Supporting Information

for

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

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I. Optical Spectra of the Dyads

Fig. S1 Concentration-dependent UV-vis absorption spectra of $L_{\text{ lipo}}$ (a) and $L_{\text{ amph}}$ (b) and comparison of normalized absorption spectra of $L_{\text{ lipo}}$ and $L_{\text{ amph}}$ in selected concentrations of $1 \times 10^{-6}$ M (c), $2 \times 10^{-6}$ M (d), $5 \times 10^{-6}$ M (e), $1 \times 10^{-5}$ 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 $1 \times 10^{-6}$ M (blue line, solvent: chloroform, 25 °C).

Fig. S2 Fluorescence spectra for dyads and reference compounds in chloroform at $2.5 \times 10^{-6}$ M excited at 331 nm. The red line represents the mixture of PDI and T2BTZ both at $2.5 \times 10^{-6}$ 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. The half-wave potential of oxidation peak of Fc was measured to be 0.427 V against Ag/AgCl. The HOMO levels of Slipo, Samphi, T2BTZ were estimated from the half-wave potentials of the oxidation peaks. The LUMO levels of Slipo, Samphi, PDI were estimated from the half-wave potentials of the oxidation peaks.

Fig. S3 Cyclic voltammetry curves of Ferrocene (a), Slipo (b), Samphi (c), PDI (d) and T2BTZ (e).

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 °C to 200 °C at a rate of 10 °C/min.

Fig. S4 Differential scanning calorimetry of Lipo (left) and Lamphi (right).

Fig. S5 Differential scanning calorimetry of Slipo (left) and Samphi (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$_2$CO$_3$ (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.

1H NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) 6.83 (d, 2H, $J = 2.1$ Hz), 6.39 (t, 1H, $J = 2.1$ Hz), 3.88 (t, 4H, $J = 6.6$ Hz), 1.74 (quint, 4H, $J = 6.6$ Hz), 1.53-1.11 (m, 36H), 0.88 (t, 6H, $J = 6.6$ 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°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.

1H NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) 6.93 (d, 2H, $J = 2.1$ Hz), 6.57 (t, 1H, $J = 2.1$ Hz), 3.88 (t, 4H, $J = 6.6$ Hz), 1.74 (quint, 4H, $J = 6.6$ Hz), 1.53-1.11 (m, 36H), 0.88 (t, 6H, $J = 6.6$ Hz).

13C NMR (100 MHz, CDCl$_3$, 40°C): $\delta$ (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°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\(_4\)Cl. 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\(_3\)): \( \delta \) (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\(_3\)): \( \delta \) (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 °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\(_2\)SO\(_4\). 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\(_3\)): \( \delta \) (ppm) 8.08 (d, 1\( H \), \( J = 3.9 \) Hz), 7.98...
(d, 1H, J = 3.9 Hz), 7.86 (d, 1H, J = 7.6 Hz), 7.82 (d, 1H, J = 7.6 Hz), 7.38 (d, 1H, J = 3.9 Hz), 7.10 (d, 1H, J = 3.9 Hz), 6.83 (d, 2H, J = 2.2 Hz), 6.43 (t, 1H, J = 2.2 Hz), 4.91 (d, 2H, J = 6.0 Hz), 4.01 (t, 4H, J = 6.6 Hz), 1.90 (quint, 4H, J = 6.9 Hz). 

13C NMR (100 MHz, CDCl3): 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_{2}O_{3}S_{3}: 774. Found: 774 (m/z). Elem. Anal.: Calcd. for C_{45}H_{62}N_{2}O_{3}S_{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 °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. 

1H NMR (300 MHz, CDCl3): δ (ppm) 7.95 (m, 2H), 7.79 (m, 2H), 6.55 (d, 2H, J = 2.2 Hz), 6.49 (t, 1H, J = 2.2 Hz), 3.94 (t, 4H, J = 6.4 Hz), 1.77 (quint, 4H, J = 6.9 Hz), 1.52-1.03 (m, 36H), 0.88 (t, 6H, J = 6.8 Hz). 

