## **Electronic Supplementary Information for:**

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## A. Synthesis and characterization of chiral dopants.

**General**. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded using Bruker Avance 300, 400 or 500 spectrometers; chemical shifts ( $\delta$ ) are reported in parts per million (ppm) relative to TMS as internal standard. Lowand high-resolution EI mass spectra were recorded on a Waters/Micromass GC-TOF instrument; highresolution ESI mass spectra were recorded on a Applied Biosystems/MDS Sciex QSTAR XL QTOF instrument. Peaks are reported as *m/z* (% intensity relative to base peak). UV-vis and CD spectra were recorded either on a JASCO J-715 or OLIS DSM 20 spectrophotometers in hexanes. Melting points were obtained on a Fisher-Johns apparatus and are uncorrected. Flash chromatography was performed using 60 Å silica gel (Silicycle Inc.). Preparative chiral phase HPLC separations were performed using either a Daicel Chiralpak AS or a Chiralpak IC column (both 50 cm x 5 cm i.d.) using mixtures of EtOH/hexanes (AS) or EtOAc/hexanes (IC) as eluant with a flow rate of 50 mL min<sup>-1</sup>.

**Materials**. All chemicals and reagents were obtained from commercial sources unless otherwise noted. (*RS*)-2-Ethoxycarbonyl-5-methoxy-1-indanone (**3a**),<sup>1</sup> ethyl 2-(bromomethyl)-4-methoxybenzoate (**4a**),<sup>1</sup> 4-bromo-2-chloro-5-methylanisole (**8**),<sup>2</sup> (*RS*)-5,5'-diheptyloxy-2,2'-spirobiindan-1,1'-dione ((*RS*)-**2-H**),<sup>1</sup> and 1-bromoheptane-1,1- $d_2^{-1}$  were prepared according to literature procedures and shown to have the expected physical and spectral properties. The liquid crystal hosts 4-(4'-heptyl[1,1'-biphen]-4-yl)-1-hexylcyclohexanecarbonitrile (**NCB76**) and 2-(4-butyloxyphenyl)-5-octyloxypyrimidine (**PhP1**) were obtained from commercial sources; (*RS*)-4-[(4-methylhexyl)oxy]phenyl 4-decyloxybenzoate (**PhB**) and 2',3'-difluoro-4-heptyl-4''-nonyl-*p*-terphenyl (**DFT**) were synthesized according to published procedures and shown to have the expected physical and spectral properties.<sup>3,4</sup>

**5-Chloro-4-methoxy-2-methylbenzoic acid (9).** Under an argon atmosphere, a solution of *n*-BuLi (10 mL, 13.6 mmol, 1.36 M in *n*-hexane) was added dropwise to a solution **8** (3.0 g, 12.8 mmol) in THF-hexane-Et<sub>2</sub>O (6:1:1, 80 mL) cooled to -98 °C. Stirring was continued for 10 min at -98 °C, excess dry ice was added, and the mixture was allowed to warm to room temperature over 12 h. The solution was concentrated to 30 mL and acidified with 3% aq HCl and extracted with CHCl<sub>3</sub> (3×100 mL). The combined organic extracts were washed with H<sub>2</sub>O (2×100 mL), brine (2×100 mL), dried (MgSO<sub>4</sub>), filtered through a short pad of silica gel, and concentrated to give a white solid. The crude product was washed with hot hexane to give 2.0 g (78%) of **9** as white needles: mp 221-222 °C (lit. 220-222 °C);<sup>5 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.11 (s, 1H), 6.77 (s, 1H), 3.96 (s, 3H), 2.65 (s, 3H).

**Ethyl 5-chloro-4-methoxy-2-methylbenzoate (10).** A solution of **9** (4.0 g, 20 mmol) in absolute ethanol (150 mL) and H<sub>2</sub>SO<sub>4</sub> (2.5 mL) was heated to reflux overnight. The ethanol was then removed *in vacuo*, the residue was neutralized with sat aq NaHCO<sub>3</sub> and extracted with EtOAc (3×50 mL). The combined organic layers were washed brine (2×50 mL), dried (MgSO<sub>4</sub>), filtered through a short pad of silica gel and concentrated to give a white solid. The crude product was recrystallized from hexane to give 3.95 g (87%) of **10** as white needles: mp 104-105 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ 7.99 (s, 1H), 6.74 (s, 1H), 4.32 (q, *J* = 7 Hz, 2H), 3.94 (s, 3H), 2.61 (s, 3H), 1.38 (t, *J* = 7 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  14.6, 22.4, 56.5, 61.0, 114.9, 119.7, 122.7, 133.0, 142.0, 157.6, 166.4; MS (EI) *m/z* 230 ([M+2]<sup>+</sup>, 14), 228 (M<sup>+</sup>, 100), 199 (24), 183 (99), 155 (16); HRMS (EI) calcd for C<sub>11</sub>H<sub>13</sub>O<sub>3</sub>Cl: 228.0553, found 228.0548.

**Ethyl 2-(bromomethyl)-5-chloro-4-methoxybenzoate (4b).** Under an argon atmosphere, a solution of **10** (5.0 g, 21.9 mmol), *N*-bromosuccinimide (4.34 g, 24.1 mmol) and AIBN (10 mg, 0.06 mmol) in dry CCl<sub>4</sub> (200 mL) was heated to reflux. The reaction was monitored by <sup>1</sup>H NMR every 24 h to determine the percent of conversion, and an additional amount of NBS in equivalent proportion to the remaining starting material and AIBN (10 mg, 0.06 mmol) were added. The solution was heated to reflux under argon until a 5:1:1 ratio of **4b**:10:dibromo product was achieved. After cooling, the mixture was washed with sat aq NaHCO<sub>3</sub> (2×100 mL), brine (2×100 mL), dried (MgSO<sub>4</sub>) and concentrated. The crude product was purified by flash chromatography on silica gel (19:1 hexanes/EtOAc) to give 4.51 g (67%) of **4b** as white needles: mp 99-100 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ 8.04 (s, 1H), 6.98 (s, 1H), 4.97 (s, 2H), 4.38 (q, *J* = 7 Hz, 2H), 3.98 (s, 3H), 1.42 (t, *J* = 7 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ 14.3, 31.2, 56.4, 61.3, 114.5, 121.8, 122.4, 133.4, 140.2, 157.8, 165.1; MS (EI) *m/z* 308 ([M+2]<sup>+</sup>, 4), 306 (M<sup>+</sup>, 3), 263 (2), 261 (1), 227 (17), 199 (100), 182 (3), 154 (2); HRMS (EI) calcd for C<sub>11</sub>H<sub>12</sub>O<sub>3</sub>ClBr: 305.9658, found 305.9656.

