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SUPPORTING INFORMATION

Liquid-crystalline and nonlinear-optical properties of bent-shaped compounds derived from 3,4'-biphenylene

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EXPERIMENTAL SECTION

Commercial chemicals (reagent grade) were purchased from Aldrich Chemical Co., Fluka or Panreac and used without further purification. Solvents were purified and dried by standard techniques.

The synthetic procedures leading to the target compounds are outlined in Schemes 2–4.

Some of the intermediates used in these syntheses were prepared according to procedures already reported in the literature: 3,4'-dihydroxybiphenyl (**1**),¹ *p*-*n*-tetradecyloxyaniline (**3**),² 3-[4''-(4'''-*n*-tetradecyloxybenzoyloxy)benzoyloxy]biphenyl-4'-ol (**4**),³ *p*-*n*-tetradecyloxybenzoic acid (**10**)³ and 4-(4'-*n*-tetradecyloxyphenylazoxy)benzoic acid (**15**).

3,4'-[Bis-(4''-formyl)benzoyloxy]biphenyl (2). DCC (1.65 g, 7.5 mmol) was added to a cooled (0 °C), stirred mixture of 3,4'-dihydroxybiphenyl (**1**) (0.5 g, 2.7 mmol), *p*-formylbenzoic acid (1.05 g, 6.9 mmol) and DMAP (0.065 mg, 0.27 mmol) and dichloromethane (25 mL) under an argon atmosphere. The reaction mixture was stirred for 30 min at 0 °C and was allowed to warm up to room temperature. The mixture was stirred for 24 h, the resulting solid was filtered off and the filtrate was evaporated to give a solid, which was purified by flash chromatography on silica gel, using dichloromethane as the eluent, gave the product in 62% yield. Mp 185 °C. ¹H-NMR (400 MHz, CDCl₃): δ 10.8 (s, 2H), 8.34–8.25 (m, 4H), 8.00–7.96 (m, 4H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 5.2 Hz, 2H), 7.4 (s, 1H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.24–7.17 (m, 1H). IR (Nujol, NaCl) 1732, 1688, 1460, 1290 cm⁻¹.

3-[4''-(4'''-*n*-Tetradecyloxybenzoyloxy)benzoyloxy]-4'-(4^{iv}-formylbenzoyloxy)

biphenyl (5). The synthetic method followed was analogous to that described for compound **2**, using 3-[4''-(4'''-*n*-tetradecyloxybenzoyloxy)benzoyloxy]biphenyl-4'-ol (**4**) (0.7 g, 1.12 mmol), *p*-formylbenzoic acid (0.202 g, 1.35 mmol), DCC (0.279 g, 1.35 mmol), DMAP (0.014 g, 0.112 mmol) and dichloromethane (30 mL). Purification was carried out by flash chromatography on silica gel, using dichloromethane as the eluent, followed by recrystallization from ethyl acetate. The product was obtained in 71% yield. Mp 143 °C. ¹H-NMR (400 MHz, CDCl₃): δ 10.16 (s, 1H), 8.39 (d, *J* = 8.7 Hz, 2H), 8.30 (d, *J* = 9.2 Hz, 2H), 8.15 (d, *J* = 8.7 Hz, 2H), 8.04 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 9.0 Hz, 2H), 7.52 (d, *J* = 8.7 Hz, 2H), 7.49 (s, 1H), 7.38 (d, *J* = 9.2 Hz, 2H), 7.33 (d, *J* = 8.7 Hz, 2H), 7.26–7.22 (m, 1H), 7.00 (d, *J* = 9.0 Hz, 2H), 4.05 (t, *J* = 6.6 Hz, 2H), 1.85–1.81 (m, 2H), 1.487–1.27 (m, 22H), 0.88 (t, *J* = 6.6 Hz, 3H). IR (Nujol, NaCl) 1726, 1598, 1249, 1157 cm⁻¹.

Methyl 2,4-dihydroxybenzoate (6). Concentrated sulfuric acid (6.5 mL) was slowly added to a solution of 2,4-dihydroxybenzoic acid (5.0 g, 32 mmol) in methanol (300 mL). The reaction mixture was stirred and heated under reflux for 4 h. A proportion of the solvent was evaporated and water (50 mL) was added. The precipitate was filtered off, washed with water and recrystallized from ethanol (96%) to give a white solid in 84% yield. Mp 118.5 °C. ¹H-NMR (300 MHz, CDCl₃): δ 10.94 (s, 1H), 7.70 (d, *J* = 8.7 Hz, 1H), 6.39–6.33 (m, 2H), 5.35 (s, 1H), 3.89 (s, 3H). IR (Nujol, NaCl) 3300–2960, 1644, 1269 cm⁻¹.