13C NMR (100 MHz, CDCl3): 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}N_{3}O_{4}: 591. Found: 591 (m/z). Elem. Anal.: Calcd. for C_{38}H_{57}N_{3}O_{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 °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$_3$N, 10/1/0.2 v/v) to afford n-4 (586 mg, 82%) as a pale yellow solid. \(^1\)H NMR (300 MHz, CDCl$_3$): \(\delta\) (ppm) 5.91 (t, 1H, \(J = 2.1\) Hz), 5.85 (d, 2H, \(J = 2.1\) Hz), 3.88 (t, 4H, \(J = 6.6\) Hz), 3.63 (br, 2H), 1.74 (quint, 4H, \(J = 6.9\) Hz), 1.50-1.14 (m, 36H), 0.88 (t, 6H, \(J = 6.8\) Hz). \(^{13}\)C NMR (100 MHz, CDCl$_3$): \(\delta\) (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 °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$_2$SO$_4$. 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$_3$): \(\delta\) (ppm) 8.32 (br, 2H), 7.89 (br, 6H), 6.56 (br, 3H), 4.37 (t, 4H, \(J = 7.0\) Hz), 3.96 (t, 4H, \(J = 6.8\) Hz), 1.86 (quint, 4H, \(J = 7.0\) Hz), 1.75 (quint, 4H, \(J = 6.8\) Hz), 1.55-1.04 (m, 72H), 0.97-0.76 (m, 6H). \(^{13}\)C NMR (100 MHz, CDCl$_3$): \(\delta\) (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 °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, CHCl₃) to afford n-6 (310 mg, 88%) as a red solid. ¹H NMR (300 MHz, CDCl₃): δ (ppm). 8.72 (m, 8H), 6.59 (t, 1H, J = 2.1 Hz), 6.46 (d, 2H, J = 2.1 Hz), 3.95 (t, 4H, J = 2.1 Hz), 3.90 (t, 4H, J = 6.3 Hz), 1.76 (quint, 4H, J = 6.3 Hz), 1.48 - 1.15 (m, 36H), 0.87 (t, 6H, J = 6.6 Hz). MS (ESI): Calcd. for C₅₄H₆₁NO₇: 835.4. Found: 835.4 (m/z). Elem. Anal.: Calcd. for C₅₄H₆₁NO₇: 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 °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 °C to afford n-7 (419 mg, 89%) as a brown solid. ¹H NMR (300 MHz, CDCl₃): δ (ppm) 8.56 (d, 2H, J = 8.1 Hz), 8.45 (d, 2H, J = 8.1 Hz), 8.39-8.18 (m, 4H), 6.57 (br, 1H), 6.56 (br, 2H), 4.19 (t, 2H, J = 7.5 Hz), 3.90 (t, 4H, J = 6.3 Hz), 2.43 (t, 2H, J = 6.9 Hz), 2.00-1.62 (m, 8H), 1.62-1.00 (m, 38H), 0.86 (t, 6H, J = 6.6 Hz). MS (ESI): Calcd. for C₆₀H₇₂N₂O₈: 948.5. Found: 949.5 (m/z + H⁺). Elem. Anal.: Calcd. for C₃₀H₃₆NO₄: C, 75.92; H, 7.65; N, 2.95. Found: C, 76.04; H, 7.62; N, 2.97.

Dyad Llippe p-7 (150 mg, 0.194 mmol), n-7 (184 mg, 0.194 mmol) and PPh₃ (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 °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 Llippe (171 mg, 52%) as a brown solid. ¹H NMR (400 MHz, CDCl₃): δ (ppm) 8.33 (d, 2H, J = 7.6 Hz), 8.07 (d, 2H, J = 7.6 Hz), 7.98 (d, 2H, J = 8.0 Hz), 7.89 (d, 2H, J = 8.0 Hz), 7.84 (d, 2H, J = 3.6 Hz), 7.68 (d, 2H, J = 3.6 Hz), 7.49 (d, 2H, J = 7.6 Hz), 7.33 (d, 2H, J = 7.6 Hz), 7.12 (d, 2H, J = 3.6 Hz), 7.04 (d, 2H, J = 3.6 Hz), 6.60 (d, 2H, J = 2.0 Hz), 6.59 (t, 1H, J = 2.0 Hz), 6.47 (d, 2H, J = 2.0 Hz), 6.31 (t, 1H, J = 2.0 Hz),
5.32 (s, 2H), 3.94 (t, 4H, J = 6.4 Hz), 3.90 (t, 4H, J = 6.4 Hz), 3.84 (t, 2H, J = 7.2 Hz), 2.44 (t, 2H, J = 6.8 Hz), 1.83-1.56 (m, 14H), 1.52-1.20 (m, 72H), 0.88 (t, 6H, J = 6.4 Hz), 0.87 (t, 6H, J = 6.4 Hz).