**5-Bromo-6-methoxyindan (12).**<sup>6</sup> A solution of Br<sub>2</sub> (3.5 mL, 67.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (100 mL) was slowly added at 0 °C to a solution of 5-methoxyindan (**11**, 10.0 g, 67.5 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (100 mL). The solution was stirred at 0 °C for 2 h, then washed with sat aq NaHCO<sub>3</sub> ( $3 \times 100$  mL), brine ( $2 \times 100$  mL) and dried (MgSO<sub>4</sub>). Activated charcoal was added to decolorize the solution, which was filtered through a short pad of silica gel and concentrated to give 15.5 g (100%) of **12** as a colorless oil: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ 7.38 (s, 1H), 6.81 (s, 1H), 3.88 (s, 3H), 2.86 (m, 4H), 2.10 (m, 2H).

**5-Methoxy-6-methylindan (13).**<sup>7</sup> Under an argon atmosphere, a solution of *n*-BuLi (33 mL, 44.9 mmol, 1.36 M in *n*-hexane) was added dropwise to a solution of **12** (8.57g, 37.8 mmol) in dry THF (100 mL) cooled to -78 °C. Stirring was continued for 10 min at -78 °C and excess MeI (10 mL, 184 mmol) was added. The mixture was allowed to warm to room temperature and then concentrated, diluted with H<sub>2</sub>O (80 mL) and extracted with CHCl<sub>3</sub> (3×100 mL). The combined extracts were washed with brine (2×100 mL), dried (MgSO<sub>4</sub>) and concentrated. The crude product was distilled under reduced pressure to give 5.56 g (91%) of **13** as a colorless liquid: <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$ 7.02 (s, 1H), 6.76 (s, 1H), 3.83 (s, 3H), 2.90 (t, *J* = 7 Hz, 2H), 2.85 (t, *J* = 7 Hz, 2H), 2.21 (s, 3H), 2.09 (m, 2H).

**5-Methoxy-6-methyl-1-indanone (14).** Equal amounts of KMnO<sub>4</sub> and CuSO<sub>4</sub>·5H<sub>2</sub>O were ground together in a mortar. The resulting purple powder (32 g) was added to a solution of **13** (2.27 g, 14.0 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (200 mL) and the mixture was stirred vigorously under gentle reflux for 48 h. The resulting mixture was filtered through a pad of Celite and the residue washed successively with CH<sub>2</sub>Cl<sub>2</sub> (3×200 mL) and Et<sub>2</sub>O (3×200 mL). The combined solution and extracts were concentrated and the product was recrystallized from hexane to give 0.86 g (35%) of **14** as light yellow needles: mp 114-116 °C (lit. 114-115 °C);<sup>8 1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ 7.53 (s, 1H), 6.84 (s, 1H), 3.92 (s, 3H), 3.08 (t, *J* =

6 Hz, 2H), 2.66 (t, J = 6 Hz, 2H), 2.23 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  16.7, 26.1, 36.7, 55.9, 106.7, 125.5, 127.5, 130.1, 156.6, 164.1, 205.8; MS (EI) *m/z* 176 (M<sup>+</sup>, 100), 148 (27), 133 (25), 117 (7), 105 (7); HRMS (EI) calcd for C<sub>11</sub>H<sub>12</sub>O<sub>2</sub>: 176.0837, found 176.0835.



**Fig. S1**. NOESY spectrum of compound **14**. The peak at 6.9 ppm (aromatic proton at C-4) correlates to the peak at 3.9 ppm (methoxy protons) and the multiplet at 3.1 ppm (benzylic methylene protons).

(*RS*)-2-Ethoxycarbonyl-5-methoxy-6-methyl-1-indanone (3b). Under an argon atmosphere, a 60% dispersion of NaH (0.90 g, 22 mmol) was washed three times with Et<sub>2</sub>O. Diethyl carbonate (2 mL, 17 mmol) in dry toluene (20 mL) were then added and the resulting mixture was stirred and heated to reflux. A solution of 14 (1.20 g, 6.8 mmol) in dry toluene (20 mL) was added slowly to the refluxing mixture over 4h and the mixture was refluxed overnight. Acetic acid and H<sub>2</sub>O were added until all the solid dissolved and the aqueous layer reached a pH of 5. The aqueous layer was extracted with EtOAc (3×50 mL), the combined extracts were washed with brine (2×100 mL) and dried (MgSO<sub>4</sub>). Activated charcoal was added to decolorize the solution, which was filtered through a short pad of silica gel and concentrated to give 1.55 g (92%) of 3b as a yellow solid: mp 93-95 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.52 (s, 1H), 6.85 (s, 1H), 4.23 (q, *J* = 7 Hz, 2H), 3.92 (s, 3H), 3.67 (dd, *J* = 8 Hz, 2 Hz, 1H), 3.47 (dd, *J* = 17 Hz, 4Hz, 1H), 3.28 (dd, *J* = 17 Hz, 8Hz, 1H), 2.21 (s, 3H), 1.30 (t, *J* = 7 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  114.5, 16.7, 30.6, 53.8, 56.0, 61.9, 106.6, 126.3, 128.1, 128.2, 155.3, 164.6, 169.9, 198.1; MS (EI) *m/z* 248 (M<sup>+</sup>, 46), 202 (21), 174 (100), 147 (10), 115 (7); HRMS (EI) calcd for C<sub>14</sub>H<sub>16</sub>O<sub>4</sub>: 248.1049, found 248.1055.

