Electronic Supplementary Material (ESI) for Molecular BioSystems

Supporting Information: Synthesis of the Light Driven Molecular Motors Conjugated with Peptide and Evaluation of the DNA Binding Properties

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Contents

General Methods. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. The ^{1}H-NMR (400 MHz or 600 MHz) spectra were recorded on a JEOL LAMBDA 400 or 600. ESIMSs were recorded using a BioTOF II mass spectrometer or APEX III (Bruker Daltonics). MALDI-TOF mass spectra were measured by negative mode using the laser at 337 nm and 3-hydroxypicolinic acid as the matrix. The ultraviolet-visible (UV-vis) absorption spectra were recorded by a JASCO V-550 UV-VIS or GENESIS 10uv scanning system (Thermo Electron Corporation). HPLC was performed on cosmosil 5C18AR and 5C18MS columns (nacalai tesque) monitoring at 254 nm. Densitometric analysis of the gel was carried out on the 20% denaturing polyacrylamide gel plates, and visualized, quantified with use of a FLA-5100 Fluor Imager.

X-ray Crystallography. In each case a single crystal was selected for data collection and mounted on a Mac Science MXC18 automated four-circle diffractometer: radiation, Cu-$K\alpha$ (1.54178 Å); monochromator, graphite crystal. The crystal system, space group, unit cell parameters, and orientation matrix were determined. Data collection was carried out with a 2θ-θ scan: temperature, 20 °C; scan speed, 14°min–1; scan range, 1.29–2.92° + 0.2° tan θ; 2θ scan limits, 2°–136°; standard reflections, 3 per 100 reflections; crystal stability, no indication of standard reflection decay during data collection.

6-Bromo-2-naphthalenemethanol (3)

To a stirred suspension of $LiAlH_4$ (298.9 mg, 7.88 mmol) in dry ether (50 ml) was added dropwise a solution of methyl 6-bromo-2-naphthoate (2.09 g, 7.88 mmol) in ether (50 ml) at 0 ºC, and stirred at room temperature for 1 h. After addition of water, the supernatant organic layer was filtered, dried with anhydrous $MgSO_4$, and evaporated to dryness to yield methyl alcohol **15** as white solid (1.86 g, 99%): mp 133-135 ºC, white solid; IR (film on KBr) ν_{max} 3267, 2922, 2865, 1590, 1457, 1364, 1339, 1128, 1038, 1013, 950, 885, 822, 735, 694, 667, 628, 593 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.90 (1H, s), 4.83 (1H, s), 7.48 (1H, dd, J = 8.6, 1.7 Hz), 7.54 (1H, dd, J = 8.8, 1.9 Hz), 7.68 (1H, d, J = 8.8 Hz), 7.73 (1H, d, J = 8.6 Hz), 7.76 (1H, br s), 7.98 (1H, d, J = 1.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 65.2, 119.8, 125.2, 126.1, 127.4, 129.51, 129.55, 129.7, 131.7, 133.9, 138.8. Anal. Calcd for $C_{11}H_9BrO$: C, 55.72; H, 3.83; Br, 33.70. Found: C, 55.72; H, 3.87; Br, 33.76.

2-Methyl-3-(6-bromo(2-naphthyl))propionic acid (4)

