Supporting Information for

Excited Singlet States of Covalently Bound, Cofacial Dimers and Trimers of Perylene-3,4:9,10-bis(dicarboximide)s

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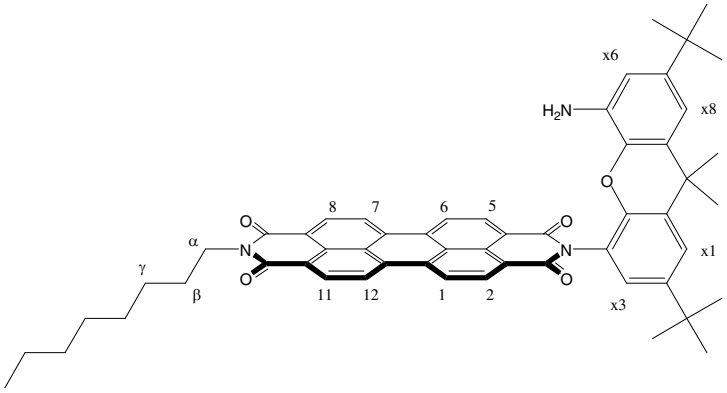
General Information

Proton nuclear magnetic resonance spectra were recorded on a Varian 400 spectrometer with TMS as an internal standard, and the chemical shifts are given in ppm downfield from TMS. Laser desorption mass spectra were obtained with a PE Voyager DE-Pro MALDI-TOF mass spectrometer using dithranol as a matrix. High resolution electrospray and fast atom bombardment mass spectra were obtained with the 70-SE-4F and Q-Tof Ultima mass spectrometers at the University of Illinois at Champaign-Urbana. Commercially available reagents were purchased from Sigma-Aldrich Co. and used without further purification. All solvents were spectrophotometric grade unless otherwise noted. Flash chromatography was performed using Sorbent Technologies (Atlanta, GA) silica gel.

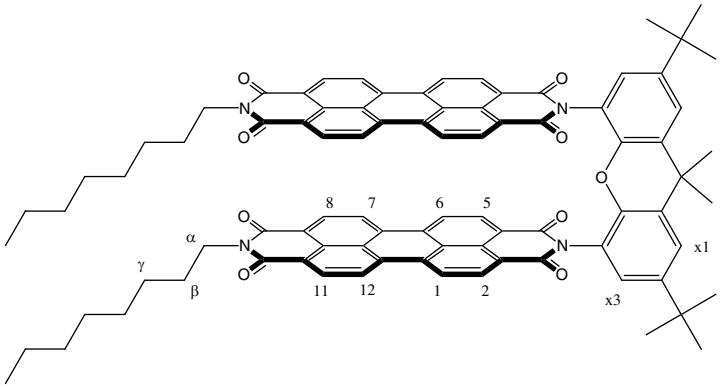
Synthesis



(PDI-C_8)-xan-NH_2

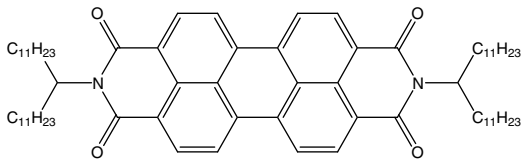
Crude *N*-octyl-perylene-3,4-dicorboxyanhydride-9,10-dicarboximide[1] (45 mg, 0.089 mmol) was dissolved in ~35 mL of chloroform and placed in a high boiling bump flask. The bump flask was placed atop a 50 mL, 14/20 round bottom flask that contained ~10 mL chloroform, imidazole (4 g), and 2,7-Di-*tert*-butyl-9,9-dimethyl-4,5-xanthenediamine (143 mg, 0.406 mmol). A condenser was placed atop the high boiling bump flask. The entire system was evacuated and flushed with nitrogen three times then heated to reflux for ~21 hours. The solution in the round bottom flask was diluted with 50 mL chloroform, washed with water (3 x 100 mL aliquots), dried with magnesium sulfate, filtered, and evaporated. The resulting brown residue was purified using silica gel chromatography and a mobile phase of 5% acetone in chloroform. The product was in the first fractions (R_f ~0.9) and was further purified using silica gel chromatography with 2% methanol in chloroform, yielding the product C_8-PDI-xan-NH_2 as a brown solid (23 mg, 0.027 mmol), 31% yield. $C_{55}H_{55}N_3O_5$ HRMS-FAB (m/z): 837.4144 (calc. 837.4142). ^{1}H NMR δ ($CDCl_3$): 8.68 ($H_{7,12}$, 2H, d, *J* = 7.9 Hz); 8.56 ($H_{1,6}$, 2H, d, *J* = 7.9 Hz); 8.50 ($H_{8,11}$, 2H, d, *J* = 7.9 Hz); 8.43 ($H_{2,5}$, 2H, d, *J* = 7.9 Hz); 7.59 (H_{x3}, 1H, d, *J* = 1.8 Hz); 7.37 (H_{x1}, 1H, d, *J* = 1.8 Hz); 6.84 (H_{x6}, 1H, d, *J* = 1.8 Hz); 6.55 (H_{x8}, 1H, d, *J* = 1.8 Hz); 4.18 (H_{α}-