**5-Iodo-6-methoxyindan (16).**<sup>9</sup> Under an argon atmosphere, a solution of *n*-BuLi (50 mL, 80 mmol, 1.6 M in *n*-hexane) was added dropwise to a solution of **12** (16 g, 70 mmol) in dry THF (100 mL) cooled to -78 °C. Stirring was continued for 10 min at -78 °C and the solution was quenched with a solution of I<sub>2</sub> (22 g, 87 mmol) in dry THF (50 mL). The mixture was allowed to warm to room temperature overnight, and then concentrated, diluted with 2% aq HCl (80 mL) and extracted with Et<sub>2</sub>O (3×100 mL). The combined extracts were washed with aq Na<sub>2</sub>SO<sub>3</sub> (2×80 mL), then washed with brine (2×100 mL), dried (MgSO<sub>4</sub>) and concentrated. The crude product was purified by flash chromatography on silica gel (5:1 hexanes/CHCl<sub>3</sub>) to give 19 g (96%) of **16** as a colorless oil: <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$ 7.62 (s, 1H), 6.76 (s, 1H), 3.87 (s, 3H), 2.87 (m, 4H), 2.11(m, 2H).

**6-Iodo-5-methoxy-1-indanone (17).** Equal amounts of KMnO<sub>4</sub> and CuSO<sub>4</sub>·5H<sub>2</sub>O were ground together in a mortar. The resulting purple powder (32 g) was added to a solution of **16** (7.67 g, 28.0 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (500 mL) and the mixture was stirred vigorously under gentle reflux for 72 h. More oxidant (32 g) was added and stirring was continued under gentle reflux. The reaction was monitored by <sup>1</sup>H NMR every 24 h to determine the extent of conversion until it reached approximately 60% and no further conversion was observed over 24 h. The mixture was filtered through Celite and the residue washed successively with CH<sub>2</sub>Cl<sub>2</sub> (3×300 mL) and Et<sub>2</sub>O (3×300 mL). The combined solution and extracts were concentrated and the product was purified by flash chromatography on silica gel (4:1 hexanes/EtOAc) to give 4.18 g (52%) of **17** as a white solid: mp 160-162 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.18(s, 1H), 6.86 (s, 1H), 3.98 (s, 3H), 3.08 (m, 2H), 2.70 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  26.2, 36.8, 57.1, 86.5, 107.9, 132.5, 135.6, 158.4, 163.1, 204.0; MS (EI) *m/z* 288 (M<sup>+</sup>, 100), 287 (12), 260 (21), 245 (14), 127 (13), 118 (13), 103 (21), 102 (12); HRMS (EI) calcd for C<sub>10</sub>H<sub>9</sub>O<sub>2</sub>I: 287.9647, found 287.9656.



**Fig. S2**. NOESY spectrum of compound **17**. The peak at 6.9 ppm (aromatic proton at C-4) correlates to the peak at 3.9 ppm (methoxy protons) and the multiplet at 3.1 ppm (benzylic methylene protons).

(*RS*)-2-Ethoxycarbonyl-6-iodo-5-methoxy-1-indanone (3c). The procedure used for the synthesis of **3b** was repeated with **17** (5.80 g, 20 mmol). Purification by flash chromatography on silica gel (4:1 hexanes/EtOAc) gave 6.30 g (87%) of **3c** as a pale yellow solid: mp 99-101 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.20 (s, 1H), 6.89 (s, 1H), 4.26 (q, *J* = 7 Hz, 2H), 4.00 (s, 3H), 3.73 (dd, *J* = 8 Hz, 4 Hz, 1H), 3.49 (dd, *J* = 17 Hz, 4Hz, 1H), 3.29 (dd, *J* = 17 Hz, 8Hz, 1H), 1.32 (t, *J* = 7 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  14.6, 30.6, 53.9, 57.2, 62.2, 87.0, 107.7, 130.5, 136.4, 157.1, 163.8, 169.4, 196.4; MS (EI) *m/z* 361 ([M+1]<sup>+</sup>, 11), 360 (M<sup>+</sup>, 97), 315 (17), 314 (49), 288 (16), 287 (48), 286 (100), 272 (12), 259 (14), 129 (14), 102 (18); HRMS (EI) calcd for: C<sub>13</sub>H<sub>13</sub>O<sub>4</sub>I: 359.9859, found 359.9843.

(*RS*)-2-Ethoxycarbonyl-2-(2-(ethoxycarbonyl)-4-chloro-5-methoxybenzyl)-5-methoxy -1-indanone (5a). Under an argon atmosphere, a 60% dispersion of NaH (0.36 g, 8.9 mmol) was washed three times with Et<sub>2</sub>O and suspended in dry DMF (10 mL). A solution of **3a** (1.61 g, 6.9 mmol) in dry DMF (10 mL) was then slowly added to the stirred suspension at room temperature. After gas evolution ceased, the mixture was heated to 60 °C for 1 h, and a solution of **4b** (2.21 g, 7.2 mmol) in dry DMF (30 mL) was slowly added. The mixture was stirred at 60 °C for 96 h, then quenched with water and extracted with CHCl<sub>3</sub> (3×100 mL). The combined extracts were washed with brine (3×100 mL), dried (MgSO<sub>4</sub>), filtered through a short pad of silica gel and concentrated to a yellow solid. The crude product was washed with hot hexane (20 mL) to give 2.38 g (75%) of **5a** as a white solid: mp 115-117 °C; <sup>1</sup>H NMR

