Supplementary information

The role of a terminal chain in promoting the twist-bend nematic phase: the synthesis and characterisation of the 1-(4-cyanobiphenyl-4'-yl)-6-(4-alkyloxyanilinebenzylidene-4'-oxy)hexanes

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Section 1: Materials/ General methods/ Instrumentation

All reagents and solvents were available commercially and purchased from Sigma Aldrich, TCI Chemicals or Alfa Aesar and were used as received unless otherwise stated. Anhydrous solvents were purchased as anhydrous (over molecular sieves).

Solvents were evaporated at approximately 20 mm Hg using a water aspirator pump connected to a Buchi rotary evaporator and trace solvents in a Thermo Scientific vacuum oven at 1.0 mm Hg and 50 °C.

Column chromatography, was performing using silica gel grade 60A 40-63 micron, purchased from Flurochem and a small neutral alumina plug was used at the base of the column to remove ionic impurities where stated. Reactions were monitored using Thin Layer Chromatography (TLC) carried out on aluminium-backed plates with a coating of Merck Kieselgel 60 F254 silica and an appropriate solvent system. Silica gel coated aluminium plates were purchased from Merck KGaA. Spots were visualised using UV light (254 nm) or by oxidation with either an aqueous permanganate dip or iodine.

Infrared spectra were recorded on a Thermo Scientific Nicolet IR100 FT-IR spectrometer with an ATR diamond cell.

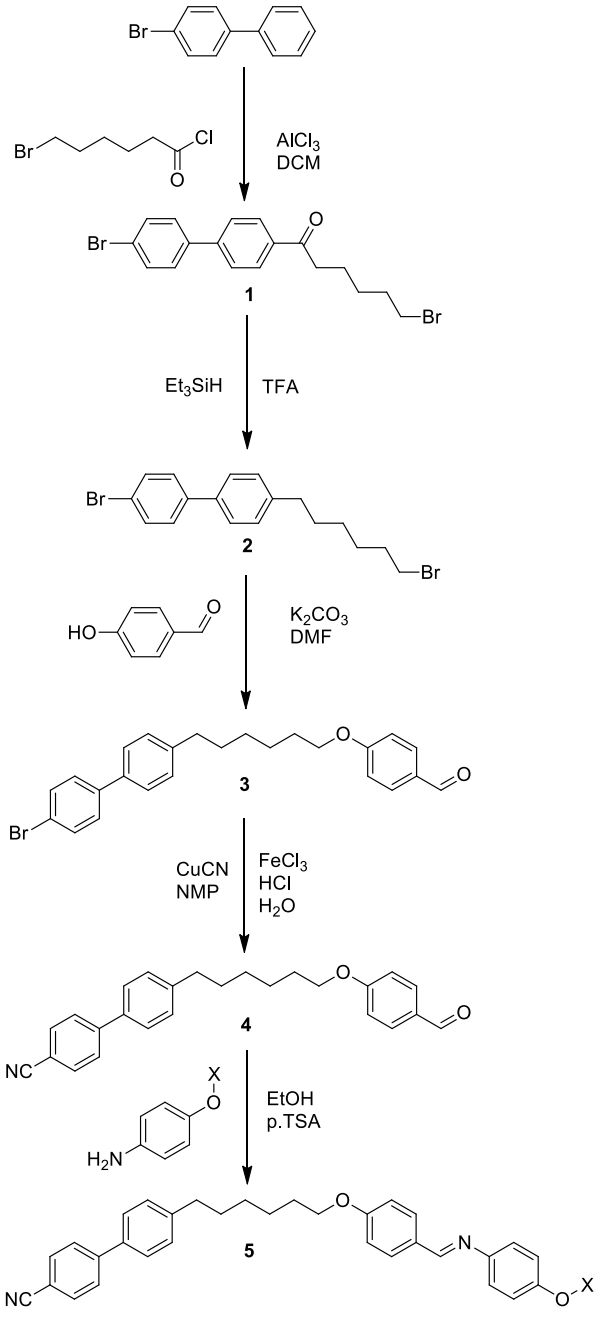
Mass spectra were recorded on a Waters QTOF Xevo G2 spectrometer.

