Supporting Information

Stereochemical Control of Nonamphiphilic Lyotropic Liquid Crystals: Chiral Nematic Phase of Assemblies Separated by Six Nanometers of Aqueous Solvents

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**Chemicals.** All reagent grade starting materials were obtained from commercial supplies and used as received. Anhydrous solvents were purchased from Sigma-Aldrich. Water used to prepare all buffers and solutions had resistivity of 18 MΩ cm (Millipore, Billerica, MA).

**General procedure.** All air sensitive reactions were performed in oven dried glassware under an atmosphere of argon unless otherwise notified. Analytical thin layer chromatography was performed on EM silica gel 60 F254 glass plates (0.25 mm). Visualization of analytical thin layer chromatography was achieved using UV absorbance (254 nm), KMnO₄, and ceric ammonium molybdate stains. Flash column chromatography was performed using SiliaFlash P60 silica gel (40-60 Å) from SiliCycle, Inc (Quebec City, Quebec, Canada). 1D ¹H and ¹³C NMR spectra were recorded on a Bruker Advance DPX-300 spectrometer. Chemical shifts are reported in ppm, using tetramethylsilane as the internal standard. ROESY spectra were acquired at a temperature of 300 K recorded on a Bruker AVANCE-II NMR spectrometer equipped with a 5 mm TCI CryoProbe with z-gradient operating at a frequency of 600.13 MHz for ¹H. For 2D ROESY experiments,¹ a total of 320 t₁ increments of 2048 complex points with 16 scans each were collected. The ROESY spin lock was set to 250 ms for all experiments. NMR data were processed using Bruker TOPSPIN 3.0 software by setting forward linear prediction to 640 points and zero filled to 2048 points for F1 dimension. Mass spectra were measured using a MAT 95 XP mass spectrometer, carried out by the Mass Spectroscopy Facility at Indiana University.

**Synthetic procedures and spectral data:**

![1,2:4,5-bis-acetal 2a](image)

1,2:4,5-bis-acetal 2a: To a stirred suspension of L-arabitol (3.008 g, 19.37 mmol) in anhydrous THF (48 mL) was added 2,2-dimethoxypropane (5.1 mL, 41 mmol) at ambient temperature. The
reaction mixture was refluxed for 15 min and then L-(-)-camphorsulfonic acid (461.2 mg, 1.946 mmol) was added at refluxing temperature. After refluxing for another 5 minutes, the reaction was quenched with 2 M NaOH (11 mL) at refluxing temperature. The solvent was removed under reduced pressure and residue was extracted with diethyl ether (11 mL x 3). The combined organic layer was dried over MgSO$_4$, filtered, and concentrated under reduced pressure. The colorless oil residue was dissolved in anhydrous methylene chloride (48 mL) and treated with Et$_3$N (2.9 mL). The reaction mixture was then brought to reflux and succinic anhydride (488.9 mg, 4.837 mmol) was added. After refluxing for 5 h, the reaction was quenched with saturated aqueous NaHCO$_3$ (7.0 mL) at refluxing temperature. The mixture was cooled to ambient temperature and the organic layer was dried over MgSO$_4$, filtered, and concentrated under reduced pressure. Flash column chromatography (SiO$_2$; hexane:ethyl acetate, 5:1 to 3:1) provided 1,2:4,5-bis-acetal 2a: (3.0925 g, 69 %) as a pale yellow oil. TLC $R_f = 0.48$ (hexane:ethyl acetate, 1:1). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 4.26 (ddd, 1H, $J = 6.6, 6.6, 4.2$ Hz), 4.13 (m, 2H), 4.04 (dd, 1H, $J = 10.8, 5.4$ Hz), 3.98 (m, 1H), 3.92 (dd, 1H, $J = 8.3, 6.6$ Hz), 3.43 (m, 1H), 2.29 (d, 1H, $J = 6.3$ Hz), 1.45 (s, 3H), 1.41 (s, 3H), 1.39 (s, 3H), 1.36 (s, 3H).

Imidazolyl thiocarbonyl derivative 3a: To a stirred solution of 1,2:4,5-bis-acetal 2a (2.241 g, 9.657 mmol) in anhydrous 1,2-dichloroethane (32 mL) was added 1,1’-thiocarbonyl diimidazole (2.249 g, 11.36 mmol) at ambient temperature. The resulting brown solution was refluxed for 7 h and then cooled to ambient temperature. The solvent was removed under reduced pressure. Flash column chromatography (SiO$_2$; hexane:ethyl acetate 3:1) provided imidazolyl thiocarbonyl
derivative 3a (3.17 g, 96 %) as a yellow oil. TLC R<sub>f</sub> = 0.36 (hexane:ethyl acetate, 1:1). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 8.34 (m, 1H), 7.63 (m, 1H), 7.03 (m, 1H), 5.88 (dd, 1H, J = 5.7, 3.3 Hz), 4.45(m, 2H), 4.08 (m, 3H), 3.83 (dd, 1H, J = 9.0, 6.0 Hz), 1.39 (s, 3H), 1.33 (s, 9H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 184.5, 137.0, 131.0, 118.0, 109.7, 109.6, 80.9, 74.8, 74.5, 65.8, 65.2, 26.3, 26.1, 25.0, 24.9. HRMS: Cacl. for (M + H)<sup>+</sup>: 343.1328, found: 343.1314.

1,2:4,5-bis-acetal 4a: Benzoyl peroxide (202.9 mg, 0.821 mmol) was added to a stirred solution of imidazolyl thiocarbonyl derivative 3a (1.444 g, 4.223 mmol) in triethylsilane (25 mL) at refluxing temperature. The reaction mixture was refluxed for 2 h during which similar amount of benzoyl peroxide was added after 30 min, 60 min, and 90 min, respectively. The solvent was then removed under reduced pressure. Flash column chromatography (SiO<sub>2</sub>; hexane:ethyl acetate, 3:1) provided 1,2:4,5-bis-acetal 4a as a light yellow oil which contained some aromatic impurities but was good enough to be carried on. TLC R<sub>f</sub> = 0.75 (hexane:ethyl acetate, 1:1). <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>): δ 4.21 (m, 2H), 4.11 (dd, 2H, J = 8.1, 6.0 Hz), 3.57 (dd, 2H, J = 7.8, 7.2 Hz), 1.80 (t, 2H, J = 6.4 Hz), 1.41 (s, 6H), 1.36 (s, 6H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ 108.8, 73.6, 69.8, 38.4, 27.0, 25.7. HRMS: Cacl. for (M + H)<sup>+</sup>: 217.1434, found: 217.1445.