A mixture of alcohol **3** (4.74 g, 20.0 mmol), PPh_3 (6.30 g, 2.57 mmol), CBr_4 (7.96 g, 24.0 mmol) in dry THF (100 ml) was stirred at room temperature for 24 h. The reaction mixture was filtered, dried with anhydrous $MgSO_4$, and evaporated to dryness to yield crude bromide (8.20 g). To a stirred suspension of NaH (60% in oil, 1.6 g, 40.0 mmol) in dry THF (100 ml) was added diethyl methylmalonate (6.97 g, 7.88 mmol), and stirred at 0 ºC for 1 h. The crude bromide (8.20 g) was added at 0 ºC, and stirred at room temperature for 12 h. The reaction mixture was treated with a saturated aqueous solution of NH_4Cl (10 ml), and extracted with AcOEt (3 x 50 mL). The combined organic layers were washed with brine, dried with anhydrous $MgSO_4$, and evaporated to dryness to yield crude ester. To the crude ester in EtOH (50 ml), 3 M aqueous solution of KOH (2 ml) was added, and stirred at 80 ºC for 12 h. After being cooled to room temperature, the reaction mixture was evaporated to dryness, added water (50 ml), and extructed with ether (50 ml). The aqueous layer was cooled at 0 °C and acidified with 3 M HCl, giving a white precipitate, which was collected by filtration. The crystalline solid obtained was heated at 190 ºC for 1 h. After being cooled to room temperature, acid (±)-**4** was obtained as pale brown solid (4.40 g, 76%): mp 119-121 ºC; IR (film on KBr) ν_{max} 3047, 2974, 2935, 2615, 1703, 1590, 1498, 1462, 1414, 1337, 1291, 1237, 1208, 1186, 1119, 1062, 963, 878, 808, 748, 723, 693, 664, 603, 564 cm^{-1}; ^1H NMR (400 MHz, CD_3OD) δ 1.17 (3H, d, J = 6.8 Hz), 2.77-2.86 (2H, m), 3.13 (1H, m), 7.40 (1H, dd, J = 8.3, 1.5 Hz), 7.52 (1H, dd, J = 8.8, 2.0 Hz), 7.67 (1H, s), 7.71 (1H, d, J = 8.8 Hz), 7.73 (1H, d, J = 8.3 Hz), 8.01 (1H, d, J = 1.5 Hz); ^{13}C NMR (100 MHz, CD_3OD) δ 17.4, 40.8, 42.5, 120.1, 128.1, 128.4, 129.7, 130.2, 130.5, 130.6, 133.4, 134.7, 139.3, 179.8. Anal. Calcd for $C_{14}H_{13}BrO_2$: C, 57.36; H, 4.47; Br, 27.26. Found: C, 57.35; H, 4.47; Br, 27.25.

2-(±)Methyl-2,3-dihydro-1*H*-cyclopenta[*a*]-(6-bromonaphthalen)-1-one (5)

A mixture of acid (±)-**4** (2.58 g, 8.81 mmol), polyphosphoric acid (prepared by the addition of 25 g of P_2O_5 to 25 mL of phosphoric acid and heating to 110 °C with stirring until all of the P_2O_5 is dissolved) was stirred at 110 ºC for 12 h. After being cooled, the reaction mixture is diluted with 100 mL of water and extracted with chloroform (3 x 100 mL). The combined organic layers were washed with brine, dried with anhydrous $MgSO_4$, and evaporated to dryness. The clued product was purified by a short column chromatography on silica gel (hexane/AcOEt = 10:1) to yield ketone (±)-**5** as white solid (2.21mg, 91%): mp 78-80 °C, white solid; IR (film on KBr) ν_{max} 2963, 2929, 2870, 1698, 1624, 1583, 1505, 1455, 1432, 1405, 1345, 1311, 1208, 1190, 1166, 1115, 1066, 992, 917, 887, 835, 808, 757, 628, 571 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.37

(3H, d, *J* = 7.3 Hz), 2.78-2.84 (2H, m), 3.46 (1H, dd, *J* = 18.3, 8.3 Hz), 7.52 (1H, d, *J* = 8.6 Hz), 7.72 (1H, dd, *J* = 9.0, 2.0 Hz), 7.93 (1H, d, *J* = 8.6 Hz), 8.04 (1H, d, *J* = 2.0 Hz), 9.02 (1H, d, *J* = 9.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.5, 35.3, 42.4, 120.6, 125.1, 125.7, 127.9, 130.1, 130.3, 132.0, 133.9, 134.5, 156.7, 209.7. Anal. Calcd for $C_{14}H_{11}BrO$: C, 61.11; H, 4.03; Br, 29.04. Found: C, 61.27; H, 4.11; Br, 29.15.

