Supporting Information

“Helical Conformational Dynamics and Photoisomerism of Alternating Pyridinedicarboxamide / m-(Phenylazo)azobenzene Oligomers.”

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Part A 1D- selective TOCSY NMR spectra

Experimental Data S2-S11

NMR Peak Assignment S12

1D- selective TOCSY NMR spectra of 7d S13

1D- selective TOCSY NMR spectra of 7e S14

1D- selective TOCSY NMR spectra of 8 S15

1D- selective TOCSY NMR spectra of 9 (part1) S16

1D- selective TOCSY NMR spectra of 9 (Part 2) S17

1D- selective TOCSY NMR spectra of 9 (All) S18

NOESY NMR Spectrum of 9 in DMSO-d6 S19

Part B Comparison of UV spectra before and after irradiation

5a, 6 S20

7a, 7b, 7d S21

7e, 8, 9 S22

Part C The temperature dependent $^1$HNMR spectra of oligomer 8 S23

Part D The $^1$H-$^1$HNMR 2D COESY spectra of oligomer 8 and 9 S23

Temperature-dependent rates of thermal $Z \rightarrow E$ isomerization of 7-9. S24

Part E Crystallographic information file of 8 S25-48
Experimental Section.

**General.** $^1$H and $^{13}$C-NMR spectra were collected on a Bruker DRX-400 or DRX-500 spectrometers. MS analysis was performed by electrospray or MALDI-TOF mass spectrometry as indicated by The Ohio State University Campus Chemical Instrumentation Center. All starting materials were from commercial suppliers and used as received unless otherwise noted. Tetrahydrofuran (THF) was distilled from sodium/benzophenone; CH$_2$Cl$_2$ and pyridine was distilled from calcium hydride. All melting points are recorded in glass capillaries and are uncorrected. Thin-layer chromatography was performed on aluminum-backed Merck Silica gel 60 F254 plates. Separation by flash chromatography were performed with Merck silica gel 9385 (100 mesh).

**2-Nitroacetanilide.** $^1$ 2-Nitroaniline 1 (41 g, 0.3 mol, 1.0 equiv) and acetic anhydride (32 mL, 33 g, 0.33 mol, 1.1 equiv) were mixed in 150 mL acetic acid and heated at reflux for 4 h. After cooling the mixture to rt, water (1 L) was added and the mixture was filtered. The filtrate was extracted with CHCl$_3$ (500 mL), providing 6.3 g of 2 as a yellow solid after drying over Na$_2$SO$_4$ and concentrating. The precipitate was dissolved in CHCl$_3$ (500 mL), washed with water (500 mL), dried (Na$_2$SO$_4$) and concentrated to yellow solid (47.7 g). The combined products weighed 53.7 g (yield 99 %) and was used without further purification. Mp 84-88 °C (CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 10.33 (bs, 1 H), 8.78 (d, $J = 8.4$ Hz, 1H), 8.22 (d, $J = 8.4$ Hz, 1H), 7.66 (t, $J = 8.0$ Hz, 1H), 7.19 (t, $J = 8.0$ Hz, 1H), 2.31 (s, 3H).


\[ \text{S3} \]

\[^{13}\text{C NMR (100MHz, CDCl}_3\] \( \delta \) 169.0, 136.3, 136.0, 134.9, 125.7, 123.2, 122.2, 25.6 ppm. IR (CHCl\(_3\)) \( \nu \) 3369, 1706, 1521, 1499 cm\(^{-1}\).