(500 MHz, CDCl<sub>3</sub>)  $\delta$  7.91 (s, 1H), 7.67 (d, J = 9 Hz, 1H), 6.87 (dd, J = 9 Hz, 2 Hz, 1H), 6.81 (s, 1H), 6.76 (d, J = 2 Hz, 1H), 4.26 (m, 2H), 4.18 (m, 3H), 3.85 (s, 3H), 3.77 (s, 3H), 3.68 (d, J = 14 Hz, 1H), 3.54 (d, J = 18 Hz, 1H), 2.98 (d, J = 18 Hz, 1H), 1.37 (t, J = 7 Hz, 3H), 1.22 (t, J = 7 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  14.4, 14.7, 35.6, 35.7, 56.0, 56.5, 61.5, 62.2, 62.4, 109.6, 115.2, 116.3, 120.9, 124.3, 126.4, 128.9, 133.0, 140.1, 157.5, 157.6, 166.2, 166.6, 171.5, 201.5; MS (EI) *m/z* 460 (M<sup>+</sup>, 6), 414 (7), 369 (38), 357 (63), 341 (97), 313 (58), 306 (54), 297 (10), 233 (91), 187 (100); HRMS (EI) calcd for C<sub>24</sub>H<sub>25</sub>O<sub>7</sub>Cl: 460.1289, found 460.1292.

(*RS*)-6-Chloro-5,5'-dimethoxy-2,2'-spirobiindan-1,1'-dione (6a). A mixture of 5a (2.20 g, 4.78 mmol) in a 1:2:2 (v/v/v) solution of H<sub>2</sub>SO<sub>4</sub>, H<sub>2</sub>O, and EtOH (150 mL) was heated to reflux overnight, then cooled to room temperature and filtered. The solid residue was taken up in CHCl<sub>3</sub>, washed with sat aq NaHCO<sub>3</sub> (2×100 mL), brine (2×100 mL), dried (MgSO<sub>4</sub>) and concentrated to give a pale yellow solid. The crude product was washed with hot EtOAc to give 1.50 g (91%) of **6a** as a white solid: mp 243-244°C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ 7.75 (s, 1H), 7.68 (d, *J* = 8 Hz, 1H), 7.04 (s, 1H), 6.98 (d, *J* = 2 Hz, 1H), 6.94 (dd, *J* = 8 Hz, 2 Hz, 1H), 4.02 (s, 3H), 3.91 (s, 3H), 3.65 (d, *J* = 17 Hz, 1H), 3.63 (d, *J* = 17 Hz, 1H), 3.12 (d, *J* = 17 Hz, 1H), 3.11 (d, *J* = 17 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ 38.0, 38.1, 56.0, 56.9, 66.1, 108.6, 109.8, 116.3, 124.2, 126.5, 126.9, 128.7, 129.1, 155.2, 157.1, 160.9, 166.2, 200.5, 200.8; MS (EI) *m*/z 344 ([M+2]<sup>+</sup>, 16), 342 (M<sup>+</sup>, 100), 314 (39), 313 (13), 271 (4); HRMS (EI) calcd for C<sub>19</sub>H<sub>15</sub>O<sub>4</sub>Cl: 342.0659, found 342.0650.

(*RS*)-5,5'-Dimethoxy-6-methyl-2,2'-spirobiindan-1,1'-dione (6b). The procedure used for the synthesis of 5a was repeated with 3b (1.55 g, 6.2 mmol) and 4a (1.86 g, 6.8 mmol) to give (*RS*)-2-ethoxycarbonyl-2-(2-(ethoxycarbonyl)-5-methoxybenzyl)-5-methoxy-6-methyl-1-indanone (5b) as a crude product, which was used directly in the next step. The procedure used for the synthesis of 6a was then repeated and the crude yellow solid was washed with hot hexanes to give 1.15 g (57% over two steps) of 6b as a white solid: mp 223-225 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$ 7.69 (d, *J* = 8 Hz, 1H), 7.53 (s, 1H), 6.98 (d, *J* = 2 Hz, 1H), 6.94 (dd, *J* = 8 Hz, 2Hz, 1H), 3.95 (s, 3H), 3.92 (s, 3H), 3.66 (d, *J* = 17 Hz, 1H), 3.63 (d, *J* = 17 Hz, 1H), 3.11 (d, *J* = 17 Hz, 1H), 3.09 (d, *J* = 17 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ 16.7, 38.2, 38.3, 56.0 (2C), 65.9, 106.6, 109.8, 116.2, 126.5, 126.7, 128.1, 128.3, 129.1, 155.4, 157.3, 164.5, 166.0, 201.5, 201.6; MS (EI) *m*/*z* 322 (M<sup>+</sup>, 100), 294 (39), 279 (12), 251 (10); HRMS (EI) calcd for C<sub>20</sub>H<sub>18</sub>O<sub>4</sub>: 322.1205, found 322.1212.

