Supplementary Information

Synthesis and mesomorphism of triphenylene-based dimers with a highly ordered columnar plastic phase

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1. Synthesis and characterization

**1, 2-dibutoxybenzene (1)**

1-Bromobutane (25.7g, 0.187mol) was added to a vigorously stirred solution of catechol (5.5 g, 0.05mol) and potassium carbonate (27.6g) in ethanol (500ml) under nitrogen. The reaction mixture was stirred under reflux for 24 h and filtered with copious washings of ethanol. The filtrate was concentrated in vacuo and subjected to a silica gel column chromatography on silica, eluting with 1:2 dichloromethane: light petroleum to give the product as pale yellow oil. (10.6 g, 95%); TLC Rf: 0.55 (dichloromethane-hexane 1:1); IR (KBr): $\nu_{\text{max}}/\text{cm}^{-1}$ 1263 (C-O-C); $\delta_H$ (300MHz, CDCl$_3$) 6.90 (4H, s, ArH), 3.99-4.10 (4H, t, OCH$_2$), 1.77-1.86 (4H, m, OCH$_2$CH$_2$), 1.46-1.58 (m, 4H, OCH$_2$CH$_2$CH$_2$), 0.97-1.07 (t, 6H, CH$_3$).

**2, 3, 6, 7, 10, 11-hexabutoxytriphenylene (2)**

Compound 1 (7.5g, 0.0337mol) was added to a vigorously stirred suspension of Iron(III) chloride (16.42g, 0.0101mol) in dichloromethane (50ml). The reaction occurred with vigorous evolution of gas and was quenched with methanol (150ml) after 70 min. The reaction mixture was filtered and the filtrate concentrated in vacuo to give a black solid which was subjected to a silica gel column chromatography, eluting with 1:1 dichloromethane: light petroleum to give 2 as pale yellow solid.
which was recrystallized from ethanol. (16.95g, 76%); TLC Rf: 0.65 (dichloromethane-hexane 1:1); IR (KBr): v_{max}/cm^{-1} 1261 (C-O-C); δ_{H} (300MHZ, CDCl_{3}) 7.85 (6H, s, ArH), 4.22-4.26 (12H, t, OCH_{2}), 1.89-1.98 (12H, m, OCH_{2}CH_{2}), 1.62-1.67 (12H, m, OCH_{2}CH_{2}CH_{2}), 1.02-1.07 (18H, t, CH_{3}).

2-hydroxy -3, 6, 7, 10, 11-Pentabutoxytriphenylene (3)

To a cooled suspension of catechol (11g, 0.1mol) in CH_{2}Cl_{2} (50 mL), a solution (0 °C) of BBr_{3} (28.6g, 0.11mol) in CH_{2}Cl_{2} (10 ml) was added slowly with stirring 3h under nitrogen. The mixture was brought to room temperature, the solvent removed and the product distilled under vacuum to give B-Bromocatecholboronane as white solid (16g, 80%). The solid was then used to make a 0.5 M solution by mixing with CH_{2}Cl_{2} (160 ml) and this was used for next ether cleavage reactions.

A solution of 2 (15g, 0.0227mol) was dissolved in anhydrous CH_{2}Cl_{2} (150 ml) and cooled to 0°C. To this was added (64ml, 0.032mol) of B-Bromocatecholboronane solution in CH_{2}Cl_{2} under argon and the mixture was stirred at room temperature for 24h. After that it was poured over ice-water and extracted with CH_{2}Cl_{2}, the combined extract was dried with anhydrous Na_{2}SO_{4} overnight, solvent was removed under vacuum and the crude product was purified by a silica gel column chromatography, eluting with 1: 30 ethyl acetate: light petroleum to give 3 as white solid which was recrystallized from ethanol. (6.5g, 49%); TLC Rf: 0.56 (ethyl acetate- hexane 1:8); IR (KBr): v_{max}/cm^{-1} 3456 (O-H), 1261 (C-O-C); δ_{H} (300MHZ, CDCl_{3}) 7.78-7.97 (6H, m, ArH), 5.91 (1H, s, OH) 4.20-4.38 (10H, t, OCH_{2}), 1.89-1.96 (10H, m, OCH_{2}CH_{2}), 1.57-1.67 (10H, m, OCH_{2}CH_{2}CH_{2}), 1.05-1.09 (18H, t, CH_{3}).