(2R,4R)-pentane-1,2,4,5-tetraol 5a: 0.5 M H<sub>2</sub>SO<sub>4</sub> (5.4 mL, 2.7 mmol) was added to a stirred solution of 1,2:4,5-bis-acetal 4a (1.508 g, contained some aromatic impurites) in ethanol (5.4 mL) at ambient temperature. The reaction mixture was refluxed for 4.5 h and then BaCO<sub>3</sub> was added to adjust the pH to 7. The resulting milky mixture was filtered and the white solid thus collected.
was heated with MeOH (5 mL) at 50 °C for 10 min. The combined filtrate was concentrated under reduced pressure. Flash column chromatography (SiO₂; DCM:MeOH, 7:3) provided (2R,4R)-pentane-1,2,4,5-tetraol 5a (546.7 mg, 95 % over 2 steps) as a white solid. TLC Rf = 0.25 (DCM:MeOH, 7:3). ¹H NMR (300 MHz, MeOH-d₄): δ 3.85 (m, 2H), 3.47 (m, 4H), 1.52 (dd, 2H, J = 7.1, 5.6 Hz); ¹³C NMR (75 MHz, MeOH-d₄): δ 70.1, 67.9, 38.1. HRMS: Cacld. for (M + Na)+: 159.0633, found: 159.0631.

(2R,4R)-2,4-Dihydroxypentane-1,5-diyl Bis-(2,4,6-triisopropyl-1-benzenesulfonate) 6a: To a stirred solution of (2R,4R)-Pentane-1,2,4,5-tetraol 5a (92.2 mg, 0.678 mmol) in anhydrous pyridine (0.55 mL) was added 2,4,6-triisopropyl benzenesulfonyl chloride (529.2 mg, 1.695 mmol) at 0 °C. The pale yellow cloudy mixture was stirred at ambient temperature for 13 h and then the solvent removed under reduced pressure. Flash column chromatography (SiO₂; hexane:acetone, 3:1) provided (2R,4R)-2,4-Dihydroxypentane-1,5-diyl bis-(2,4,6-triisopropyl-1-benzenesulfonate) 6a (325.5 mg, 72 %) as a white fluffy solid. TLC Rf = 0.70 (hexane:ethyl acetate, 1:1). ¹H NMR (300 MHz, CDCl₃): δ 7.20 (s, 4H), 4.23 (m, 2H), 4.10 (m, 6H), 3.98 (dd, 2H, J = 10.2, 6.9 Hz), 2.92 (septet, 2H, J = 6.9 Hz), 2.87 (br, 2H), 1.64 (t, 2H, J = 5.7 Hz), 1.27 (d, 24H, J = 6.9 Hz), 1.26 (d, 12H, J = 6.9 Hz).

Acetonide 7a: To a stirred solution of (2R,4R)-2,4-Dihydroxypentane-1,5-diyl bis-(2,4,6-triisopropyl-1-benzenesulfonate) 6a (472.8 mg, 0.708 mmol) in acetone (7.1 mL) was added 2,2-dimethoxypropane (0.89 mL, 7.1 mmol) and p-toluenesulfonic acid (18.2 mg, 0.094 mmol) at ambient temperature. The resulting colorless solution was stirred at ambient temperature...
overnight (23 h). The reaction mixture was quenched with aqueous saturated NaHCO₃ (8 mL) and the solvent removed under reduced pressure. The residue was extracted with methylene chloride (4 mL × 3) and the combined organic layer dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash column chromatography (SiO₂; hexane:ethyl acetate, 3:1) provided acetonide 7a (473.4 mg, 94 %) as a pale yellow solid. TLC Rf = 0.64 (hexane:ethyl acetate, 3:1). ¹H NMR (300 MHz, CDCl₃): δ 7.19 (s, 4H), 4.13 (septet, 4H, J = 6.9 Hz), 4.03 (m, 6H), 2.92 (septet, 2H, J = 6.9 Hz), 1.66 (t, 2H, J = 7.7 Hz), 1.26 (d, 36H, J = 6.9 Hz), 1.22 (s, 6H); HRMS: Cacld. for (M + Na)⁺: 731.3627, found: 731.3651.

Diiodide 8a: To a stirred solution of (2R,4R)-2,4-Dihydroxypentane-1,5-diyl bis-(2,4,6-triisopropyl-1-benzenesulfonate) 7a (938.1 mg, 1.325 mmol) in 2-butanone (13 mL) was added sodium iodide (1.985 g, 13.24 mmol) followed by anhydrous pyridine (0.54 mL, 6.7 mmol) at ambient temperature. The resulting light yellow suspension was refluxed overnight (25 h). The reaction mixture was cooled to ambient temperature and the solvent was removed under reduced pressure. The residue was treated with water (10 mL), extracted with ethyl acetate (10 mL × 4), dried over MgSO₄, filtered and concentrated under reduced pressure. Flash column chromatography (SiO₂; hexane to hexane:ethyl acetate, 15:1) provided diiodide 8a (484.7 mg, 92 %) as a colorless oil. TLC Rf = 0.55 (hexane:ethyl acetate, 10:1). ¹H NMR (300 MHz, CDCl₃): δ 3.85 (m, 2H), 3.18 (m, 4H), 1.78 (t, 2H, J = 3.3 Hz), 1.39 (s, 6H); ¹³C NMR (75 MHz, CDCl₃): δ 101.6 (C), 66.9 (CH), 38.6 (CH₂), 24.6 (CH₃), 8.9 (CH₂).
**Diiodo diol 9a:** To a stirred solution of diiodide 8a (100.9 mg, 0.255 mmol) in THF (2.5 mL) was added 1N HCl (2.6 mL) at ambient temperature. The reaction mixture was stirred at ambient temperature for 45 min. NaHCO₃ (powder) was added to adjust pH to 7 and then the organic solvent was removed under reduced pressure. The residue was extracted with ethyl acetate (3 mL × 4), dried over MgSO₄, filtered and concentrated under reduced pressure. Diiodo diol 9a was obtained as a white solid which was in good enough quality to be carried on without further purification. TLC Rₓ = 0.14 (hexane:ethyl acetate, 3:1). \(^1\)H NMR (300 MHz, CDCl₃): δ 3.94 (m, 2H), 3.39 (dd, 2H, \(J = 10.2, 4.5\) Hz), 3.28 (dd, 2H, \(J = 10.2, 6.9\) Hz), 2.54 (d, 2H, \(J = 4.5\) Hz), 1.86 (dd, 2H, \(J = 6.3, 5.4\) Hz); \(^1^3\)C NMR (75 MHz, CDCl₃): δ 68.4, 41.6, 15.0.