Proton (^{1}H) and carbon (^{13}C) NMR spectra were recorded on a Varian Unity INOVA 400 MHz NMR spectrometer with pulsed field gradients and waveform generator or a 300 MHz Bruker Ultrashield NMR. The chemical shifts δ are quoted in parts per million (ppm) ($SiMe_4$, δ = 0), using residual non-deuterated solvent signals as reference. Coupling constants (J values) are quoted in Hertz (Hz) and are vicinal ^{3}J, unless otherwise indicated. The splitting patterns are reported using the following abbreviations: b (broad), s (singlet), d (doublet), t (triplet), q (quartet), quin (quintet), m (multiplet), and combinations thereof. ^{13}C Spectra are proton decoupled unless otherwise stated. Ar refers to an aromatic ring.

The purity of final products were verified using C,H,N microanalysis performed by the Micro Analytical Laboratory in the School of Chemistry at the University of Manchester or the Centre for Chemical Instrumental Analysis and Services at the University of Sheffield.

Section 2: Synthetic procedures

The synthetic route used to obtain the 1-(4-cyanobiphenyl-4'-yl)-6-(4-alkyloxyaniline-benzylidene-4'-oxy)hexanes, CB6O.O*m*, series is shown in scheme S1. The syntheses of 4-bromo-4'-(6-bromohexanoyl)biphenyl, **1** [1]**,** 1-bromo-6-(4'-bromobiphenyl-4-yl)hexane*,* **2** [1], 4-((6-(4'-bromo-[1,1'-biphenyl]-4-yl)hexyl)oxy)benzaldehyde, **3** [2], and 4'-(6-(4-formylphenoxy)hexyl)-[1,1'-biphenyl]-4-carbonitrile, **4** [2] have been described in detail elsewhere.



Scheme S1. The synthetic route for the CB6O.O*m* series; X=C_mH_{2m+1}.

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The synthesis of 1-(4-cyanobiphenyl-4'-yl)-6-(4-methoxyaniline-benzylidene-4'-oxy)hexane, CB6O.O1 (**5**, X=CH_3), is described in detail. Thus, a mixture of **4** (0.20 g, 0.5 mmol), *p*-anisidine (0.06 g, 0.5 mmol), *p*TSA (a few crystals) and EtOH (30 ml) was heated at reflux overnight. The reaction mixture was cooled to room temperature; the resulting precipitate was collected via vacuum filtration and washed with EtOH (100 ml). The crude product thus obtained was recrystallised from EtOH to give the title compound as a white solid. Yield: 0.24 g, 98%. Elemental analysis: Calculated for $C_{33}H_{32}N_2O_2$: C, 81.12%, H, 6.60%, N, 5.73%. Found: C, 81.19%, H, 6.63%, N, 5.63%. Infrared *v* cm^{-1}: 2925 (C-H), 2852 (C-H), 2223 (C≡N), 1606, 1571, 1510, 1464, 1235, 1163, 842, 831, 803, 546, 535. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, ArCHN), 7.84 (2H, d, *J* 8.6 Hz, Ar), 7.73 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.23 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.95 (2H, d, *J* 8.6 Hz, Ar), 4.04 (2H, t, *J* 6.4 Hz, O$CH_2$$CH_2$), 3.85 (3H, s, O$CH_3$), 2.71 (2H, t, *J* 7.6 Hz, Ar$CH_2$$CH_2$), 1.85 (2H, quin, *J* 7.3 Hz, OCH$_2$$CH_2$$CH_2$), 1.73 (2H, quin, *J* 7.4 Hz, ArCH$_2$$CH_2$$CH_2$), 1.56 (2H, m, $CH_2$$CH_2$$CH_2$$CH_2$), 1.49 (2H, m, $CH_2$$CH_2$$CH_2$$CH_2$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.56, 157.98, 157.89, 145.58, 145.31, 143.50, 136.55, 132.57, 130.24, 129.36, 129.21, 127.49, 127.12, 122.06, 119.04, 114.67, 114.38, 110.58, 68.03, 55.51, 35.49, 31.24, 29.09, 28.93, 25.87. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{33}H_{33}N_2O_2$: 489.2542. Found: 489.2538.