$_{CH2}$, 2H, t, J = 7.9 Hz); 1.75 ($H_{\beta\text{-}CH2}$, 2H, m); 1.71 ($H_{xan2(CH3)}$, 6H, s); 1.45-1.40 ($H_{\gamma\text{-}CH2}$, 2H, m); 1.41 ($H_{xan(t\text{-}butyl\ on\ X2)}$, 9H, s); 1.40-1.27 ($H_{4(CH2)}$, 8H, m); 0.89 ($H_{CH3}$, 3H, t, J = 7.3 Hz).



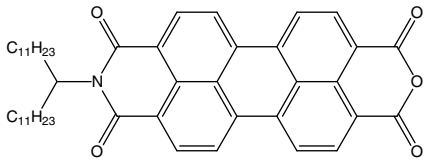
2a

Crude *N*-octyl-perylene-3,4-dicorboxyanhydride-9,10-dicarboximide[1] (717 mg, 1.42 mmol), 2,7-di-*tert*-butyl-9,9-dimethyl-4,5-xanthenediamine[2] (100 mg, 0.284 mmol), and imidazole (4 g), were places in a 10 mL 14/20 round bottom flask. The reaction mixture was heated to 160°C for 26 hours. The reaction mixture was dumped hot into 75 mL of chloroform. The solution was then washed with water (3 x 200 mL aliquots). The organic layer was dried over magnesium sulfate, filtered, and evaporated. The resulting orange/brown solid was dissolved in 15 mL of chloroform precipitated in 100 mL acetone, centrifuged, and decanted. The resulting material was orange/brown and totaled 200 mg (0.151 mmol, 55% yield). Melting point >300°C. $C_{87}H_{78}N_4O_9$ HRMS-FAB (m/z): 1322.5773 (calc. 1322.5769). ^{1}H NMR δ ($CDCl_3$): 8.28 ($H_{1,6}$, 4H, d, J = 7.7 Hz); 8.23 ($H_{7,12}$, 4H, d, J = 7.7 Hz); 8.11 ($H_{2,5}$, 4H, d, J = 7.9 Hz); 8.03 ($H_{8,11}$, 4H, d, J = 7.9 Hz); 7.61 (H_{x1}, 2H, s); 7.11 (H_{x3}, 2H, s); 4.21 ($H_{\alpha\text{-}CH2}$, 4H, t, J = 8.0 Hz); 1.87 ($H_{xan(CH3)}$, 6H, s); 1.76 ($H_{\beta\text{-}CH2}$, 4H, m); 1.49 and 1.41 ($H_{\gamma\text{-}CH2}$, 4H, m); 1.37 ($H_{xan(t\text{-}butyl)}$, 18H, s); 1.32 and 1.28 ($H_{(CH2)4}$, 16H, bs); 0.92 (H_{CH3}, 6H, m).



***N, N'*-bis(12-tricosanyl)-perylene-3,4:9,10-bis(dicarboximide)**[3]

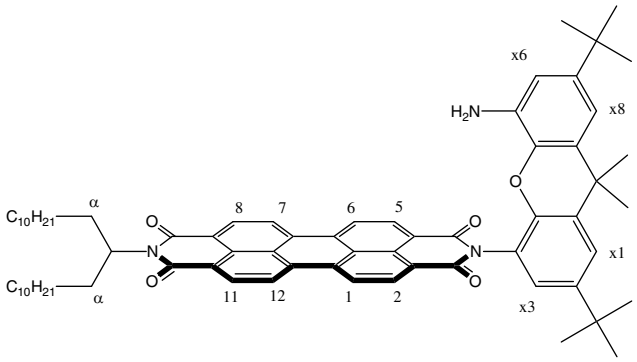
12-aminotricosane (2.486 g, 7.31 mmol), perylene-3,4:9,10-tetracarboxylicdianhydride (1.483 g, 3.78 mmol), and imidazole (10 g, 146.89 mmol) were heated to 180˚C under a nitrogen atmosphere for 4.5 hours. The reaction mixture was cooled to room temperature and 75 ml ethanol added. This solution was poured into 300 ml of 2M HCl and stirred at room temperature for 4 hours. The red precipitate was filtered, washed with water, and dried to give 3.788 g (1) (96.8 %). $C_{70}H_{102}N_2O_4$ HRMS-FAB (m/z): 1034.7835 (calc. 1034.7840). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.617 (m, 8H), 5.180 (m, 2H), 2.234 (m, 4H), 1.863 (m, 4H), 1.19-1.25 (m, 72H), 0.830 (t, 12H, J = 6.9 Hz).