Methyl 2-hydroxy-4-*n*-tetradecyloxybenzoate (7). A mixture of **6** (1.80 g, 10.7 mmol), KHCO₃ (1.07 g, 10.7 mmol) and dry acetone (100 mL) was heated under reflux under an argon atmosphere. *n*-Tetradecylbromide (2.97 g, 10.7 mmol) was added slowly

by syringe and, after 6 h, the reaction mixture was cooled to room temperature and poured into cold water (100 mL). The product was extracted using hexane/ethyl acetate (2:1) (3 × 50 mL) and the organic layer washed with water (3 × 60 mL), saturated aqueous NaCl (1 × 60 mL) and dried over anhydrous Na₂SO₄. The solvent was removed and the solid was recrystallized from ethanol to give the product in 85% yield. Mp 66 °C. ¹H-NMR (300 MHz, CDCl₃): δ 7.96 (d, *J* = 9.0 Hz, 2H), 6.88 (d, *J* = 9.0 Hz, 2H), 3.98 (d, *J* = 6.0 Hz, 2H), 3.87 (s, 3H), 1.75–1.71 (m, 2H), 1.41–1.22 (m, 22H), 0.86 (t, *J* = 6.0 Hz, 3H). IR (Nujol, NaCl) 1723, 1610, 1259 cm⁻¹.

2-Hydroxy-4-*n*-tetradecyloxybenzoic acid (8). LiOH·H₂O (1.44 g, 34.3 mmol) was added in small amounts to a cooled (0 °C) mixture of compound **7** (3.0 g, 8.2 mmol) in methanol/water (10:3.5, 135 mL). After the addition was complete, the mixture was stirred and heated under reflux for 2 h and allowed to cooled to room temperature. 3.5% aqueous NaOH (150 mL) was added to give the sodium salt of the organic acid. The solution was acidified with concentrated hydrochloric acid and the precipitate was filtered off, washed with water and purified by recrystallization from ethanol to give a white solid in 91% yield. Mp 107 °C. ¹H-NMR (300 MHz, CDCl₃): δ 10.60 (s, 1H), 7.77 (d, *J* = 8.7 Hz, 1H), 6.47–6.42 (m, 2H), 3.96 (t, *J* = 6.6 Hz, 2H), 1.79–1.74 (m, 2H), 1.35–1.23 (m, 22H), 0.85 (t, *J* = 6.6 Hz, 3H). IR (Nujol, NaCl) 3300 (broad), 2900 (broad), 1645, 1625, 1174 cm⁻¹.

Benzyl 2,4-dihydroxybenzoate (9). A mixture of 2,4-dihydroxybenzoic acid (5.0 g, 32 mmol) and NaHCO₃ (8.64 g, 96 mmol) in dry DMF (40 mL) was heated at 90 °C for 1 h under an argon atmosphere. Benzyl bromide (5.47 g, 32 mmol) was added dropwise and the mixture was stirred for 7 h after the addition was complete. The reaction mixture was cooled to room temperature and poured into water (200 mL). The product was

extracted with hexane/ethyl acetate (1:1) (2×100 mL) and the organic layer was washed with water (2×50 mL), dried over MgSO_4 and the solvent removed. The resulting yellow oil was purified twice by flash chromatography on silica gel using dichloromethane as eluent in the first purification and diethyl ether in the second. The resulting oil solidified slowly to give the product in 70% yield. $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 10.98 (s, 1H), 7.75 (d, $J = 8.4$ Hz, 1H), 7.42–7.34 (m, 5H), 6.38 (d, $J = 2.4$ Hz, 1H), 6.33 (dd, $J_1 = 2.4$ Hz, $J_2 = 8.4$ Hz, 1H), 5.35 (s, 1H). IR (Nujol, NaCl) 3347, 1661, 1623, 1143 cm^{-1} .

