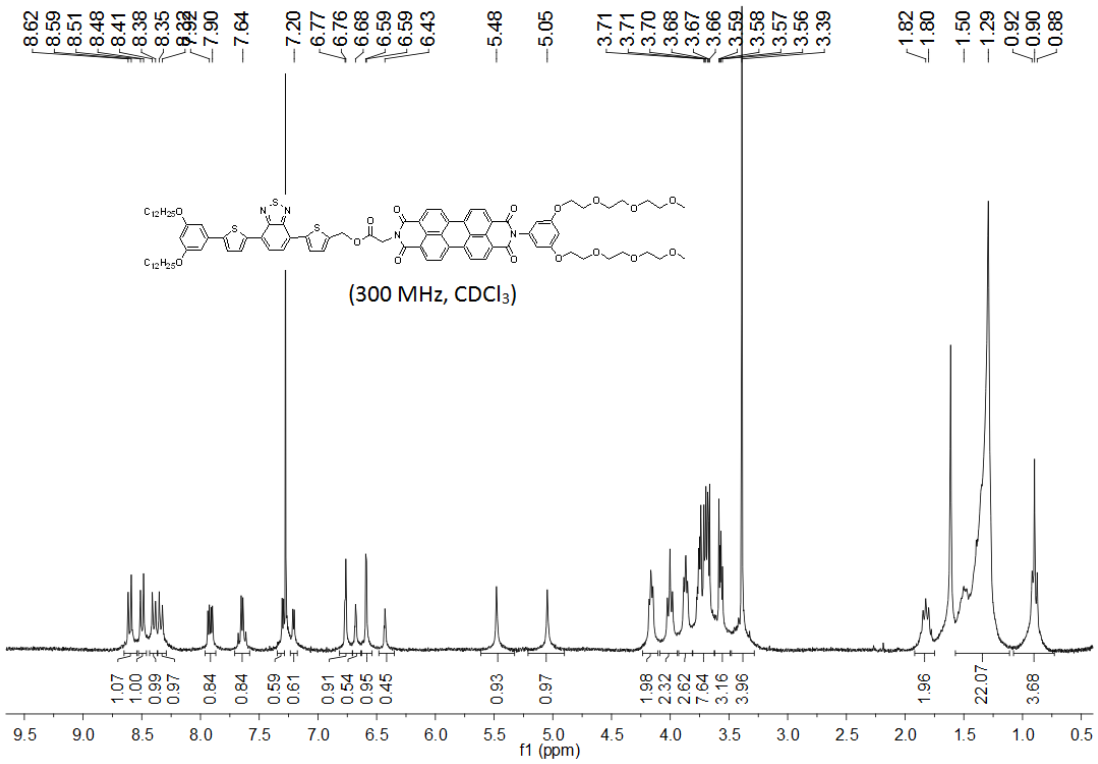


Fig. S14 Scanning electron microscopy images of four dyads.

VII. ^{1}H and ^{13}C NMR Spectra of the final compounds