2-Nitrosoacetanilide (2). 2-Nitroacetanilide (6.22 g, 34.6 mmol, 1.0 equiv) and 250 mg Rh/C were combined in 36 mL of THF. The solution was cooled to 0 °C and hydrazine (1.19 mL, 1.22 g, 38.0 mmol, 1.1 eq.) was added over 3 h by syringe pump. After addition, the catalyst was removed by filtration through celite and the filtrate was evaporated in vacuo. The resultant yellow solid was dissolved in 1.8 L of THF. After cooling to -78 °C, \( \tau \)-butyl hypochlorite (4.53 mL, 4.12 g, 38.0 mmol, 1.1 eq.) was added in one portion. The mixture was stirred for 1 h in the dark, then was concentrated. The resulting residue was purified by column chromatography (1:5 EtOAc/Hex) affording nitroso 2 as a green solid (4.97 g, 88 %). Mp 106-107 °C (EtOAc/Hex). \(^{1}\text{H NMR (400 MHz, CDCl}_3\] \( \delta \) 10.79 (bs, 1H), 8.84 (d, \( J \) = 8.8 Hz, 1H), 7.69 (m, 1H), 7.30 (bs, 1H), 7.15 (t, \( J \) = 7.6 Hz, 1H), 2.35 (s, 3H). \(^{13}\text{C NMR (100 MHz, CDCl}_3\] \( \delta \) 169.5, 156.0, 139.1, 135.7, 122.6, 121.0, 118.6, 25.4 ppm. IR (CHCl\(_3\)) \( \nu \) 3396, 1705, 1482 cm\(^{-1}\).

2-(3-Aminophenyl)isoindole-1,3-dione (4c).

Part A. 2-(3-Nitrophenyl)isoindole-1,3-dione (4c). A mixture of 3-nitroaniline (4a) (20 g, 145 mmol, 1.0 equiv), phthalic anhydride (21.4 g, 145 mmol, 1.0 equiv) and dry triethylamine (1.5 mL, 10 mmol, 0.068 eq.) in 600 mL toluene was heated at reflux in a flask equipped with a Dean-Stark apparatus. After 5h, the mixture was cooled to rt and concentrated on a rotary evaporator. The resulting residue was partially dissolved in hot ethanol. After 12 h, the suspension was filtered, providing 2-(3-nitrophenyl)isoindole-1,3-dione as a pale white
solid (32 g, yield 82 %). Mp 237-240 °C (EtOH). \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 8.46 (t, \(J = 1.9\) Hz, 1H), 8.30 (m, 1H), 8.02 (m, 2H), 7.92- 7.86 (m, 3H), 7.72 (t, \(J = 8.2\) Hz, 1H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 166.51, 148.58, 134.93, 132.96, 132.00, 131.39, 129.88, 124.16, 122.55, 121.45 ppm. IR (CHCl\(_3\)) \(\nu\) 1793, 1728 cm\(^{-1}\). HRMS (ES) \(m/z [\text{Na}]^+\) 291.0374 (calcd.

**Part B. 2-(3-Aminophenyl)isoindoline-1,3-dione (4c).**

2-(3-nitrophenyl)isoindoline-1,3-dione (5 g, 18.6 mmol) and tin chloride dihydrate (29.4 g, 130 mmol) was mixed in 100 mL EtOAc/MeOH (9:1). The reaction mixture was heated at reflux for 2 h, cooled to room temperature and the pH was adjusted to 7.0 with sat. aq. sodium bicarbonate. The mixture was filtered and the resulant filter cake was washed with ethyl acetate (2 x 50 ml). The ethyl acetate extracts were evaporated and the resultant residue was dissolved in hot 20% aqueous ethanol solution. After crystallization overnight, filtration provided 4c (15.2 g, 47 %) as a pale white solid. Mp 178-179 °C (H\(_2\)O/EtOH). \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.96 (dd, \(J = 3.0, 5.3\) Hz, 2H), 7.80 (dd, \(J = 3.0, 5.3\) Hz, 2H), 7.28 (m, 1H), 6.83 (d, \(J = 7.6\) Hz, 1H), 6.74 (t, \(J = 8.0\) Hz, 2H), 3.98 (s, 2H). \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 167.30, 147.18, 134.29, 132.56, 132.83, 129.85, 123.68, 116.63, 114.94, 113.20 ppm. IR (CHCl\(_3\)) \(\nu\) 3410, 1722, 1621 cm\(^{-1}\). HRMS (ES) \(m/z [\text{Na}]^+\) 261.0622 (calcd. for C\(_{14}\)H\(_{10}\)N\(_2\)O\(_2\)Na: 261.0634).