**Hydroxyacetophenone 10a:** To a stirred solution of diiodo diol 9a (90.7 mg, 0.255 mmol, assuming quantative yield from previous step) in 2-propanol (5.1 mL) was added 2,6-dihydroxyacetophenone (240.3 mg, 1.532 mmol) at ambient temperature. The resulting yellow solution was heated to reflux and a mixture of KOH (118.5 mg, 1.901 mmol) in water (0.20 mL) and 2-propanol (0.26 mL) was added. The reaction mixture was refluxed for 24 h, cooled to ambient temperature, and then concentrated under reduced pressure. The dark brown residue was treated with ethyl acetate (5 mL) and filtered. The filtrate was concentrated under reduced pressure. Flash column chromatography (SiO₂; hexane:ethyl acetate, 5:1 to 3:1 to 1:1 to 1:2) provided hydroxyacetophenone 10a (87.9 mg, 85 %) as a light yellow solid. TLC Rₓ = 0.24 (hexane:ethyl acetate, 1:2). \(^1\)H NMR (300 MHz, DMSO-d6): δ 12.21 (br, 2H), 7.34 (t, 2H, \(J = 8.3\) Hz), 6.56 (d, 2H, \(J = 8.4\) Hz), 6.49 (d, 2H, \(J = 8.1\) Hz), 4.97 (d, 2H, \(J = 5.4\) Hz), 4.08 (m, 2H),...
3.99 (m, 4H), 2.62 (s, 6H), 1.64 (t, 2H, \( J = 6.3 \) Hz); HRMS: Cacl. for (M + Na): 427.1369, found: 427.1354.

![Diester](image)

Diester 11a: To a freshly prepared NaOMe solution (206.8 mg of sodium cube in 18 mL of anhydrous MeOH) was added anhydrous diethyl ether (36 mL) followed by dimethyl oxalate (1.065 g, 8.931 mmol). After the solid disappeared, hydroxylacetophenone 10b (360.3 mg, 0.892 mmol) was added to the reaction mixture. The resulting yellow solution was refluxed for 24 h, cooled to ambient temperature, and then concentrated under reduced pressure. The yellow solid residue was treated with water (45 mL) and acidified with concentrated HCl (0.20 mL) until pH ~ 2. The yellow precipitated was filtered off, dissolved in anhydrous MeOH, and treated with concentrated HCl (2.7 mL). The reaction mixture was refluxed for 40 min and then cooled to ambient temperature. The pale brown precipitate was filter off and found to be pure to be carried on (191.7 mg, 40 %). TLC \( R_f = 0.10 \) (ethyl acetate:ethanol, 9:1). \(^1\)H NMR (300 MHz, DMSO-d6): \( \delta \) 7.73 (t, 2H, \( J = 8.4 \) Hz), 7.22 (d, 2H, \( J = 8.4 \) Hz), 7.09 (d, 2H, \( J = 8.4 \) Hz), 6.75 (s, 2H), 4.07 (m, 6H), 3.92 (s, 6H), 3.50 (br, 2H), 1.78 (t, 2H, \( J = 6.0 \) Hz); \(^{13}\)C NMR (75 MHz, DMSO-d6): \( \delta \) 176.6, 160.6, 158.6, 157.1, 150.1, 135.4, 115.4, 114.8, 110.4, 109.7, 74.4, 65.2, 53.4, 37.2. HRMS: Cacl. for (M + Na): 563.1165, found: 563.1149.

![Diester](image)

5´DSCG-(R,R)-diviol 1a: To a stirred suspension of diester 11a (101.5 mg, 0.188 mmol) in ethanol (9.4 mL) was added aqueous NaOH solution (2.5 M, 0.15 mL, 0.38 mmol) at ambient temperature. The reaction mixture was refluxed for 10 min, cooled to ambient temperature, and
then at 0 °C for 20 min. After filtration and drying under high vacuum for 24 h, 1a was obtained as brown solid (91.2 mg, 89%). $^1$H NMR (300 MHz, D$_2$O): $\delta$ 7.60 (t, 2H, $J = 8.4$ Hz), 7.08 (d, 2H, $J = 8.4$ Hz), 6.89 (d, 2H, $J = 8.4$ Hz), 6.64 (s, 2H), 4.37 (m, 2H), 4.13 (m, 4H), 1.97 (t, 2H, $J = 6.2$ Hz); $^{13}$C NMR (75 MHz, D$_2$O): $\delta$ 181.4, 165.1, 157.2, 157.2, 156.2, 135.2, 113.0, 112.0, 110.6, 108.2, 73.1, 65.5, 35.5. HRMS: Cacl. for (M + Na)$^+$: 579.0483, found: 579.0491.

1,2:4,5-bis-acetal 2b: To a stirred suspension of D-arabitol (3.004 g, 19.55 mmol) in anhydrous THF (49 mL) was added 2,2-dimethoxypropane (5.1 mL, 41 mmol) at ambient temperature. The reaction mixture was refluxed for 15 min and then L-(-)-camphorsulfonic acid (463.7 mg, 1.956 mmol) was added at refluxing temperature. After refluxing for another 3 minutes, the reaction was quenched with 2 M NaOH (11 mL) at refluxing temperature. The solvent was removed under reduced pressure and residue was extracted with diethyl ether (11 mL $\times$ 3). The combined organic layer was dried over MgSO$_4$, filtered, and concentrated under reduced pressure. The colorless oil residue was dissolved in anhydrous methylene chloride (49 mL) and treated with Et$_3$N (3.0 mL). The reaction mixture was then brought to reflux and succinic anhydride (504.6 mg, 4.891 mmol) was added. After refluxing for 6.5 h, the reaction was quenched with saturated aqueous NaHCO$_3$ (8.0 mL) at refluxing temperature. The mixture was cooled to ambient temperature and the organic layer was dried over MgSO$_4$, filtered, and concentrated under reduced pressure. Flash column chromatography (SiO$_2$; hexane:ethyl acetate, 5:1 to 3:1) provided 1,2:4,5-bis-acetal 2b: (2.7085 g, 60 %) as a pale yellow oil. TLC $R_f = 0.49$ (hexane:ethyl acetate, 1:1). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 4.26 (ddd, 1H, $J = 6.6, 6.6, 4.2$ Hz), 4.13 (m, 2H), 4.04 (dd,
1H, J = 10.8, 5.4 Hz), 3.98 (m, 1H), 3.92 (dd, 1H, \( J = 8.3, 6.6 \) Hz), 3.43(m, 1H), 2.29 (d, 1H, \( J = 6.3 \) Hz), 1.45 (s, 3H), 1.41 (s, 3H), 1.39 (s, 3H), 1.36 (s, 3H).

