SUPPLEMENTARY INFORMATION (11 Pages)

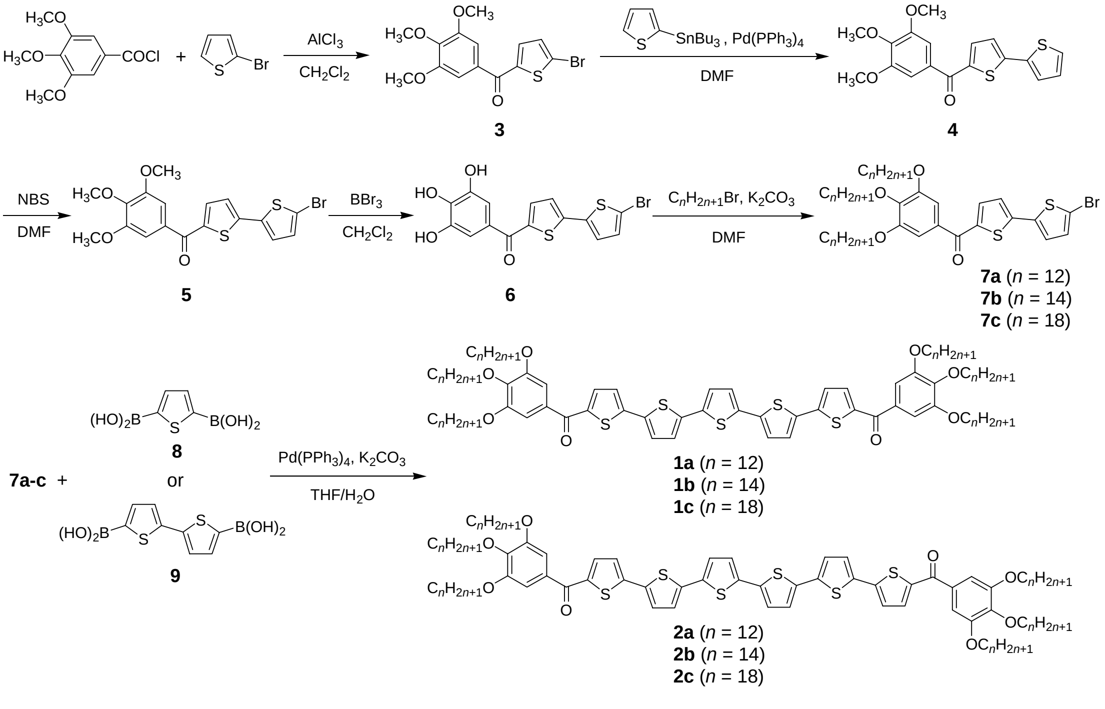
Columnar Liquid Crystalline π-Conjugated Oligothiophenes

Takuma Yasuda, Kenji Kishimoto and Takashi Kato*

Experimental Details:

Materials and Syntheses. All reagents and solvents were purchased from Aldrich or Tokyo Kasei, and used as received. $Pd(PPh_3)_4$ was obtained according to the literature.[1] Thiophene-2,5-diboronic acid (**8**) and 2,2'-bithiophene-5,5'-diboronic acid (**9**) were prepared by similar procedures reported in the literature.[2] The synthetic routes used to obtain compounds **1a-c** and **2a-c** are shown in Scheme S1. All reactions were performed under an Ar atmosphere using standard Schlenk techniques.

Scheme S1. Synthetic routes of liquid crystalline oligothiophenes.