(*RS*)-2-Ethoxycarbonyl-2-(2-(ethoxycarbonyl)-5-methoxybenzyl)-6-iodo-5-methoxy-1-indanone (5c). The procedure used for the synthesis of 5a was repeated with 3c (6.30 g, 17.5 mmol) and 4a (5.80 g, 20 mmol). Purification by flash chromatography on silica gel (4:1 hexanes/EtOAc) gave 8.89 g (92%) of 5c as a colorless oil: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  8.16 (s, 1H), 7.86 (d, *J* = 8 Hz, 1H), 6.72 (m, 3H), 4.30 (m, 2H), 4.17 (m, 3H), 3.91 (s, 3H), 3.72 (s, 3H), 3.68 (d, *J* = 14 Hz, 1H), 3.52 (d, *J* = 18 Hz, 1H), 1.35 (t, *J* = 7 Hz, 3H), 1.22 (t, *J* = 7 Hz, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  14.4, 14.7, 35.6, 36.1, 55.6, 57.1, 61.1, 62.3, 62.8, 86.6, 107.5, 113.2, 116.7, 123.7, 130.5, 133.3, 136.2, 141.4, 157.6, 162.4, 163.5, 167.6, 171.2, 199.8; MS (EI) *m/z* 553 ([M+1]<sup>+</sup>, 13), 552 (M<sup>+</sup>, 45), 507 (35), 506 (56), 479 (11), 461 (29), 460 (12), 449 (16), 434 (25), 433 (60), 432 (39), 405 (16), 359 (54), 314 (13), 313 (100), 307 (21), 306 (37), 277 (13), 263 (24), 235 (11), 233 (12), 205 (11), 194 (15), 192 (10), 187 (13), 165 (29), 163 (23), 149 (16), 148 (22); HRMS (EI) calcd for C<sub>24</sub>H<sub>25</sub>O<sub>7</sub>I: 552.0645, found 552.0663.

(*RS*)-6-Iodo-5,5'-dimethoxy-2,2'-spirobiindan-1,1'-dione (6c). The procedure used for the synthesis of 6a was repeated with 5c. The crude yellow solid was washed with hot EtOAc to give 5.95 g (85%) of 6c as a white solid: mp 235-237 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.16 (s, 1H), 7.67 (d, *J* = 8 Hz, 1H),

6.98 (d, J = 2 Hz, 1H), 6.93 (s, 1H), 6.93 (dd, J = 8 Hz, 2 Hz, 1H), 3.99 (s, 3H), 3.91 (s, 3H), 3.64 (d, J = 17 Hz, 1H), 3.61 (d, J = 17 Hz, 1H), 3.11 (d, J = 17 Hz, 1H), 3.07 (d, J = 17 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  38.1 (2C), 56.1, 57.1, 66.1, 86.9, 107.6, 109.8, 116.4, 126.9, 128.7, 130.6, 136.4, 157.1, 157.2, 163.5, 166.2, 200.0, 200.9; MS (EI) m/z 435 ([M+1]<sup>+</sup>, 19), 434 (M<sup>+</sup>, 100), 406 (20), 308 (25), 128 (11); HRMS (EI) calcd for C<sub>19</sub>H<sub>15</sub>O<sub>4</sub>I: 434.0015, found 434.0007.

(*RS*)-6-Chloro-5,5'-dihydroxy-2,2'-spirobiindan-1,1'-dione (7a). Under an argon atmosphere, 6a (0.79 g, 2.30 mmol) was added to a stirred solution of AlCl<sub>3</sub> (1.53 g, 11.50 mmol) in dry toluene (200 mL) and the mixture was refluxed overnight. After cooling, the mixture was quenched with H<sub>2</sub>O and the solid precipitate collected by filtration to give 0.51 g (71%) of 7a as a beige solid: mp > 300 °C (dec.); <sup>1</sup>H NMR (400 MHz, MeOD)  $\delta$ 7.64 (s, 2H), 7.52 (d, *J* = 8 Hz, 1H), 7.06 (s, 1H), 6.92 (d, *J* = 2 Hz, 1H), 6.86 (dd, *J* = 8 Hz, 2 Hz, 1H), 3.50 (d, *J* = 17 Hz, 1H), 3.47 (d, *J* = 17 Hz, 1H), 3.14 (d, *J* = 17 Hz, 1H), 3.13 (d, *J* = 17 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$ 37.5, 37.6, 65.9, 111.7, 112.6, 116.5, 122.6, 125.8, 126.4, 127.3, 127.9, 155.9, 158.2, 160.5, 165.4, 202.0, 202.5; MS (EI) *m/z* 316 ([M+2]<sup>+</sup>, 13), 314 (M<sup>+</sup>, 100), 286 (39), 285 (18), 251 (6), 223 (11); HRMS (EI) calcd for C<sub>17</sub>H<sub>11</sub>O<sub>4</sub>Cl: 314.0346, found 314.0358.

(*RS*)-5,5'-Dihydroxy-6-methyl-2,2'-spirobiindan-1,1'-dione (7b). The procedure described for the synthesis of 7a was repeated with 6b (0.50 g, 1.55 mmol) to give 420 mg (92%) of 7b as an off-white solid: mp > 300 °C (dec.); <sup>1</sup>H NMR (400 MHz, MeOD)  $\delta$ 7.56 (d, *J* = 8 Hz, 1H), 7.43 (s, 1H), 6.90 (d, *J* = 2 Hz, 1H), 6.89 (s, 1H), 6.84 (dd, *J* = 8 Hz, 2 Hz, 1H), 3.48 (d, *J* = 17 Hz, 1H), 3.45 (d, *J* = 17 Hz, 1H), 3.13 (d, *J* = 17 Hz, 1H), 3.10 (d, *J* = 17 Hz, 1H); MS (EI) *m*/*z* 294 (M<sup>+</sup>, 100), 267 (4), 249 (11), 238 (2), 194 (3); HRMS (EI) calcd for C<sub>18</sub>H<sub>14</sub>O<sub>4</sub>: 294.0892, found 294.0900.

**Purification of CuI.** CuI (2 g) was dissolved in sat aq KI (10 mL) and stirred with activated charcoal for 2 h. The solution was filtered and the residue washed with sat aq KI (2 mL). Water (50 mL) was added to the combined filtrate and the precipitated CuI was collected by filtration, washed with  $H_2O$ , EtOH and Et<sub>2</sub>O, and dried under vacuum overnight.