The remaining homologues in the CB6O.O*m* series were prepared using the method described for CB6O.O1.

CB6O.O2 (**5**, X=C_2H_5)

Elemental analysis: Calculated for $C_{34}H_{34}N_2O_2$: C, 81.24%, H, 6.82%, N, 5.57%. Found: C, 81.48%, H, 6.85%, N, 5.53%. Infrared *v* cm^{-1}: 2928 (C-H), 2858 (C-H), 2225 (C≡N), 1604, 1571, 1508, 1476, 1243, 1170, 1046, 835, 813, 549. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, ArCHN), 7.84 (2H, d, *J* 8.6 Hz, Ar), 7.73 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.22 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.94 (2H, d, *J* 8.6 Hz, Ar), 4.09 (2H, t, *J* 7.0 Hz, O$CH_2$$CH_3$), 4.04 (2H, t, *J* 6.8 Hz, O$CH_2$$CH_2$),

2.71 (2H, t, *J* 7.6 Hz, Ar$\underline{CH_2}CH_2$), 1.85 (2H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.73 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.54 (4H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.45 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.53, 157.76, 157.36, 145.58, 145.16, 143.50, 136.55, 132.57, 130.21, 129.39, 129.20, 127.49, 127.12, 122.06, 119.03, 114.97, 114.66, 110.58, 68.02, 63.70, 35.49, 31.23, 29.09, 28.93, 25.87, 14.90. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{34}H_{35}N_2O_2$: 503.2699. Found: 503.2685.

CB6O.O3 (**5**, X=C_3H_7)

Elemental analysis: Calculated for $C_{35}H_{36}N_2O_2$: C, 81.36%, H, 7.02%, N, 5.42%. Found: C, 81.59%, H, 7.06%, N, 5.39%. Infrared *v* cm^{-1}: 2929 (C-H), 2856 (C-H), 2225 (C≡N), 1605, 1569, 1509, 1473, 1247, 1170, 1016, 838, 814, 548. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, Ar$\underline{CH}$N), 7.84 (2H, d, *J* 8.6 Hz, Ar), 7.74 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.22 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.95 (2H, d, *J* 8.6 Hz, Ar), 4.04 (2H, t, *J* 6.4Hz, $O\underline{CH_2}CH_2$), 3.96 (2H, t, *J* 6.6 Hz, $O\underline{CH_2}CH_3$), 2.71 (2H, t, *J* 7.7 Hz, Ar$\underline{CH_2}CH_2$), 1.86 (2H, quin, *J* 7.0 Hz, $OCH_2\underline{CH_2}CH_2$), 1.83 (2H, quin, *J* 7.0 Hz, $OCH_2\underline{CH_2}CH_3$), 1.73 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.56 (2H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.49 (2H, m, $CH_2CH_2\underline{CH_2}CH_2$), 1.07 (3H, t, *J* 7.4 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.52, 157.75, 157.56, 145.58, 145.10, 143.51, 136.55, 132.58, 130.21, 129.39, 129.21, 127.49, 127.13, 122.04, 119.05, 114.98, 114.65, 110.57, 69.79, 68.01, 35.50, 31.25, 29.09, 28.93, 25.88, 22.66, 10.55. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{35}H_{37}N_2O_2$: 517.2855. Found: 517.2845.