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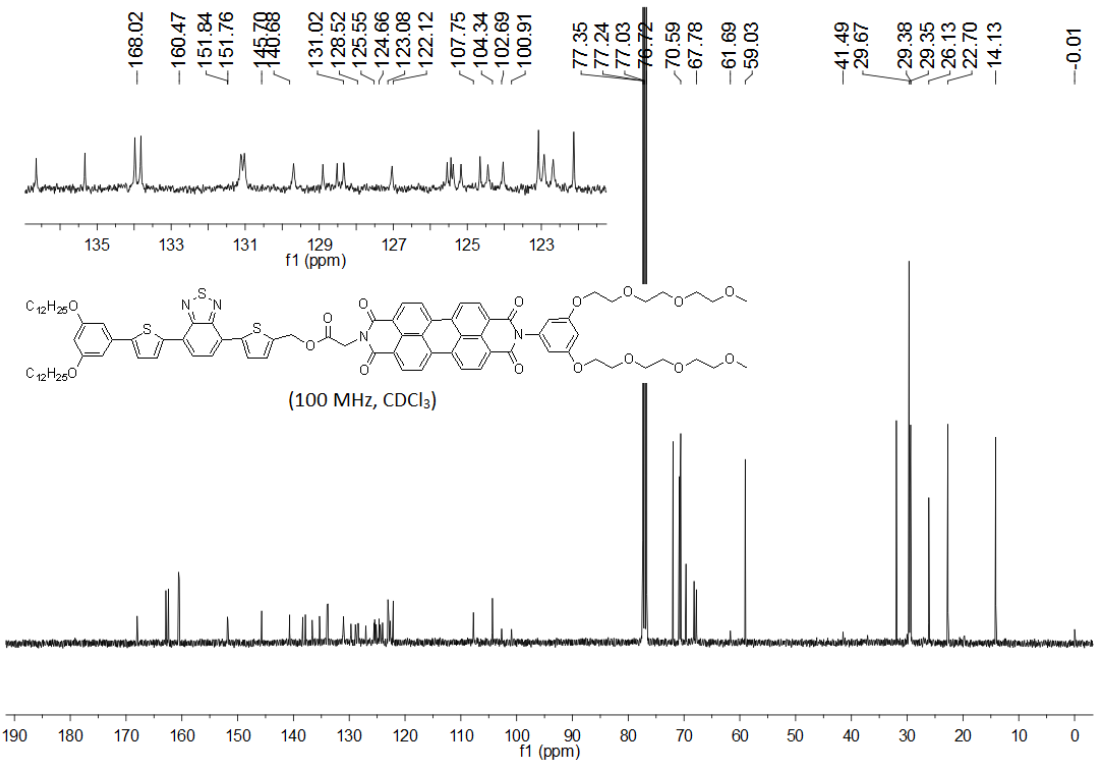
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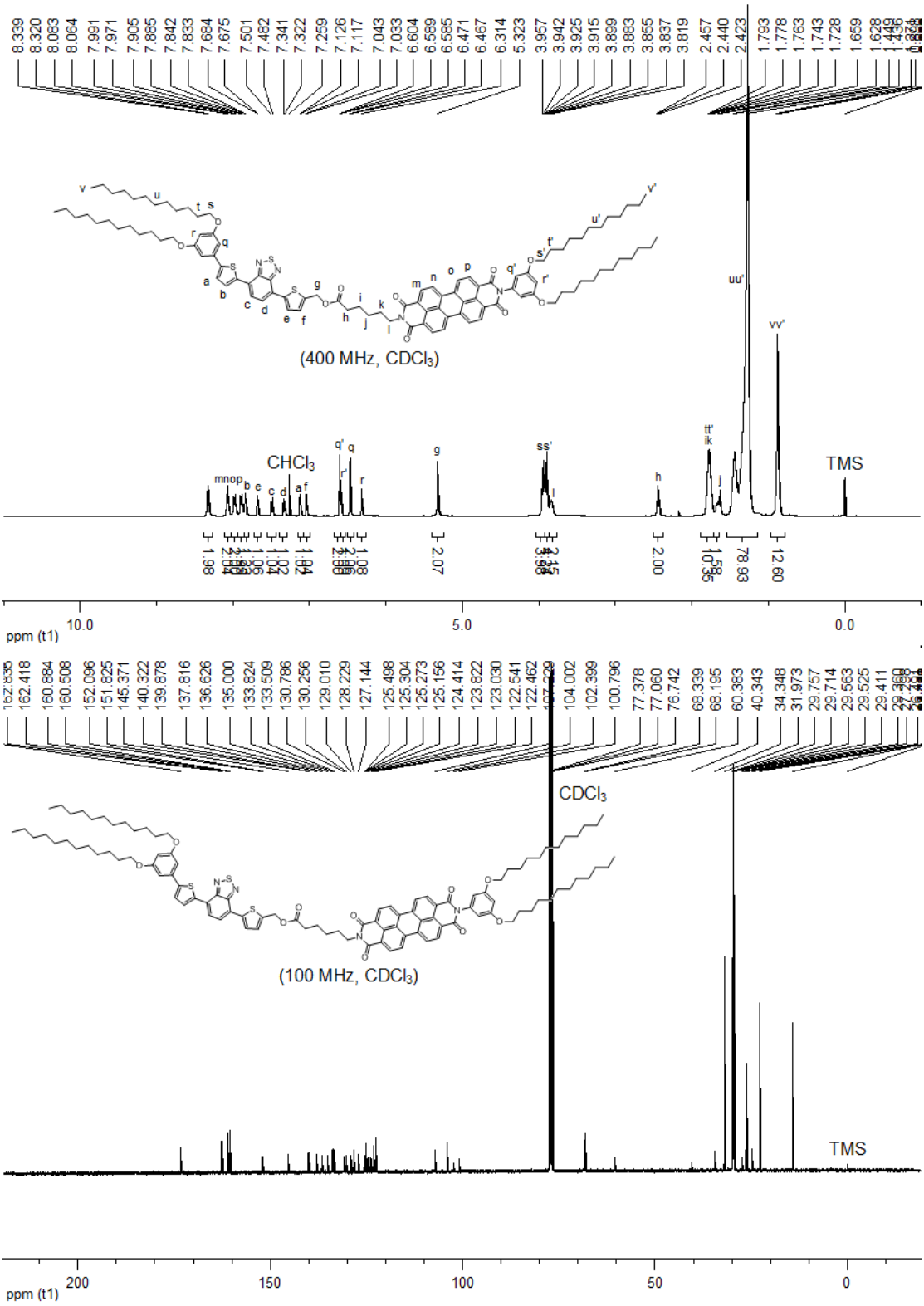