***N,N'*-bis-(12-tricosanyl)-perylene-3,4-dicarboxyanhydride-9,10-dicarboximide**

A 0.19 M KOH in *t*-butanol (18 ml, 3.4 mmol) solution was made by placing KOH (0.42 g, 7.5 mmol) in 40 mL of *t*-butanol and refluxing until the KOH was completely dissolved. *N,N'*-bis-(12-tricosanyl)-perylene-3,4:9,10-bis(dicarboximide) (1.008 g, 0.97 mmol) was added to the a refluxing solution of 0.19 M KOH in *t*-butanol (18 ml, 3.4 mmol) and heated for 22 minutes. This hot mixture was immediately poured into a stirring solution of 28 mL acetic acid and 14 mL hydrochloric acid. This was stirred until the solution returned to room temperature after which the solution was diluted with 50 ml chloroform and washed with water (3 X 100 mL aliquots). The organic fraction was concentrated and column chromatographed on silica using 100 %

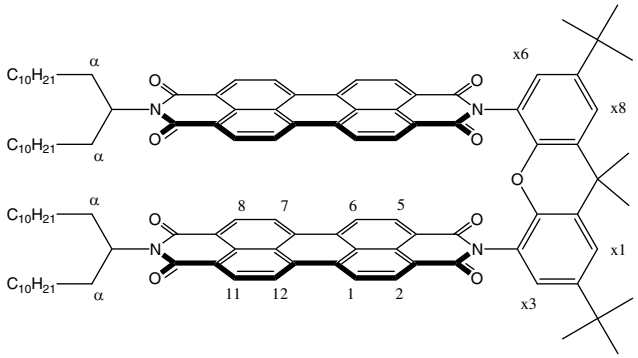
chloroform as the eluent to yield 0.271 g of the product (39 %). $C_{47}H_{55}NO_5$ HRMS-ESI (m/z): $[M+H]^+$ 714.4161 (calc. 714.4158) ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 8.697 (m, 8H), 5.178 (m, 1H), 2.238 (m, 2H), 1.855 (m, 2H), 1.15-1.30 (m, 36H), 0.835 (t, J = 6.9 Hz, 6H).



(PDI-ST)-xan-NH_2

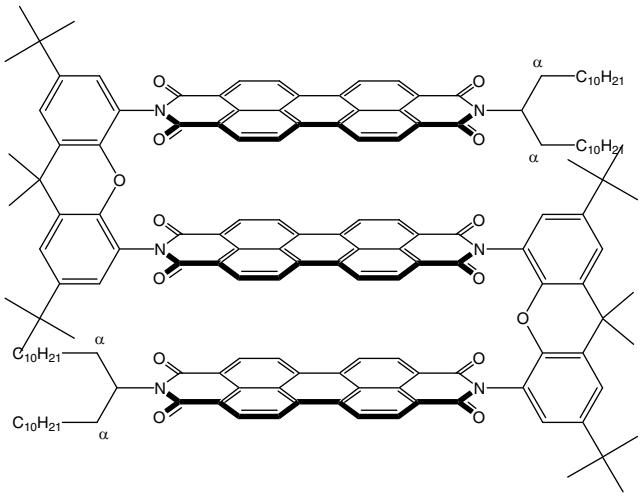
N-(12-tricosanyl)-perylene-3,4-dicarboxyanhydride-9,10-dicarboximide (125 mg, 0.175 mmol) was dissolved in 35 mL of toluene and placed in a high boiling bump flask. The bump flask was placed atop a 50 mL, round bottom flask that contained 35 mL toluene, imidazole (4 g), and 2,7-Di-*tert*-butyl-9,9-dimethyl-4,5-xanthenediamine (204 mg, 0.579 mmol). A condenser was placed atop the high boiling bump flask. The entire system was evacuated and flushed with nitrogen three times then heated to reflux for 21 hours. The solution in the round bottom flask was diluted with 50 mL chloroform, washed with water (3 x 75 mL aliquots), dried with magnesium sulfate, filtered, and evaporated. The resulting brown residue was purified using silica gel chromatography and a mobile phase of 0.5% acetone in chloroform and yielded the product **(PDI-ST)-xan-NH_2** as a brown solid (121 mg, 0.116 mmol), 66% yield. $C_{70}H_{85}N_3O_5$ HRMS-FAB (m/z): 1047.6492 (calc. 1047.6489). ^{1}H NMR δ ($CDCl_3$): 8.74 ($H_{perylene}$, 2H, d, J = 7.9 Hz); 8.69 ($H_{perylene}$, 2H, m); 8.63 ($H_{perylene}$, 2H, d, J = 9.2 Hz); 8.61 ($H_{perylene}$, 2H, d, J = 9.2 Hz); 7.60 (H_{x3}, 1H, s); 7.29 (H_{x1}, 1H, s); 6.85 (H_{x6}, 1H, s); 6.56 (H_{x8}, 1H, s); 5.21 (H_{NCH}, 1H, m);