**N-[2-(3-Aminophenylazo)phenyl]acetamide (6).** 2-Nitrosoacetanilide (2b) (2.5 g, 15 mmol, 1.0 eq.) and 4.0 g 4c (17 mmol, 1.1 equiv) was dissolved in 50 mL ethanol and 20 mL glacial acetic acid was added to the resulting solution. The reaction mixture was heated at 80
°C for 12 h then cooled to room temperature and filtered. The filter cake was washed sequentially with acetic acid (10 mL) and water (50 mL) then dissolved in a mixture of THF (100 mL), methanol (100 mL) and 40% aq. methylamine (50 mL). The reaction mixture was stirred for 10 h at room temperature and concentrated on rotovap. Chromatography (Ethyl acetate/hexane =1/5) provided 6 as an orange solid (2.4 g, yield 68%). Mp 119-120 °C. 

$^1$H NMR (400 MHz, CDCl$_3$) δ 10.23 (bs, 1H), 8.69 (d, $J$ = 8.4 Hz, 1H), 7.85 (dd, $J$ = 1.6 Hz, 8.0 Hz, 1H), 7.47 (td, $J$ = 1.6 Hz, 8.0 Hz, 1H), 7.35 (t, $J$ = 8.0 Hz, 1H), 7.30 (t, $J$ = 1.0 Hz, 1H), 7.18 (m, 2H), 6.85 (m, 1H), 3.90 (bs, 2H), 2.29 (s, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.63, 153.47, 147.39, 138.71, 135.61, 132.70, 123.35, 121.87, 120.19, 118.14, 114.15, 107.67, 25.39 ppm. IR (CHCl$_3$) v 3394, 2253, 1690 cm$^{-1}$. HRMS (ES) $m/z$ [Na]$^+$ 277.1055 (calcd. for C$_{14}$H$_{14}$N$_4$ONa, 277.1060).

N-{2-[3-(2-acetylamino-phenylazo)-phenylazo]-phenyl}-acetamide (7a).

2-Nitrosoacetanilide (2b) (0.80 g, 4.87 mmol, 1.05 eq.) and 5c (1.18g, 4.64 mmol, 1.0 equiv) was dissolved in 40 mL glacial acetic acid and heated at reflux. After 6 h, the mixture was cooled to room temperature and CHCl$_3$ (400 mL) was added. The solution was then washed with water (4×400 mL) and dried (Na$_2$SO$_4$) and concentrated. Chromatography (CH$_2$Cl$_2$/EtOAc = 50/1) provided 7a (1.00 g, 54 %) as an orange solid. Mp 210-211 °C (CH$_2$Cl$_2$/EtOAc). $^1$H NMR (400 MHz, CDCl$_3$) δ 10.12 (bs, 2H), 8.71 (d, $J$ = 8.4 Hz, 2H), 8.33 (s, 1H), 7.99 (d, $J$ = 8.0 Hz, 2H), 7.95 (d, $J$ = 7.6 Hz, 2H), 7.73 (t, $J$ = 8.0 Hz, 1H), 7.52 (t, $J$ = 8.0 Hz, 2H), 7.22 (t, $J$ = 7.6 Hz, 2H), 2.32 (s, 6H). $^{13}$C NMR (100 MHz, CDCl$_3$) δ 168.6, 153.2, 138.7, 136.1, 133.6, 130.0, 124.5, 123.5, 121.6, 120.4, 117.2, 25.4 ppm. IR
(CHCl₃) ν 2259, 1698 cm⁻¹. HRMS (ES) m/z [Na]⁺ 423.1528 (calcd. for C₂₂H₂₀N₆O₂Na, 423.1540).