(2*R,2'*R**)-(*P**,*P**)-(*E*)-(±)- and (2*R**,2'*R**)-(*P**,*P**)-(*Z*)-(±)-2,2'-Dimethyl-2,2',3,3'-tetrahydro-1,1'-bicyclopenta[*a*]-9,9'-bromonaphthalenylidene (6)**

To a stirred suspension of zinc powder (197.3 mg, 3.02 mmol) in dry THF (2 ml) was added slowly $TiCl_4$ (0.17 ml, 1.51 mmol) at 0 °C. The reaction mixture was stirred at 0 ºC for 0.5 h and then refluxed for 1.5 h. After a solution of methyl ketone (±)-**20** (200.0 mg, 0.75 mmol) in dry THF (2 ml) was added, the reaction mixture was refluxed for 3 h. The reaction mixture was filtrated with Celite, treated with a saturated aqueous solution of NH_4Cl (10 ml), and extracted with AcOEt (3 x 10 mL). The combined organic layers were washed with brine, dried with anhydrous $MgSO_4$, and evaporated to dryness. The crude product was purified by a short column chromatography on silica gel (hexane/AcOEt = 20:1) and further purified by HPLC (ODS, MeOH). From the less polar fraction, *trans*-dimethyl olefine (2*R**,2'*R**)-(*P**,*P**)-(*E*)-(±)-**6a** was obtained as colorless solid (13.2 mg, 14%) which was recrystallized from hexane/AcOEt giving yellow prisms: mp 197-198 ºC; IR (film on KBr) ν_{max} 3047, 2962, 2926, 2852, 1578, 1561, 1503, 1455, 1374, 1349, 1258, 1217, 1149, 1070, 969, 911, 883, 822, 802, 757, 707, 670, 650, 640 cm^{-1}; ^{1}H NMR (600 MHz, CD_2Cl_2) δ 1.26 (6H, d, *J* = 6.4 Hz, Me2ax), 2.36 (2H, d, *J* = 14.6 Hz, H3eq), 2.93 (2H, dd, *J* = 14.6, 6.4 Hz, H3ax), 2.99 (2 H, dq, *J* = 6.4, 6.4 Hz, H2eq), 7.45 (2H, d, *J* = 8.3 Hz, H4), 7.61 (2H, dd, *J* = 8.8, 2.0 Hz, H8), 7.69 (2H, d, *J* = 8.3 Hz, H5), 8.07 (2H, d, *J* = 2.0 Hz, H6), 8.11 (2H, d, *J* = 8.8 Hz, H9); ^{13}C NMR (150 MHz, CD_2Cl_2) δ 19.3 (Me2ax), 41.5 (C3), 43.5 (C2), 118.9 (C7), 125.7 (C4), 127.4 (C5), 128.7 (C8), 128.8 (C9), 129.0 (C9a), 130.7 (C6), 134.6 (C5a), 139.0 (C9b), 141.9 (C1), 143.0 (C3a); high resolution mass spectrum (HRMS), calcd for $C_{28}H_{22}Br_2$ 516.0088, found: 516.0080 (M^+).

From the more polar fraction, (±)-*cis*-dimethyl olefin (2*R**,2'*R**)-(*P**,*P**)-(*Z*)-(±)-**6b** was obtained as yellow solid (10.1 mg, 7%) which was recrystallized from AcOEt giving yellow prisms: mp 225-226 ºC; IR (film on KBr) ν_{max} 3042, 2961, 2925, 2860, 1730, 1577, 1504, 1455, 1345, 1188, 1073, 912, 883, 821, 801, 767, 737, 705, 675, 664, 642 cm^{-1}; ^{1}H NMR (600 MHz, CD_2Cl_2) δ 1.20 (6H, d, *J* = 6.8 Hz, Me2ax), 2.68 (2H, d, *J* = 15.1 Hz, H3eq), 3.55 (2H, dd, *J* = 15.1, 6.8 Hz, H3ax), 3.62 (2H, dq, *J* = 6.8, 6.8 Hz,

H2eq), 6.42 (2H, d, *J* = 8.9 Hz, H9), 6.45 (2H, dd, *J* = 8.9, 2.0 Hz, H8), 7.54 (2H, d, *J* = 8.2 Hz, H4), 7.65 (2H, d, *J* = 8.2 Hz, H5), 7.86 (2 H, d, *J* = 2.0 Hz, H6); ^{13}C NMR (150 MHz, CD_2Cl_2) δ 20.7 (Me2ax), 40.6 (C3), 42.2 (C2), 123.4 (C4), 124.0 (C7 and C8), 126.6 (C9), 127.6 (C6), 128.4 (C5), 129.8 (C9a), 132.2 (C5a), 136.9 (C9b), 139.9 (C1), 144.0 (C3a); high resolution mass spectrum (HRMS), calcd for $C_{28}H_{22}Br_2$ 516.0088, found: 516.0082 (M^+).

