Supplementary information for

Progression from Nano to Macro Science in Soft Matter Systems:

Dimers to Trimers and Oligomers in Twist-Bend Liquid Crystals

Richard J. Mandle and John W. Goodby

Department of Chemistry, University of York, York, UK, YO10 5DD

1.1. General Methods

Bis 1,9-(4-Hydroxyphenyl)nonane (1) was prepared as described previously. [1] 1-Ethyl-3-(3dimethylaminopropyl)carbodiimide (EDAC) was purchased from Carbosynth UK, *N*,*N*dimethylaminopyridine (DMAP) was purchased from Sigma Aldrich. Solvents were purchased from Fisher Scientific UK and were dried *via* passage over activated alumina prior to use. Reactions were monitored by thin layer chromatography (TLC) with DCM as the eluent. Silica coated aluminium TLC plates used were purchased from Merck (Kieselgel 60 F-254) and visualised using either UV light (254 nm and 365 nm), or by oxidation with either iodine or aqueous potassium permanganate solution. Yields refer to chromatographically (HPLC) and spectroscopically (¹H NMR, ¹³C NMR) homogenous material.

NMR spectra were recorded on a JEOL ECX spectrometer operating at 400 MHz (¹H), 100.5 MHz (¹³C) and 376.4 MHz (¹⁹F). Mass spectra were recorded on a Bruker micrOTOF MS-Agilent series 1200LC spectrometer. FT-IR spectroscopy was performed using a Shimadzu IR Prestige-21 with temperature controlled Specac Golden Gate diamond ATR IR insert. Polarised optical microscopy was performed on a Zeiss Axioskop 40Pol microscope using a Mettler FP82HT hotstage controlled by a Mettler FP90 central processor. Photomicrographs were captured *via* an InfinityX-21 digital camera mounted atop the microscope. Differential scanning calorimetry was performed on a Mettler DSC822^e calibrated before use against indium and zinc standards under an atmosphere of dry nitrogen. DSC thermograms were processed in Matlab. Computational chemistry was performed using the using Gaussian G09 revision d01 on the York Advanced Research Computing Cluster (YARCC) as described in the text. [2]

Small angle X-ray diffraction was performed using a Bruker D8 Discover equipped with a temperature controlled, bored graphite rod furnace, custom built at the University of York. The radiation used was copper K α (λ = 0.154056 nm) from a 1 µS microfocus source. Diffraction patterns were recorded on a 2048x2048 pixel Bruker VANTEC 500 area detector.

Samples were filled into 1mm capillary tubes and aligned with a pair of 1T magnets. Diffraction patterns were collected as a function of temperature and the data processed using Matlab. Raw data are available upon request from the University of York data catalogue.

1.2. Synthetic Details



1: 4-(9-(4-Hydroxyphenyl)nonyl)phenyl 4-cyanobenzoate

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2: Nonane-1,9-diylbis(4,1-phenylene) bis(4-cyanobenzoate)

A suspension of *bis* 1,9-(4-hydroxyphenyl)nonane (2 g, 6.41 mmol), 4-cyanobenzoic acid (1.9 g, 12.82 mmol), EDAC (3.67 g, 19.2 mmol) and DMAP (< 50 mg) in DCM (50 ml) was stirred for 16 h. The reaction solution was concentrated *in vacuo* and purified by flash chromatography with 3:2 hexanes/DCM as the eluent affording compounds **1** ($Rf_{DCM} = 0.35$, 28.3 % yield) and **2** ($Rf_{DCM} = 0.66$, 54.7% Yield). Spectral data for **2** was in keeping with that reported previously. [1] Compound **1** was obtained as a white powder following recrystalisation from methanol/1,4-dioxane (8:1).