2-[3-(2-amino-phenylazo)-phenylazo] aniline (7b). (1.00 g, 2.50 mmol, 1.0 eq.) 7a was dissolved in 80 mL of ethanol/H₂O (1:1) and treated with KOH (1.8 g, 32.14 mmol). After heating at reflux for 10 h, the mixture was cooled to room temperature and evaporated in vacuo. The resultant residue was extracted with CH₂Cl₂ (3×200 mL) and the organic extracts were dried (Na₂SO₄) and concentrated. Purification with chromatography (EtOAc/hexane =1/5) afforded 7b (0.75 g, 80 %) as a red solid. Mp 124-125 ºC (EtOAc/hexane). ¹H NMR (400 MHz, CDCl₃) δ 8.32 (t, J = 1.6 Hz 1H), 7.92 (td, J = 1.6, 8.0 Hz, 4H), 7.63 (t, J = 8.0 Hz, 1H), 7.27 (td, J = 1.6, 8.0 Hz, 2H), 6.86 (td, J = 1.6, 8.0, 2H), 6.86 (d, J = 8.0, 2H), 6.15 (bs, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 152.68, 141.89, 135.92, 131.51, 128.50, 127.23, 122.41, 116.38, 116.02, 114.57 ppm. IR (CHCl₃) ν 3487, 1614, 1577 cm⁻¹. MS (EI) m/z⁺ 316.1421 (calcd. for C₁₈H₁₆N₆, 316.1431).

Benzyl 2-[3-(2-amino-phenylazo)-phenylazo]-phenylcarbamate (7c). Diamine 7b (330 mg, 1.0 mmol, 1.0 eq.) was dissolved in CH₂Cl₂ (15 mL) and treated with 1 M aq. NaOH (15 mL). The mixture was cooled to 0 ºC and benzylchloroformate (0.16 mL, 1.1 mmol, 1.1 eq.) was added dropwise over 30 min. After 12 h, the organic layer was separated and washed with water (50 mL) and brine (50 mL), then dried (Na₂SO₄) and concentrated. Chromatography (EtOAc/hexane =1/8) provided 7c (320 mg, yield 68 %) as a red solid. Mp 111-113 ºC (dec). ¹H NMR (400 MHz, CDCl₃) δ 9.50 (bs, 1H), 8.50 (d, J = 8.4 Hz, 1H), 8.30 (s, 1H), 7.98-7.87 (m, 4H), 7.64 (t, J= 7.6 Hz, 1H), 7.55-7.40 (m, 6H), 7.27 (t, J= 7.6
Hz, 1H), 7.16 (t, J= 7.6 Hz, 1H), 6.86 (t, J= 7.6 Hz, 1H), 6.77 (d, J= 7.6 Hz, 1H), 5.93 (bs, 2H), 5.32 (s, 2H) \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) 153.61, 153.03, 143.13, 138.72, 136.84, 136.49, 136.05, 133.09, 132.69, 129.62, 128.60, 128.34, 128.32, 127.74, 125.03, 124.05, 122.55, 120.19, 118.80, 117.36, 116.99, 115.41, 67.06 ppm. IR (CHCl\(_3\)) \(\nu\) 3485, 3399, 1732, 1596, cm\(^{-1}\). HRMS(ES) \(m/z\) [Na]+ 473.1687 (calcld for C\(_{26}\)H\(_{22}\)N\(_6\)O\(_2\)Na\(^+\), 473.1696).