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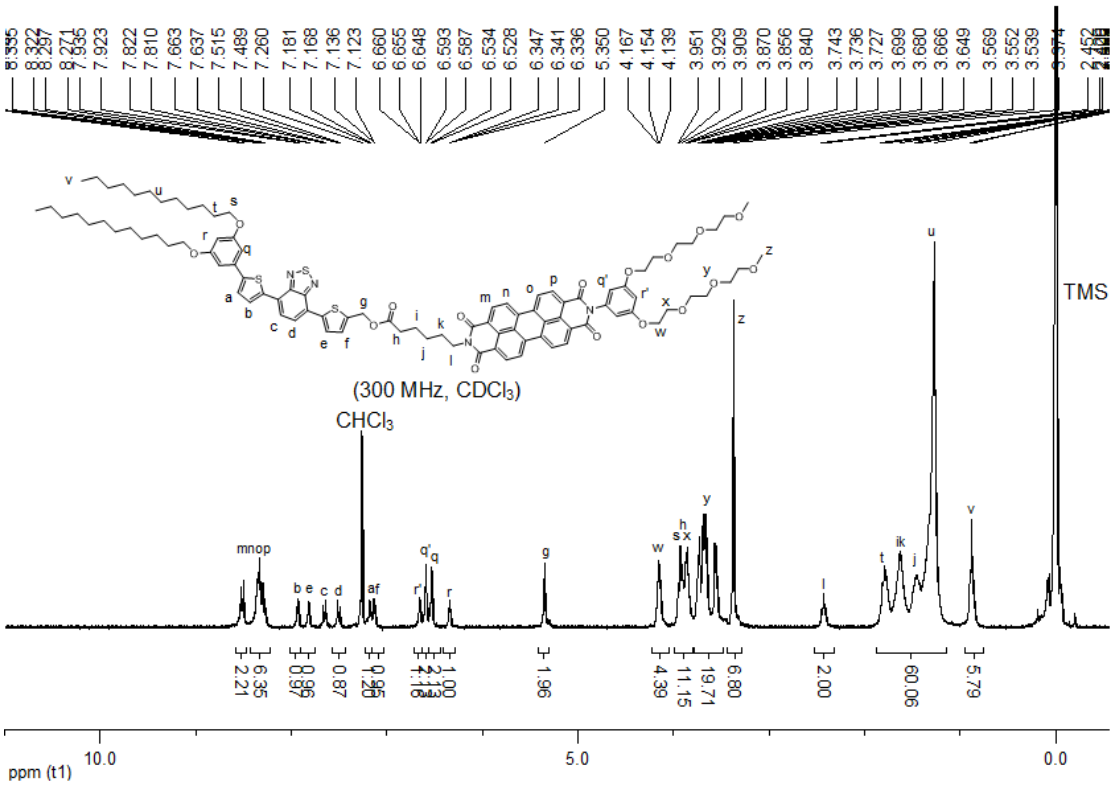
ppm (t1)
(100 MHz, CDCl3)
CDCl3
TMS
ppm (t1)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