2-Bromo-5-(3,4,5-trimethoxybenzoyl)thiophene (3). To a stirred mixture of 3,4,5-trimethoxybenzoyl chloride (20.8 g, 90 mmol) and 2-bromothiophene (15.3 g, 94 mmol) in dry CH_2Cl_2 (200 mL) was slowly added $AlCl_3$ (13.2 g, 99 mmol) at 0 °C under an Ar atmosphere. Thereafter, the mixture was stirred for 3 h at room temperature. The reaction mixture was added into an aqueous hydrochloric acid (ca. 5%), and the product was extracted with $CHCl_3$ three times. The combined organic layers were washed with water, and dried over anhydrous Na_2SO_4. After filtration and evaporation, the crude product was purified by column chromatography (silica, $CHCl_3$) and dried under vacuum to afford **3** as a light-yellow solid (yield = 21.8 g, 68%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, J = 4.0 Hz, 1H), 7.16 (d, J = 4.0 Hz, 1H), 7.10 (s, 2H), 3.94 (s, 3H), 3.92 (s, 6H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 185.87, 153.06, 144.77, 142.05, 134.54, 132.42, 131.08, 122.92, 106.65, 60.99, 56.32.

5-(3,4,5-Trimethoxybenzoyl)-2,2’-bithiophene (4). To a solution of **3** (14.3 g, 40 mmol) and 2-tributylstannylthiophene (16.4 g, 44 mmol) in dry DMF (160 mL) was added $Pd(PPh_3)_4$ (1.85 g, 1.6 mmol) at room temperature under an Ar atmosphere. The mixture was stirred for 10 h at 80 °C. After cooling to room temperature, the reaction mixture was poured into an aqueous solution (ca. 5%) of KF to obtain a precipitate. The precipitate was filtered off, dissolved in $CHCl_3$, and then purified by column chromatography (silica, $CHCl_3$). Recrystallization from $CHCl_3$/hexane gave **4** as a yellow solid (yield = 12.8 g, 89%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, J = 4.0 Hz, 1H), 7.38-7.34 (m, 2H), 7.23 (d, J = 4.0 Hz, 1H), 7.14 (s, 2H), 7.09 (dd, J = 4.8 Hz and 3.6 Hz, 1H), 3.95 (s, 3H), 3.93 (s, 6H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.74, 153.02, 146.06, 141.78, 141.39, 136.24, 135.39, 133.14, 128.31, 126.63, 125.76, 124.07, 106.65, 61.00, 56.33.

5-Bromo-5’-(3,4,5-trimethoxybenzoyl)-2,2’-bithiophene (5). To a solution of **4** (7.21 g, 20 mmol) in dry DMF (150 mL) was slowly added *N*-bromosuccinimide (3.56 g, 20 mmol) at 0 °C under an Ar atmosphere. The mixture was stirred for 10 h at room temperature, and then poured into a large amount of water. The product was extracted with $CHCl_3$ three times. The combined organic layers were washed with water, and dried over anhydrous Na_2SO_4. After filtration and evaporation, the crude product was purified by column chromatography (silica, $CHCl_3$/hexane/ethyl acetate = 5:5:1), and dried under vacuum to afford **5** as a yellow solid (yield = 7.64 g, 87%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 3.6 Hz, 1H), 7.15 (d, *J* = 3.6 Hz, 1H), 7.13 (s, 2H), 7.11 (d, *J* = 4.0 Hz, 1H), 7.04 (d, *J* = 4.0 Hz, 1H), 3.95 (s, 3H), 3.92 (s, 6H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.60, 153.01, 146.62, 144.68, 141.78, 137.62, 135.22, 132.91, 131.11, 125.77, 124.17, 122.54, 113.55, 106.64, 60.98, 56.31.

5-Bromo-5’-(3,4,5-trihydroxybenzoyl)-2,2’-bithiophene (6). To a stirred solution of **5** (7.03 g, 16 mmol) in dry CH_2Cl_2 (150 mL) was added dropwise BBr_3 (1.0 M in CH_2Cl_2, 53 mL) at 0 °C under an Ar atmosphere. The mixture was allowed to warm to room temperature and stirred for further 4 h. The reaction mixture was then quenched with methanol, and concentrated under reduced pressure. The resulting residue was added into water to obtain a yellow precipitate. The product was collected by filtration, washed with cold methanol, $CHCl_3$ and hexane in this order, and dried under vacuum to give **6** as a yellow solid (yield = 6.17 g, 97%). ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.43 (br, 2H), 9.13 (br, 1H), 7.63 (d, *J* = 4.0 Hz, 1H), 7.43 (d, *J* = 4.0 Hz, 1H), 7.39 (d, *J* = 4.0 Hz, 1H), 7.29 (d, *J* = 4.0 Hz, 1H), 6.88 (s, 2H). $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6): δ 185.18, 145.75, 142.36, 142.04, 138.78, 137.17, 135.05, 132.11, 127.07, 126.88, 125.29, 112.62, 108.80. MS (MALDI-TOF): *m*/*z* 397.1 $[M+H]^+$; calcd 396.9.