CB6O.O4 (**5**, X=C_4H_9)

Elemental analysis: Calculated for $C_{36}H_{38}N_2O_2$: C, 81.47%, H, 7.22%, N, 5.28%. Found: C, 81.62%, H, 7.24%, N, 5.23%. Infrared *v* cm^{-1}: 2935 (C-H), 2873 (C-H), 2225 (C≡N), 1606, 1570, 1509, 1474, 1247, 1167, 1009, 840, 814, 550. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.41 (1H, s, Ar$\underline{CH}$N), 7.83 (2H, d, *J* 8.6 Hz, Ar), 7.73 (2H, d, *J* 8.6 Hz, Ar), 7.68 (2H, d, *J* 8.6 Hz, Ar), 7.52 (2H, d, *J* 8.6 Hz, Ar), 7.31 (2H, d, *J* 8.6 Hz, Ar), 7.20 (2H, d, *J* 8.6 Hz, Ar), 6.97 (2H, d, *J* 8.6 Hz, Ar), 6.93 (2H, d, *J* 8.6 Hz, Ar), 4.04 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.98 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$),

2.70 (2H, t, *J* 7.6 Hz, Ar$\underline{CH_2}CH_2$), 1.84 (2H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.81 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.71 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.52 (6H, m, $CH_2\underline{CH_2}CH_2CH_2$), 0.99 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.51, 157.77, 157.57, 145.58, 145.07, 143.51, 136.55, 132.59, 130.21, 129.37, 129.21, 127.50, 127.13, 122.04, 119.07, 114.96, 114.64, 110.56, 68.00, 67.97, 35.50, 31.39, 31.27, 29.09, 28.93, 25.88, 19.28, 13.91. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{36}H_{39}N_2O_2$: 531.3012. Found: 531.2999.

CB6O.O5 (**5**, X=C_5H_{11})

Elemental analysis: Calculated for $C_{37}H_{40}N_2O_2$: C, 81.58%, H, 7.40%, N, 5.14%. Found: C, 81.80%, H, 7.45%, N, 5.10%. Infrared *v* cm^{-1}: 2933 (C-H), 2858 (C-H), 2221 (C≡N), 1604, 1573, 1511, 1470, 1250, 1162, 1022, 838, 813, 545. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.41 (1H, s, Ar$\underline{CHN}$), 7.83 (2H, d, *J* 8.6 Hz, Ar), 7.73 (2H, d, *J* 8.6 Hz, Ar), 7.68 (2H, d, *J* 8.6 Hz, Ar), 7.52 (2H, d, *J* 8.6 Hz, Ar), 7.31 (2H, d, *J* 8.6 Hz, Ar), 7.21 (2H, d, *J* 8.6 Hz, Ar), 6.97 (2H, d, *J* 8.6 Hz, Ar), 6.93 (2H, d, *J* 8.6 Hz, Ar), 4.02 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.98 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$), 2.70 (2H, t, *J* 7.6 Hz, Ar$\underline{CH_2}CH_2$), 1.81 (4H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.71 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.44 (8H, m, $CH_2\underline{CH_2}CH_2CH_2$), 0.95 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.51, 157.77, 157.56, 145.58, 145.05, 143.52, 136.54, 132.59, 130.22, 129.36 129.21, 127.50, 127.13, 122.06, 119.08, 114.95, 114.64, 110.54, 68.27, 68.00, 35.51, 31.28, 29.10, 29.05, 28.95, 28.24, 25.89, 22.52, 14.08. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{37}H_{42}N_2O_2$: 545.3168. Found: 545.3150.

CB6O.O6 (**5**, X=C_6H_{13})

Elemental analysis: Calculated for $C_{38}H_{42}N_2O_2$: C, 81.68%, H, 7.58%, N, 5.01%. Found: C, 81.89%, H, 7.62%, N, 4.97%. Infrared *v* cm^{-1}: 2934 (C-H), 2858 (C-H), 2224 (C≡N), 1605, 1572, 1510, 1473, 1244, 1164, 1027, 835, 814, 543. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, Ar$\underline{CHN}$), 7.84 (2H, d, *J* 8.6 Hz, Ar), 7.74 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.22 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.94 (2H, d, *J* 8.6 Hz, Ar), 4.05 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.99 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$), 2.71 (2H, t, *J* 7.6 Hz, Ar$\underline{CH_2}CH_2$), 1.82 (4H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.72 (2H, quin, *J* 7.4

Hz, $ArCH_2\underline{CH_2}CH_2$), 1.48 (6H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.36 (4H, m, $CH_2\underline{CH_2}CH_2CH_2$), 0.93 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.51, 157.78, 157.56, 145.58, 145.05, 143.52, 136.54, 132.59, 130.22, 129.35, 129.22, 127.50, 127.13, 122.06, 119.09, 114.95, 114.64, 110.54, 68.29, 68.00, 35.51, 31.64, 31.28, 29.31, 29.10, 28.95, 25.89, 25.77, 22.65, 14.10. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{38}H_{43}N_2O_2$: 559.3325. Found: 559.3300.