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<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 1.22 - 1.37 (10H, m, Ar-CH<sub>2</sub>-CH<sub>2</sub>-(CH<sub>2</sub>)<sub>5</sub>-CH<sub>2</sub>-CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>OH),
1.49 - 1.64 (4H, m, Ar-CH<sub>2</sub>-CH<sub>2</sub>-(CH<sub>2</sub>)<sub>5</sub>-CH<sub>2</sub>-CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>OH),
2.50 (2H, t, J = 7.8 Hz, Ar-CH<sub>2</sub>-CH<sub>2</sub>-(CH<sub>2</sub>)<sub>5</sub>-CH<sub>2</sub>-CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>OH),
2.60 (2H, t, J = 7.8 Hz, Ar-CH<sub>2</sub>-CH<sub>2</sub>-(CH<sub>2</sub>)<sub>5</sub>-CH<sub>2</sub>-CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>OH),
4.78 (1H, s, -CH<sub>2</sub>-C<sub>6</sub>H<sub>4</sub>-OH), 6.72 (2H, ddd, J = 1.8 Hz, J = 2.8
Hz, J = 8.2 Hz, ArH), 7.01 (2H, ddd, J = 1.8 Hz, J = 2.3 Hz, J =
8.7 Hz, ArH), 7.09 (2H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.2 Hz,
ArH), 7.22 (2H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.2 Hz, ArH),
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	7.79 (2H, ddd, <i>J</i> = (2H, ddd, <i>J</i> = 1.8 H	= 1.8 Hz, <i>J</i> = 2.3 H Iz, <i>J</i> = 2.8 Hz, <i>J</i> = 8	lz, <i>J</i> = 8.7 Hz, Ar 3.2 Hz, ArH)	H), 8.28
¹³ C NMR (400 MHz, CDCl ₃):	29.15, 29.20, 29 116.90, 117.88, 133.52, 135.06, 14	.40, 31.41, 31.68 121.01, 129.39, ⁻ 11.11, 148.37, 153.4	, 34.99, 35.35, 129.47, 130.61, 44, 163.79	115.00, 132.36,
FT-IR (vmax, cm ⁻¹ , 115 °C):	702, 761, 823, 850 1415, 1504, 1606,), 877, 1014, 1070, 1730, 2850, 2920	1165, 1178, 119	3, 1263,
MS (ESI+, m/z):	480.1696 ($C_{29}H_{31}KNO_3$, calcd. for $C_{29}H_{31}KNO_3$ 480.1936, M + K), 464.2178 (100%, $C_{29}H_{31}NNaO_3$, calcd. for $C_{29}H_{31}NNaO_3$ 464.2196, M + Na),			
Elemental Analysis:	Observed:	C 78.618%, H 7	7.139%, N 3.171%	6

General Procedure for the Esterification of Compound 1 with Benzoic Acids

A solution of compound **1** (50 mg, 0.114 mmol), benzoic acid (0.15 mmol), EDAC (28.7 mg, 0.15 mmol) and DMAP (1 mg) in anhydrous DCM (2 ml) was stirred until complete consumption of **1** as evidenced by TLC (**1** Rf_{DCM} = 0.35). The crude material was purified by flash chromatography with DCM as the eluent. Recrystalisation of the chromatographed material from ethanol/THF afforded compounds **3-7**in 60-80 % yields.

Calcd. for C₂₉H₃₁NO₃: C 78.88%, H 7.08%, N 3.17%,



3: 4-(9-(4-((4-cyanobenzoyl)oxy)phenyl)nonyl)phenyl 4-isothiocyanatobenzoate

- ¹H NMR (400 MHz, CDCl₃): 1.25 1.36 (10H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar), 1.56 1.66 (4H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar), 2.57 2.64 (4H, m [overlapping t + t], Ar-CH₂-(CH₂)₇-CH₂-Ar), 7.05 7.11 (4H, m, ArH), 7.18 7.25 (4H, m, ArH), 7.31 (2H, ddd, J = 1.8 Hz, J = 2.3 Hz, J = 8.7 Hz, ArH), 7.79 (2H, d, J = 8.7 Hz, ArH), 8.17 (2H, ddd, J = 2.3 Hz, J = 8.7 Hz, J = 8.7 Hz, ArH), 8.28 (2H, d, J = 8.7 Hz, ArH)
- ¹³C NMR (100.5 MHz, CDCl₃): 29.21, 29.43, 29.45, 31.42, 35.35, 116.88, 117.88, 121.01, 121.15, 125.81, 128.14, 129.38, 129.47, 130.59, 131.60, 132.35, 133.52, 136.20, 138.11, 140.75, 141.07, 148.37, 148.57, 163.73, 164.18
- FT-IR (vmax, cm⁻¹): 684, 721, 758, 821, 860, 931, 1014, 1070, 1165, 1230, 1261, 1369, 1506, 1598, 1647, 2079, 2233, 2358, 2850, 2924, 2970, 3028
- MS (ESI+, m/z): 625.2108 (100%, $C_{37}H_{34}N_2NaO_4S$, cacld. for $C_{37}H_{34}N_2NaO_4S$ 625.2131, M + Na)