3.34 ($H_{xan(NH2)}$, 2H, bs); 2.28 ($H_{\alpha-CH2}$, 2H, m); 1.91 ($H_{\alpha-CH2}$, 2H, m); 1.72 ($H_{xan2(CH3)}$, 6H, s); 1.41 ($H_{xan(t\text{-}butyl\ on\ X2)}$, 9H, s); 1.39-1.31 ($H_{CH2}$, 12H, m); 1.27 ($H_{xan(t\text{-}butyl\ on\ X7)}$, 9H, s); 1.22 ($H_{CH2}$, 24H, bs); 0.86 ($H_{2(CH3)}$, 6H, t, J = 6.7, 7.0).



2b

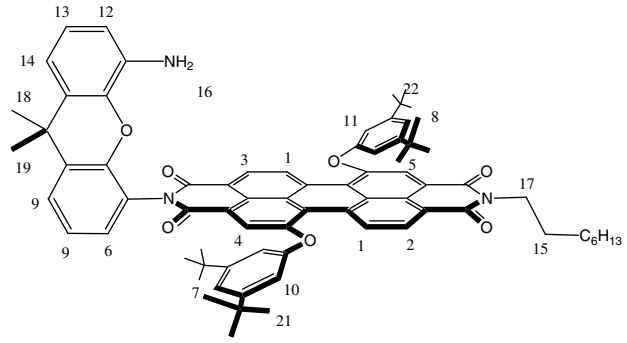
N-(12-tricosanyl)-perylene-3,4-dicarboxyanhydride-9,10-dicarboximide (320 mg, 0.449 mmol), 2,7-Di-*tert*-butyl-9,9-dimethyl-4,5-xanthenediamine (66 mg, 0.188 mmol), and imidazole (3 g), were placed in a 5 mL 14/20 round bottom flask. The reaction mixture was heated to 160°C for 18 hours. The reaction mixture was dumped hot into 75 mL of chloroform. The solution was then washed with water (3 x 100 mL aliquots). The organic layer was dried over magnesium sulfate, filtered, and evaporated. The resulting brown residue was purified using silica gel chromatography and a mobile phase of 1% acetone in chloroform yielded **2b** as a brown solid (137 mg, 0.079 mmol), 42% yield. Melting point 272°C. $C_{117}H_{138}N_4O_9$ HRMS-FAB (m/z): 1743.0456 (calc. 1743.0464). ^{1}H NMR δ ($CDCl_3$): 8.50 – 7.87 ($H_{perylene}$, 16H, m); 7.60 (H_{x1}, 2H, s); 7.07 (H_{x3}, 2H, s); 5.07 (H_{NCH}, 2H, m); 2.32 ($H_{\alpha-CH2}$, 4H, m); 2.24 ($H_{\alpha-CH2}$, 4H, m); 1.85 ($H_{xan(2CH3)}$, 6H, s); 1.34 ($H_{xan(t\text{-}butyl)}$, 18H, s); 1.30-1.00 (H_{36CH2}, 72H, m); 0.95-0.78 (H_{4CH3}, 12H, m).



3

(PDI-ST)-xan-NH_2 (107 mg, 0.102 mmol), perylene-3,4:9,10-tetracarboxylic dianhydride (18 mg, 0.046 mmol), 1.3 g imidazole, and 4 mL toluene were placed in a 10 mL 14/20 round bottom flask. The flask was evacuated and flushed with nitrogen three times. The solution was heated to reflux under nitrogen for 72 hours. The reaction mixture was cooled to room temperature and diluted with 30 mL chloroform and washed with water (3 x 75 mL aliquots). The organic layer was dried over magnesium sulfate, filtered, and evaporated. The resulting brown residue was purified using silica gel chromatography and a mobile phase beginning with 1% acetone in chloroform then ramped up to 2% acetone in chloroform. Once **(PDI-ST)-xan-NH_2** came off the column the product came off the column with a mobile phase of 10% methanol in chloroform. Product **3**, a reddish-brown shiny solid, was obtained in 69% yield (94 mg, 0.038 mmol). Melting point >300°C. $C_{164}H_{174}N_6O_{14}$ HRMS-FAB (m/z): 2451.3084 (calc. 2451.3088). ^{1}H NMR δ ($CDCl_3$): 8.31 ($H_{perylene}$, 4H, bs); 8.10-7.40 ($H_{perylene}$, 20H, bs); 7.81 (H_{x3}, 2H, s); 7.64 (H_{x1}, 2H, s); 7.53 (H_{x1}, 2H, s); 6.81 (H_{x3}, 2H, s); 4.90 (H_{NCH}, 2H, bs); 2.11 ($H_{\alpha-CH2}$, 4H, bs); 1.96 ($H_{\alpha-CH2}$, 4H, bs); 1.82 ($H_{xan4(CH3)}$, 12H, s); 1.61 ($H_{xan2(t\text{-}butyl)}$, 18H, s); 1.33 (H_{CH2},