CB6O.O7 (**5**, X=C_7H_{15})

Elemental analysis: Calculated for $C_{39}H_{44}N_2O_2$: C, 81.78%, H, 7.74%, N, 4.89%. Found: C, 81.97%, H, 7.80%, N, 4.84%. Infrared *v* cm^{-1}: 2934 (C-H), 2854 (C-H), 2227 (C≡N), 1608, 1569, 1510, 1474, 1249, 1163, 1016, 836, 824, 810, 540. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, Ar<u>CH</u>N), 7.84 (2H, d, *J* 8.6 Hz, Ar), 7.73 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.22 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.94 (2H, d, *J* 8.6 Hz, Ar), 4.05 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.99 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$), 2.71 (2H, t, *J* 7.6 Hz, $Ar\underline{CH_2}CH_2$), 1.82 (4H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.72 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.48 (6H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.34 (6H, m, $CH_2\underline{CH_2}CH_2CH_2$), 0.92 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.51, 157.74, 157.57, 145.58, 145.06, 143.51, 136.55, 132.59, 130.21, 129.38, 129.21, 127.50, 127.13, 122.04, 119.06, 114.97, 114.65, 110.57, 68.30, 68.01, 35.50, 31.81, 31.26, 29.35, 29.11, 28.94, 26.05, 25.88, 22.63, 14.12. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{39}H_{45}N_2O_2$: 573.3481. Found: 573.3469.

CB6O.O8 (**5**, X=C_8H_{17})

Elemental analysis: Calculated for $C_{40}H_{46}N_2O_2$: C, 81.87%, H, 7.90%, N, 4.77%. Found: C, 82.05%, H, 7.96%, N, 4.73%. Infrared *v* cm^{-1}: 2932 (C-H), 2852 (C-H), 2225 (C≡N), 1606, 1572, 1510, 1473, 1250, 1166, 1019, 843, 827, 814, 544. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, Ar<u>CH</u>N), 7.83 (2H, d, *J* 8.6 Hz, Ar), 7.74 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.22 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.94 (2H, d, *J* 8.6 Hz, Ar), 4.04 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.98 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$), 2.71 (2H, t, *J* 7.6 Hz, $Ar\underline{CH_2}CH_2$), 1.84 (4H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.72(2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.56 (2H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.49 (4H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.33 (8H, m,

$CH_2\underline{CH_2}CH_2CH_2$), 0.91 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.51, 157.73, 157.58, 145.58, 145.08, 143.50, 136.55, 132.57, 130.20, 129.39, 129.20, 127.49, 127.12, 122.03, 119.03, 114.98, 114.65, 110.57, 68.32, 68.01, 35.49, 31.83, 31.25, 29.39, 29.35, 29.26, 29.09, 28.93, 26.08, 25.88, 22.68, 14.12. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{40}H_{47}N_2O_2$: 587.3638. Found: 587.3621.