Benzyl 4-(2'-hydroxy-4'-*n*-tetradecyloxybenzoyloxy)benzoate (11). DCC (1.18 g) was added to a cooled (0°C), stirred mixture of compound **8** (2.0 g, 5.71 mmol), benzyl 4-hydroxybenzoate (1.30 g, 5.71 mmol) and a catalytic amount of DMAP (0.069 g, 0.571 mmol) in freshly distilled dichloromethane (50 mL) under an argon atmosphere. The reaction mixture was stirred for 30 min at 0°C and was allowed to warm up to room temperature. The mixture was stirred for 24 h, the resulting solid was filtered off and the filtrate was evaporated to give a solid, which was purified by flash chromatography on silica gel using hexane/ethyl acetate (15:1) as eluent followed by recrystallization from ethanol to give **11** in 47% yield. Mp 63°C . $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 10.53 (s, 1H), 8.14 (d, $J = 8.7$ Hz, 2H), 7.92 (d, $J = 8.7$ Hz, 1H), 7.45–7.33 (m, 5H), 7.28 (d, $J = 8.7$ Hz, 2H), 6.51–6.46 (m, 2H), 5.36 (s, 2H), 3.99 (t, $J = 6.3$ Hz, 2H), 1.80–1.72 (m, 2H), 1.47–1.24 (m, 22H), 0.88 (t, $J = 6.3$ Hz, 3H). IR (Nujol, NaCl) 2950, 1718, 1672, 1601, 1160 cm^{-1} .

4-(2'-Hydroxy-4'-*n*-tetradecyloxybenzoyloxy)benzoic acid (12). A mixture of compound **11** (1.0 g, 1.74 mmol), $\text{Pd}(\text{OH})_2/\text{C}$ (20%, 0.1 g), cyclohexene (7 mL) and ethanol (25 mL) was stirred and heated under reflux for 16 h. The reaction mixture was

allowed to cool down to room temperature, filtered through a pad of Celite[®] and the solvent evaporated to give a solid, which was purified by recrystallization from ethanol. A white solid was obtained in 98% yield. Mp: Cr 107 °C SmC 186 °C I. ¹H-NMR (300 MHz, CDCl₃): δ 10.5 (s, 1H), 8.19 (d, *J* = 8.7 Hz, 2H), 7.93 (d, *J* = 8.7 Hz, 1H), 7.31 (d, *J* = 8.7 Hz, 2H), 6.51–6.46 (m, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 1.81–1.76 (m, 2H), 1.44–1.25 (m, 22H), 0.83 (t, *J* = 6.3 Hz, 3H). IR (Nujol, NaCl) 2921 (broad), 1693, 1603, 1159 cm⁻¹.

Benzyl 2-hydroxy-4-(4'-*n*-tetradecyloxybenzoyloxy)benzoate (13). The synthetic method followed was analogous to that described for compound **11**, using 4-*n*-tetradecyloxybenzoic acid (2.0 g, 5.98 mmol), **9** (1.51 g, 5.98 mmol), DCC (1.23 g, 5.98 mmol), DMAP (0.073 g, 0.598 mmol) and dichloromethane (50 mL). Purification was carried out by flash chromatography on silica gel using hexane/ethyl acetate (15:1) as eluent. A white solid was obtained in 41% yield. Mp 66 °C. ¹H-NMR (300 MHz, CDCl₃): δ 10.87 (s, 1H), 8.09 (d, *J* = 8.7 Hz, 2H), 7.92 (d, *J* = 9.0 Hz, 1H), 7.43–7.34 (m, 5H), 6.93 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 2.4 Hz, 1H), 6.74 (dd, *J*₁ = 2.4 Hz, *J*₂ = 9.0 Hz, 1H), 5.37 (s, 2H), 4.01 (t, *J* = 6.3 Hz, 2H), 1.81–1.74 (m, 2H), 1.45–1.24 (m, 22H), 0.84 (t, *J* = 6.3 Hz, 3H). IR (Nujol, NaCl) 3065 (broad), 1737, 1670, 1605, 1160 cm⁻¹.

2-Hydroxy-4-(4'-*n*-tetradecyloxybenzoyloxy)benzoic acid (14). The synthetic method followed was the same as that described for **12**, obtaining **14** from compound **13** in 90% yield. Mp: Cr 126 °C SmC 177 °C I. ¹H-NMR (300 MHz, CDCl₃): δ 10.88 (s, 1H), 8.10 (d, *J* = 8.4 Hz, 2H), 7.94 (d, *J* = 8.4 Hz, 1H), 7.43–7.34 (m, 5H), 6.95 (d, *J* = 8.4 Hz, 2H), 6.88 (d, *J* = 2.1 Hz, 1H), 6.78 (dd, *J*₁ = 2.1 Hz, *J*₂ = 8.4 Hz, 1H), 4.02 (t, *J* =

6.6 Hz, 2H), 1.74–1.68 (m, 2H), 1.47–1.24 (m, 22H), 0.85 (t, $J = 6.6$ Hz, 3H). IR (Nujol, NaCl) 2990 (broad), 2562, 1743, 1640, 1603, 1159 cm^{-1} .