(2*R,2'*R**)-(*P**,*P**)-(*E*)-(±) -2,2'-Dimethyl-2,2',3,3'-tetrahydro-1,1'-bicyclopenta[*a*] - 9,9'-diaminonaphthalenylidene (7a)**

A mixture of *trans*-dimethyl olefine (2*R**,2'*R**)-(*P**,*P**)-(*E*)-(±)-**21** (39.4 mg, 0.0760 mmol), NaO^{t}Bu (21.9 mg, 0.228 mmol), $Pd_2(dba)_3$ (7.0 mg, 0.00760 mmol), (±)-BINAP (14.2 mg, 0.0228 mmol), and benzophenone imine (34.4 mg, 0.190 mmol) in toluene (0.5 ml) was heated at 80 ºC for 9 h. After being cooled at room temperature, the reaction mixture was filtrated with Celite, and evaporated to dryness. The crude product was purified by a short column chromatography on silica gel (hexane/AcOEt = 5:1) to yield the imine adduct. The imine adduct was dissolved in THF (3 ml), added aqueous 2.0 M HCl (0.15 ml), and stirred at room temperature for 2 h. The reaction mixture was partitioned between 1.0 M HCl (20 ml) and 2:1 hexane/AcOEt (20 ml). The aqueous layer was separated, made alkaline, extracted with AcOEt (3 x 20 mL). The combined organic layers were dried with anhydrous Na_2SO_4 and evaporated to dryness. The crude product was purified by HPLC (silica gel, hexane/AcOEt = 2:1) to yield (2*R**,2'*R**)-(*P**,*P**)-(*E*)-(±)-**7a** as yellow solid (18.6 mg, 63%), which was recrystallized from hexane/AcOEt giving yellow prisms: mp 186-188 ºC; IR (film on KBr) ν_{max} 2969, 2952, 2925, 2861, 2845, 2225, 1718, 1619, 1498, 1451, 1430, 1367, 1349, 1260, 1157, 1072, 940, 893, 831, 808, 766, 714, 704, 678, 627, 611 cm^{-1}; ^{1}H NMR (600 MHz, CD_3OD) δ 1.24 (6H, d, *J* = 6.4 Hz, Me2ax), 2.26 (2H, d, *J* = 14.3 Hz, H3eq), 2.83 (2H, dd, *J* = 14.3, 6.4 Hz, H3ax), 2.98 (2H, dq, *J* = 6.4, 6.4 Hz, H2eq), 7.08 (2H, dd, *J* = 8.8, 2.0 Hz, H8), 7.11 (2H, d, *J* = 2.0 Hz, H6), 7.24 (2H, d, *J* = 8.2 Hz, H4), 7.48 (2H, d, *J* = 8.2 Hz, H5), 7.99 (2H, d, *J* = 8.8 Hz, H9); ^{13}C NMR (150 MHz, CD_3OD) δ 19.7, 41.7, 44.1, 111.0, 119.0, 125.1, 126.1, 126.9, 128.6, 136.0, 139.0, 139.4, 142.6, 144.9; mass spectrum (FABMS), 391 ($[M+1]^+$).

(±)-**7b**: yellow solid: ^{1}H NMR (400 MHz, CD_3OD) 1.17 (6H, d, *J* = 6.8 Hz, Me2ax), 2.60 (2H, d, *J* = 15.6 Hz, H3eq), 3.47 (2H, m, H3ax), 3.56 (2H, m, H2eq), 6.01 (2H , dd, *J* = 8.8, 2.0 Hz), 6.51 (2H, d, *J* = 8.8 Hz), 6.86 (2H, d, *J* = 2.9 Hz), 7.35 (2H, d, *J* = 8.8 Hz), 7.45 (2H, d, *J* = 8.8 Hz); ^{13}C NMR (100 MHz, CD_3OD) 21.13, 41.46, 43.84, 110.7, 114.1, 118.3, 124.7, 125.9, 127.8, 128.7, 135.5, 138.1, 141.3, 141.8.