13C NMR (100 MHz, CDCl3): 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, 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 C105H132N4O10S3: 1705.91. Found: 1705.76 (m/z), 1728.74 (m/z + Na+), 1744.72 (m/z + K+).

When R=R2:

2-(3,5-Bis((2-(2-methoxy)ethoxy)ethoxy)ethoxy)phenylisoindoline-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 K2CO3 (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 °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 MgSO4, 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. 1H NMR (300 MHz, CDCl3): δ (ppm) 7.94 (m, 2H), 7.79 (m, 2H), 6.61 (d, 2H, J = 2.2 Hz), 6.55 (t, 1H, J = 2.2 Hz), 4.13 (t, 4H, J = 4.8 Hz), 3.95-3.79 (m, 4H), 3.78-3.59 (m, 12H), 3.37 (s, 6H).

13C NMR (100 MHz, CDCl3): 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 C28H37NO10: 547. Found: 547 (m/z). Elem. Anal.: Calcd. for C28H37NO10: C, 61.41; H, 6.81; N, 2.56. Found: C, 61.71; H, 6.79; N, 2.47.

3,5-Bis((2-(2-methoxy)ethoxy)ethoxy)aniline (n-4). Phthalimide n-3 (456 mg, 0.83 mmol) was dissolved in absolute ethanol (20 mL) at 60 °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. 1H NMR (300 MHz, CDCl3): δ (ppm) 5.90 (d, 2H, J = 2.1 Hz), 5.84 (t, 1H, J = 2.1 Hz), 4.02 (t, 4H, J = 4.8 Hz), 3.92-3.46 (m, 22H), 3.35 (s, 6H).

13C NMR (100 MHz, CDCl3): 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 C28H37NO10: 547. Found: 547 (m/z). Elem. Anal.: Calcd. for C28H37NO10: C, 61.41; H, 6.81; N, 2.56. Found: C, 61.71; H, 6.79; N, 2.47.
MHz, CDCl$_3$): $\delta$ (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-methoxyethoxy)ethoxy)ethoxy)phenyl)-9,10-bis(dodecyloxy carbonyl)-perylen-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 $^\circ$C 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$_2$SO$_4$, 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. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) 8.35 (m, 2H), 7.94 (m, 6H), 6.63 (t, 1H, $J = 2.1$ Hz), 6.55 (d, 2H, $J = 2.1$ Hz), 4.36 (t, 4H, $J = 6.9$ Hz), 4.15 (t, 4H, $J = 4.8$ Hz), 3.84 (t, 4H, $J = 4.8$ Hz), 3.74-3.64 (m, 12H), 3.55-3.53 (m, 4H), 3.37 (s, 6H), 1.83 (quint, 4H, $J = 7.2$ Hz), 1.52-1.14 (m, 36H), 0.87 (t, 6H, $J = 6.9$ Hz). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (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-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 $^\circ$C 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 $^\circ$C to afford n-6 (170 mg, 88%) as a red solid. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) 8.72 (br, 8H), 6.65 (br, 1H), 6.51 (br, 2H), 4.15 (br, 4H), 3.86 (br, 4H), 3.80-3.46 (br, 16H), 3.37 (s, 6H). MS (ESI): Calcd. for C$_{44}$H$_{41}$NO$_{13}$: 791.26. Found: 792.27 (m/z+H$^+$), 814.25 (m/z+Na$^+$).

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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 °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 °C to afford n-7 (419 mg, 89%) as a brown solid. $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ (ppm) 8.45 (d, 2H, $J = 8.1$ Hz), 8.25 (d, 2H, $J = 8.1$ Hz), 8.11 (d, 2H, $J = 8.1$ Hz), 8.02 (d, 2H, $J = 8.1$ Hz), 6.68 (br, 3H), 4.18 (br, 4H), 4.12 (br, 2H), 4.04-3.49 (m, 20H), 3.38 (s, 6H), 2.37 (br, 2H), 1.75 (br, 4H), 1.51 (br, 2H). MS (ESI): Calcd. for C$_{50}$H$_{52}$N$_2$O$_{14}$: 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$_3$N (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$_3$): $\delta$ (ppm) 8.51 (d, 2H, $J = 8.1$ Hz), 8.43-8.22 (m, 6H), 7.93 (d, 2H, $J = 3.6$ Hz), 7.82 (d, 2H, $J = 3.6$ Hz), 7.65 (d, 2H, $J = 3.6$ Hz), 7.50 (d, 2H, $J = 7.6$ Hz), 7.17 (d, 2H, $J = 3.6$ Hz), 7.13 (d, 2H, $J = 3.6$ Hz), 6.66 (t, 1H, $J = 2.1$ Hz), 6.59 (d, 2H, $J = 2.1$ Hz), 6.5 (d, 2H, $J = 2.1$ Hz), 6.34 (t, 1H, $J = 2.1$ Hz), 5.35 (s, 2H), 4.15 (t, 4H, $J = 4.2$ Hz), 3.99-3.80 (m, 10H), 3.79-3.48 (m, 16H), 3.37 (s, 6H), 2.43 (t, 4H, $J = 6.9$ Hz), 1.88-1.15 (m, 46H), 0.88 (t, 6H, $J = 6.3$ Hz). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (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$_2$O$_{16}$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$_{amphi}$
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 °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 °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, 1.51 mmol), 10 mL DCM and 0.5 mL Et₃N was added dropwise at 0 °C. After that, the solution was stirred overnight at room temperature. The reaction mixture was successively washed with aqueous NaHCO₃, 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%).