4: 4-(9-(4-((4-cyanobenzoyl)oxy)phenyl)nonyl)phenyl 4-nitrobenzoate

- ¹H NMR (400 MHz, CDCl₃): 1.26 1.38 (10H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar), 1.55 1.68 (4H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-Ar), 2.58 2.64 (4H, m [overlapping t + t], Ar-CH₂-(CH₂)₇-CH₂-Ar), 7.07 7.12 (4H, m, ArH), 7.20 2.25 (4H, m [m + s, CHCl₃], ArH), 7.79 (2H, ddd, J = 1.4 Hz, J = 1.8 Hz, H = 8.7 Hz, ArH), 8.28 (2H, ddd, J = 1.4 Hz, J = 1.8 Hz, H = 8.7 Hz, ArH), 8.31 8.38 (4H, m, ArH-NO₂)
- ^{13}C NMR (100.5 MHz, CDCl_3): 28.22, 28.43, 28.45, 30.42, 34.35, 115.89, 116.87, 120.00, 120.01, 122.68, 128.46, 128.48, 129.59, 130.24, 131.35, 132.51, 134.07, 140.06, 140.13, 147.36, 147.38, 149.82, 162.47, 162.73
- FT-IR (vmax, cm⁻¹): 686, 715, 761, 794, 854, 869, 1014, 1074, 1166, 1193, 1259, 1346, 1408, 1506, 1523, 1606, 1734, 2232, 2343, 2852, 2922
- MS (ESI+, m/z): 613.2311 (100%, $C_{36}H_{34}N_2NaO_6$, cacld. For $C_{36}H_{34}N_2NaO_6$ 613.2309, M + Na)



5: 4-(9-(4-((4-cyanobenzoyl)oxy)phenyl)nonyl)phenyl 4-fluorobenzoate

- ¹H NMR (400 MHz, CDCl₃): 1.26 1.38 (10H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar), 1.55 1.68 (4H, m, Ar-CH₂-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar), 2.57 2.65 (4H, m, Ar-CH₂-(CH₂)₇-CH₂-Ar), 7.09 (4H, m, J = 4.6 Hz, J = 8.0 Hz, ArH), 7.17 (2H, t, J = 8.6 Hz, ArH), 7.23 (4H, m, J = 12.7 Hz, J = 4.5 Hz, ArH), 7.80 (2H, d, J = 8.2 Hz, ArH), 8.20 (2H, m, J = 8.4 Hz, J = 5.6 Hz, ArH), 8.29 (2H, d, J = 8.2 Hz, ArH)
- ¹³C NMR (100.5 MHz, CDCl₃): 29.22, 29.43, 29.45, 31.42, 31.44, 35.35, 115.73 (d, J = 22.1 Hz), 116.87, 117.88, 121.01, 121.23, 125.88 (d, J = 2.8 Hz), 129.35, 129.47, 130.59, 132.35, 132.68, 132.73 (d, J = 9.5 Hz), 133.52, 140.63, 141.07, 148.37, 148.65, 163.73, 164.38, 164.81, 166.07 (d, J = 254.9 Hz)

¹⁹F NMR (376.4 MHz, CDCl₃): -104.54 - -104.42 (m, ArF)

- FT-IR (vmax, cm⁻¹): 684, 721, 758, 786, 881, 1016, 1076, 1153, 1168, 1201, 1263, 1415, 1467, 1504, 1604, 1726, 2233, 2360, 2850, 2920
- MS (ESI+, m/z): 586.2382 (100%, $C_{36}H_{34}FNNaO_4$, calcd. for $C_{36}H_{34}FNNaO_4$ 586.2364, M + Na)