(2*R,2'*R**)-(*P**,*P**)-(*E*)-(±) -2,2'-Dimethyl-2,2',3,3'-tetrahydro-1,1'-bicyclopenta[*a*] - 9,9'-diazidenaphthalenylidene (8a)**

trans-Diamino olefine (2*R**,2'*R**)-(*P**,*P**)-(*E*)-(±)-**7a** (7.4 mg, 0.0189 mmol) was dissolved in DMF (0.5 ml) and cooled to 0 ºC. To this stirred mixture was added ^{t}BuONO (11.1 ml, 0.0947 mmol) followed by $TMSN_3$ (9.9 ml, 0.0756 mmol) dropwise. The solution was stirred at room temperature for 15 h. The reaction mixture was purified without concentration by a short column chromatography on silica gel (hexane/AcOEt = 30:1) and further purified by HPLC (silica gel, hexane/AcOEt = 40:1) to yield (2*R**,2'*R**)-(*P**,*P**)-(*E*)-(±)-**8a** as yellow solid (7.5 mg, 89%), which was recrystallized from hexane/AcOEt giving yellow prisms: IR (film on KBr) ν_{max} 2965, 2927, 2109, 1624, 1587, 1512, 1464, 1349, 1286, 1197, 1117, 952, 864, 800, 778, 709 cm^{-1}; ^{1}H NMR (600 MHz, CD_2Cl_2) 1.28 (6H, d, *J* = 6.4 Hz, Me2ax), 2.36 (2H, d, *J* = 14.5 Hz, H3eq), 2.94 (2H, dd, *J* = 14.5, 6.4 Hz, H3ax), 3.00 (2H, dq, *J* = 6.4, 6.4 Hz, H2eq), 7.25 (2H, dd, *J* = 8.8, 2.3 Hz, H8), 7.43 (2H , d, *J* = 8.2 Hz, H4), 7.54 (2H , d, *J* = 2.3 Hz, H6), 7.69 (2H, d, *J* = 8.2 Hz, H5), 8.23 (2H, d, *J* = 8.8 Hz, H9)

(2*R,2'*R**)-(*P**,*P**)-(*Z*)-(±) -2,2'-Dimethyl-2,2',3,3'-tetrahydro-1,1'-bicyclopenta[*a*] - 9,9'-diazidenaphthalenylidene (8b)**

^{1}H NMR (400 MHz, CD_3OD) δ 1.17 (6H, d, *J* = 6.8 Hz, Me2ax), 2.60 (2H, d, *J* = 15.6 Hz, H3eq), 3.47 (2H, m, H3ax), 3.56 (2H, m, H2eq), 6.01 (2H, dd, *J* = 8.8, 2.0 Hz), 6.51 (2H, d, *J* = 8.8 Hz), 6.86 (2H, d, *J* = 2.9 Hz), 7.35 (2H, d, *J* = 8.8 Hz), 7.45 (2H, d, *J* = 8.8 Hz)

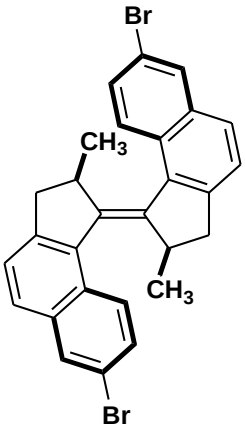
Peptide conjugated molecular motor (11a)

To the solution of 7-trans (1.9 mg, 4.3 μM) and PRGRP (11.8 mg, 17.1 μmol) or KAKAK peptide (10 mg, 14.0 μmol) in DMF (200 μL), sodium ascorbate (8.7 mg, 43 μmol) in water (20 μL) and $CuSO_4 \cdot 5H_2O$ (10.7 mg, 43 μmol) in water (20 μL) were added. After stirring for 19 h, the reaction mixture was removed microtube to centrifuge (10000 rpm, 5 min) and followed by filtered the solution. That solution was purified by HPLC to yield compound **11a** (2.7%), **12a** (2.0 %).