**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 °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 °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, 1.51 mmol), 10 mL DCM and 0.5 mL Et₃N was added dropwise at 0 °C. After that, the solution was stirred overnight at room temperature. The reaction mixture was successively washed with aqueous NaHCO₃, 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%).

**1H NMR (400 MHz, CDCl₃):** δ (ppm) 8.46 (d, 2H, J = 7.6 Hz), 8.26 (d, 2H J= 8.0 Hz), 8.14 (d, 2H, J = 8.0 Hz), 8.05 (d, 2H, J = 8.0 Hz), 7.75 (d, 1H, J = 3.2 Hz), 7.70 (d, 1H, J = 3.6 Hz), 7.40 (d, 1H, J = 7.6 Hz), 7.32 (d, 1H, J = 7.6 Hz), 7.14 (m, 2H), 6.64 (d, 2H, J = 1.6 Hz), 6.62 (t, 1H, J = 2.0 Hz), 6.58 (d, 2H, J = 1.6 Hz), 6.37 (t, 1H, J = 2.0 Hz), 5.44 (s, 2H), 5.00 (s, 2H), 3.95 (m, 8H), 1.78 (m, 8H), 1.45 (m, 8H), 1.31 (m, 64H), 0.88 (m, 12H).

**13C NMR (100 MHz, CDCl₃):** 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 C₃₁₀H₁₂₀N₄O₁₀S₅: 1649.85. Found: 1649.69 (m/z). Elem. Anal.: Calcd. for C₃₁₀H₁₂₀N₄O₁₀S₅: C, 73.51; H, 7.57; N, 3.39. Found: C, 73.59; H, 7.63; N, 3.37.
**Dyad S**<sub>amphi</sub>. 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 °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 °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<sub>3</sub>N was added dropwise at 0 °C. After dropwise, the solution was stirred overnight at room temperature. The reaction mixture was successively washed with aqueous NaHCO<sub>3</sub>, 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 S<sub>amphi</sub> (41.2 mg, 69%).

**d. Synthesis of reference compounds**

\[\text{H NMR (300 MHz, CDCl}_3\text{): }\delta (ppm) 8.65 (d, 2H, } J = 8.1 \text{ Hz), 8.59 (d, 2H } J = 7.8 \text{ Hz), 8.5 (d, 2H, } J = 8.1 \text{ Hz), 8.46 (d, 2H, } J = 8.4 \text{ Hz), 7.99 (d, 1H, } J = 4.2 \text{ Hz), 7.94 (d, 1H, } J = 3.6 \text{ Hz), 7.74 (d, 1H, } J = 7.5 \text{ Hz), 7.71 (d, 1H, } J = 7.5 \text{ Hz), 7.33 (d, 1H, } J = 3.6 \text{ Hz), 7.21 (d, 1H, } J = 3.6 \text{ Hz), 6.78 (d, 2H, } J = 2.1 \text{ Hz), 6.65 (t, 1H, } J = 2.1 \text{ Hz), 6.55 (d, 2H, } J = 2.1 \text{ Hz), 6.43 (t, 1H, } J = 2.1 \text{ Hz), 5.47 (s, 2H), 5.04 (s, 2H), 4.14 (t, 4H, } J = 4.2 \text{ Hz), 4.00 (t, 4H, } J = 6.3 \text{ Hz), 3.85 (t, 4H, } J = 4.2 \text{ Hz), 3.73 (m, 4H), 3.67 (m, 8H), 3.55 (m, 4H), 3.37 (s, 6H), 1.81 (t, 4H, } J = 6.6 \text{ Hz), 1.88-1.15 (m, 36H), 0.88 (t, 6H, } J = 6.6 \text{ Hz).} \]