(*RS*)-6-Cyano-5,5'-dimethoxy-2,2'-spirobiindan-1,1'-dione (18). An oven-dried 25-mL Schlenk flask was purged with argon and charged with freshly purified CuI (88 mg, 0.46 mmol), 1,10-phenanthroline (166 mg, 0.92 mmol), 6c (1.00 g, 2.3 mmol) and KCN (0.30 g, 4.6 mmol). After a second argon purge, DMF (3 mL) was added to the flask. After purging with argon three more times, the mixture was stirred at room temperature for 4h and then heated to 110 °C for 24 h. The reaction mixture was cooled, diluted with CHCl<sub>3</sub>, and filtered through a short pad of silica gel. The solid residue was washed with CHCl<sub>3</sub>, the combined filtrate and extract concentrated, and the crude product was purified by flash chromatography on silica gel (4:1 CHCl<sub>3</sub>/EtOAc) to give 0.28 g (36%) of 18 as a pale yellow solid: mp 265 °C (dec.); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.99 (s, 1H), 7.70 (d, *J* = 8 Hz, 1H), 7.12 (s, 1H), 7.04 (d, *J* = 2 Hz, 1H), 6.97 (dd, *J* = 8 Hz, 2 Hz, 1H), 4.08 (s, 3H), 3.94 (s, 3H), 3.73 (d, *J* = 18 Hz, 1H), 3.69 (d, *J* = 17 Hz, 1H), 3.20 (d, *J* = 18 Hz, 1H), 3.15 (d, *J* = 17 Hz, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  37.9, 38.6, 56.2, 57.2, 66.2, 103.6, 108.7, 109.9, 115.8, 116.6, 127.1, 128.5, 129.1, 131.5, 157.0, 161.1, 166.3, 166.5, 199.8, 200.2; MS (EI) *m/z* 334 ([M+1]<sup>+</sup>, 19), 333 (M<sup>+</sup>, 100), 316 (10), 305 (49), 304 (25), 290 (10), 274 (14), 262 (16), 190 (17); HRMS (EI) calcd for C<sub>20</sub>H<sub>15</sub>NO<sub>4</sub>: 333.1001, found 333.1012.

(*RS*)-6-Cyano-5,5'-dihydroxy-2,2'-spirobiindan-1,1'-dione (7c). The procedure described for the synthesis of 7a was repeated with 18 (0.20 g, 0.6 mmol) to give 0.13 g (73%) of 7c as a beige solid: mp > 300 °C (dec.); <sup>1</sup>H NMR (400 MHz, MeOD)  $\delta$ 7.92 (s, 2H), 7.56 (d, *J* = 8 Hz, 1H), 7.12 (s, 1H), 6.93 (d, *J* = 2 Hz, 1H), 6.86 (dd, *J* = 8 Hz, 2 Hz, 1H), 3.55 (d, *J* = 18 Hz, 1H), 3.52 (d, *J* = 17 Hz, 1H), 3.23

(d, J = 18 Hz, 1H), 3.15 (d, J = 17 Hz, 1H); <sup>13</sup>C NMR (100 MHz, MeOD)  $\delta$  37.4, 37.8, 65.8, 101.2, 111.7, 112.6, 115.7, 116.6, 126.5, 127.0, 127.8, 130.7, 158.2, 161.3, 165.4, 166.0, 201.3, 201.9; MS (EI) m/z 306 ([M+1]<sup>+</sup>, 21), 305 (M<sup>+</sup>, 100), 288 (15), 277 (50), 276 (32), 260 (17), 248 (11), 165 (11),; HRMS (EI) calcd for C<sub>18</sub>H<sub>11</sub>NO<sub>4</sub>: 305.0688, found 305.0679.

(R)- and (S)-6-Chloro-5,5'-bis-(heptyloxy-1,1- $d_2$ )-2,2'-spirobiindan-1,1'-dione ((R)- and (S)-2-Cl). Under an argon atmosphere, a solution of 7a (63.2 mg, 0.2 mmol), Cs<sub>2</sub>CO<sub>3</sub> (260 mg, 0.8 mmol) and 1-bromoheptane-1,1-d<sub>2</sub> (217 mg, 1.2 mmol) in dry DMF (15 mL) was stirred overnight at room temperature and heated to 70 °C for 24 h. After cooling, the reaction mixture was poured into water and extracted with EtOAc (3×30 mL). The combined extracts were washed with brine (2×50 mL), dried (MgSO<sub>4</sub>) and concentrated. The crude product was purified by flash chromatography on silica gel (9:1 hexanes/EtOAc) and recrystallized from HPLC-grade hexane to give 85 mg (83%) of (RS)-2-Cl as a white solid: mp 154-156 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.75 (s, 1H), 7.67 (d, J = 8 Hz, 1H), 6.99 (s, 1H), 6.95 (d, J = 2 Hz, 1H), 6.92 (dd, J = 8 Hz, 2 Hz, 1H), 3.64 (d, J = 17 Hz, 1H), 3.61 (d, J = 17 Hz, 1H), 3.09 (d, J = 17 Hz, 1H), 3.07 (d, J = 17 Hz, 1H), 1.89 (quintet, J = 7 Hz, 2H), 1.82 (quintet, J = 7Hz, 2H), 1.32-1.58 (m, 16H), 0.92 (t, J = 7 Hz, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  14.4, 22.9, 26.1, 26.2, 28.9, 29.1, 29.2, 29.3, 32.0, 38.1, 38.2, 66.1, 109.2, 110.3, 116.6, 124.5, 126.5, 126.8, 128.5, 128.8, 155.1, 157.0, 160.6, 165.8, 200.6, 200.9; MS (EI) m/z 516 ([M+2]<sup>+</sup>, 12), 514 (M<sup>+</sup>, 100), 414 (15), 314 (6), 286 (8); HRMS (EI) calcd for  $C_{31}H_{35}D_4O_4Cl$ : 514.2788, found 514.2785; UV (hexanes)  $\lambda_{max}$  (log  $\varepsilon$ ) 206 (4.73), 269 (4.61), 289 (4.34), 297 (4.41), 307 (4.17). The racemic mixture was resolved by preparative chiral phase HPLC (Chiralpak AS, 2% EtOH in hexanes, 50 mL/min) to give (R)-2-Cl and (S)-2-Cl (first and second eluates, respectively) in optically pure form: CD ((R)-2-Cl, hexanes)  $\lambda_{ext}$  ( $\Delta \varepsilon$ ) 208 (9.5), 221 (-21.7), 231 (25.8), 258 (-10.2), 272 (6.3), 294(-4.3), 311 (-4.4), 321 (-5.1), 332 (-5.1), 344 (-2.9). Prior to doping into liquid crystal mixtures, the resolved compound was recrystallized from HPLC-grade hexanes after filtration through a 0.45 µm PTFE filter.