**Imidazolyl thiocarbonyl derivative 3b:** To a stirred solution of 1,2:4,5-bis-acetal 2b (2.678 g, 11.54 mmol) in anhydrous 1,2-dichloroethane (38 mL) was added 1,1'-thiocarbonyl diimidazole (2.8955g, 14.62 mmol) at ambient temperature. The resulting brown solution was refluxed for 7 h and then cooled to ambient temperature. The solvent was removed under reduced pressure. Flash column chromatography (SiO\(_2\); hexane:ethyl acetate 3:1 to 1:1) provided imidazolyl thiocarbonyl derivative \( 3b \) (3.8679 g, 98 %) as a yellow oil. TLC \( R_f \) = 0.25 (hexane:ethyl acetate, 1:1). \(^1\)H NMR (300 MHz, CDCl\(_3\)): \( \delta \) 8.34 (m, 1H), 7.63 (m, 1H), 7.03 (m, 1H), 5.88 (dd, 1H, \( J = 5.7, 3.3 \) Hz), 4.45(m, 2H), 4.08 (m, 3H), 3.83 (dd, 1H, \( J = 9.0, 6.0 \) Hz), 1.39 (s, 3H), 1.33 (s, 9H); \(^13\)C NMR (75 MHz, CDCl\(_3\)): \( \delta \) 184.5, 137.0, 131.0, 118.0, 109.7, 109.6, 80.9, 74.8, 74.5, 65.8, 65.2, 26.3, 26.1, 25.0, 24.9. HRMS: Cacld. for (M + H)\(^+\): 343.1328, found: 343.1313.

**1,2:4,5-bis-acetal 4b:** Benzoyl peroxide (560.0 mg, 2.266 mmol) was added to a stirred solution of imidazolyl thiocarbonyl derivative 3b (3.8679 g, 11.31 mmol) in triethylsilane (56 mL) at refluxing temperature. The reaction mixture was refluxed for 2 h during which similar amount of benzoyl peroxide was added after 30 min, 60 min, and 90 min, respectively. The solvent was then removed under reduced pressure. Flash column chromatography (SiO\(_2\); hexane to hexane:ethyl acetate, 30:1 to 15:1) provided 1,2:4,5-bis-acetal \( 4b \) (~2.42 g) as a light yellow oil.
which contained some aromatic impurities but was good enough to be carried on. TLC Rf = 0.65 (hexane:ethyl acetate, 1:1). $^{1}$H NMR (300 MHz, CDCl$_3$): 4.21 (m, 2H), 4.11 (dd, 2H, $J$ = 8.1, 6.0 Hz), 3.57 (dd, 2H, $J$ = 7.8, 7.2 Hz), 1.80 (t, 2H, $J$ = 6.4 Hz), 1.41 (s, 3H), 1.36 (s, 3H).

(2S,4S)-pentane-1,2,4,5-tetraol 5b: $^{3}$ 0.5 M H$_2$SO$_4$ (14 mL, 7.0 mmol) was added to a stirred solution of 1,2:4,5-bis-acetal 4b (~2.42 g, 11.1 mmol) in ethanol (14 mL) at ambient temperature. The reaction mixture was refluxed for 4 h and then BaCO$_3$ was added to adjust the pH to 7. The resulting milky mixture was filtered and the white solid thus collected was heated with MeOH (15 mL) at 50 °C for 10 min. The combined filtrate was concentrated under reduced pressure. Flash column chromatography (SiO$_2$; DCM:MeOH, 7:3) provided (2S,4S)-pentane-1,2,4,5-tetraol 5b (960.5 mg, 62 % over 2 steps) as a white solid. TLC Rf = 0.25 (DCM:MeOH, 7:3). $^{1}$H NMR (300 MHz, MeOH-d$_4$): $\delta$ 3.85 (m, 2H), 3.47 (m, 4H), 1.52 (dd, 2H, $J$ = 7.1, 5.6 Hz).

(2S,4S)-2,4-Dihydroxypentane-1,5-diyl Bis-(2,4,6-triisopropyl-1-benzenesulfonate) 6b: $^{3}$ To a stirred solution of (2S,4S)-pentane-1,2,4,5-tetraol 5b (85.3 mg, 0.627 mmol) in anhydrous pyridine (0.63 mL) was added 2,4,6-triisopropyl benzenesulfonyl chloride (490.5 mg, 1.571 mmol) at 0 °C. The pale yellow cloudy mixture was stirred at ambient temperature for 10 h and then the solvent removed under reduced pressure. Flash column chromatography (SiO$_2$; hexane:acetone, 3:1) provided (2S,4S)-2,4-dihydroxypentane-1,5-diyl bis-(2,4,6-triisopropyl-1-benzenesulfonate) 6a (293.3 mg, 70 %) as a white fluffy solid. TLC Rf = 0.77 (hexane:ethyl acetate, 1:1). $^{1}$H NMR (300 MHz, CDCl$_3$): $\delta$ 7.20 (s, 4H), 4.23 (m, 2H), 4.10 (m, 6H), 3.98 (dd,
2H, J = 10.2, 6.9 Hz), 2.92 (septet, 2H, J = 6.9 Hz), 2.87 (br, 2H), 1.64 (t, 2H, J = 5.7 Hz), 1.27 (d, 24H, J = 6.9 Hz), 1.26 (d, 12H, J = 6.9 Hz).

**Acetonide 7b:** To a stirred solution of (2S,4S)-2,4-Dihydroxypentane-1,5-diyl bis-(2,4,6-triisopropyl-1-benzenesulfonate) 6b (1.115 g, 1.670 mmol) in acetone (17 mL) was added 2,2-dimethoxypropane 2.1 mL, 17 mmol) and p-toluenesulfonic acid (36.5 mg, 0.189 mmol) at ambient temperature. The resulting colorless solution was stirred at ambient temperature overnight (25 h). The reaction mixture was quenched with aqueous saturated NaHCO₃ (17 mL) and the solvent removed under reduced pressure. The residue was extracted with methylene chloride (17 mL × 3) and the combined organic layer dried over Na₂SO₄, filtered, and concentrated under reduced pressure. Flash column chromatography (SiO₂; hexane:ethyl acetate, 3:1) provided acetonide 7b (2.099 g, 80 %) as a white solid. TLC Rf = 0.73 (hexane:ethyl acetate, 3:1). ¹H NMR (300 MHz, CDCl₃): δ 7.19 (s, 4H), 4.08 (m, 10H), 2.92 (septet, 2H, J = 6.9 Hz), 1.65 (t, 2H, J = 7.8 Hz), 1.27 (d, 12H, J = 7.5 Hz), 1.26 (d, 24H, J = 6.9 Hz), 1.23 (s, 6H); ¹³C NMR (75 MHz, CDCl₃): δ 153.7, 150.8, 129.3, 123.8, 101.0, 70.1, 64.5, 34.2, 30.0, 29.6, 24.7, 24.3, 23.5. Caclcd. for (M+Na)⁺: 731.3627, found: 731.3610.