\[\text{C NMR (100 MHz, CDCl}_3\text{): }\delta (ppm) 168.0, 162.9, 162.4, 160.6, 161.5, 151.8, 145.7, 140.7, 138.3, 137.9, 136.6, 135.3, 134.0, 133.8, 131.0, 129.7, 128.9, 128.5, 128.3, 127.0, 125.5, 125.4, 125.2, 124.7, 124.4, 124.0, 123.1, 122.9, 122.7, 122.1, 107.7, 104.3, 102.7, 100.9, 72.0, 70.8, 70.7, 70.6, 69.6, 68.2, 67.8, 59.0, 31.9, 29.7, 29.6, 29.5, 29.4, 29.3, 26.1, 22.7, 14.1. \]

**MS (MALDI-TOF):** Calcd. for C<sub>91</sub>H<sub>104</sub>N<sub>4</sub>O<sub>16</sub>S<sub>3</sub>: 1605.7. Found: 1605.48 (m/z), 1627.48 (m/z + Na<sup>+</sup>), 1644.45 (m/z + K<sup>+</sup>). Elem. Anal.: Calcd. for C<sub>101</sub>H<sub>124</sub>N<sub>10</sub>O<sub>10</sub>S<sub>3</sub>: C, 68.06; H, 6.53; N, 3.49. Found: C, 67.74; H, 6.86; N, 3.22.
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\(_3\)): \(\delta\) (ppm) 8.45 (d, 2H, \(J = 7.8\) Hz), 8.12 (d, 2H, \(J = 8.1\) Hz), 8.02 (d, 2H, \(J = 8.1\) Hz), 7.86 (d, 2H, \(J = 8.4\) Hz), 6.62 (d, 2H, \(J = 2.1\) Hz), 6.56 (t, 1H, \(J = 1.8\) Hz), 4.92 (s, 2H), 3.90 (t, 4H, \(J = 6.0\) Hz), 3.85 (s, 3H), 1.71 (m, 4H), 1.25 (m, 36H), 0.86 (m, 6H). \(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (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\(_2\)O\(_8\): 906.48. Found: 907.37 (m/z + H\(^+\)). Elem. Anal.: Calcd. for C\(_{57}\)H\(_{66}\)N\(_2\)O\(_8\): 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\(_3\)N (44 \(\mu\)L, 0.318 mmol) and acetic anhydride (20 \(\mu\)L, 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\(_2\)SO\(_4\), 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\(_3\)): \(\delta\) (ppm) 8.05 (d, 1H, \(J = 3.6\) Hz), 7.94 (d, 1H, \(J = 3.6\) Hz), 7.79 (br, 2H), 7.35 (d, 1H, \(J = 3.6\) Hz), 7.15 (d, 1H, \(J = 3.6\) Hz), 6.81 (d, 2H, \(J = 3.6\) Hz).
2.0 Hz), 6.42 (t, 1H, J = 2.0 Hz), 5.30 (s, 2H), 3.99 (t, 4H, J = 6.4 Hz), 2.12 (s, 3H), 1.80 (quint, 4H, J = 6.4 Hz), 1.53-1.16 (m, 36H), 0.88 (t, 6H, J = 6.4 Hz). 13C NMR (100 MHz, CDCl3): 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.

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. $\Delta\delta$ 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. $\Delta\delta$ 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 $^\circ$C)

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<th></th>
<th>S$_{\text{lipo}}$</th>
<th>S$_{\text{amphi}}$</th>
<th>L$_{\text{lipo}}$</th>
<th>L$_{\text{amphi}}$</th>
<th>PDI</th>
<th>T2BTZ</th>
<th>PDI&amp;T2BTZ</th>
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<td>$\Delta\delta$ of PDI-H$^a$</td>
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<td>0.07</td>
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Fig. S7 Concentration-dependent $^1$H NMR spectra of S$_{\text{lipo}}$

Fig. S8 Concentration-dependent $^1$H NMR spectra of S$_{\text{amphi}}$
Fig. S9 Concentration-dependent $^1$H NMR spectra of L-amphi

Fig. S10 Concentration-dependent $^1$H NMR spectra of 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