**Benzyl 2-[3-(2-benzyloxycarbonylamino-phenylazo)-phenylazo]-phenylcarbamate (7d).** Diamine 7b (250 mg, 0.79 mmol, 1.0 eq.) was dissolved in CH\(_2\)Cl\(_2\) (15 mL) and treated with 1 M aq. NaOH (15 mL). The mixture was cooled to 0 °C and benzylchloroformate (0.25 mL, 1.74 mmol, 2.2 eq.) was added dropwise over 30 min. After 12 h, the organic layer was separated and washed with water (50 mL) and brine (50 mL), then dried (Na\(_2\)SO\(_4\)) and concentrated. Chromatography (EtOAc/hexane =1/8) provided 7d (350 mg, yield 76 %) as an orange solid. Mp 146-147 °C (CH\(_2\)Cl\(_2\)). \(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 9.48 (bs, 2H), 8.48 (d, J = 8.0 Hz, 2H), 8.35 (t, J = 1.5 Hz, 1H), 7.97 (dd, J = 1.5, 8.0 Hz, 2H), 7.88 (dd, J = 1.0, 8.0 Hz, 2H), 7.68 (t, J = 8.0 Hz, 1H), 7.53 (td, J = 1.5, 8.5 Hz, 2H), 7.47 (d, J = 7.0 Hz, 4H), 7.42-7.36 (m, 6H), 7.16 (td, J = 1.5, 8.0 Hz, 2H), 5.29 (s, 4H). \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) \(\delta\) 153.19, 153.09, 138.83, 136.54, 136.03, 133.34, 129.86, 128.61, 128.37, 128.31, 124.48, 122.63, 120.59, 118.94, 117.73, 67.12. IR (CHCl\(_3\)) \(\nu\) 3055, 1735,1596 cm\(^{-1}\). HRMS (ES) \(m/z\) [Na]+ 607.2051 (calcld for C\(_{34}\)H\(_{28}\)N\(_6\)O\(_4\)Na\(^+\), 607.2064).

**N-{2-[3-(2-picolinoylamino-phenylazo)-phenylazo]-phenyl}-picolinamide (7e).** Picolinic acid (130 mg, 1.0 mmol, 3.0 equiv) was dissolved in 3.0 ml SOCl\(_2\). After heated at reflux for 2 h, the solvent was removed by evaporation with dry benzene (3 x10 ml). To this
residue 108 mg 7b (0.34 mmol, 1.0 equiv), 5 mL dichloromethane and 2 mL pyridine were added. After stirring for 10 h, the mixture was concentrated and chromatography (CH$_2$Cl$_2$/ethyl acetate = 50/1) provided 7e (113 mg, yield 63%) as an orange solid. Mp 220-221 ºC (CH$_2$Cl$_2$). $^1$H NMR (500 MHz, CDCl$_3$) δ 12.51 (bs, 2H), 8.92 (m, 3H), 8.49 (d, $J$ = 4.6 Hz, 2H), 8.31 (dd, $J$ = 1.5, 7.8 Hz, 2H), 8.12 (d, $J$ = 7.8 Hz, 2H), 8.04 (d, $J$ = 7.5 Hz, 2H), 7.85-7.75 (m, 3H), 7.63 (t, $J$ = 7.8 Hz, 2H), 7.3 (m, 4H). $^{13}$C NMR (125 MHz, CDCl$_3$) δ 162.20, 153.70, 149.74, 148.18, 139.78, 137.41, 136.74, 133.45, 127.41, 126.27, 123.64, 122.13, 120.23, 118.96. IR (CHCl$_3$) ν 3691, 3306, 1685, 1594, 1520, 1465, 1431, 1311, 1264, 1155, 1098, 998, 726, 687 cm$^{-1}$. HRMS (ES) m/z [Na$^+$] 549.1777 (calcd for C$_{30}$H$_{22}$N$_8$O$_2$Na$^+$, 549.1758).