3,4'-Bis[4-(4-*n*-tetradecyloxyphenyliminomethyl)benzoyloxy]biphenyl (III). A solution of 4-*n*-tetradecyloxyaniline (0.35 g, 1.14 mmol), **2** (0.235, 0.52 mmol) and 5 drops of acetic acid in ethanol (30 mL) was stirred and heated under reflux under an argon atmosphere for 16 h in the dark. Thin layer chromatography on neutral alumina (to avoid hydrolysis of the imine linkage) was performed to monitor the progress of the reaction. The reaction mixture was cooled to room temperature, the solid filtered off and recrystallized twice (first from toluene and then from ethyl acetate) to give a yellow solid (75% yield). $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 8.59 (s, 2H), 8.30 (dd, $J_1 = 2.7$, $J_2 = 8.4$ Hz, 4H), 8.04 (d, $J = 8.7$ Hz, 4H), 7.67 (d, $J = 8.4$ Hz, 2H), 7.53–7.50 (m, 3H), 7.34 (d, $J = 8.4$ Hz, 2H), 7.33–7.27 (m, 5H), 6.94 (d, $J = 8.7$ Hz, 4H), 4.00 (t, $J = 6.0$ Hz, 4H), 1.83–1.76 (m, 4H), 1.48–1.29 (m, 44H), 0.89 (t, $J = 6.0$ Hz, 6H). $^{13}\text{C-NMR}$ (300 MHz, CDCl_3) δ 164.6, 158.6, 156.0, 151.5, 150.8, 144.2, 142.1, 141.2, 138.0, 131.4, 131.3, 130.4, 129.7, 128.4, 128.2, 124.6, 122.3, 121.9, 120.5, 120.3, 115.3, 68.5, 31.8, 29.5, 29.4, 29.3, 29.2, 26.0, 22.5, 13.8. IR (Nujol, NaCl) 1729, 1623, 1461, 1247, 1176 cm^{-1} . EA for $\text{C}_{68}\text{H}_{84}\text{N}_2\text{O}_6$ (calculated: 79.65% C, 8.26% H, 2.73% N) found 79.83% C, 8.27% H, 2.68% N. MS (FAB $^+$): m/z : 420, 1026.

3,4'-Bis[4-(2-hydroxy-4-*n*-tetradecyloxybenzoyloxy)benzoyloxy]biphenyl (IV). DCC (0.184 g, 0.89 mmol) was added to a cooled (0 $^\circ\text{C}$), stirred mixture of compound **12** (0.400 g, 0.85 mmol), 3,4'-dihydroxybiphenyl (0.059 g, 0.33 mmol) and DMAP (0.020 g, 0.17 mmol) in freshly distilled dichloromethane (25 mL) under an argon atmosphere. The reaction mixture was stirred for 30 min at 0 $^\circ\text{C}$ and was then allowed

to warm up to room temperature. The mixture was stirred for 24 h and the solid was filtered off, washed and the filtrate evaporated to dryness. The resulting solid was purified by flash chromatography on silica gel using dichloromethane/hexane (10:1) as eluent followed by recrystallization twice (first from toluene and then from ethyl acetate) to give **IV** in 39% yield. $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 10.5 (s, 2H), 8.30 (d, $J = 9.0$ Hz, 4H), 7.95 (d, $J = 9.0$ Hz, 2H), 7.65 (d, $J = 8.4$ Hz, 2H), 7.50 (d, $J = 4.8$ Hz, 2H), 7.44 (s, 1H), 7.36 (d, $J = 9.0$ Hz, 4H), 7.29 (d, $J = 8.4$ Hz, 2H), 7.24–7.20 (m, 1H), 6.54–6.48 (m, 4H), 4.00 (t, $J = 6.0$ Hz, 4H), 1.84–1.75 (m, 4H), 1.40–1.25 (m, 44H), 0.86 (t, $J = 6.6$ Hz, 6H). $^{13}\text{C-NMR}$ (300 MHz, CDCl_3) δ 168.1, 166.2, 164.7, 164.3, 154.5, 153.4, 150.6, 142.1, 138.1, 131.9, 131.7, 129.9, 128.4, 127.3, 124.8, 122.1, 122.0, 108.7, 104.1, 101.3, 68.5, 31.9, 29.7, 29.6, 29.5, 29.3, 29.0, 25.9, 22.7, 14.1. IR (Nujol, NaCl) 1736, 1676, 1626, 1602, 1261, 1162 cm^{-1} . EA for $\text{C}_{68}\text{H}_{82}\text{O}_{12}$ (calculated: 74.83% C, 7.57% H) found 74.86% C, 7.36% H. MS (FAB $^+$): m/z : 333, 453, 665, 786.