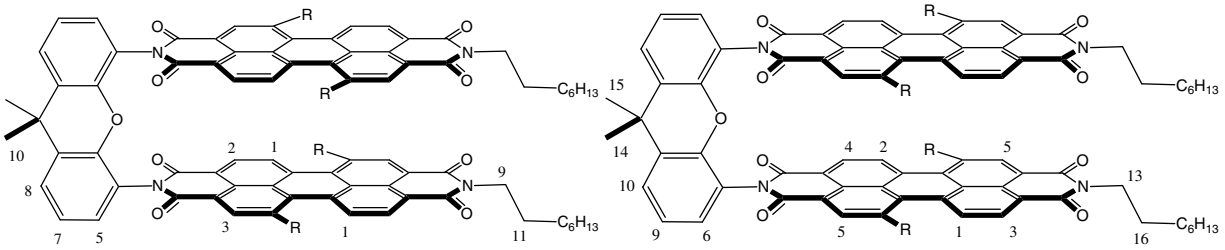
30, bs); 1.26 ($H_{xan2(t\text{-}butyl)}$, 18H, s); 1.20-1.00 (H_{CH2}, 40H, m); 0.95 ($H_{2(CH3)}$, 6H, m); 0.78 ($H_{2(CH3)}$, 6H, m).



(C_8-PPDI)-xan-NH_2

N-(octyl)-1,7-(3',5'-di-t-butylphenoxy)perylene-3,4-dicarboxyanhydride-9,10-dicarboximide (100 mg, 0.11 mmol), 9,9-dimethyl-4,5-xanthenediamine[4] (87 mg, 0.37 mmol), 130 mg imidazole, and 12 mL toluene were placed in a 25 mL 14/20 round bottom flask. This reaction mixture was evacuated and backfilled with nitrogen three times. The solution was heated to 110° C under nitrogen for 10 hours. The reaction mixture was cooled to room temperature and diluted with 50 mL chloroform and washed with a brine solution (3 x 100 mL aliquots). The organic layer was dried over magnesium sulfate, filtered, and evaporated. The resulting red residue was purified using silica gel chromatography and a mobile phase of ethanol free chloroform (R_f = 0.37). The product was obtained as a red solid (49 mg, 0.043 mmol) in 39% yield. HRMS-FAB (m/z): 1133.5918 (calc. 1133.5918). ^{1}H NMR δ ($CDCl_3$): H_1, 9.73 (2H, d, *J* = 8.55 Hz); H_2, 8.71 (1H, d, *J* = 7.33 Hz); H_3, 8.66 (1H, d, *J* = 7.33 Hz); H_4, 8.40 (1H, s); H_5, 8.37 (1H, s); H_6, 7.55 (1H, m); H_7, 7.37 (1H, s); H_8, 7.35 (1H, s); H_9, 7.23 (2H, m); H_{10}, 7.06 (2H, s); H_{11}, 7.05 (2H, s); H_{12}, 6.84 (1H, t, *J* = 8.55 Hz); H_{13}, 6.79 (1H, d, *J* = 7.94 Hz); H_{14}, 6.48 (1H, d, *J* = 7.94 Hz); H_{15}, 4.16 (2H, t, *J* = 7.94 Hz); H_{16}, 3.31 (2H, s); H_{17}, 1.73 (2H, m); H_{18}, 1.67 (3H, s); H_{19},

1.63 (3H, s); H_{21}, 1.35 (18H, s); H_{22}, 1.33 (18H, s); H (octyl methylenes), 1.4-1.2 (10H, m); H (octyl methyls), 0.87 (3H, t, J = 7.33 Hz).