5-Bromo-5’-(3,4,5-tri-*n*-dodecyloxybenzoyl)-2,2’-bithiophene (7a). A mixture of **6** (2.38 g, 4.0 mmol), 1-bromododecane (4.98 g, 20 mmol), and K_2CO_3 (4.15 g, 30 mmol) in dry DMF (30 mL) was vigorously stirred for 20 h at 80 °C under an Ar atmosphere. After cooling to room temperature, the reaction mixture was added into an aqueous hydrochloric acid (ca. 5%), and the product was extracted with $CHCl_3$ three times. The combined organic layers were washed with brine and water, and dried over anhydrous Na_2SO_4. After filtration and evaporation, the product was purified by column chromatography (silica, $CHCl_3$/hexane = 2:1, v/v), recrystallized from $CHCl_3$/methanol, and dried under vacuum to give **7a** as a light-yellow solid (yield = 4.81 g, 89%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 4.0 Hz, 1H), 7.14 (d, *J* = 4.0 Hz, 1H), 7.09 (d, *J* = 4.0 Hz, 1H), 7.08 (s, 2H), 7.04 (d, *J* = 4.0 Hz, 1H), 4.07-4.00 (m, 6H), 1.84-1.74 (m, 6H), 1.50-1.22 (m, 54H), 0.88 (t, *J* = 6.8 Hz, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 186.73, 152.86, 144.40, 142.19, 142.00, 137.74, 135.10, 132.44, 131.07, 125.65, 124.11, 113.40, 107.94, 73.57, 69.27, 31.92, 31.90, 30.32, 29.73, 29.71, 29.68, 29.63, 29.55, 29.38, 29.35, 29.29, 26.06, 22.66, 14.09. MS (MALDI-TOF): *m*/*z* 901.6 $[M+H]^+$; calcd 901.5.

5-Bromo-5'-(3,4,5-tri-*n*-tetradecyloxybenzoyl)-2,2'-bithiophene (7b). This compound was prepared in a similar manner to **7a**, and obtained as a light-yellow solid (yield = 91%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 4.0 Hz, 1H), 7.14 (d, *J* = 4.0 Hz, 1H), 7.09 (d, *J* = 4.0 Hz, 1H), 7.08 (s, 2H), 7.04 (d, *J* = 4.0 Hz, 1H), 4.07-4.00 (m, 6H), 1.84-1.76 (m, 6H), 1.50-1.22 (m, 66H), 0.88 (t, *J* = 6.8 Hz, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 186.81, 152.88, 144.44, 142.19, 142.00, 137.74, 135.15, 132.45, 131.09, 125.68, 124.14, 113.43, 107.95, 73.61, 69.29, 31.91, 30.32, 29.76, 29.73, 29.70, 29.66, 29.63, 29.57, 29.39, 29.36, 29.29, 26.06, 22.68, 14.11. MS (MALDI-TOF): *m*/*z* 985.8 $[M+H]^+$; calcd 985.6.