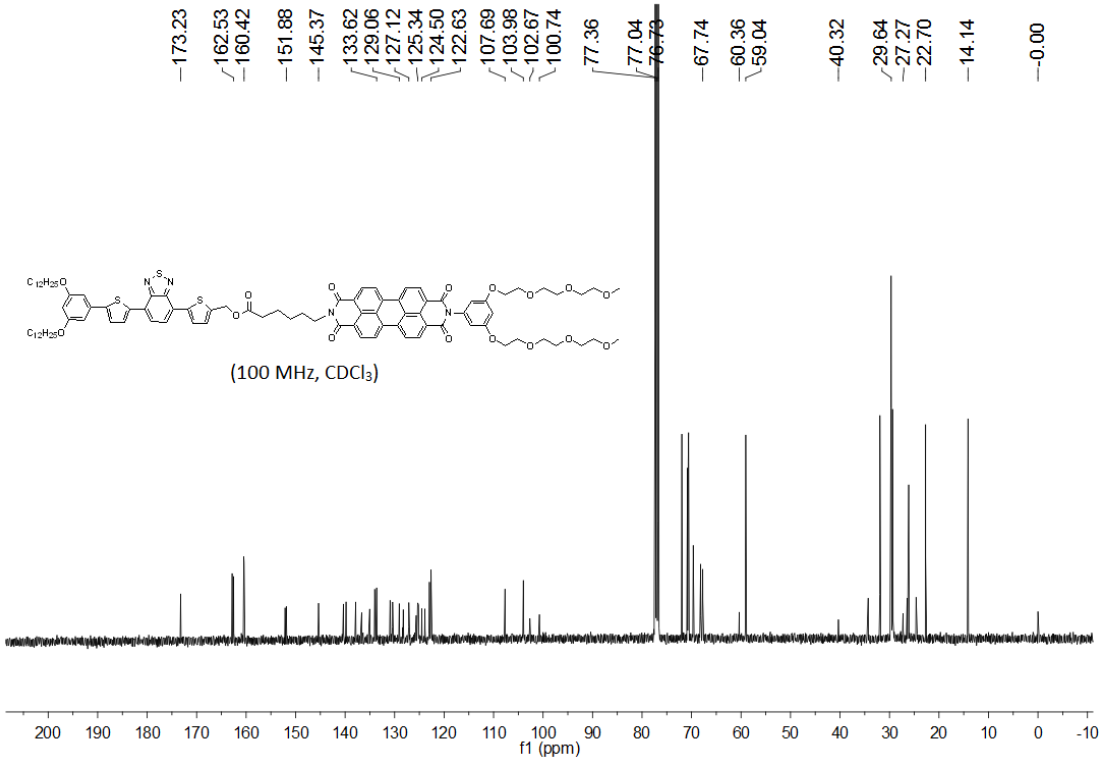
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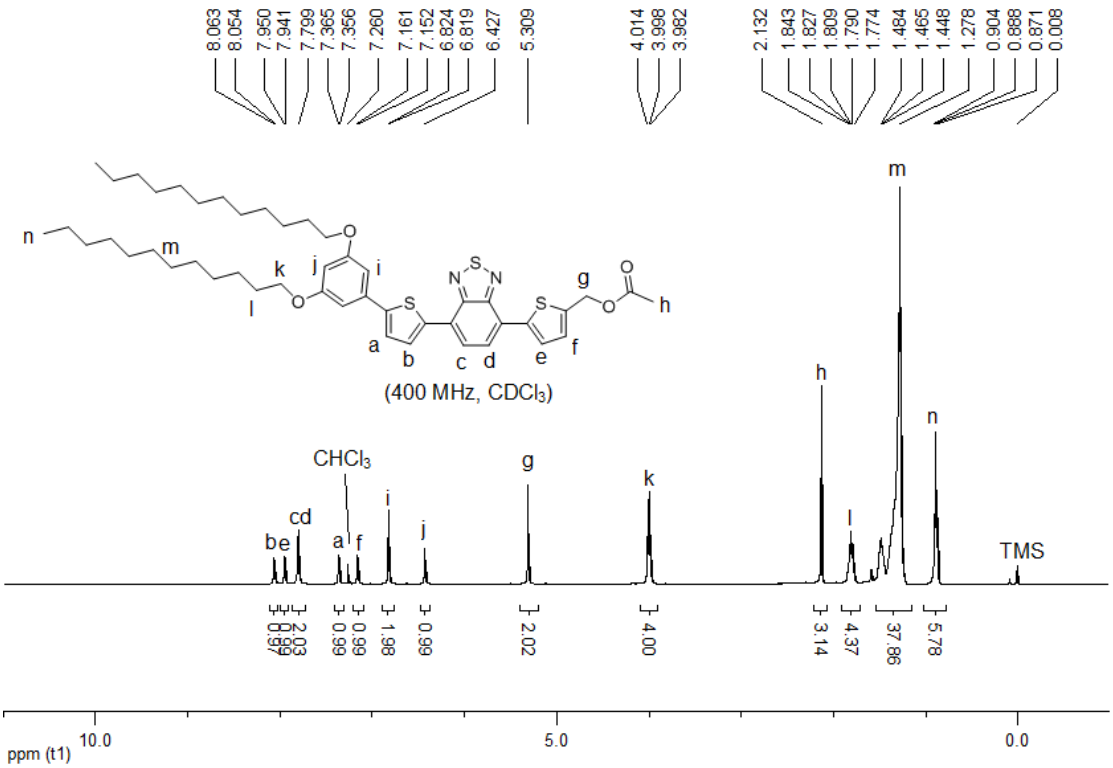
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