(R)- and (S)-5.5'-Bis-(heptyloxy-1,1- $d_2$ )-6-methyl-2.2'-spirobiindan-1,1'-dione ((R)- and (S)-2-Me). The procedure described for the synthesis of (RS)-2-Cl was repeated with 7b (150 mg, 0.51 mmol) and 1-bromoheptane-1,1- $d_2$  (320 mg, 1.77 mmol) to give 140 mg (55%) of (RS)-2-Me as a white solid: mp 139-141 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.67 (d, J = 8 Hz, 1H), 7.52 (s, 1H), 6.95 (d, J = 2 Hz, 1H), 6.92 (dd, J = 8 Hz, 2 Hz, 1H), 6.89 (s, 1H), 3.64 (d, J = 17 Hz, 1H), 3.61 (d, J = 17 Hz, 1H), 3.09 (d, J = 17 Hz, 1H),17 Hz, 1H), 3.07 (d, J = 17 Hz, 1H), 2.23 (s, 3H), 1.80-1.87 (m, 4H), 1.33-1.50 (m, 16H), 0.92 (t, J = 7Hz, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  14.4, 16.8, 22.9, 26.2, 26.3, 29.2, 29.3, 32.1, 38.3, 38.4, 65.8, 107.2, 110.3, 116.5, 126.5, 126.7, 128.1, 128.2, 128.9, 155.4, 157.2, 164.1, 165.7, 201.6, 201.7; MS (EI) m/z 494 (M<sup>+</sup>, 100.0), 394 (2.8), 366 (1.5), 266 (2.9); HRMS (EI) calcd for C<sub>32</sub>H<sub>38</sub>D<sub>4</sub>O<sub>4</sub>: 494.3334, found 494.3346; UV (hexanes)  $\lambda_{max}$  (log  $\varepsilon$ ) 208 (4.68), 221 (4.58), 269 (4.58), 290 (4.35), 296 (4.39). The racemic mixture was resolved by preparative chiral phase HPLC (Chiralpak AS, 2% EtOH in hexanes, 50 mL/min) to give (R)-2-Me and (S)-2-Me (first and second eluates, respectively) in optically pure form: CD ((R)-2-Me, hexanes)  $\lambda_{ext}$  ( $\Delta \epsilon$ ) 207 (10.4), 221 (-21.1), 230 (21.8), 258 (-9.8), 271 (5.4), 294 (-3.0), 301 (-0.9), 308 (-4.3), 318 (-5.4), 332 (-5.3), 343 (-3.1). Prior to doping into liquid crystal mixtures, the resolved compound was recrystallized from HPLC-grade hexanes after filtration through a 0.45 µm PTFE filter.

(*R*)- and (*S*)-6-Cyano-5,5'-diheptyloxy-2,2'-spirobiindan-1,1'-dione ((*R*)- and (*S*)-2-CN). The procedure described with the synthesis of (*RS*)-2-Cl was repeated with 7c (100 mg, 0.33 mmol) and 1-bromoheptane (500 mg, 2.76 mmol). The crude product was purified by flash chromatography on silica gel (5:1 hexanes/EtOAc) and recrystallized from HPLC-grade hexanes/EtOAc to give 115 mg (70%) of (*RS*)-2-CN as a white solid: mp 182-184 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$ 7.97 (s, 1H), 7.67

(d, J = 8 Hz, 1H), 7.08 (s, 1H), 6.97 (s, 1H), 6.94 (d, J = 8 Hz), 4.22 (t, J = 6 Hz), 4.08 (t, J = 6 Hz), 3.70 (d, J = 17 Hz, 1H), 3.67 (d, J = 17 Hz, 1H), 3.17 (d, J = 17 Hz, 1H), 3.12 (d, J = 17 Hz, 1H), 1.94 (quintet, J = 7 Hz, 2H), 1.85 (quintet, J = 7 Hz, 2H), 1.32-1.56 (m, 16H), 0.94 (t, J = 7 Hz, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  14.5, 23.0, 26.2, 26.3, 29.1, 29.3, 29.4, 32.08, 32.15, 37.9, 38.6, 66.1, 69.1, 70.4, 103.6, 109.3, 110.4, 115.8, 116.9, 127.0, 128.2, 128.8, 131.4, 157.1, 161.0, 165.9, 166.1, 200.0, 200.3; MS (EI) *m*/*z* 502 ([M+1]<sup>+</sup>, 28), 501 (M<sup>+</sup>, 100), 404 (13), 403 (47), 306 (11), 305 (40), 277 (30), 276 (18); HRMS (EI) calcd for C<sub>32</sub>H<sub>39</sub>NO<sub>4</sub>: 501.2879, found 501.2865; UV (hexanes)  $\lambda_{max}$  (log  $\varepsilon$ ) 226 (4.50), 271 (4.53), 290 (4.19), 298 (4.24), 309 (3.87). The racemic mixture was resolved by preparative chiral phase HPLC (Chiralpak IC, 40% EtOAc/hexanes, 50 mL/min) to give (*S*)-**2-CN** and (*R*)-**2-CN** (first and second eluates, respectively) in optically pure form: CD ((*R*)-**2-CN**, hexanes)  $\lambda_{ext}$  ( $\Delta \varepsilon$ ) 223 (– 18.1), 234 (20.3), 260 (–9.2), 274 (2.1), 299(–6.6), 310 (–4.7), 322 (–3.8), 328 (–3.6), 338 (–3.6). Prior to doping into liquid crystal mixtures, the resolved compound was recrystallized from HPLC-grade hexanes/EtOAc after filtration through a 0.45 µm PTFE filter.