Diiodide 8b: To a stirred solution of (2S,4S)-2,4-Dihydroxypentane-1,5-diyl bis-(2,4,6-triisopropyl-1-benzenesulfonate) 7b (1.343 g, 1.897 mmol) in 2-butanone (19 mL) was added sodium iodide (2.815 g, 18.78 mmol) followed by anhydrous pyridine (0.77 mL, 9.5 mmol) at ambient temperature. The resulting light yellow suspension was refluxed overnight (22 h). The
reaction mixture was cooled to ambient temperature and the solvent was removed under reduced pressure. The residue was treated with water (30 mL), extracted with ethyl acetate (30 mL × 3), dried over MgSO₄, filtered and concentrated under reduced pressure. Flash column chromatography (SiO₂; hexane to hexane:ethyl acetate, 15:1) provided diiodide 8b (1.632 g, 85 %) as a light yellow oil. TLC Rf = 0.53 (hexane:ethyl acetate, 10:1). ¹H NMR (300 MHz, CDCl₃): δ 3.85 (m, 2H), 3.18 (m, 4H), 1.78 (t, 2H, J = 3.3 Hz), 1.39 (s, 6H); ¹³C NMR (75 MHz, CDCl₃): δ 101.6 (C), 66.9 (CH), 38.6 (CH₂), 24.6 (CH₃), 8.9 (CH₂).

Diiodo diol 9b: To a stirred solution of diiodide 8b (972.0 mg 2.455 mmol) in THF (25 mL) was added 1N HCl (25 mL) at ambient temperature. The reaction mixture was stirred at ambient temperature for 2 h. NaHCO₃ (powder) was added to adjust pH to 7 and then the organic solvent was removed under reduced pressure. The residue was extracted with ethyl acetate (20 mL × 3), dried over MgSO₄, filtered and concentrated under reduced pressure. Diiodo diol 9b (1.339 g, 92 %) was obtained as a white solid which was in good enough quality to be carried on without further purification. TLC Rf = 0.14 (hexane:ethyl acetate, 3:1). ¹H NMR (300 MHz, CDCl₃): δ 3.94 (m, 2H), 3.39 (dd, 2H, J = 10.2, 4.5 Hz), 3.28 (dd, 2H, J = 10.2, 6.9 Hz), 2.53 (d, 2H, J = 5.1 Hz), 1.86 (dd, 2H, J = 6.3, 5.4 Hz); ¹³C NMR (75 MHz, CDCl₃): δ 68.4, 41.6, 15.0. HRMS: Caclded. for M⁺: 355.8765, found: 355.8768.

Hydroxyacetophenone 10b: To a stirred solution of diiodo diol 9b (651.1 mg, 1.829 mmol) in 2-propanol (37 mL) was added 2,6-dihydroxyacetophenone (1.7231 g, 10.99 mmol) at ambient
temperature. The resulting yellow solution was heated to reflux and a mixture of KOH (843.8 mg, 13.53 mmol) in water (1.0 mL) and 2-propanol (1.9 mL) was added. The reaction mixture was refluxed for 22 h, cooled to ambient temperature, and then concentrated under reduced pressure. The dark brown residue was treated with ethyl acetate (50 mL) and filtered. The collected solid was washed with ethyl acetate and the combined filtrate was concentrated under reduced pressure. Flash column chromatography (SiO$_2$; hexane:ethyl acetate, 1:1 to 1:2 to 1:5, and then ethyl acetate) provided hydroxyacetophenone 10b (g, 35 %) as a light yellow solid. TLC R$_f$ = 0.18 (hexane:ethyl acetate, 1:2). $^1$H NMR (300 MHz, DMSO-d$_6$): δ 12.21 (s, 2H), 7.34 (t, 2H, $J$ = 7.8 Hz), 6.56 (d, 2H, $J$ = 7.8 Hz), 6.50 (d, 2H, $J$ = 7.8 Hz), 4.97 (br, 2H), 4.11 (m, 2H), 3.99 (m, 4H), 2.62 (s, 6H), 1.64 (m, 2H); $^{13}$C NMR (75 MHz, DMSO-d$_6$): δ 204.4, 161.1, 159.5, 134.9, 113.2, 109.5, 103.0, 73.7, 65.2, 38.1, 33.5. HRMS: Cacld. for (M + Na)$^+$: 427.1369, found: 427.1354.

Diester 11b: To a freshly prepared NaOMe solution (206.8 mg of sodium cube in 18 mL of anhydrous MeOH) was added anhydrous diethyl ether (36 mL) followed by dimethyl oxalate (1.0653 g, 8.931 mmol). After the solid disappeared, hydroxylacetophenone 10b (360.3 mg, 0.892 mmol) was added to the reaction mixture. The resulting yellow solution was refluxed for 24 h, cooled to ambient temperature, and then concentrated under reduced pressure. The yellow solid residue was treated with water (45 mL) and acidified with concentrated HCl (0.20 mL) until pH ~ 2. The yellow precipitated was filtered off, dissolved in anhydrous MeOH, and treated with concentrated HCl (2.7 mL). The reaction mixture was refluxed for 40 min and then cooled to ambient temperature. The pale brown precipitate was filter off and found to be pure to be
carried on (191.7 mg, 40 %). TLC R<sub>f</sub> = 0.10 (ethyl acetate:ethanol, 9:1).<sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>): δ 7.73 (t, 2H, J = 8.4 Hz), 7.22 (d, 2H, J = 8.4 Hz), 7.09 (d, 2H, J = 8.4 Hz), 6.75 (s, 2H), 4.85 (d, 2H, J = 4.8 Hz), 4.07 (m, 6H), 3.92 (s, 6H), 1.78 (t, 2H, J = 6.0 Hz);<sup>13</sup>C NMR (75 MHz, DMSO-d<sub>6</sub>): δ 176.6, 160.6, 158.6, 157.1, 150.1, 135.4, 115.4, 114.8, 110.4, 109.7, 74.4, 65.2, 53.4, 37.2. MS: Cacl. for (M + Na)<sup>+</sup>: 563.1165, found: 563.1178.

