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New five-ring symmetrical bent-core mesogens exhibiting the fascinating B₇ phase

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Three new homologous series of symmetrical five-ring bent-core compounds have been synthesized and investigated for their mesomorphic properties. The laterally unsubstituted parent compounds exhibit a metastable SmCP_A phase. However, the two series of compounds containing a strongly polar cyano or nitro group at the angular position of the central phenyl unit show the fascinating classical B₇ phase. The mesophases have been characterized using standard techniques.

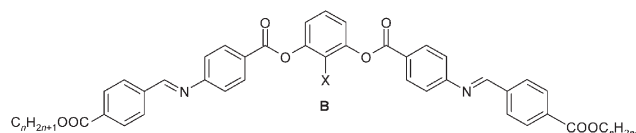
1. Introduction

Compounds composed of bent-core (BC) molecules, which exhibit certain mesophases, are also known as banana-shaped mesogens, and have been the subject of increasing current research activity. Although several hundred such BC compounds exhibiting mesophases have been synthesized, a complete understanding of the structure–property relationships is still elusive. The symbol B_n, where n=1,2,3,... was assigned [1] to the various mesophases exhibited by such BC compounds, and a total of seven phases were identified initially. A brief summary of all these phases can be found in an excellent review [2]. The B₇ phase was first observed in a series of Schiff's base compounds that are derived from 2-nitroresorcinol [3]. This phase has a helical superstructure and exhibits a variety of fascinating textures. It has also been reported that this phase does not respond to an applied electric field, at least up to about 18 V μm⁻¹ [4].

One of the characteristic textures exhibited by the B₇ phase is a helical pattern resembling that of telephone wires. This particular texture has been seen in a number of other systems [5–8] which contain no substituent in the angular position. In all these cases electro-optical switching was reported and, on the basis of textural features, the phase was characterized as a B₇ mesophase. However, the X-ray diffraction (XRD) patterns differ from that obtained for the original B₇ phase [2]. In addition, the X-ray scattering diagrams of the B₇ phase exhibited by compounds belonging to different homologous series, including those containing a nitro or

cyano group in the angular position, are quite similar [9]. Further, Coleman *et al.* [10] have carried out extensive studies on these different phases designated as B₇, using a combination of synchrotron-based XRD, freeze fracture transmission electron microscopy, and depolarized transmission and reflection light microscopy to establish the structural differences.

We reported the occurrence of B₇ mesophase in two homologous series of seven-ring esters as well as in a series of five-ring Schiff's base esters, all derived from 2-cyanoresorcinol [11–13]. The various fascinating textural patterns, the XRD intensity profiles, and the effect of an applied electric field on the B₇ phase exhibited by these compounds are not only similar but match well with those observed for the B₇ phase exhibited by the standard *n*-OPIMB-NO₂ compounds [2]. The first observation of a transition from the non-switchable B₇ mesophase to an antiferroelectric subphase, designated as B_{7AF1}, has also been reported [14]. Indeed, this transition was observed in compounds where the orientation of the azomethine linkage was reversed, in comparison with the standard *n*-OPIMB-NO₂ compounds. In continuation of our studies on the influence of the orientation of bridging groups on the mesomorphic properties of bent-core compounds, we report here the synthesis and characterization of three homologous series of compounds. The compounds have the general structure **B** shown below.



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2. Experimental

2.1. General

All intermediate compounds were purified by column chromatography on silica gel (100–200 mesh), and the target Schiff's base compounds by column chromatography on basic alumina followed by repeated crystallization using appropriate analytical grade solvents. The compounds were characterized using a combination of infrared spectroscopy (Shimadzu FTIR-8400 spectrophotometer), ^1H and ^{13}C NMR (Bruker AMX 400 spectrometer), and elemental analysis (Carlo-Erba 1106 analyser). The phase behaviour was investigated by textural observation under a polarizing optical microscopy (Leitz Laborlux 12 POL/Olympus BX50) using a heating stage and a central processor (Mettler FP82HT and Mettler FP90, respectively). The transition temperatures and associated enthalpy changes were obtained from thermograms recorded by differential scanning calorimetry (DSC) (Perkin-Elmer calorimeter, Model Pyris 1D). The calorimeter was calibrated using pure indium as a standard. XRD experiments on powder samples was carried out using Cu-K_α ($\lambda=1.54\text{ \AA}$) radiation from a 4kW rotating anode generator (Rigaku Ultrax 18) with a flat graphite crystal monochromator. The diffraction pattern was collected on a two-dimensional image plate (Marresearch). The electro-optical studies were carried out using standard triangular-wave method [15], employing a Universal Waveform Generator (Wavetek, model 39), an amplifier (Trek, Model 601B-3) and an oscilloscope (Agilent 54621A). Commercially obtained polyimide coated ITO cells (EHC, Japan) were used for these experiments.