1, 2-Bis (3′, 6′, 7′, 10′, 11′-pentabutoxytriphenylen-2′-yloxy)-ethane (4a)

A mixture of 3 (500mg, 0.827mmol), 1, 2-Dibromoethane (78mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4a. (0.25g, 49%); TLC Rf: 0.15 (dichloromethane-hexane 2:1); (Found: C, 75.98; H, 8.87. C_{78}H_{106}O_{12} requires: C, 75.82 ;H, 8.65%); IR (KBr): v_{max}/cm^{-1} 1260 (C-O-C); δ_{H} (300MHZ, CDCl_{3}) 7.81-8.09 (12H, m, ArH), 4.74 (4H, t, OCH_{2}CH_{2}O), 4.08-4.22 (20H, m, OCH_{2}), 1.74-1.96 (20H, m, OCH_{2}CH_{2}), 1.43-1.69 (20H, m, OCH_{2}CH_{2}CH_{2}), 1.02-1.07 (30H, m, CH_{3}); HRMS (ESI) : calc. m/z 1234.7679 (C_{78}H_{106}O_{12}), found
m/z 1234.7652 (M$^+$).

1, 3-Bis (3$^\prime$, 6$^\prime$, 7$^\prime$, 10$^\prime$, 11$^\prime$-pentabutyloxytriphenylen-2$^\prime$-yloxy)-propane (4b)

A mixture of 3 (500mg, 0.827mmol), 1, 3-Dibromopropane (83mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2 × 50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4b. (0.32g, 63%); TLC Rf: 0.17 (dichloromethane-hexane 2:1); (Found: C, 75.76; H, 8.71. C$_{79}$H$_{108}$O$_{12}$ requires: C, 75.93; H, 8.71%); IR (KBr): $\nu_{\text{max}}$/cm$^{-1}$ 1261 (C-O-C); $\delta_H$ (300MHZ, CDCl$_3$) 7.81-7.96 (12H, s, ArH), 4.58 (4H, t, OCH$_2$), 4.13-4.27 (20H, t, OCH$_2$), 2.57 (2H, m, OCH$_2$CH$_2$), 1.79-1.93 (20H, m, OCH$_2$CH$_2$), 1.47-1.62 (20H, m, OCH$_2$CH$_2$CH$_2$), 0.89-1.07 (30H, m, CH$_3$); HRMS (ESI) : calc. m/z 1248.7835 (C$_{79}$H$_{108}$O$_{12}$), found m/z 1248.7822 (M$^+$).

1, 4-Bis (3$^\prime$, 6$^\prime$, 7$^\prime$, 10$^\prime$, 11$^\prime$-pentabutyloxytriphenylen-2$^\prime$-yloxy)-butane (4c)

A mixture of 3 (500mg, 0.827mmol), 1, 4-Dibromobutane (89mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2 × 50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4c. (0.30g, 59%); TLC Rf: 0.20 (dichloromethane-hexane 2:1); (Found: C, 75.90; H, 8.82. C$_{80}$H$_{110}$O$_{12}$ requires: C, 76.03; H, 8.77%); IR (KBr): $\nu_{\text{max}}$/cm$^{-1}$ 1261 (C-O-C); $\delta_H$ (300MHZ, CDCl$_3$) 7.85 (12H, s, ArH), 4.40 (4H, t, OCH$_2$), 4.18-4.25 (20H, t, OCH$_2$), 2.26 (4H, m, OCH$_2$CH$_2$), 1.82-1.95 (20H, m, OCH$_2$CH$_2$), 1.53-1.65 (20H, m, OCH$_2$CH$_2$CH$_2$), 1.05 (30H, t, CH$_3$); HRMS (ESI) : calc. m/z 1262.7992 (C$_{80}$H$_{110}$O$_{12}$), found m/z 1262.7968 (M$^+$).

1, 5-Bis (3$^\prime$, 6$^\prime$, 7$^\prime$, 10$^\prime$, 11$^\prime$-pentabutyloxytriphenylen-2$^\prime$-yloxy)-pentane (4d)

A mixture of 3 (500mg, 0.827mmol), 1, 5-Dibromopentane (95mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2 × 50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4d. (0.27g, 51%); TLC Rf:
0.28 (dichloromethane-hexane 2:1); (Found: C, 76.14; H, 8.85. C₈₁H₁₁₂O₁₂ requires: C, 76.14; H, 8.84%); IR (KBr): ν max/cm⁻¹ 1261 (C-O-C); δ H (300MHZ, CDCl₃) 7.86 (12H, s, ArH), 4.24 (24H, t, OCH₂), 2.11 (4H, t, OCH₂), 1.89-2.08 (22H, m, CH₂), 1.53-1.65 (24H, m, OCH₂CH₂CH₂), 1.05 (30H, t, CH₃); HRMS (ESI) : calc. m/z 1276.8148 (C₈₁H₁₁₂O₁₂), found m/z 1276.8123 (M)+.