3,4'-Bis[2-hydroxy-4-(4-*n*-tetradecyloxybenzoyloxy)benzoyloxy]biphenyl (V). An experimental procedure similar to that used for the synthesis of compound **IV** was followed, using in this case **14** (0.300 g, 0.62 mmol), 3,4'-dihydroxybiphenyl (0.044 g, 0.24 mmol), DCC (0.138 g, 0.67 mmol), DMAP (0.020 g, 0.17 mmol) and dichloromethane (25 mL). The purification was carried by flash chromatography on silica gel using dichloromethane/hexane (10:1) as eluent followed by recrystallization twice (first from toluene and then from ethyl acetate). (46% yield). $^1\text{H-NMR}$ (300 MHz, CDCl_3): δ 10.65 (s, 2H), 8.16 (d, $J = 4.5$ Hz, 2H), 8.11 (d, $J = 9.0$ Hz, 4H), 7.67 (d, $J = 8.4$ Hz, 2H), 7.53 (d, $J = 4.5$ Hz, 2H), 7.44 (s, 1H), 7.30 (d, $J = 9.0$ Hz, 2H), 7.24–7.20 (m, 1H), 6.96 (d, $J = 9.0$ Hz, 4H), 6.92–6.86 (m, 4H), 4.04 (t, $J = 6.6$ Hz, 4H), 1.84–1.78 (m, 4H), 1.46–1.25 (m, 44H), 0.87 (t, $J = 6.4$ Hz, 6H). $^{13}\text{C-NMR}$ (300 MHz, CDCl_3) δ 168.1, 166.1, 164.3, 163.8, 155.5, 151.3, 150.5, 142.0, 138.2, 132.4, 131.9,

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131.8, 130.0, 129.8, 128.4, 128.3, 122.1, 122.0, 120.5, 109.3, 104.3, 101.2, 68.4, 31.9, 29.7, 29.5, 29.4, 29.3, 29.2, 29.1, 27.2, 27.2, 26.0, 22.7, 14.1. IR (Nujol, NaCl) 1733, 1686, 1606, 1253, 1168 cm^{-1} . EA ($\text{C}_{68}\text{H}_{82}\text{O}_{12}$ calculated: 74.83% C, 7.57% H) found 74.98% C, 7.35% H. MS (FAB^+): m/z : 317, 453, 635, 770.

3-[4''-(4'''-*n*-tetradecyloxybenzoyloxy)benzoyloxy]-4'-[4^{iv}-(4^v-*n*-

tetradecyloxyphenylazoxy)benzoyloxy]biphenyl (VII). The acid chloride (**15**) of 4-(4'-*n*-tetradecyloxyphenylazoxy)benzoic acid was prepared using the acid (0.328 g, 0.72 mmol), oxalyl chloride (0.188 g, 1.44 mmol), toluene (60 mL) and 5 drops of dimethylformamide. The acid chloride was reacted with 3-[4''-(4'''-*n*-tetradecyloxybenzoyloxy)benzoyloxy]biphenyl-4'-ol (0.375 g, 0.60 mmol), triethylamine (0.12 mL, 0.843 mmol) and dichloromethane (60 mL). The product was purified by flash chromatography using a dichloromethane/hexane (8:2) as eluent followed by recrystallization twice (first from toluene and then from ethyl acetate). (70% yield of a pale yellow solid). $^1\text{H-NMR}$ (500 MHz, CDCl_3): δ 8.50–8.16 (m, 10H), 7.74–.69 (m, 2H), 7.56–7.53 (m 2H), 7.51–7.49 (m, 1H), 7.41 (d, $J = 8.6$ Hz, 2H), 7.39–7.34 (m, 2H), 7.28–7.24 (m, 1H), 7.05–7.99 (m, 4H), 4.11–4.05 (m, 4H), 1.89–1.91 (m, 4H), 1.55–1.47 (m, 4H), 1.44–1.24 (m, 40H), 0.92 (t, $J = 6.8$ Hz, 6H). $^{13}\text{C-NMR}$ (500 MHz, CDCl_3) δ 164.5, 164.4, 164.1, 163.9, 162.4, 160.8, 155.5, 151.6, 151.4, 150.7, 150.5, 148.2, 142.1, 142.0, 141.2, 138.3, 138.1, 137.6, 132.5, 131.9, 131.7, 130.9, 130.8, 129.9, 129.0, 128.5, 128.4, 128.4, 126.9, 125.1, 124.7, 124.3, 122.5, 122.2, 122.1, 122.0, 121.0, 120.7, 120.7, 120.5, 114.5, 114.3, 68.7, 68.4, 68.4, 32.0, 29.7, 29.6, 29.6, 29.4, 29.2, 29.1, 26.0, 22.7, 14.2. IR (Nujol, NaCl) 1732, 1461, 1255, 1162 cm^{-1} . EA for $\text{C}_{68}\text{H}_{84}\text{N}_2\text{O}_6$ (calculated: 75.96% C, 7.80% H, 2.64% N) found 75.93% C, 7.85% H, 2.77% N. MS (MALDI^+): m/z : 860, 965, 1017, 1044, 1060.