5'DSCG-(S,S)-diviol 1b: To a stirred suspension of diester 11b (120.5 mg, 0.223 mmol) in ethanol (11 mL) was added aqueous NaOH solution (2.5 M, 0.18 mL, 0.45 mmol) at ambient temperature. The reaction mixture was refluxed for 10 min, cooled to ambient temperature, and then at 0 °C for 20 min. After filtration and drying under high vacuum for 24 h, 1b was obtained as brown solid (108.1 mg, 87%).<sup>1</sup>H NMR (300 MHz, D<sub>2</sub>O): δ 7.59 (t, 2H, J = 8.4 Hz), 7.06 (d, 2H, J = 8.7 Hz), 6.87 (d, 2H, J = 8.4 Hz), 6.62 (s, 2H), 4.38 (m, 2H), 4.11 (m, 4H), 1.97 (t, 2H, J = 6.2 Hz);<sup>13</sup>C NMR (75 MHz, D<sub>2</sub>O): δ 181.4, 165.1, 157.2, 157.1, 156.2, 135.2, 113.0, 112.0, 110.5, 108.1, 73.1, 65.5, 35.4. HRMS: Cacl. for (M + Na)<sup>+</sup>: 579.0483, found: 579.0491.

1,2:4,5-bis-acetal 2c: To a stirred suspension of xylitol (2.590 g, 16.85 mmol) in anhydrous THF (48 mL) was added 2,2-dimethoxypropane (4.6 mL, 37 mmol) at ambient temperature. The reaction mixture was refluxed for 15 min and then L(-)-camphorsulfonic acid (398.7 mg, 1.682 mmol) was added at refluxing temperature. After refluxing for another 5 minutes, the reaction was quenched with 2 M NaOH (10 mL) at refluxing temperature. After removing the majority of the solvent under reduced pressure, the aqueous layer was extracted with diethyl ether (10 mL ×
3). The combined organic layer was dried over MgSO₄, filtered, and concentrated under reduced pressure. The colorless oil residue was dissolved in anhydrous DCM (50 mL) and Et₃N (2.5 mL) was added. The reaction mixture was then brought to reflux and succinic anhydride (427.6 mg, 4.203 mmol) was added. After refluxing for another hour, the reaction was quenched with saturated aqueous NaHCO₃ (5 mL) at refluxing temperature. The mixture was cooled to ambient temperature and the organic layer was dried over MgSO₄, filtered, and concentrated under reduced pressure. Flash column chromatography (SiO₂; diethyl ether:hexane) provided 1,2:4,5-bis-acetal 5: (1.720 g, 44 %) as a pale yellow oil. TLC Rf = 0.23 (diethyl ether:hexane, 1:1). ¹H NMR (300 MHz, CDCl₃): δ 4.18 (ddd, 2H, J = 6.6, 6.6, 5.1 Hz), 4.07 (dd, 2H, J = 8.1, 6.6 Hz), 3.89 (dd, 2H, J = 8.3, 6.8 Hz), 3.60 (dt, 1H, J = 6.0, 5.1 Hz), 2.44 (d, 1H, J = 6.0 Hz), 1.45 (s, 6H), 1.38 (s, 6H).

![Image of compound 3c]

**Imidazolyl thiocarbonyl derivative 3c:** To a stirred solution of 1,2:4,5-bis-acetal 5 (265.2 mg, 1.143 mmol) in anhydrous 1,2-dichloroethane (4.1 mL) was added N,N´-thiocarbonyldiimidazole (396.1 mg, 2.000 mmol) at ambient temperature. The resulting brown solution was refluxed for 4 h and then cooled to ambient temperature. The solvent was removed under reduced pressure. Flash column chromatography (SiO₂; hexane:ethyl acetate) provided imidazolyl thiocarbonyl derivative 6 (371.8 mg, 95 %) as a yellow oil. TLC Rf = 0.30 (diethyl ether:hexane, 3:2). ¹H NMR (300 MHz, CDCl₃): δ 8.38 (t, 1H, J = 0.9 Hz), 7.68 (t, 1H, J = 1.5 Hz), 7.06 (dd, 1H, J = 1.8, 0.9 Hz), 5.84 (t, 1H, J = 5.4 Hz), 4.13 (m, 4H), 3.92 (dd, 2H, J = 8.9, 5.9 Hz), 1.43 (s, 6H), 1.35 (s, 6H). HRMS: Caclcd. for (M + H): 343.1328, found: 343.1312.
1,2:4,5-bis-acetal 4c: Benzoyl peroxide (53.4 mg, 0.216 mmol) was added to a stirred solution of imidazolyl thiocarbonyl derivative 6 (371.8 mg, 1.087 mmol) in triethylsilane (8.0 mL) at refluxing temperature. The reaction mixture was refluxed for 2h during which similar amount of benzoyl peroxide was added after 30 min, 60 min, and 90 min, respectively. The solvent was then removed under reduced pressure. Flash column chromatography (SiO$_2$, hexane:ethyl acetate) provided 1,2:4,5-bis-acetal 7 (199.7 mg, 85%) as a yellow oil. TLC $R_f$ = 0.71 (diethyl ether:hexane, 1:1). $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ 4.19 (m, 2H), 4.09 (dd, 2H, $J = 8.1$, 6.0 Hz), 3.62 (dd, 2H, $J = 7.8$, 7.4 Hz), 2.01 (m, 1H), 1.79 (dt, 1H, $J = 13.8$, 6.0 Hz), 1.42 (s, 6H), 1.36 (s, 6H).

(2S,4R)-pentane-1,2,4,5-tetraol 5c: 0.5 M H$_2$SO$_4$ (1.0 mL) was added to a stirred solution of 1,2:4,5-bis-acetal 7 (170.0 mg, 0.787 mmol) in ethanol (1 mL) at ambient temperature. The reaction mixture was refluxed for 3.5 h and then BaCO$_3$ was added to adjust the pH to 7. The resulting milky mixture was refluxed for another 10 min, cooled to ambient temperature, and then filtered. The solid was treated with MeOH (5 mL) and the mixture was stirred at 50 °C for 10 min. Filtered again and washed the solid with another 5 mL of MeOH. The combined filtrated was concentrated under reduced pressure. Flash column chromatography (SiO$_2$, DCM:MeOH) provided (2S,4R)-pentane-1,2,4,5-tetraol 5c (119.4 mg, 97%) as a white solid. TLC $R_f$ = 0.01 (ethyl acetate). $^1$H NMR (300 MHz, MeOH-d4): $\delta$ 3.82 (m, 4H), 3.49 (m, 2H), 1.73 (dt, 1H, $J =$
14.1, 4.8 Hz), 1.53 (dt, 1H, J = 14.1, 8.1 Hz); $^{13}$C NMR (75 MHz, MeOH-d4): δ 71.7 (CH), 67.4 (CH$_2$), 37.7 (CH$_2$).