1, 6-Bis (3′, 6′, 7′, 10′, 11′-pentabutyloxytriphenylen-2′-yloxy)-hexane (4e)

A mixture of 3 (500mg, 0.827mmol), 1, 6-Dibromohexane (101mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4e. (0.26g, 49%); TLC Rf: 0.30 (dichloromethane-hexane 2:1); (Found: C, 76.17; H, 8.94. C₈₂H₁₁₄O₁₂ requires: C, 76.24; H, 8.90%); IR (KBr): ν max/cm⁻¹ 1261 (C-O-C); δ H (300MHZ, CDCl₃) 7.84 (12H, s, ArH), 4.24 (24H, t, OCH₂), 1.92-2.03 (24H, m, OCH₂CH₂), 1.57-1.73 (24H, m, OCH₂CH₂CH₂), 1.05 (30H, t, CH₃); HRMS (ESI) : calc. m/z 1290.8305 (C₈₂H₁₁₄O₁₂), found m/z 1290.8314 (M)+.

1, 7-Bis (3′, 6′, 7′, 10′, 11′-pentabutyloxytriphenylen-2′-yloxy)-heptane (4f)

A mixture of 3 (500mg, 0.827mmol), 1, 7-Dibromoheptane (107mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4f. (0.31g, 58%); TLC Rf: 0.34 (dichloromethane-hexane 2:1); (Found: C, 76.26; H, 9.02. C₈₃H₁₁₆O₁₂ requires: C, 76.34; H, 8.95%); IR (KBr): ν max/cm⁻¹ 1261 (C-O-C); δ H (300MHZ, CDCl₃) 7.84 (12H, s, ArH), 4.24 (24H, t, OCH₂), 1.89-1.98 (24H, m, OCH₂CH₂), 1.56-1.68 (26H, m, CH₂), 1.05 (30H, t, CH₃); HRMS (ESI) : calc. m/z 1304.8461 (C₈₃H₁₁₆O₁₂), found m/z 1304.8436 (M)+.

1, 8-Bis (3′, 6′, 7′, 10′, 11′-pentabutyloxytriphenylen-2′-yloxy)-octane (4g)

A mixture of 3 (500mg, 0.827mmol), 1, 8-Dibromoctane (112mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting
with dichloromethane and finally recrystallized from ethanol to give pure 4g. (0.22g, 41%); TLC Rf: 0.35 (dichloromethane-hexane 2:1); (Found: C, 76.38; H, 8.95. C_{84}H_{118}O_{12} requires: C, 76.44; H, 9.01%); IR (KBr): \nu_{max}/cm^{-1} 1261 (C-O-C); \delta_{H} (300MHZ, CDCl_{3}) 7.84 (12H, s, ArH), 4.24 (24H, t, OCH_{2}), 1.89-1.96 (24H, m, OCH_{2}CH_{2}), 1.51-1.70 (28H, m, CH_{2}), 1.05 (30H, t, CH_{3}); HRMS (ESI) : calc. m/z 1318.8618 (C_{84}H_{118}O_{12}), found m/z 1318.8598 (M)^+.

1, 9-Bis (3', 6', 7', 10', 11'-pentabutyloxytriphenylen-2'-yloxy)-nonane (4h)
A mixture of 3 (500mg, 0.827mmol), 1, 9-Dibromonane (118mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4h. (0.29g, 52%); TLC Rf: 0.40 (dichloromethane-hexane 2:1); (Found: C, 76.57; H, 9.0. C_{85}H_{120}O_{12} requires: C, 76.54; H, 9.07%); IR (KBr): \nu_{max}/cm^{-1} 1260 (C-O-C); \delta_{H} (300MHZ, CDCl_{3}) 7.84 (12H, s, ArH), 4.25 (24H, t, OCH_{2}), 1.83-1.98 (24H, m, OCH_{2}CH_{2}), 1.46-1.68 (30H, m, CH_{2}), 1.05 (30H, t, CH_{3}); HRMS (ESI) : calc. m/z 1332.8774 (C_{85}H_{120}O_{12}), found m/z 1332.8784 (M)^+.