2.2. Synthesis

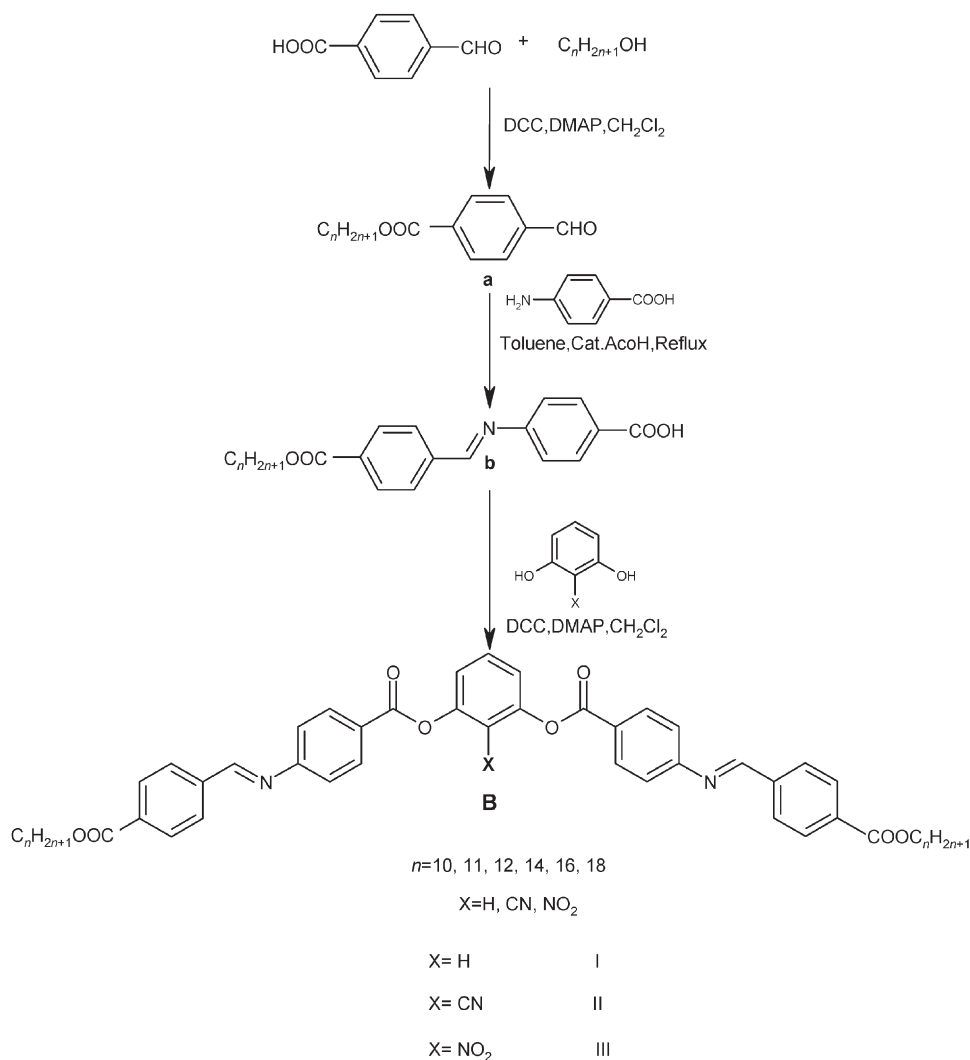
The general synthetic pathway used to prepare the new symmetrical bent-core compounds under investigation is shown in scheme 1. 4-Formylbenzoic acid and 4-aminobenzoic acid were obtained commercially and used without further purification. 2-Cyanoresorcinol was prepared following a procedure described by us previously [12]. Resorcinol and 2-nitroresorcinol were commercial compounds; resorcinol was purified by column chromatography followed by crystallization, while 2-nitroresorcinol was used without further purification. 4-Carboxybenzaldehyde was reacted with an appropriate *n*-alcohol in the presence of *N,N'*-dicyclohexylcarbodiimide (DCC) as dehydrating agent and 4-(*N,N*-dimethylamino)pyridine (DMAP) as catalyst to obtain the *n*-alkyl 4-formylbenzoate, which was then condensed with 4-aminobenzoic acid to obtain 4-(4-*n*-

alkyloxycarbonylbenzylideneamino)benzoic acid. The five-ring bent-core compounds were prepared by esterification of this acid with a 2-substituted resorcinol. A detailed procedure for the preparation of compound **IId** and physical data are given below.