**Bromoacetate 6c**: To a stirred suspension of (2S,4R)-pentane-1,2,4,5-tetraol 5c (597.7 mg, 4.395 mmol) in anhydrous 1,4-dioxane (11 mL) was added acetyl bromide (0.80 mL, 10 mmol) dropwise at 0 °C. The reaction mixture was stirred at ambient temperature overnight (22 h) and then the solvent was removed under reduced pressure. The residue was taken up in diethyl ether (20 mL) and washed with saturated aqueous NH$_4$Cl. The aqueous layer was extracted with diethyl ether (20 mL × 3), dried over MgSO$_4$, filtered, and concentrated under reduced pressure. $^1$H NMR of the crude indicated the presence of the desired product bromoacetate 6c, which was unstable and carried on immediately without further purification.

**Diepoxide 7c**: The crude bromoacetate 6c obtained from previous step was dissolved in anhydrous diethyl ether (44 mL) and then cooled to 0 °C. Freshly ground KOH (904.7 mg, 14.51 mmol) was added to the reaction mixture in one portion. The reaction mixture was then warmed to ambient temperature while stirred vigorously. Another portion of freshly ground KOH (905.8 mg, 14.53 mmol) was added to the reaction mixture after 1 h at 0 °C. After stirring at ambient temperature for 5 h, the reaction mixture was filter through a pad of MgSO$_4$. The filtrate was carefully distilled from an ice-water bath under reduce pressure with the receiving flask immersed in a bath at -78 °C. The pale yellow oil left in the distilling flask was in good enough purity to be carried on. TLC $R_f = 0.40$ (hexane:ethyl acetate, 3:1).
**Diiodo diol 8c:** The crude diepoxide 7c obtained from previous step was dissolved in anhydrous acetonitrile (11 mL). To the reaction mixture was added sodium iodide (1.352 g, 9.022 mmol) followed by cerium (III) chloride heptahydrate (1.643 g, 4.409 mmol) at ambient temperature. The resulting yellow suspension was refluxed for 2 h and then cooled to ambient temperature. The reaction mixture was washed with water (15 mL), extracted with methylene chloride (15 mL × 3), dried over MgSO₄, filtered, and then concentrated. The crude yellow oil was carried on without further purification. TLC R$_f$ = 0.15 (hexane:ethyl acetate, 3:1).

**Hydroxyacetophenone 9c:** To a stirred solution of the crude diiodo diol 8c in 2-propanol (88 mL) was added 2,6-dihydroxyacetophenone (3.616 g, 23.05 mmol) at ambient temperature. The resulting yellow solution was heated to reflux and a mixture of KOH (1.7024 g, 27.31 mmol) in water (1.5 mL) and 2-propanol (4.4 mL) was added. The reaction mixture was refluxed for 15 h, cooled to ambient temperature, and then concentrated under reduced pressure. Flash column chromatography (SiO$_2$; hexane:ethyl acetate, 1:1 to 1:2 to 1:5 to 1:8, and then ethyl acetate) provided hydroxyacetophenone 9c (438.5 mg, 25% over 4 steps) as a light yellow solid. TLC R$_f$ = 0.26 (hexane:ethyl acetate, 1:2). $^1$H NMR (300 MHz, CDCl$_3$): δ 12.2 (s, 2H), 7.34 (t, 2H, J = 8.3 Hz), 6.56 (d, 2H, J = 8.4 Hz), 6.49 (d, 2H, J = 8.1 Hz), 5.02 (d, 2H, J = 4.8 Hz), 4.05 (m, 6H), 2.61 (s, 6H), 1.83 (m, 1H), 1.71 (m, 1H); $^{13}$C NMR (75 MHz, DMSO-d6): δ 204.4, 161.0, 159.5, 134.9, 113.2, 109.5, 103.0, 73.0, 66.0, 37.8, 33.4. HRMS: Cacld. for M$^+$: 404.1466, found: 404.1458.
**Diester 10c**: To a freshly prepared NaOMe solution (202.3 mg of sodium cube in 17.6 mL of anhydrous MeOH) was added anhydrous diethyl ether (35.2 mL) followed by dimethyl oxalate (1.060 g, 8.886 mmol). After the solid disappeared, hydroxylacetophenone 9c (355.7 mg, 0.880 mmol) was added to the reaction mixture. The resulting yellow solution was refluxed for 24 h, cooled to ambient temperature, and then concentrated under reduced pressure. The yellow solid residue was treated with water (45 mL) and acidified with concentrated HCl (0.22 mL) until pH ~ 2. The yellow precipitated was filtered off, dissolved in anhydrous MeOH, and treated with concentrated HCl (2.7 mL). The reaction mixture was refluxed for 1.5 h, cooled to ambient temperature and then 0 °C for 1 h. The brown precipitate was filter off and found to be the desired product diester 10c (8.6 mg, 2 %). However, the majority of the diester 10c remained in the filtrate as oily substance. The filtrate was concentrated to about half the volume and carried on without further purification. 

$^1$H NMR (300 MHz, DMSO-d6): $\delta$ 7.71 (t, 2H, $J = 8.4$ Hz), 7.20 (d, 2H, $J = 8.4$ Hz), 7.08 (d, 2H, $J = 8.4$ Hz), 6.73 (s, 2H), 4.91 (br, 2H), 4.08 (m, 6H), 3.92 (s, 6H), 2.03 (m, 1H), 1.81 (m, 1H). HRMS: Cacld. for (M + Na)$^+$: 563.1165, found: 563.1167.