6: 4-(9-(4-((4-cyanobenzoyl)oxy)phenyl)nonyl)phenyl 4-(trifluoromethyl)benzoate

- ¹H NMR (400 MHz, CDCl₃): 1.26 1.38 (10H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar'), 1.61 (4H, quintet, J = 6.9 Hz, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar'), 2.62 (4H, t, J = 6.9 Hz, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar'), 7.10 (4H, ddd, J = 2.8 Hz, J = 3.2 Hz, J = 8.7 Hz, ArH), 7.23 (4H, d, J = 8.2 Hz, ArH), 7.76 (2H, d, J = 8.2 Hz, ArH), 7.80 (2H, ddd, J = 1.4, J = 1.8 Hz, J = 8.2 Hz, ArH), 8.27 8.32 (4H, m, ArH)
- ¹³C NMR (100.5 MHz, CDCl₃): 29.22, 29.44, 29.46, 31.43, 35.36, 116.89, 117.88, 121.01, 121.12, 125.57 (quartet, *J* = 3.5 Hz), 129.43, 129.47, 130.52, 130.59, 132.36, 132.90, 133.52, 135.08, 140.91, 141.08, 148.38, 148.50, 163.74, 164.15,
- ¹⁹F NMR (376.4 MHz, CDCl₃): -63.01 (S, Ar-CF₃)
- FT-IR (vmax, cm⁻¹): 686, 700, 721, 758, 769, 860, 1016, 1078, 1134, 1166, 1263, 1327, 1411, 1506, 1728, 2233, 2360, 2850, 2922
- MS (ESI+, m/z): 636.2316 (100%, $C_{37}H_{34}F_3NNaO_4$, cacld. for $C_{37}H_{34}F_3NNaO_4$ 626.2332, M + Na)



7: 4-(9-(4-((4-cyanobenzoyl)oxy)phenyl)nonyl)phenyl 4-(pentafluoro-I6sulfanyl)benzoate

- ¹H NMR (400 MHz, CDCl₃): 1.26 1.39 (10H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar'), 157 166 (4H, m, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar'), 2.62 (4H, t, J = 6.7 Hz, Ar-CH₂-CH₂-(CH₂)₅-CH₂-CH₂-Ar'), 7.09 (4H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 7.23 (4H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 7.80 (2H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 7.80 (2H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 7.89 (2H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 8.26 8.30 (4H, m, ArH)
- ¹³C NMR (100.5 MHz, CDCl₃): 29.21, 29.43, 29.45, 31.42, 35.35, 116.88, 117.87, 121.01, 121.05, 126.34 (t, *J* = 4.6 Hz), 129.45, 130.58, 132.35, 132.55, 133.51, 141.01, 141.06, 148.38, 148.41, 157.10 (m), 163.62, 163.73
- ¹⁹F NMR (376.4 MHz, CDCl₃): 62.47 (4F, d, *J* = 151.7 Hz, SF_{eq.}) 83.37 (1F, quintet, *J* = 151.7 Hz, SF_{ax.})
- FT-IR (vmax, cm⁻¹): 665, 686, 721, 758, 829, 879, 1016, 1078, 1166, 1190, 1265, 1408, 1465, 1506, 1728, 2233, 2358, 2850, 2922





Quantities used: Compound *i1-1* (44 mg, 100 μ mol), terephthalic acid (8.3 mg, 50 μ mol) EDAC (19.1 mg, 100 μ mol), DMAP (1 mg), DCM (3 ml). The experimental procedure was as described for the unsymmetrical compounds. Column chromatography with DCM (Rf_{DCM} = 0.75) followed by recrystalisation from ethanol/THF (3:1) afforded compound **7** as an amorphous white powder.

Yield: 35 mg (70 %)