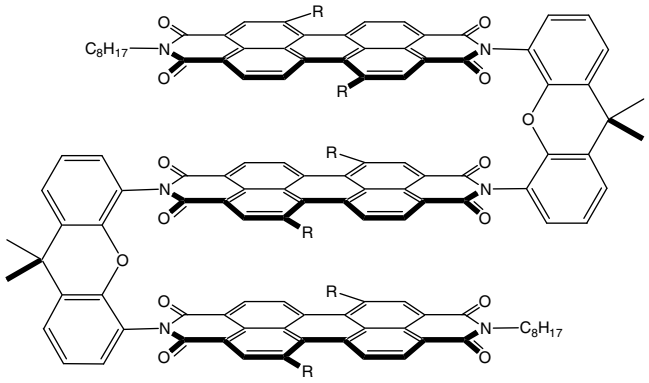


5a **R = 3,5-di-*tert*-butylphenoxy** **5b**

5a and **5b**

N-(octyl)-1,7-(3',5'-di-t-butylphenoxy)perylene-3,4-dicarboxyanhydride-9,10-dicarboximide (100 mg, 0.11 mmol), 9,9-dimethyl-4,5-xanthenediamine (87 mg, 0.37 mmol), 130 mg imidazole, and 12 mL toluene were placed in a 25 mL 14/20 round bottom flask. This reaction mixture was evacuated and backfilled with nitrogen three times. The solution was heated to 110° C under nitrogen for 10 hours. The reaction mixture was cooled to room temperature and diluted with 50 mL chloroform and washed with a brine solution (3 x 100 mL aliquots). The organic layer was dried over magnesium sulfate, filtered, and evaporated. The resulting red residue was purified using silica gel chromatography and a mobile phase of ethanol free chloroform (R_f = 0.50. Isomer **5a** was obtained as a red solid (50 mg, 0.025 mmol) in 23% yield. Melting point >300°C. $C_{135}H_{142}N_4O_{13}$ HRMS-FAB (m/z): 2027.0575 (calc. 2027.0573). ^{1}H NMR δ ($CDCl_3$): H_1, 9.66 (2H, d, J = 8.55 Hz); H_1, 9.65 (2H, d, J = 8.55 Hz); H_2, 8.28 (2H, d, J = 8.55 Hz); H_2, 8.27 (2H, d, J = 8.55 Hz); H_3, 7.94 (2H, s); H_4, 7.90 (2H, s); H_5, 7.52 (2H, dd, J = 7.94, 1.22 Hz); H_6, 7.29 (4H, s); H_7, 7.15 (2H, t, J = 7.94 Hz); H_8, 7.04 (4H, d, J = 1.22 Hz); H_8, 7.02 (4H, d, J = 1.22 Hz); H_8, 7.02 (2H, dd, J = 7.94, 1.22 Hz); H_9, 4.09 (4H, m); H_{10}, 1.76 (6$H_{xan(2CH3)}$, s); H_{11}, 1.64 (4H, m); H(octyl methylenes), 1.26 (20H, s); H_{13}, 1.23 (36H, s); H_{14}, 1.22 (36H, s); H(octyl methyls)$_{15}$, 0.85 (6H, m).

Isomer **5b** was obtained as a red solid (18 mg, 0.089 mmol) in 8% yield. Melting point >300°C. HRMS-FAB (m/z): 2027.0575 (calc. 2027.0573). ^{1}H NMR δ ($CDCl_3$): H_1, 9.30 (2H, d, *J* = 8.55 Hz); H_2, 9.26 (2H, d, *J* = 8.55 Hz); H_3, 8.25 (2H, d, *J* = 8.55 Hz); H_4, 8.18 (2H, d, *J* = 8.55 Hz); H_5, 8.10 (2H, s); H_5, 8.09 (2H, s); H_6, 7.54 (2H, d, *J* = 7.94 Hz); H_7, 7.30 (2H, s); H_8, 7.25 (2H, s); H_9, 7.18 (2H, t, *J* = 7.94 Hz); H_{10}, 7.04 (2H, d, *J* = 7.94 Hz); H_{11}, 6.92 (4H, s); H_{12}, 6.88 (4H, s); H_{13} 4.13 (4H, m); H_{14}, 1.80 ($3H_{xan(CH3)}$, s); H_{15}, 1.76 ($3H_{xan(CH3)}$, s); H_{16}, 1.70 (4H, m); H(octyl methylenes), 1.25, 1.30 (20H, two s); H_{17}, 1.28 (36H, s); H_{18}, 1.27 (36H, s); H(octyl methyls), 0.86 (6H, m).