**5'DSCG-meso-diviol 1c**: To the filtrate from the previous step was added aqueous NaOH solution (2.5 M, 5.0 mL, 13 mmol) at ambient temperature. Some pale brown precipitate formed and the pH of the liquid was approximately 1. The reaction mixture was warmed to reflux and treated with NaOH pellet in 10 portions. The resulting suspension was refluxed for 1.5 h, cooled to ambient temperature, and then 0 °C for 30 min. The pale brown precipitate was collected by
centrifugation and dried under vacuum overnight. 5′DSCG-meso-diviol 1c was obtained as pale brown powder (332.3 mg, 73% over 2 steps). 1H NMR (300 MHz, D2O): δ 7.58 (t, 2H, J = 8.4 Hz), 7.06 (d, 2H, J = 8.4 Hz), 6.91 (d, 2H, J = 8.4 Hz), 6.60 (s, 2H), 4.25 (m, 4H), 4.09 (m, 2H), 2.36 (m, 1H), 1.92 (m, 1H); 13C NMR (75 MHz, D2O): 181.1, 165.1, 157.0, 156.9, 156.2, 135.2, 113.1, 112.1, 110.7, 108.4, 72.5, 65.9, 37.6. HRMS Cacld. for (M + Na)+: 579.0491, found: 579.0471.

Cleaning of glass substrates. Substrates used for gold films were Fisher’s Finest premium microscope slides purchased from Fisher Scientific (Pittsburgh, PA). Prior to gold deposition, the glass slides were cleaned with Piranha solution. The slides were soaked in Piranha solution (7 parts of 35% aqueous hydrogen peroxide solution and 3 parts of concentrated sulfuric acid) for 45 min at 70 °C. Warning! Piranha solution is extremely corrosive and can potentially detonate when mixed with significant amounts of oxidizable materials. It is advised to neutralize the Piranha solution with sodium hydroxide before disposal. After cooling to ambient temperature, the Piranha solution was poured off and the glass slides rinsed 20 times sequentially with deionized water, followed by 10 times with ethanol, and then 10 times with methanol. The cleaned slides were dried individually with a stream of nitrogen gas and kept in an 80 °C oven overnight.

Deposition of Gold Films. Deposition of gold films was done following literature procedure. Semitransparent gold films were deposited onto the glass substrate using an electron beam evaporation system (Thermionics, Port Townsend, WA). A layer of titanium (~70 Å) was deposited first to enhance the adhesion of the gold. A layer of gold (~280 Å) was then deposited at an oblique angle of 45° to the surface normal of the substrate. The rate of deposition was kept
at 0.2 Å/s for both gold and titanium and the pressure was maintained at no higher than $2 \times 10^{-6}$ Torr throughout the deposition.

**Preparation of SAMs on obliquely deposited gold films.** Gold films deposited on microscope glass slides were cut into strips (8 mm × 2.5 mm) along the direction of gold deposition. The strips were rinsed with 200 proof ethanol (3 x), dried with a stream of nitrogen gas and then immersed in ethanolic solutions containing 2 mM of thiols overnight (~15 h). Each strip was dried with a stream of nitrogen gas and used immediately.

**Assembly of optical cells and characterization of birefringence.** Aqueous solutions of 5´DSCG and 5´DSCG-diviols were prepared by dissolution in deionized water in vials. The vials were subjected to vortex mixing for at least 1 min before use. The liquid crystal samples were assembled in a sandwiched cell composed of two microscope glass slides or gold films supporting SAMs with one sheet of Saran Wrap® (13 – 15 µm) inserted in between as a spacer. A square hole was cut on the sheet of Saran Wrap® to accommodate the liquid crystal sample. The liquid crystal samples were loaded between the slides in the square hole and sealed with binder clips on each side to prevent the evaporation of solvent. The samples were viewed under crossed polarizers on an Olympus BX51 polarizing microscope and were rotated from an arbitrary starting point to record the birefringence.

**Circular dichroism.** Wavelength scan and thermal study of chiral 5´DSCG-diviols were performed on an AVIV 420 CD spectrometer (AVIV Biomedical, Inc., Lakewood, NJ) using a 1 mm path length cuvette. For thermal study, the temperature was increased at a rate of 2 °C/min and samples were equilibrated for 60 s prior to each data collection.

**UV.** UV measurements were performed on an Agilent 8453 spectrometer (Agilent Technologies, Santa Clara, CA) using a 1mm or 1cm path length cuvette at ambient temperature.
Sample preparation for cryo-TEM. The samples were prepared by applying \( \sim 5 \) µL droplet of the sample on the microperforated cryo-TEM grid, followed by blotting the grid with a filter paper to produce a thin layer. The grids were then vitrified by plunging into liquid. Grids were mounted in a Gatan 626 cryo holder and examined in a JEM-2100 transmission electron microscope (JEOL Ltd., Tokyo, Japan).

**Figure S1.** \(^1\)H NMR of \((R,R)\)-, \((S,S)\)-, and meso-5’DSCG-diviol at the linker region. All three samples were 0.50 wt% (mole ratio of water/5’DSCG-diviol was 5503:1).

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Figure S2. ROESY spectrum of 0.503 wt% 5’DSCG-(S,S)-diviol and 5’DSCG-meso-diviol in D$_2$O at 25 °C.
Figure S3. $^1$H NMR of 0.02 wt% (0.4 mM) to 3.5 wt% (72 mM) 5′DSCG-(S,S)-diviol in D$_2$O.
Figure S4. Cryogenic transmission electronic microscopic image of 5.5 wt% (488) 5´DSCG. (Mole ratio) indicates water/5´DSCG.

Figure S5. Cryogenic transmission electronic microscopic image of 5.0 wt% (587) 5´DSCG-\((R,R)\)-diviol. (Mole ratio) indicates water/5´DSCG-\((R,R)\)-diviol.
Figure S6. Cryogenic transmission electronic microscopic image of 8.2 wt% (321) 5´DSCG. (Mole ratio) indicates water/5´DSCG.

Figure S7. Cryogenic transmission electronic microscopic image of 8.8 wt% (321) 5´DSCG-(R,R)-diviol. (Mole ratio) indicates water/5´DSCG-(R,R)-diviol.
Figure S8. Optical images (cross polarizers) of (A) 16.8 wt% 5´DSCG, and (B) 14.1 wt% 5´DSCG mixed with 14.5 wt% xylitol in water sandwiched between obliquely deposited gold films supporting HS(CH2)10(O CH2CH2)3OH. Numbers in the parentheses indicate mole ratio of water and mesogens in the sample. Scale bar = 152 µm. The arrows indicate direction of gold deposition projected onto the glass slides, and the orientation of the sample relative to the cross polarizers.
Figure S9. Optical images of 13.4 wt% 5^DSCG mixed with 16.2 wt% D-mannitol in a wedge cell composed of plain glass slides when the thickness of the cell was 178 µm. Schematic representation of homogenous and homeotropic alignment of liquid crystals are shown under the images.

References