(R)- and (S)-5,5'-Diheptyloxy-6-nitro-2,2'-spirobiindan-1,1'-dione ((R)- and (S)-2-NO<sub>2</sub>). A mixture of (RS)-2-H (476 mg, 1.0 mmol) and Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (242 mg, 1.0 mmol) in acetic anhydride (10 mL) was stirred at room temperature, producing an exothermic reaction. After the evolution of heat ended, the mixture was stirred overnight at 40 °C. The mixture was then poured onto ice and extracted with Et<sub>2</sub>O (3×100 mL). The combined extracts were washed with H<sub>2</sub>O (2×100 mL), sat aq NaHCO<sub>3</sub> (2×100 mL), brine (2×50 mL), dried (MgSO<sub>4</sub>) and concentrated. The crude product was purified by flash chromatography on silica gel (6:1 hexanes/EtOAc, third eluant) and recrystallized from HPLC-grade hexanes/EtOAc to give 170 mg (33%) of (RS)-2-NO<sub>2</sub> as a white solid: mp 166-168 °C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta 8.17$  (s, 1H), 7.69 (d, J = 8 Hz, 1H), 7.18 (s, 1H), 6.98 (s, 1H), 6.96 (d, J = 8 Hz, 1H), 4.23 (t, J = 6 Hz, 2H), 4.08 (t, J = 6 Hz, 2H), 3.71 (d, J = 17 Hz, 1H), 3.68 (d, J = 17 Hz, 1H), 3.18 (d, J = 17 Hz, = 17 Hz, 1H), 3.14 (d, J = 17 Hz, 1H), 1.90 (quintet, J = 7 Hz, 2H), 1.85 (quintet, J = 7 Hz, 2H), 1.35-1.55 (m, 16H), 0.94 (t, J = 7 Hz, 6H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  14.5, 23.0, 26.1, 26.3, 29.1, 29.3, 29.4, 32.1, 32.2, 37.9, 38.4, 66.3, 69.1, 70.8, 110.4, 111.0, 117.0, 122.5, 127.1, 127.8, 128.2, 141.1, 157.0, 157.9, 159.5, 166.1, 200.2, 200.3; MS (EI) *m/z* 522 ([M+1]<sup>+</sup>, 34), 521 (M<sup>+</sup>, 100), 423 (28), 325 (26), 297 (16); HRMS (EI) calcd for  $C_{31}H_{39}NO_6$ : 521.2777, found 521.2763; UV (hexanes)  $\lambda_{max}$  (log  $\varepsilon$ ) 220 (4.18), 272 (4.42), 290 (4.10), 298 (4.11). The racemic mixture was resolved by preparative chiral phase HPLC (Chiralpak IC, 40% EtOAc/hexanes, 50 mL/min) to give (S)-2-NO<sub>2</sub> and (R)-2-NO<sub>2</sub> (first and second eluates, respectively) in optically pure form: CD ((R)-2-NO<sub>2</sub>, hexanes)  $\lambda_{ext}$  ( $\Delta\epsilon$ ) 218 (-9.6), 236 (4.9), 258 (-5.0), 266 (-5.1), 274 (2.3), 299(-5.0), 314 (-4.3), 320 (-4.3). Prior to doping into liquid crystal mixtures, the resolved compound was recrystallized from HPLC-grade hexanes/EtOAc after filtration through a 0.45 µm PTFE filter.

## **B.** Ferroelectric polarization measurements.

Texture analyses were performed using a Nikon Eclipse E600 POL polarized microscope fitted with a Linkam LTS 350 hot stage and TMS 93 temperature controller. Spontaneous polarization measurements were performed as a function of temperature by the triangular wave method ( $6V/\mu$ m, 100 Hz) using a LC Vision LCAS-1 analysis system in conjunction with the Linkam hot stage.<sup>10</sup> Parallel rubbed polyimide-coated ITO glass cells (4 µm spacing, 0.16 cm<sup>2</sup> addressed area) supplied by E.H.C. Co. (Tokyo, Japan) were used for the measurements. Good alignment was obtained by slow cooling (0.5-2 K/min) of the filled cells from the isotropic phase while applying a 100 Hz AC field ( $6V/\mu$ m). The sign of  $P_S$  along the polar axis was assigned from the relative configuration of the electrical field and the switching position of the sample according to the established convention.<sup>11</sup> Tilt angles ( $\theta$ ) were measured as a function of temperature between crossed polarizers as half the rotation between two extinction positions corresponding to opposite polarization orientations.



**Fig. S3**. Circular dichroism spectra of (a) (*R*)-**2-Me**, (b) (*R*)-**2-CN**, and (c) (*R*)-**2-NO**<sub>2</sub>; all spectra were acquired at a concentration of  $2.0 \times 10^{-5}$  M in hexanes.



**Fig. S4**. Reduced polarization  $P_0$  vs. dopant mole fraction  $x_d$  in the liquid crystal hosts **PhP1** (•), **PhB** ( $\bigcirc$ ), **NCB76** (•) and **DFT** ( $\square$ ) measured at  $T-T_C = -10$  K: (a) (R)-2-Cl, (b) (R)-2-Me, (c) (R)-2-CN, and (d) (R)-2-NO<sub>2</sub>.



**Fig. S5**. Polarization inversion temperature  $T_i - T_C$  vs. dopant mole fraction  $x_d$  for (*R*)-2-Me in the liquid crystal host NCB76.

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