- ¹H NMR (400 MHz, CDCl₃): 1.24 1.38 (20H, m, -CH₂-), 1.56 1.66 (8H, m, -CH₂-), 2.61 (8H, t, J = 7.3 Hz, Ar-CH₂- x4), 7.08 (4H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 7.12 (4H, ddd, J = 1.8 Hz, J = 2.8 Hz, J = 8.7 Hz, ArH), 7.22 7.25 (4H, m, ArH), 7.79 (4H, ddd, J = 1.4 Hz, J = 1.8 Hz, J = 8.7 Hz, ArH), 8.27 (4H, ddd, J = 1.4 Hz, J = 1.8 Hz, J = 8.7 Hz, ArH), 8.29 (4H, s, ArH)
- ^{13}C NMR (100.5 MHz, CDCl_3): 28.22. 28.43, 28.46, 30.43, 34.36, 115.88, 116.87, 120.01, 120.15, 128.41, 128.47, 129.21, 129.59, 131.35, 132.52, 132.94, 139.85, 140.07, 147.38, 147.58, 162.73, 163.47
- FT-IR (vmax, cm⁻¹): 685, 721, 759, 824, 861, 1016, 1073, 1166, 1199, 1265, 1365, 1408, 1466, 1506, 1559, 1609, 1653, 1729, 2233, 2848, 2918, 3028
- MS (ESI+, m/z): 1013.4752 (100%, $C_{66}H_{64}N_2O_8$, M+H, calcd. for $C_{66}H_{64}N_2O_8$ 1013.4735)



Bis(4-(9-(4-((4-cyanobenzoyl)oxy)phenyl)nonyl)phenyl)4,4'-(nonane-1,9-diyl)dibenzoate (3) (RM1697)

Quantities used: Compound **1** (88 mg, 0.201 mmol), 4,4'-(nonane-1,9-diyl)dibenzoic acid (37 mg, 0.101 mmol) EDAC (42 mg, 0.221 mmol), DMAP (1 mg), DCM (3.5 ml). The experimental procedure was as described for compound **1**. Column chromatography with DCM ($Rf_{DCM} = 0.70$) followed by recrystalisation from ethanol/THF (5:2) afforded compound **4** as an amorphous white powder.

90 mg	(74%)
	90 mg (

- ¹H NMR (400 MHz, CDCl₃): 1.25 1.37 (30 H, m, $-CH_{2}$ -), 1.55 1.66 (12H, m, $-CH_{2}$ -), 2.56 2.65 (8H, m [t + t], Ar- CH_{2} x4), 2.67 (4H, t, *J* = 7.3 Hz, Ar-CH₂-), 7.07 (4H, d, *J* = 8.2 Hz, Ar*H*), 7.09 (4H, d, *J* = 8.2 Hz, Ar*H*), 7.19 (4H, d, *J* = 8.2 Hz, Ar*H*), 7.22 (4H, d, *J* = 8.2 Hz, Ar*H*), 7.28 (4H, d, *J* = 8.2 Hz, Ar*H*), 7.79 (4H, d, *J* = 8.2 Hz, Ar*H*), 8.08 (4H, d, *J* = 8.2 Hz, Ar*H*), 8.28 (4H, d, *J* = 8.2 Hz, Ar*H*)
- ^{13}C NMR (100.5 MHz, CDCl_3): 29.21, 29.24, 29.44, 31.13, 31.42, 31.46, 35.36, 36.06, 116.88, 117.89, 121.01, 121.35, 127.09, 128.60, 129.29, 129.48, 130.20, 130.60, 132.35, 133.55, 140.39, 141.09, 148.38, 148.86, 149.21, 163.73, 165.42,

FT-IR (vmax, cm⁻¹): 686, 720, 759,828, 860, 940, 1016, 1072, 1112, 1166, 1195, 1264, 1408, 1467, 1506, 1609, 1729, 2232, 2848, 2918, 3053

- MS (ESI+, m/z): 1215.6446 (100%, $C_{81}H_{87}N_2O_8$, M+H, calcd. for $C_{81}H_{87}N_2O_8$ 1215.6457)
- Elemental Analysis: Observed: C 79.643%, H 7.133%, N 2.218% Calcd. for C₈₁H₈₆N₂O₈: C 80.03%, H 7.13%, N 2.30%