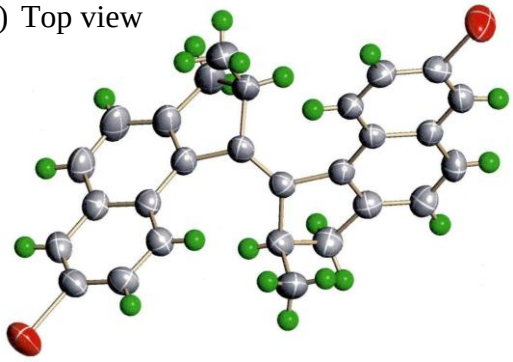
MALDI-TOF-MS (positive mode): (±)-**11a**: calcd:1792.2[M-H], found:1792.9[M-H]

(±)-**12a**: calcd:1633.9[M-H], found:1634.0[M-H]

Fig.1S X-ray stereo view of (2*R,2'*R**)-(*P**,*P**)-(±) –trans-(6a)**



(a) Top view



(b) Side view

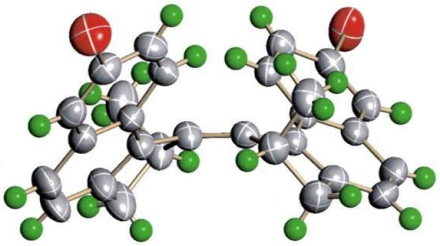
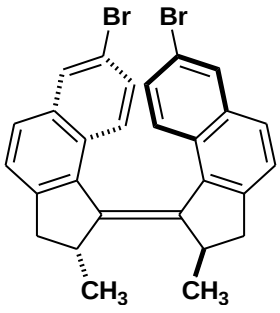
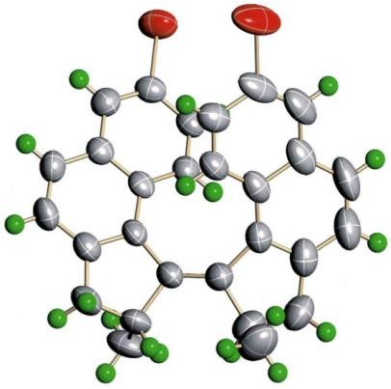


Fig.2S X-ray stereo view of (2*R,2'*R**)-(*P**,*P**)-(±) –cis-(6b)**



(a) Top view



(b) Side view

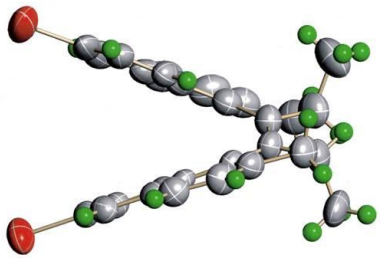
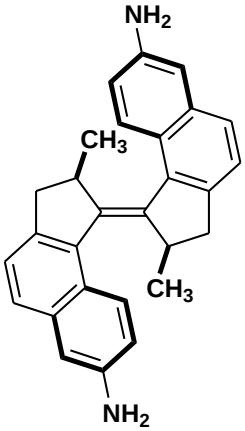
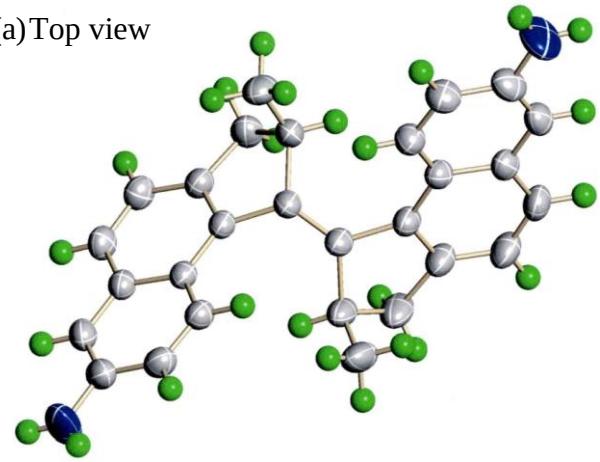