5-Bromo-5'-(3,4,5-tri-*n*-octadecyloxybenzoyl)-2,2'-bithiophene (7c). This compound was prepared in a similar manner to **7a**, and obtained as a light-yellow solid (yield = 87%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 4.0 Hz, 1H), 7.14 (d, *J* = 4.0 Hz, 1H), 7.09 (d, *J* = 4.0 Hz, 1H), 7.08 (s, 2H), 7.04 (d, *J* = 4.0 Hz, 1H), 4.07-4.00 (m, 6H), 1.84-1.76 (m, 6H), 1.50-1.22 (m, 90H), 0.88 (t, *J* = 6.8 Hz, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 186.81, 152.89, 144.45, 142.20, 142.01, 137.76, 135.16, 132.46, 131.10, 125.69, 124.14, 113.44, 107.96, 73.61, 69.30, 31.92, 30.33, 29.76, 29.74, 29.71, 29.66, 29.63, 29.58, 29.40, 29.36, 29.30, 26.07, 22.69, 14.12. MS

(MALDI-TOF): *m*/*z* 1154.1 [M+H]$^+$; 1153.8.

1a. To a solution of **7a** (1.17 g, 1.3 mmol) and **8** (0.10 g, 0.6 mmol) in dry THF (10 mL) were added Pd(PPh$_3$)$_4$ (0.03 g, 0.03 mmol) and aqueous K_2CO_3 (2.0 M, 5 mL; Ar bubbled before use) under an Ar atmosphere. The mixture was stirred for 28 h at 60 °C. After cooling to room temperature, the reaction mixture was poured into water, and extracted with $CHCl_3$ three times. The combined organic layers were washed with brine and water, and dried over anhydrous Na_2SO_4. After filtration and evaporation, the product was purified by column chromatography (silica, $CHCl_3$), recrystallized from $CHCl_3$/acetone, and dried under vacuum to provide **1a** as an orange solid (yield = 0.86 g, 83%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 4.0 Hz, 2H), 7.28 (d, *J* = 4.0 Hz, 2H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.16-7.14 (m, 4H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.86-1.75 (m, 12H), 1.49-1.25 (m, 108H), 0.88 (t, *J* = 6.8 Hz, 18H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 186.74, 152.88, 145.14, 142.15, 141.80, 137.80, 136.09, 135.31, 135.28, 132.58, 126.47, 125.04, 124.82, 123.99, 107.95, 73.60, 69.29, 31.94, 31.92, 30.34, 29.75, 29.70, 29.66, 29.63, 29.58, 29.40, 29.36, 29.31, 26.08, 22.69, 14.12. MS (MALDI-TOF): *m*/*z* 1725.7 [M+H]$^+$; calcd 1726.1. Anal. calcd for $C_{106}H_{164}O_8S_5$: C, 73.73; H, 9.57%; found: C, 73.49; H, 9.72%.

1b. This compound was prepare from **7b** (1.28 g, 1.3 mmol), **8** (0.10 g, 0.6 mmol), and Pd(PPh$_3$)$_4$ (0.03 g, 0.03 mmol) by adopting the procedure used for **1a**, and was obtained as an orange solid (yield = 1.05 g, 92%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 4.0 Hz, 2H), 7.28 (d, *J* = 4.0 Hz, 2H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.16-7.14 (m, 4H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.85-1.75 (m, 12H), 1.50-1.24 (m, 132H), 0.88 (t, *J* = 6.8 Hz, 18H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 186.69, 152.90, 145.13, 142.15, 141.79, 137.80, 136.07, 135.29, 135.26, 132.57, 126.45, 125.02, 124.79, 123.97, 107.94, 73.58, 69.28, 31.92, 30.34, 29.74, 29.71, 29.68, 29.67, 29.63, 29.58, 29.40, 29.38, 29.36, 29.31, 26.08, 22.68, 14.11. MS (MALDI-TOF): *m*/*z* 1894.4 [M+H]$^+$; calcd 1894.3. Anal. calcd for $C_{118}H_{188}O_8S_5$: C, 74.79; H, 10.00%; found: C, 74.63; H, 10.20%.