Bis-{2-[3-(2-benzoxycarbonylamino-phenylazo)-phenylazo]-phenyl}-pyridine-2,6-dicarboxamide (8). Amine 7c (320 mg, 0.71 mmol, 1.0 eq.) was dissolved in a mixture of CH$_2$Cl$_2$ (5 mL) and pyridine (2 mL), and 4 Å molecular sieves (300 mg) were added to the mixture. After stirring for 30 min., pyridine-2,6-dicarbonyl dichloride (72 mg, 0.36 mmol, 0.6 equiv) was added. After 12 h at room temperature, the sieves were removed by filtration and the filtrate was diluted with CH$_2$Cl$_2$ (30 mL), washed with 50 mL water and 50 mL brine, then dried (Na$_2$SO$_4$) and concentrated. Chromatography (CH$_2$Cl$_2$) provided 8 (180 mg, 50 %) as a yellow solid. Mp 204-205 ºC (CH$_2$Cl$_2$). $^1$H NMR (500 MHz, CDCl$_3$, see supp. info. for detailed peak assignments) δ 11.74 (bs, 2 H), 9.37 (s, 2 H), 8.51-8.40 (m, 6H), 8.16 (t, $J$ = 7.6 Hz, 1H), 7.99 (s, 2H), 7.78 (d, $J$ = 7.2 Hz, 2H), 7.64 (d, $J$ = 7.6 Hz, 2H), 7.56 (t, $J$ = 7.2 Hz, 2H), 7.39-7.24 (m, 16 H), 7.20 (t, $J$ = 7.6 Hz, 2H), 7.17 (t, $J$ = 7.6 Hz, 2H), 6.95 (t, $J$ = 7.6
Hz, 2H), 5.16 (s, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 160.75, 153.02, 152.83, 152.22, 149.68, 138.96, 138.37, 136.47, 135.86, 133.40, 133.05, 129.26, 128.61, 128.36, 128.18, 125.83, 125.44, 124.48, 123.31, 122.63, 120.90, 119.92, 118.68, 117.23, 114.37, 67.03 ppm.

IR (CHCl$_3$) $\nu$ 3347, 1732, 1688 cm$^{-1}$. HRMS (ES) $m/z$ [Na]$^+$ 1054.3522 (calcd. for C$_{59}$H$_{45}$N$_{13}$O$_6$Na, 1054.3508).

**Benzyl-2-{3-[2-{N2-[3-(2-benzyloxy carbonylamino-phenylazo)-phenylazo]-phenyl-pyridine-2,6-dicarboxamido]-phenylazo}--phenylazo}-phenylcarbamate (9)**

**Part A. Bis-{2-[3-(2-aminophenylazo)-phenylazo]-phenyl}-pyridine-2,6-dicarboxamide (8a).** Oligomer 8 (110 mg, 0.1 mmol, 1.0 eq.) was dissolved in thioanisole (0.66 g, 0.63 mL, 5.33 mmol, 50 eq.) and trifluoroacetic acid (3.28 g, 2.2 mL, 28 mmol, 270 eq.) was added. After stirring at room temperature for 10 h, ethyl acetate (50 mL) was added and the mixture was washed with water (50 mL), dried (MgSO$_4$) and concentrated. Purification by column chromatography (EtOAc/hexane =1/2) provided diamine 8a (77 mg, 95%) as a red solid. Mp: 133-135 °C (dec); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 11.73 (s, 2H), 8.55 ($d, \ J = 8.0 \ Hz, \ 2H$), 8.42 ($d, \ J = 8.0 \ Hz, \ 2H$), 8.06 ($t, \ J = 7.6 \ Hz, \ 1H$), 7.90 (s, 2H), 7.67 ($d, \ J = 8.0 \ Hz, \ 2H$), 7.45 ($d, \ J = 7.6 \ Hz, \ 2H$), 7.26 (m, 8H), 6.95 (m, 4H) 6.78 (m, 4H), 5.83 (bs, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 160.85, 152.64, 152.62, 149.46, 142.88, 139.19, 138.98, 136.46, 135.80, 132.48, 132.38, 128.72, 128.33, 125.33, 123.97, 123.23, 122.97, 119.85, 117.96, 116.99, 116.88, 115.26 ppm. IR (CHCl$_3$) $\nu$ 3687, 1601 cm$^{-1}$. HRMS (ES) $m/z$ [Na]$^+$ 786.2740 (calcd. for C$_{44}$H$_{33}$N$_{13}$O$_2$Na, 786.2772).
Part B.