1.3. NMR Data



Figure SI1: ¹H NMR spectra (400 MHz, CDCI₃) of compound 3



Figure SI2: ¹³C NMR spectra (100.5 MHz, CDCl₃) of compound **3**



Figure SI3: ¹H NMR spectra (400 MHz, CDCI₃) of compound **4**



Figure SI4: ¹³C NMR spectra (100.5 MHz, CDCl₃) of compound 4



Figure SI5: ¹H NMR spectra (400 MHz, CDCI₃) of compound 5



Figure SI6: ¹³C NMR spectra (100.5 MHz, CDCl₃) of compound 5



Figure SI7: ¹⁹F NMR spectra (376.4 MHz, CDCl₃) of compound 5



Figure S8: ¹H NMR spectra (400 MHz, CDCl₃) of compound 6



Figure SI9: ¹³C NMR spectra (100.5 MHz, CDCl₃) of compound 6



Figure SI10: ¹⁹F NMR spectra (376.4 MHz, CDCI₃) of compound 6





Figure SI12: ¹³C NMR spectra (100.5 MHz, CDCl₃) of compound 7



Figure SI13: ¹⁹F NMR spectra (376.4 MHz, CDCI₃) of compound 7



Figure SI14: ¹H NMR (400 MHz, CDCI₃) of compound 8



Figure SI15: Gradient DQF-COSY ¹H-¹H NMR (400 MHz, CDCI₃) of compound 8



Figure SI16: ¹³C NMR (100.5 MHz, CDCl₃) of compound 8



Figure SI17: ¹H NMR (400 MHz, CDCI₃) of compound 9



Figure SI18: ¹³C NMR (100.5 MHz, CDCl₃) of compound 9

1.4. DSC Thermograms



Figure SI19: DSC Thermogram (10 °C min⁻¹, 1st cool) of compound **3** with expansions showing the nematic to isotropic (bottom right) and nematic to N_{TB} transitions (bottom left)



Figure SI20: DSC Thermogram (10 °C min⁻¹, 1st cool) of compound **4** with expansions showing the nematic to isotropic (bottom right) and nematic to N_{TB} transitions (bottom left)



Figure SI21: DSC Thermogram (10 °C min⁻¹, 1st cool) of compound with expansions showing the nematic to 5 isotropic (bottom right) and nematic to N_{TB} transitions (bottom left)



Figure SI22: DSC Thermogram (10 °C min⁻¹, 1st cool) of compound **6** with expansions showing the nematic to isotropic (bottom right) and nematic to N_{TB} transitions (bottom left)



Figure SI23: DSC Thermogram (10 °C min⁻¹, 1st cool) of compound **7** with expansions showing the nematic to isotropic (bottom right) and nematic to N_{TB} transitions (bottom left)



Figure SI24: DSC thermogram of compound **8** on cooling. The insets show the twistbend nematic to nematic phase transition (top left) and nematic to isotropic transition (top right).

1.5. Small angle X-ray



Figure SI25: Plot of integrated X-ray diffraction intensity as a function of temperature (range 207-141 °C, 3 ° steps) for compound **8**.

	Small Angle	Wide Angle
	2θ = 3 - 15	2θ = 15 - 25
T (°C)		
		1
147	21.4	4.5
150	21.4	4.6
153	21.4	4.5
156	21.3	4.7
159	21.3	4.6
162	21.3	4.7
165	21.5	4.6
168	21.4	4.7
171	21.4	4.6
174	21.8	4.7
177	21.8	4.8
180	22	4.8
183	22	4.7
186	22	4.8
189	22.1	4.7
192	22	4.8
195	21.6	4.8
198	21.6	4.9
201	21.8	4.7
204	21.8	4.8
207	21.8	4.8

 Table SI1:
 Tabulated d-spacings for wide-angle and small-angle SAXS peaks as a function of temperature for compound 8.



Figure SI26: Plot of integrated X-ray diffraction intensity as a function of temperature (range 145-103 °C, 2 ° steps) for compound **9**.

	Small Angle	Wide Angle
	2θ = 3 - 15	2θ = 15 - 25
T (°C)		
101	17 /	47
101	17.4	4.7
103	17.0	4.8
105	17.3	4.8
107	17.5	4.7
109	17.7	4.8
111	17.5	4.8
113	17.2	4.8
115	17.1	4.7
117	17.1	4.7
119	17.1	4.7
121	17	4.7
123	17	4.8
125	16.9	4.7
127	17	4.6
129	17	4.6
131	16.8	4.6
133	16.9	4.6
135	17.1	4.7
137	17	4.6
139	17	4.7
141	17.1	4.6
143	16.9	4.6
145	16.8	4.6

 Table SI1:
 Tabulated d-spacings for wide-angle and small-angle SAXS peaks as a function of temperature for compound 9.

References

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