1, 10-Bis (3', 6', 7', 10', 11'-pentabutyloxytriphenylen-2'-yloxy)-decane (4i)
A mixture of 3 (500mg, 0.827mmol), 1, 10-Dibromodecane (124mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4i. (0.23g, 42%); TLC Rf: 0.45 (dichloromethane-hexane 2:1); (Found: C, 76.52; H, 9.09. C_{86}H_{122}O_{12} requires: C, 76.63; H, 9.12%); IR (KBr): \nu_{max}/cm^{-1} 1263 (C-O-C); \delta_{H} (300MHZ, CDCl_{3}) 7.85 (12H, s, ArH), 4.25 (24H, t, OCH_{2}), 1.83-1.98 (24H, m, OCH_{2}CH_{2}), 1.26-1.67 (32H, m, CH_{2}), 1.05 (30H, t, CH_{3}); HRMS (ESI) : calc. m/z 1346.8931 (C_{86}H_{122}O_{12}), found m/z 1346.8922 (M)^+.

1, 11-Bis (3', 6', 7', 10', 11'-pentabutyloxytriphenylen-2'-yloxy)-undecane (4j)
A mixture of 3 (500mg, 0.827mmol), 1, 11-Dibromoundecane (130mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml),
the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4j. (0.26g, 47%); TLC Rf: 0.45 (dichloromethane-hexane 2:1); (Found: C, 76.55; H, 9.15. C87H124O12 requires: C, 76.73; H, 9.18%); IR (KBr): ν\text{max}/cm\textsuperscript{-1} 1263 (C-O-C); δ\textsubscript{H} (300MHZ, CDCl\textsubscript{3}) 7.84 (12H, s, ArH), 4.25 (24H, t, OCH\textsubscript{2}), 1.89-1.96 (24H, m, OCH\textsubscript{2}CH\textsubscript{2}), 1.23-1.67 (34H, m, CH\textsubscript{2}), 1.05 (30H, t, CH\textsubscript{3}); HRMS (ESI) : calc. m/z 1360.9087 (C87H124O12), found m/z 1360.9038 (M)+.

1, 12-Bis (3′, 6′, 7′, 10′, 11′-pentabutyloxytriphenylene-2′-yloxy)-dodeane (4k)

A mixture of 3 (500mg, 0.827mmol), 1, 12-Dibromododecane (135mg, 0.413mmol) and anhydrous potassium carbonate (1.0g) in ethanol (20ml) was heated under reflux for 72h. The mixture was cooled to 0 °C, filtered, washed with water(50ml), and extracted with dichloromethane (2×50ml), the solvent removed in vacuo, and the residue purified by column chromatography on silica eluting with dichloromethane and finally recrystallized from ethanol to give pure 4k. (0.21g, 37%); TLC Rf: 0.48 (dichloromethane-hexane 2:1); (Found: C, 76.54; H, 9.10. C88H126O12 requires: C, 76.82; H, 9.23%); IR (KBr): ν\text{max}/cm\textsuperscript{-1} 1261 (C-O-C); δ\textsubscript{H} (300MHZ, CDCl\textsubscript{3}) 7.84 (12H, s, ArH), 4.25 (24H, t, OCH\textsubscript{2}), 1.89-1.96 (24H, m, OCH\textsubscript{2}CH\textsubscript{2}), 1.26-1.67 (36H, m, CH\textsubscript{2}), 1.05 (30H, t, CH\textsubscript{3}); HRMS (ESI) : calc. m/z 1374.9118 (C88H126O12), found m/z 1374.9148 (M)+.
2. $^1$H-NMR and HRMS spectra

![ESI Fig. 1 $^1$H-NMR spectra of 1]

![ESI Fig. 2 $^1$H-NMR spectra of 2]
ESI Fig. 3 $^1$H-NMR spectra of 3
ESI Fig. 4 $^1$H-NMR spectra of 4a