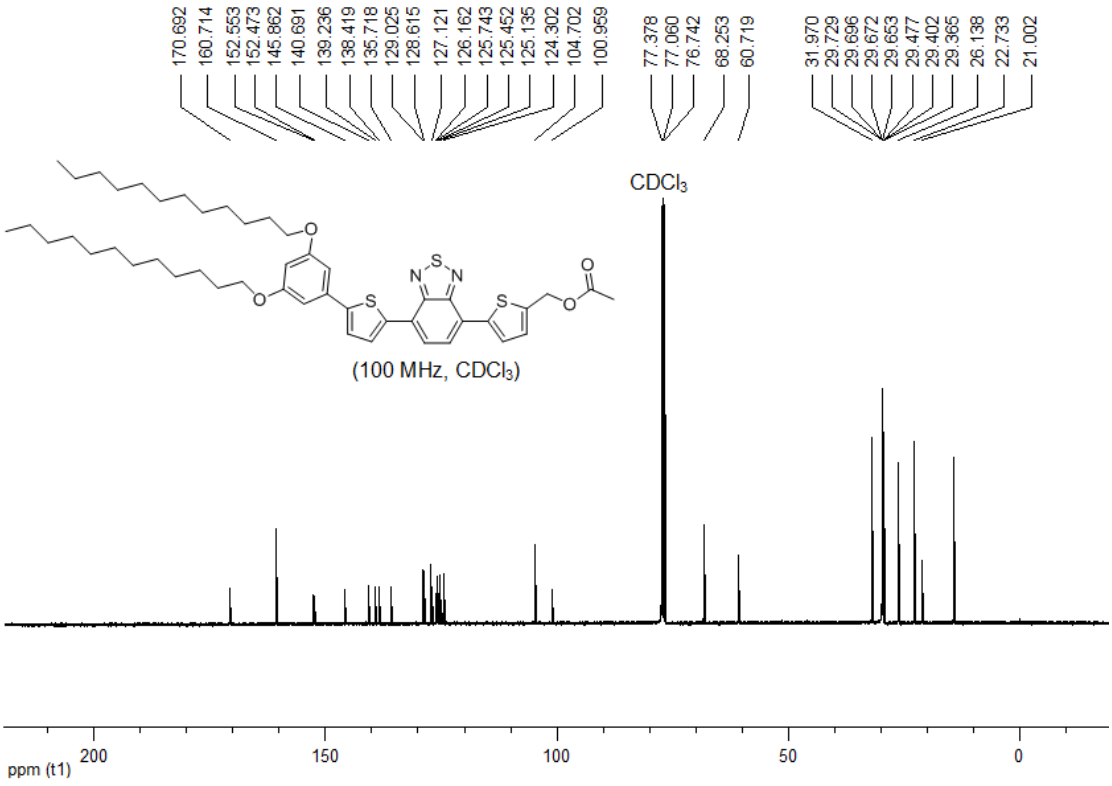
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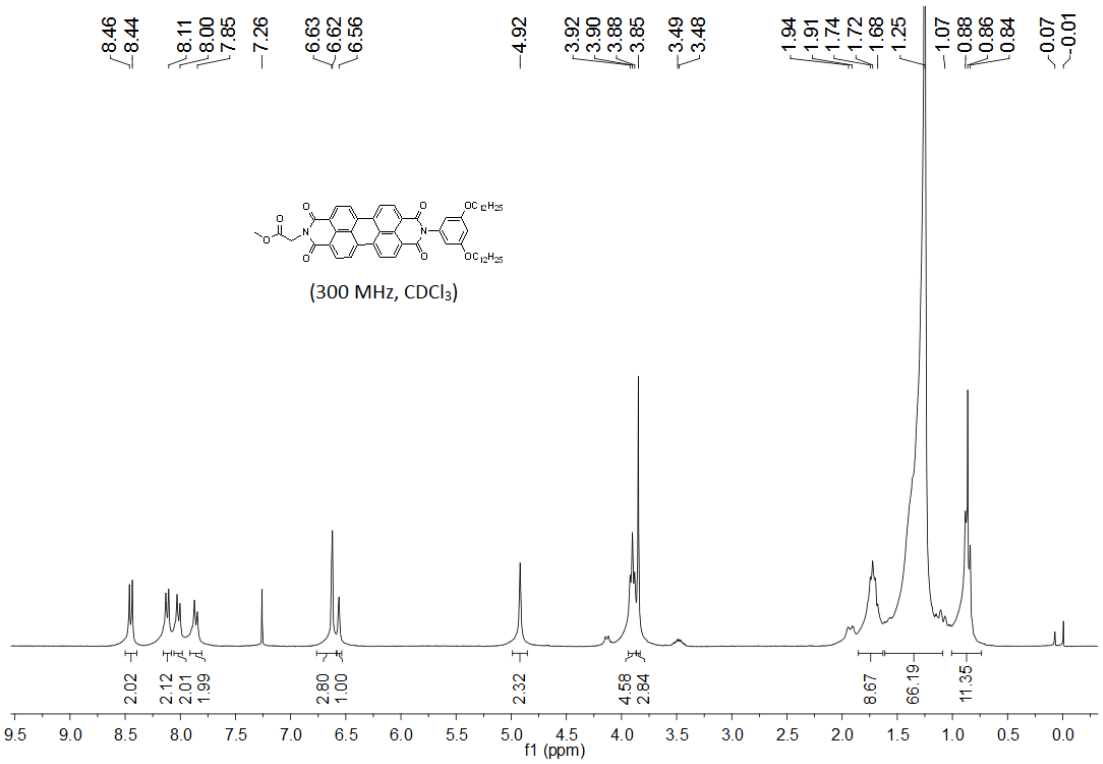
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