**Bis-2-{3-[2-{N2-[3-(2-aminophenylazo)-phenylazo]phenylpyridine-2,6-dicarboxamido}-phenylazo]phenylazo}phenylpyridine-2,6-dicarboxamide (8b)** Diamine 8a (77 mg, 0.1 mmol, 1.0 eq.) was dissolved in CH$_2$Cl$_2$ (2 mL) and treated with 1 M aqueous NaOH (2 mL). The mixture was cooled to 0 °C and benzylchloroformate (0.016 mL, 0.11 mmol, 1.1 eq.) was added dropwise over 30 min. After stirring for 12 h, the organic layer was separated and washed with water (50 mL) and brine (50 mL), then dried (MgSO$_4$) and concentrated. Chromatography (EtOAc/hexane =1/5) provided 8b (84 mg, 93 %) as a red solid. Mp: 120-121 °C (CH$_2$Cl$_2$); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 11.73 (d, $J = 17.2$ Hz, 2H), 9.37 (s, 1H), 8.61 (d, $J= 17.6$ Hz, 1H), 8.56-8.38 (m, 4H), 8.11 (t, $J = 7.2$ Hz, 1H), 7.94 (d, $J = 14.4$ Hz, 2H), 7.77 (m, 2H), 7.60- 7.41 (m, 3H), 7.39-7.20 (m, 12H), 7.17 (m, 1H), 7.10 (t, $J = 7.6$ Hz, 1H), 7.04-6.88 (m, 3H), 6.82 (dd, $J = 6.4$, 8.8 Hz, 2H), 5.97 (bs, 2H), 5.18 (s, 2H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 160.79, 152.89, 152.74, 152.71, 152.09, 149.57, 142.76, 139.33, 138.98, 138.92, 138.29, 136.57, 136.32, 136.27, 135.39, 135.78, 133.19, 132.88, 132.57, 132.48, 129.06, 128.83, 128.49, 128.22, 128.06, 125.42, 125.30, 124.24, 124.03, 123.35, 123.28, 123.19, 122.49, 120.79, 119.96, 119.76,118.56, 117.86, 117.35, 117.18, 116.99, 115.05, 114.68, 66.90 ppm. IR (CHCl$_3$) $\nu$ 3395, 3336, 1732, 1688, 1597, cm$^{-1}$. HRMS (ES) $m/z$ [Na]$^+$ 920.3125 (calcd. for C$_{51}$H$_{39}$N$_{13}$O$_4$Na, 920.3140).