ESI Fig. 5 HRMS spectra of 4a
ESI Fig. 6 $^1$H-NMR spectra of 4b

ESI Fig. 7 HRMS spectra of 4b
ESI Fig. 8 $^1$H-NMR spectra of 4c

ESI Fig. 9 HRMS spectra of 4c
ESI Fig. 10 $^1$H-NMR spectra of 4d

ESI Fig. 11 HRMS spectra of 4d
ESI Fig. 12 $^1$H-NMR spectra of 4e

ESI Fig. 13 HRMS spectra of 4e
ESI Fig. 14: $^1$H-NMR spectra of 4f

ESI Fig. 15: HRMS spectra of 4f
ESI Fig. 16 $^1$H-NMR spectra of 4g

ESI Fig. 17 HRMS spectra of 4g
ESI Fig. 18 $^1$H-NMR spectra of 4h

ESI Fig. 19 HRMS spectra of 4h
ESI Fig. 20 $^1$H-NMR spectra of 4i

ESI Fig. 21 HRMS spectra of 4i
ESI Fig. 22 $^1$H-NMR spectra of 4j

ESI Fig. 23 HRMS spectra of 4j
ESI Fig. 24 $^1$H-NMR spectra of 4k

ESI Fig. 25 HRMS spectra of 4k
3. Mesomorphism

Mesomorphism of 4a

ESI Fig. 26 Crystalline texture observed by POM with 90° angle of compound 4a sandwiched between clean glass slides on cooling from isotropic phase at 140 °C (left); DSC trace of compound 4a run at 10 °C/min under N₂ (right).

Mesomorphism of 4b

ESI Fig. 27 Crystalline texture observed by POM with 90° angle of compound 4b sandwiched between clean glass slides on cooling from isotropic phase at 145 °C (left); DSC trace of compound 4b run at 10 °C/min under N₂ (right).

Mesomorphism of 4c

ESI Fig. 28 Crystalline texture observed by POM with 90° angle of compound 4c sandwiched between clean glass slides on cooling from isotropic phase at 127 °C (left); DSC trace of compound 4c run at 10 °C/min under N₂ (right).
Mesomorphism of 4d

ESI Fig. 29 texture of compound 4d observed by POM when cooling from isotropic phase at (a) 85°C, (b)75°C and (c)70°C

ESI Fig. 30 Focal conic texture of compound 4d sandwiched between clean glass slides on cooling from isotropic phase at 110 ºC (a); sample in (a) is rotated by 30° (b) and 60° (c) around the optical axis between the crossed polarizers. This highly birefringent texture with extinction branches is characteristic of an edge-on alignment, when sample is rotated between the crossed polarizers, the extinction branches rotate respect to the sample as well. (White solid curves indicate the same grain)

Mesomorphism of 4e

ESI Fig. 31 Dendritic texture of compound 4e sandwiched between clean glass slides on cooling from isotropic phase at 120 ºC (left), this texture which indicated a Colh phase was observed by POM with 45° angle and did not show any changes when cooled to room temperature; DSC trace of compound 4e run at 10 ºC/min under N² (right).
Mesomorphism of 4f

**ESI Fig. 32** DSC trace of compound 4f run at 10 °C/min under N².

Mesomorphism of 4g

**ESI Fig. 33** DSC trace of compound 4g run at 10 °C/min under N².

Mesomorphism of 4h

**ESI Fig. 34** DSC trace of compound 4h run at 10 °C/min under N².
Mesomorphism of 4i

ESI Fig. 35 Optical textures of compound 4i sandwiched between clean glass slides on cooling from isotropic phase: (left) dendritic texture which indicated a Col₇h phase observed by POM with 45° angle at 148 ºC; (middle) mosaic texture which indicated a Col₇hp phase observed by POM with 45° angle at 140 ºC; (right) mosaic texture which indicated a Col₇hp phase observed by POM with 90° angle at 140 ºC. Phase transition between Col₇h and Col₇hp was clearly observed for the colours and areas of domain were changed while the birefringence increases.

ESI Fig. 36 DSC trace of compound 4i run at 10 ºC/min under N₂.

Mesomorphism of 4j

ESI Fig. 37 Optical textures of compound 4j sandwiched between clean glass slides on cooling from isotropic phase: (left) dendritic texture which indicated a Col₇h phase observed by POM with 45° angle at 140 ºC; (right) mosaic texture which indicated a Col₇hp phase observed by POM with 90° angle at 100 ºC. Phase transition between Col₇h and Col₇hp was clearly observed for the colours and areas of domain were changed while the birefringence increases.
ESI Fig. 38 DSC trace of compound 4j run at 10 ºC/min under N².

Mesomorphism of 4k

ESI Fig. 39 Dendritic texture of compound 4k sandwiched between clean glass slides on cooling from isotropic phase at 125 ºC (left), this texture which indicated a Col₈ phase was observed by POM with 45° angle and did not show any changes when cooled to room temperature; DSC trace of compound 4k run at 10 ºC/min under N² (right).