Fig 3S. X-ray stereo view of (2*R, 2'*R**)-(*P**,*P**)-(±) –trans-(7a)**



(a) Top view



(b) Side view

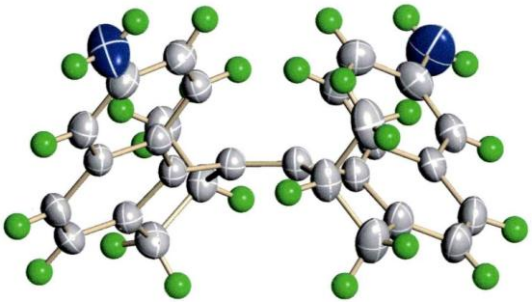


Table S1. Crystal data and structure refinement for **6a**.

Empirical formula	C28 H22 Br2	
Formula weight	518.28	
Temperature	293 K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.8972(16) Å	α= 90°.
	b = 13.900(2) Å	β= 91.130(13)°.
	c = 17.980(3) Å	γ = 90°.
Volume	2223.2(6) $Å^3$	
Z	4	
Density (calculated)	1.548 Mg/m^3	
Absorption coefficient	4.702 mm^{-1}	
F(000)	1040	
Crystal size	0.21 x 0.21 x .018 mm^3	
Theta range for data collection	4.02 to 67.99°.	
Index ranges	-10<=h<=10, 0<=k<=16, 0<=l<=21	
Reflections collected	4186	
Independent reflections	4050 [R(int) = 0.0257]	
Completeness to theta = 67.99°	100.0 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4050 / 0 / 359	
Goodness-of-fit on F^2	1.062	
Final R indices [I>2sigma(I)]	R1 = 0.0420, wR2 = 0.1142	
R indices (all data)	R1 = 0.0502, wR2 = 0.1200	
Largest diff. peak and hole	0.614 and -0.753 e.$Å^{-3}$	

Table S2. Crystal data and structure refinement for **6b**.

Empirical formula	C28 H22 Br2	
Formula weight	518.28	
Temperature	293 K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.3211(15) Å	α= 90°.
	b = 15.332(3) Å	β= 97.666(17)°.
	c = 18.052(4) Å	γ = 90°.
Volume	2282.4(8) $Å^3$	
Z	4	
Density (calculated)	1.508 Mg/m^3	
Absorption coefficient	4.580 mm^{-1}	
F(000)	1040	
Crystal size	0.23 x 0.12 x 0.11 mm^3	
Theta range for data collection	3.80 to 68.00°.	
Index ranges	-10<=h<=9, 0<=k<=18, 0<=l<=21	
Reflections collected	4280	
Independent reflections	4144 [R(int) = 0.0372]	
Completeness to theta = 68.00°	100.0 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4144 / 0 / 273	
Goodness-of-fit on F^2	1.046	
Final R indices [I>2sigma(I)]	R1 = 0.0662, wR2 = 0.1820	
R indices (all data)	R1 = 0.0889, wR2 = 0.2069	
Largest diff. peak and hole	1.357 and -1.208 $e.Å^{-3}$	

Table S3. Crystal data and structure refinement for **7a**.

Empirical formula	C28 H26 N2	
Formula weight	390.51	
Temperature	293 K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.4916(15) Å	α= 90°.
	b = 13.520(3) Å	β= 93.152(15)°.
	c = 18.343(3) Å	γ = 90°.
Volume	2102.8(7) $Å^3$	
Z	4	
Density (calculated)	1.234 Mg/m^3	
Absorption coefficient	0.548 mm^{-1}	
F(000)	832	
Crystal size	0.25 x 0.21 x 0.17 mm^3	
Theta range for data collection	4.06 to 67.97°.	
Index ranges	-10<=h<=10, -16<=k<=0, 0<=l<=22	
Reflections collected	4151	
Independent reflections	3828 [R(int) = 0.0223]	
Completeness to theta = 67.97°	100.0 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3828 / 0 / 367	
Goodness-of-fit on F^2	1.076	
Final R indices [I>2sigma(I)]	R1 = 0.0463, wR2 = 0.1305	
R indices (all data)	R1 = 0.0553, wR2 = 0.1368	
Largest diff. peak and hole	0.174 and -0.248 e.$Å^{-3}$	