1c. This compound was prepared from **7c** (1.50 g, 1.3 mmol), **8** (0.10 g, 0.6 mmol), and $Pd(PPh_3)_4$ (0.03 g, 0.03 mmol) by adopting the procedure used for **1a**, and was obtained as an orange solid (yield = 0.96 g, 72%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 4.0 Hz, 2H), 7.28 (d, *J* = 4.0 Hz, 2H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.16-7.14 (m, 4H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.85-1.75 (m, 12H), 1.49-1.22 (m, 180H), 0.88 (t, *J* = 6.8 Hz, 18H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.73, 152.89, 145.15, 142.14, 141.80, 137.80, 136.09, 135.32, 135.27, 132.59, 126.47, 125.04, 124.81, 123.99, 107.94, 73.61, 69.32, 31.92, 30.35, 29.76, 29.74, 29.72, 29.66, 29.64, 29.59, 29.41, 29.36, 29.32, 26.07, 22.69, 14.12. MS (MALDI-TOF): *m*/*z* 2230.9 $[M+H]^+$; calcd 2230.7. Anal. calcd for $C_{142}H_{236}O_8S_5$: C, 76.42; H, 10.66%; found: C, 76.30; H, 10.81%.

2a. This compound was prepared from **7a** (1.17 g, 1.3 mmol), **9** (0.15 g, 0.6 mmol), and $Pd(PPh_3)_4$ (0.03 g, 0.03 mmol) by adopting the procedure used for **1a**, and was obtained as a reddish purple solid (yield = 0.86 g, 80%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 4.0 Hz, 2H), 7.27 (d, *J* = 4.0 Hz, 2H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.15-7.11 (m, 6H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.85-1.75 (m, 12H), 1.50-1.25 (m, 108H), 0.88 (t, *J* = 6.8 Hz, 18H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.71, 152.88, 145.21, 142.15, 141.73, 137.94, 136.38, 135.68, 135.32, 135.13, 132.59, 126.46, 125.02, 124.66, 123.95, 107.96, 73.60, 69.30, 31.94, 31.92, 30.34, 29.75, 29.70, 29.66, 29.63, 29.58, 29.39, 29.36, 29.31, 26.08, 22.68, 14.11. MS (MALDI-TOF): *m*/*z* 1808.4 $[M+H]^+$; calcd 1808.1. Anal. calcd for $C_{110}H_{166}O_8S_6$: C, 73.04; H, 9.25%; found: C, 72.81; H, 9.35%.

2b. This compound was prepared from **7b** (1.28 g, 1.3 mmol), **9** (0.15 g, 0.6 mmol), and $Pd(PPh_3)_4$ (0.03 g, 0.03 mmol) by adopting the procedure used for **1a**, and was obtained as a reddish purple solid (yield = 1.08 g, 91%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 4.0 Hz, 2H), 7.27 (d, *J* = 4.0 Hz, 2H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.16-7.11

(m, 6H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.85-1.75 (m, 12H), 1.50-1.24 (m, 132H), 0.88 (t, *J* = 6.8 Hz, 18H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.74, 152.89, 145.22, 142.16, 141.75, 137.95, 136.40, 135.69, 135.32, 135.15, 132.60, 126.47, 125.03, 124.69, 123.96, 107.98, 73.61, 69.31, 31.93, 30.35, 29.75, 29.72, 29.69, 29.67, 29.64, 29.59, 29.41, 29.39, 29.37, 29.31, 26.08, 22.69, 14.12. MS (MALDI-TOF): *m*/*z* 1976.9 $[M+H]^+$; calcd 1976.3. Anal. calcd for $C_{122}H_{190}O_8S_6$: C, 74.11; H, 9.69%; found: C, 74.00; H, 9.85%.

2c. This compound was prepared from **7c** (1.50 g, 1.3 mmol), **9** (0.15 g, 0.6 mmol), and $Pd(PPh_3)_4$ (0.03 g, 0.03 mmol) by adopting the procedure used for **1a**, and was obtained as a reddish purple solid (yield = 1.02 g, 73%). ^1H NMR (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 4.0 Hz, 2H), 7.28 (d, *J* = 4.0 Hz, 2H), 7.21 (d, *J* = 4.0 Hz, 2H), 7.16-7.12 (m, 6H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.85-1.75 (m, 12H), 1.49-1.24 (m, 180H), 0.88 (t, *J* = 6.4 Hz, 18H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.73, 152.89, 145.21, 142.19, 141.76, 137.95, 136.41, 135.69, 135.31, 135.16, 132.62, 126.47, 125.04, 124.69, 123.96, 107.99, 73.61, 69.32, 31.92, 30.35, 29.76, 29.74, 29.72, 29.67, 29.64, 29.58, 29.40, 29.36, 29.31, 26.07, 22.69, 14.12. MS (MALDI-TOF): *m*/*z* 2313.2 $[M+H]^+$; calcd 2312.7. Anal. calcd for $C_{146}H_{238}O_8S_6$: C, 75.79; H, 10.37%; found: C, 75.64; H, 10.51%.