R = 3,5-di-tert-butylphenoxy

6

(PPDI-C_8)-xan-NH_2 (102 mg, 0.104 mmol), 1,7-(3',5'-di-t-butylphenoxy)perylene-3,4;9,10-tetracarboxyanhydride (31 mg, 0.105 mmol), 1.0 g imidazole, and 4 mL toluene were placed in a 5 mL 14/20 round bottom flask. This solution was evacuated and backfilled with nitrogen three times. The solution was then heated with an oil bath whose temperature was 130° C under nitrogen for 48 hours. The reaction mixture was allowed to cool to room temperature, diluted with 25 mL chloroform and washed with water (3 x 75 mL aliquots). The organic layer was dried over magnesium sulfate, filtered, and evaporated. The resulting red residue was purified

using preparatory thin layer chromatography with neat chloroform (amylene stabilized) as the eluent (R_f ~ 0.4). This product was further purified using HPLC using a Phenomenex Luna Si 5µ (2) 100A 4.6 X 250 mm column affording 1 mg of product (1.2% yield), Figure S1. MALDI-MS (m/z): 3032.2 (calc. 3031.5). ^{1}H NMR δ ($CDCl_3$): 9.95 (1H, d, J = 8.6 Hz); 9.93 (1H, d, J = 8.6 Hz); 9.05 (1H, d, J = 8.6 Hz); 9.01 (1H, d, J = 8.6 Hz); 9.00 (1H, d, J = 8.6 Hz); 8.82 (1H, d, J = 8.6 Hz); 8.74 (1H, d, J = 8.6 Hz); 8.52 (1H, d, J = 8.6 Hz); 8.25 (1H, s); 8.10 (1H, s); 8.05 (1H, s); 7.99 (1H, s); 7.81 (1H, d, J = 8.6 Hz); 7.75 (1H, dd, J = 8.6,1.2 Hz); 7.69 (1H, d, J = 8.6 Hz); 7.58-7.46 (7H, m); 7.40-7.27 (9H, m); 7.24-7.04 (7H, m); 7.05 (2H, d, J = 1.2Hz); 6.91 (1H, dd, J = 8.6,1.2 Hz); 6.87 (1H, dd, J = 8.6,1.2 Hz); 6.70 (2H, d, J = 1.2 Hz); 6.59 (2H, d, J = 1.2 Hz); 6.36 (2H, d, J = 1.2 Hz); 3.83 (4H, m); 1.85 (3H $_{xan(CH3)}$, s); 1.80 (3H $_{xan(CH3)}$, s); 1.70 (6H $_{xan(2CH3)}$, s); 1.57 (4H, m); 1.56 (18H, s); 1.30-1.26 (20H, broad multiplet); 1.22 (36H, s); 1.14 (18H, s); 1.08 (18H, s); 1.06 (18H, s); 0.86 (6H, m).

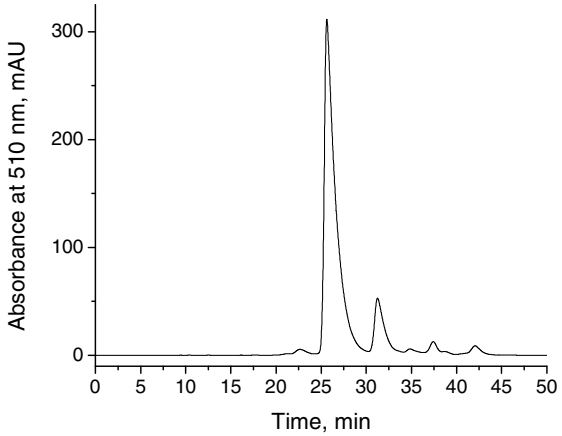


Figure S1. HPLC chromatogram of crude **6**. Mobile phase: 18% chloroform/82% hexanes (v/v).

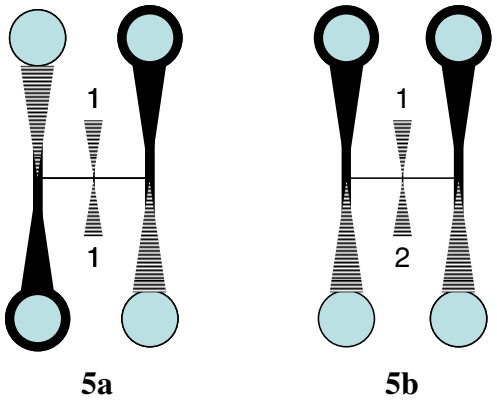


Figure S2. Representation of PPDI dimers, view along the PPDI N-N axis, with PPDIs staggered (left) and eclipsed (right). Phenoxy groups are represented by circles, perylenes are represented by thick vertical wedges and xanthene is represented by a horizontal line that runs perpendicular to the perylenes. The thin vertical wedges represent the methyl groups at the 9-position of the xanthene while the numbers are labels for the different types of methyl groups. Bold objects are "up" (closer to the reader) while normal objects are "down" (away from the reader). Methyl groups are numbered based on environment. In the staggered representation each methyl group is numbered "1" because each methyl group has the same physical proximity between the phenoxy groups on its side of the xanthene plane. In the eclipsed representation, the "1" methyl group has the phenoxy groups on its side up, while the "2" methyl group has both of its phenoxy groups down.