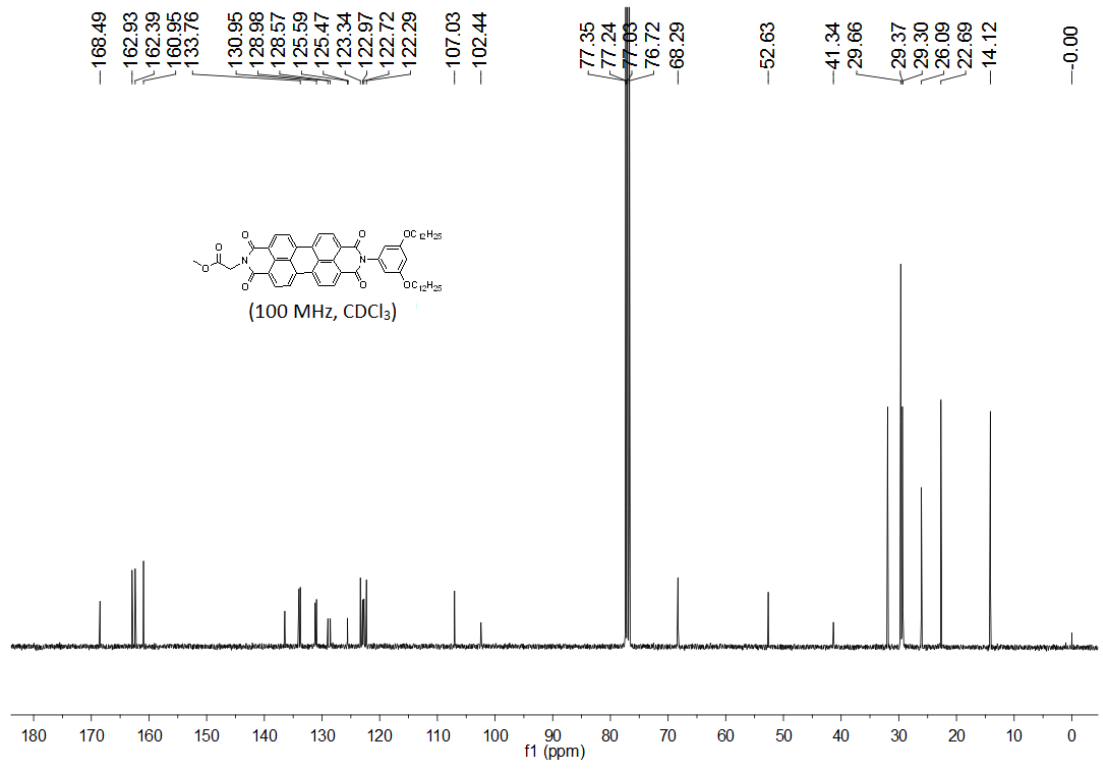
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76.72
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f1 (ppm)