Instrumentation. ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded on a JEOL JNM-LA400 spectrometer. Mass spectra were obtained with a PerSeptive Biosystems Voyager-DE STR spectrometer. Elemental analyses were carried out with a Yanaco MT-6 CHN autocorder. Differential scanning calorimetry (DSC) measurements were performed on a NETZSCH DSC204 Phoenix calorimeter at a scanning rate of 5 °C min^{-1}. A polarizing optical microscope Olympus BH-51 equipped with Mettler FP82 HT hot stage was used for visual observation. FT-IR measurements were conducted on a JASCO FT/IR-660 Plus spectrometer equipped with a JASCO IRT-30 microscope and a Mettler FP82 HT hot-stage. Oriented samples were prepared by mechanically shearing polydomain samples in sandwiched KBr crystals or glass pates.[3] X-ray diffraction (XRD) patterns were obtained using a Rigaku RINT-2500 diffractmeter with a heating stage using Ni-filtered CuKα radiation. UV-vis absorption and photoluminescence (PL) spectra were measured with a Agilent 8453 and a JASCO FP-777W spectrometers, respectively. The excitation wavelength used was that of the UV-vis absorption maximum of each sample.

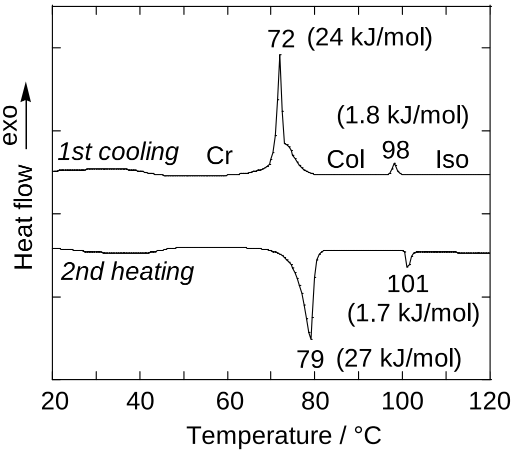


Fig. S1 DSC thermogram of **1a** at a scanning rate of 5 °C min^{-1}.