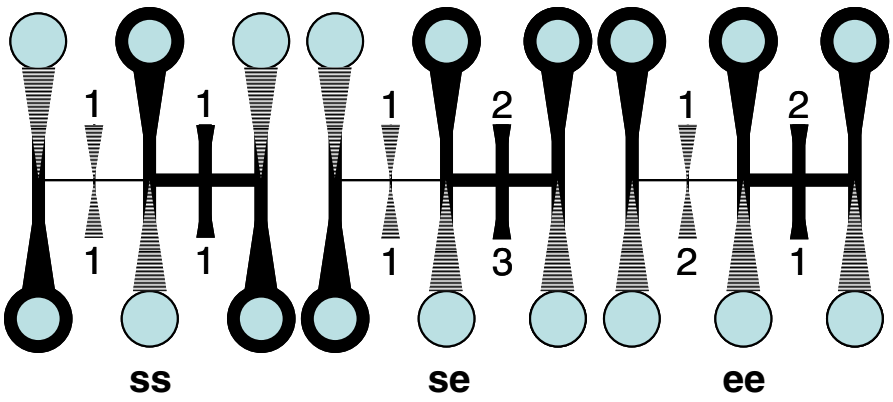


Figure S3. Representative diagrams of the possible isomers of **6**. Methyl groups are numbered based on different environments. The all-staggered isomer (ss) should result in the xanthene methyl groups having only one resonance while the methyl groups in the all eclipsed isomer (ee) should only have two resonances. The staggered-eclipsed isomer (se) should result in three different resonances.

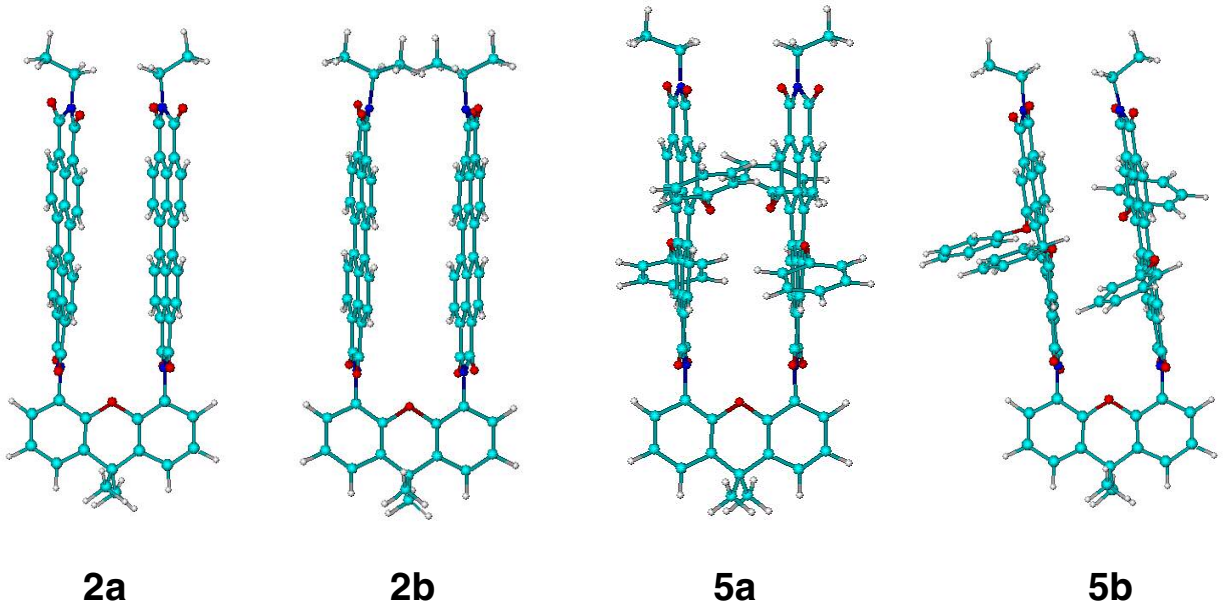


Figure S4. Energy-minimized ground state structures of the indicated molecules using the PM3 semi-empirical MO method. The t-butyl groups on the xanthenes were removed in **2a** and **2b**, the t-butyl groups on the phenoxy groups of **5a** and **5b** were removed, and the alkyl tails on the PDI and PPDI imides were truncated to ethyl and isopropyl groups to increase computational speed.

References:

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