3-[4''-(4'''-*n*-tetradecyloxybenzoyloxy)benzoyloxy]-4'-[4^{iv}-(4^v-*n*-

tetradecyloxyphenyliminomethyl)benzoyloxy]biphenyl (VIII). The synthetic method

followed to obtain this imine was similar to that described for the synthesis of compound **III**. In this case 4-*n*-tetradecyloxyaniline (0.16 g, 0.52 mmol), **5** (0.36 g, 0.48 mmol), ethanol (60 mL) and acetic acid (5 drops) were used. Purification was carried out by recrystallization twice (first from toluene and then from ethyl acetate). (70% yield of a pale yellow solid). ¹H-NMR (400 MHz, CDCl₃) δ 8.59 (s, 1H), 8.31 (d, *J* = 8.6 Hz, 4H), 8.16 (d, *J* = 8.9 Hz, 2H), 8.04 (d, *J* = 8.6 Hz, 2H), 7.68 (d, *J* = 8.7 Hz, 2H), 7.52 (d, *J* = 5.3 Hz, 2H), 7.48–7.45 (m, 1H), 7.39 (d, *J* = 8.6 Hz, 2H), 7.34 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 8.9 Hz, 2H), 7.26–7.22 (m, 1H), 6.99 (d, *J* = 8.9 Hz, 2H), 6.95 (d, *J* = 8.9 Hz, 2H), 4.06 (t, *J* = 6.6 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 2.88–2.77 (m, 4H), 1.53–1.20 (m, 44H), 0.89 (t, *J* = 6.8 Hz, 6H). ¹³C-NMR (300 MHz, CDCl₃) δ 164.7, 164.5, 164.3, 163.8, 158.5, 156.3, 155.4, 151.3, 150.6, 143.9, 142.0, 141.0, 138.1, 132.4, 131.8, 131.2, 130.5, 129.9, 128.5, 128.3, 126.8, 124.7, 122.4, 122.1, 122.0, 120.9, 120.6, 120.4, 115.1, 114.4, 68.4, 68.3, 38.1, 31.9, 29.6, 29.6, 29.4, 29.3, 29.1, 26.0, 25.9, 22.7, 14.1. IR (KBr) 2918, 2850, 1732, 1626, 1605, 1286, 1253 cm⁻¹. EA for C₆₈H₈₃NO₈ (calculated 78.35% C, 8.03% H, 1.34% N) found 78.66% C 7.93% H, 1.30% N. UV-vis (CHCl₃, 4.8·10⁻⁵ M): λ_{max}: 264 nm (ε: 66.270·10³ M⁻¹cm⁻¹), λ_{max}: 359 nm (ε: 19.125·10³ M⁻¹cm⁻¹). MS (FAB⁺): *m/z*: 317, 420, 460, 1043.

Techniques. UV-Vis measurements were made using an ATI Unicam UV4 spectrophotometer. Infrared spectra for all the compounds were obtained using ATI Mattson (FTIR) and Nicolet Avatar 360 (FTIR) spectrophotometers in the 400–4000 cm⁻¹ spectral range. ¹H and ¹³C NMR spectra were recorded on Varian Unity 300 MHz,

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Bruker ARX-300 MHz, Bruker AV-400 MHz and AV-500 MHz spectrometers and samples were in solution. Elemental analyses were performed on a Perkin-Elmer 240 analyzer. Mass spectrometry studies (FAB⁺, MALDI⁺) were performed with a VG AutoSpec EBE and a Microflex (MALDI-ToF).

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