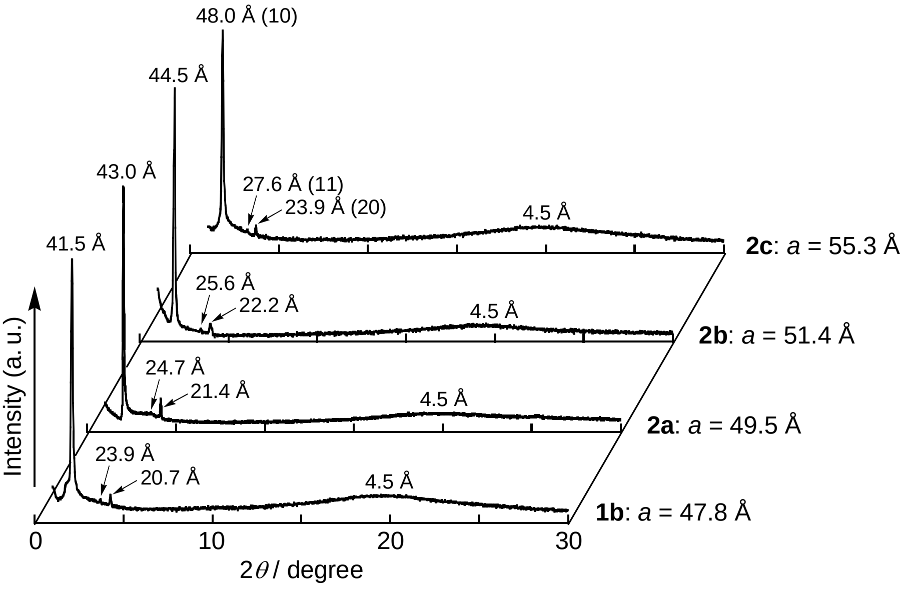


Fig. S2 X-ray diffraction patterns of **1b** at 90 °C and **2a-c** at 105 °C in $\mathrm{Col_h}$ phases.

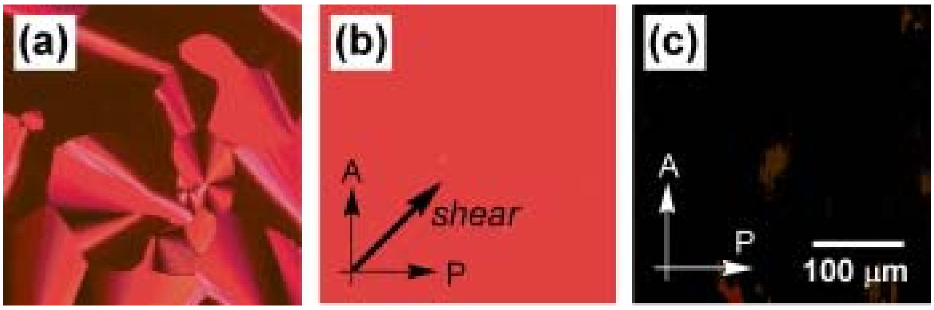


Fig. S3 Polarised optical photomicrographs of **2c** in the Col$_h$ phase at 105 °C: (a) before shearing; (b) and (c) after shearing. The birefringence of the sample is extinguished when the shearing direction is aligned with the polariser (P) and analyzer (A) axes.

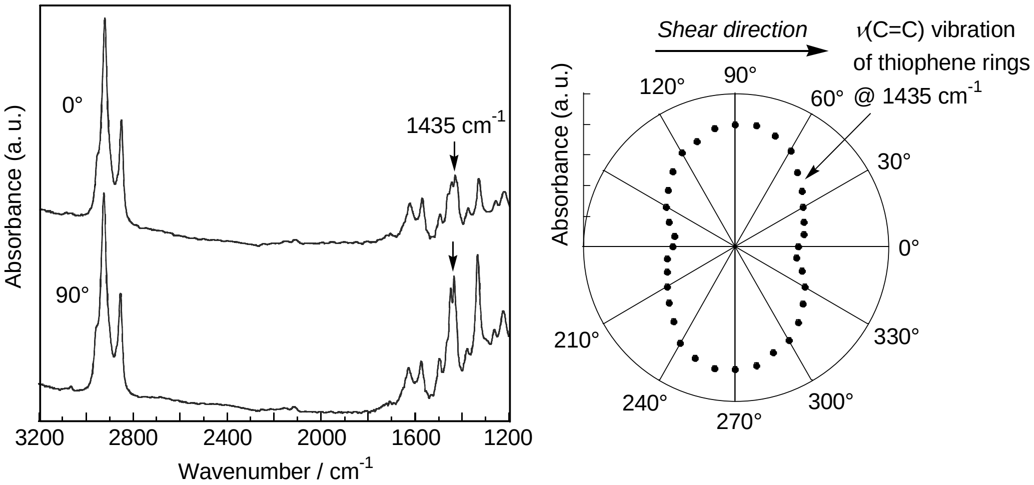
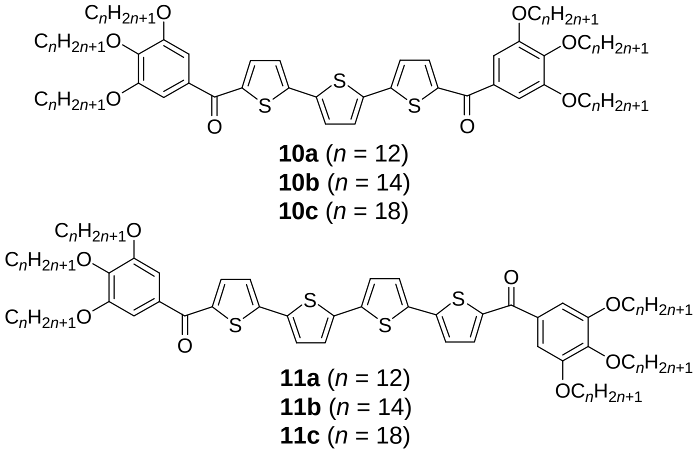