CB6O.O9 (**5**, X=C_9H_{19})

Elemental analysis: Calculated for $C_{41}H_{48}N_2O_2$: C, 81.96%, H, 8.05%, N, 4.66%. Found: C, 82.10%, H, 8.13%, N, 4.64%. Infrared *v* cm^{-1}: 2921 (C-H), 2852 (C-H), 2234 (C≡N), 1606, 1574, 1510, 1475, 1247, 1170, 1017, 841, 813, 547. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.42 (1H, s, Ar<u>CH</u>N), 7.84 (2H, d, *J* 8.6 Hz, Ar), 7.74 (2H, d, *J* 8.6 Hz, Ar), 7.69 (2H, d, *J* 8.6 Hz, Ar), 7.53 (2H, d, *J* 8.6 Hz, Ar), 7.32 (2H, d, *J* 8.6 Hz, Ar), 7.22 (2H, d, *J* 8.6 Hz, Ar), 6.98 (2H, d, *J* 8.6 Hz, Ar), 6.94 (2H, d, *J* 8.6 Hz, Ar), 4.05 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.99 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$), 2.71 (2H, t, *J* 7.6 Hz, $Ar\underline{CH_2}CH_2$), 1.83 (4H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.73 (2H, quin, *J* 7.4 Hz, $ArCH_2\underline{CH_2}CH_2$), 1.51 (6H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.31 (10H, m, $CH_2\underline{CH_2}CH_2CH_2$), 0.91 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.51, 157.74, 157.58, 145.58, 145.07, 143.51, 136.54, 132.58, 130.22, 129.38, 129.21, 127.50, 127.13, 122.04, 119.06, 114.97, 114.65, 110.57, 68.30, 60.01, 35.50, 31.91, 31.26, 29.57, 29.45, 29.35, 29.30, 29.10, 28.94, 26.08, 25.89, 22.70, 14.15. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{41}H_{49}N_2O_2$: 601.3794. Found: 601.3787.

CB6O.O10 (**5**, X=$C_{10}H_{21}$)

Elemental analysis: Calculated for $C_{42}H_{50}N_2O_2$: C, 82.04%, H, 8.20%, N, 4.56%. Found: C, 82.23%, H, 8.24%, N, 4.53%. Infrared *v* cm^{-1}: 2920 (C-H), 2852 (C-H), 2228 (C≡N), 1607, 1574, 1510, 1475, 1248, 1170, 1021, 840, 813, 547. ^{1}H NMR (300 MHz $CDCl_3$)δ: 8.41 (1H, s, Ar<u>CH</u>N), 7.83 (2H, d, *J* 8.6 Hz, Ar), 7.72 (2H, d, *J* 8.6 Hz, Ar), 7.68 (2H, d, *J* 8.6 Hz, Ar), 7.52 (2H, d, *J* 8.6 Hz, Ar), 7.31 (2H, d, *J* 8.6 Hz, Ar), 7.21 (2H, d, *J* 8.6 Hz, Ar), 6.97 (2H, d, *J* 8.6 Hz, Ar), 6.93 (2H, d, *J* 8.6 Hz, Ar), 4.03 (2H, t, *J* 7.0 Hz, $O\underline{CH_2}CH_3$), 3.98 (2H, t, *J* 6.8 Hz, $O\underline{CH_2}CH_2$), 2.70 (2H, t, *J* 7.6 Hz, $Ar\underline{CH_2}CH_2$), 1.83 (4H, quin, *J* 7.3 Hz, $OCH_2\underline{CH_2}CH_2$), 1.71 (2H, quin, *J* 7.4

Hz, $ArCH_2\underline{CH_2}CH_2$), 1.48 (6H, m, $CH_2\underline{CH_2}CH_2CH_2$), 1.32 (12H, m, $CH_2\underline{CH_2}CH_2CH_2$), 0.90 (3H, t, *J* 7.0 Hz, $CH_2\underline{CH_3}$). ^{13}C NMR (75 MHz $CDCl_3$)δ: 161.53, 157.70, 157.59, 145.58, 145.08, 143.51, 136.54, 132.57, 130.22, 129.41, 129.21, 127.48, 127.12, 122.04, 119.04, 114.99, 114.66, 110.58, 68.32, 68.02, 35.50, 31.92, 31.25, 29.61, 29.59, 29.44, 29.35, 29.11, 28.94, 26.09, 25.88, 22.71, 14.14. MS (ESI+, m/z): $[M+H]^+$ Calculated for $C_{42}H_{51}N_2O_2$: 615.3951. Found: 615.3933.

Section 3: Reference

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