**Benzyl-2-{3-[2-{N2-[3-(2-benzyloxycarbonylamino-phenylazo)-phenylazo]phenylazo-phenylpyridine-2,6-dicarboxamido}-phenylazo]phenylazo}phenylcarbamate (9).** Amine 8b (77 mg, 0.09 mmol, 1.0 eq.) was dissolved in a mixture of CH$_2$Cl$_2$ (2 mL) and pyridine (1 mL)
and 4 Å molecular sieves (30 mg) were added. After stirring at room temperature for 30 min., pyridine-2,6-dicarbonyl dichloride (10 mg, 0.05 mmol, 0.55 eq.) was added. After 12 h, the sieves were removed by filtration and the resultant filtrate was diluted with CH$_2$Cl$_2$ (30 mL), washed with water (50 mL) and brine (50 mL), then dried (Na$_2$SO$_4$) and concentrated. Chromatography (CH$_2$Cl$_2$) provided 9 (49 mg, 59 %) as a yellow solid. Mp 180-181 ºC (CH$_2$Cl$_2$). $^1$H NMR (500 MHz, CDCl$_3$, see supp. info. for detailed peak assignments) $\delta$ 11.08 (bs, 2H), 10.83 (bs, 2 H), 10.59 (bs, 2 H), 9.15 (bs, 2H), 8.34 (d, $J = 7.8$ Hz, 2H), 8.15 (d, $J = 7.8$ Hz, 2H), 7.85 - 7.75 (m, 9H), 7.69 (d, $J = 6.9$ Hz, 2H), 7.62 (s, 2H), 7.58 (d, $J = 7.9$ Hz, 2H), 7.53 (d, $J = 7.6$ Hz, 2H), 7.48 (t, $J = 7.9$ Hz, 2H), 7.39 (s, 2H), 7.35 (m, 4H), 7.31 (m, 2H), 7.26-7.23 (m, 10H), 7.22-6.98 (m, 24H), 6.84 (t, $J = 7.6$ Hz, 2H), 5.02 (br, 4H). $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 159.08, 158.88, 157.90, 152.81, 152.55, 151.94, 151.87, 148.63, 148.05 147.71, 139.40, 138.46, 138.40, 138.34, 138.05, 136.74, 136.33, 136.22, 135.85, 135.71, 133.21, 133.05, 132.89, 132.56, 131.03, 129.05, 128.80, 128.44, 128.16, 128.05, 127.94, 126.35, 124.91, 124.77, 122.98, 122.40, 120.80, 119.60, 119.12, 118.42, 115.96, 115.46, 114.88, 112.52, 66.74 ppm. IR (CHCl$_3$) 3334, 3155, 1816, 1784, 1731, 1688, 1597 cm$^{-1}$. MALDI-TOF MS for C$_{109}$H$_{79}$N$_{27}$O$_{10}$ calcd. 1926 ; obsd. 1926 .
The example of assigning peaks by choosing various mixing time in 1D selective TOCSY NMR.

Excited peaks were labelled with their chemical shifts.
1D selective TOCSY
NMR spectra of 7d
(500MHz)

Excited peaks were labelled with their chemical shifts.
1D selective TOCSY
NMR spectra of 7e
(500MHz)

Excited peaks were labelled with their chemical shifts.
1D selective TOCSY NMR spectra of 8 (500MHz)

Excited peaks were labelled with their chemical shifts.
Excited peaks were labelled with their chemical shifts.
1D selective TOCSY NMR spectra of 9 (500 MHz) - Part 2

Excited peaks were labelled with their chemical shifts.
1D selective TOCSY NMR spectra of 9 (500 MHz) - all
2D $^1$H-$^1$H NOESY spectrum of 9 in DMSO solution
UV-Vis spectra of compounds before irradiation and at 350 nm for 1 h
$^1$H-NMR lineshape analysis of resonances for CBz methylene (Hd) protons used to determine the interconversion barrier of 8.

The $^1$H-$^1$HNMR 2D COESY spectra of oligomer 8 (left) and 9 (right)
Temperature-dependent rates of thermal $Z \rightarrow E$ isomerization of 7-9.

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Crystallographic details for 8.

The data collection crystal was an orange plate. The data set was measured at 150 K on a Nonius Kappa CCD diffractometer equipped with an Oxford Cryosystems Cryostream Cooler. Phi and omega scans with a frame width of 1.0˚ were used for data collection. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.043. The structure was solved by the direct methods procedure in SHELXS-97(2). Full-matrix least-squares refinements based on F² were performed in SHELXL-97(3), as incorporated in the WinGX package(4). The asymmetric unit consists of the oligomer, 8, and a molecule of CHCl₃.

The hydrogen atoms bonded to the nitrogen atoms were refined isotropically. The remaining hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final difference electron density map contains maximum and minimum peak heights of 0.54 and -0.54 e/Å³. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

References

Table 1. Crystallographic details for compound 8

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Table 2. Atomic coordinates ($x \times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for compound 8. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

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Table 4. Anisotropic displacement parameters (Å² x 10³) for compound 8. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2a^*U_{11} + \ldots + 2hkab^*U_{12}]$

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