Fig. S4 Polarised IR spectra and polar plots of absorbance at 1435 cm^{-1} (aromatic C=C stretching vibration) with different polarisation angles for the uniaxially oriented **1a** in the Col$_h$ phase.

Scheme S2. Structures of analogous non-mesomorphic compounds.



10a (n = 12)
10b (n = 14)
10c (n = 18)

11a (n = 12)
11b (n = 14)
11c (n = 18)

Table S1 Thermal properties of **10a-c** and **11a-c**

Compound	Phase transition behaviour [a]
10a	Cr 88 Iso
10b	Cr 71 Cr′ 79 Iso
10c	Cr 84 Iso
11a	Cr 94 Cr′ 101 Iso
11b	Cr 103 Iso
11c	Cr 102 Iso

[a] Transition temperatures (°C) determined by DSC (second heating; 5 °C min^{-1}). Cr: crystalline; Iso: isotropic.

Selected data: 10a. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, J = 4.0 Hz, 2H), 7.31 (s, 2H), 7.25 (d, J = 4.0 Hz, 2H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.86-1.75 (m, 12H), 1.49-1.25 (m, 108H), 0.88 (t, J = 6.8 Hz, 18H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 186.74, 152.91, 144.63, 142.33, 142.26, 137.07, 135.22, 132.47, 126.58, 124.45, 108.00, 73.61, 69.32, 31.94, 31.92, 30.34, 29.75, 29.70, 29.66, 29.63, 29.58, 29.40, 29.36, 29.31, 26.08, 22.69, 14.12. Anal. calcd for $C_{98}H_{160}O_8S_3$: C, 75.33; H, 10.32%; found: C, 75.17; H, 10.46%.

11a. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, J = 4.0 Hz, 2H), 7.29 (d, J = 4.0 Hz, 2H), 7.22 (d, J = 4.0 Hz, 2H), 7.18 (d, J = 4.0 Hz, 2H), 7.10 (s, 4H), 4.08-4.01 (m, 12H), 1.85-1.75 (m, 12H), 1.49-1.25 (m, 108H), 0.88 (t, J = 6.8 Hz, 18H). $^{13}C\{^1H\}$ NMR

(100 MHz, $CDCl_3$): δ 186.73, 152.89, 145.00, 142.19, 141.94, 137.49, 135.68, 135.28, 132.54, 126.47, 125.17, 124.10, 107.98, 73.61, 69.31, 31.93, 30.91, 30.34, 29.75, 29.69, 29.65, 29.63, 29.57, 29.39, 29.36, 29.31, 26.08, 22.68, 14.11. Anal. calcd for $C_{102}H_{162}O_8S_4$: C, 74.49; H, 9.93%; found: C, 74.34; H, 10.08%.

References

1 D. R. Coulson, *Inorg. Synth.*, 1972, **13**, 121.

2 (*a*) T. Olinga, S. Destri and W. Porzio, *Macromol. Chem. Phys.*, 1997, **198**, 1091; (*b*) M. Jayakannan, J. L. J. van Dongen and R. A. J. Janssen, *Macromolecules*, 2001, **34**, 5386.

3 M. Yoshio, T. Mukai, H. Ohno and T. Kato, *J. Am. Chem. Soc.*, 2004, **126**, 994.