2.2.1. 4-*n*-Tetradecyl 4-formylbenzoate, a. 4-Formylbenzoic acid (2 g, 13.3mmol) and *n*-tetradecanol (2.8 g, 13.3 mmol) in dry dichloromethane (25 ml) were treated with a catalytic amount of DMAP and stirred for 15 min. To this stirred mixture, DCC (3.0 g, 14.6mmol) was added and stirring was continued for 4 h at room temperature. The precipitated *N,N'*-dicyclohexylurea was filtered off and washed thoroughly with chloroform. The solvent was evaporated from the filtrate, and the product chromatographed on silica gel using a 2/3 mixture of chloroform/hexane (b.p. 60–80°C) as eluant. Removal of solvent from the eluate provided a white product, which was crystallized from methanol; yield 3 g (66%), m.p.40°C. IR (nujol) ν_{max} : 2922, 2852, 2729, 1714, 1695, 1460, 1284 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ (ppm): 10.1 (s, Ar-CHO), 8.19 (d, 3J 8.2 Hz, 2H, Ar-H), 7.94 (d, 3J 8.3 Hz, 2H, Ar-H), 4.35 (t, 3J 6.7 Hz, 2H, Ar-CO-O-CH₂-), 1.82–1.75 (quin, 3J 6.76, 2H, Ar-CO-O-CH₂-CH₂-), 1.55–1.34 (m, (-CH₂)₁₁, 22H), 0.88 (t, 3J 6.7Hz, -CH₃, 3H). Elemental analysis: $\text{C}_{22}\text{H}_{34}\text{O}_3$ requires C 76.23, H 9.89, found C 75.78, H 9.72 %.

2.2.2. 4-(4-*n*-Tetradecyloxycarbonylbenzylideneamino)benzoic acid, b. A mixture of compound **a** (1.8 g, 5.2 mmol), 4-aminobenzoic acid (0.7 g, 5.2 mmol) and a few drops of acetic acid in toluene was heated under reflux using a Dean–Stark apparatus for 4 h. The solution was cooled to room temperature and the precipitated product filtered off. This was crystallized from toluene; yield 2.0 g (83%), Cr 129.0 SmX 156.5 SmC 225.5 I (°C). IR (KBr) ν_{max} : 2952, 2918, 2848, 2675, 2555, 1708, 1693, 1681, 1687, 1596, 1571, 1429, 1282 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.5 (s, -CH=N-), 8.17–8.15 (m, 4H, Ar-H), 7.99 (d, 3J 7.52 Hz, 2H, Ar-H), 7.26 (d, 3J 6 Hz, 2H, Ar-H), 4.34 (t, 3J 6.16 Hz, 2H, Ar-CO-O-CH₂-), 1.79–1.45 (m, (-CH₂)₁₂, 24H), 0.86 (t, 3J 6.52, -CH₃, 3H). Elemental analysis: $\text{C}_{29}\text{H}_{39}\text{NO}_4$ requires C 74.8, H 8.4, N 3.0; found C 74.4, H 8.3, N 3.0%.

2.2.3. 2-Cyano-1, 3-phenylene bis[4-(4-*n*-tetradecyloxycarbonylbenzylideneamino)benzoate], IId. Compound **b** (0.4 g, 0.85 mmol) was reacted with 2-cyanoresorcinol (0.058 g, 0.43 mmol) in dry dichloromethane in the presence of DCC (0.194 g, 0.94 mmol) and a catalytic amount of DMAP. The mixture was stirred at room



Scheme 1. Synthetic route for the preparation of bent-core compounds.

temperature for 24 h. The precipitated *N,N'*-dicyclohexylurea was filtered off and the solvent from the filtrate evaporated. The residue was dissolved in chloroform and filtered through a column of basic alumina. The product thus obtained on removal of the solvent was further purified by repeated crystallization using a mixture of chloroform and acetonitrile; yield 0.34 g (79%), m.p. 111.5°C. IR (KBr) ν_{\max} : 3101, 3064, 2954, 2918, 2850, 2241, 1714, 1737, 1732, 1593, 1571, 1469, 1240, 1120, 1016 cm^{-1} . ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.51 (s, 2H, $2 \times -\text{CH}=\text{N}-$), 8.30 (d, 3J 8.52 Hz, 4H, Ar-H), 8.17 (d, 3J 8.28 Hz, 4H, Ar-H), 8.01 (d, 3J 8.32 Hz, 4H, Ar-H), 7.74 (t, 3J 8.44 Hz, 1H, Ar-H), 7.46 (d, 3J 8.4 Hz, 2H, Ar-H), 7.32 (d, 3J 8.52 Hz, 4H, Ar-H), 4.35 (t, 3J 6.64 Hz, 4H, $2 \times \text{Ar}-\text{CO}-\text{O}-\text{CH}_2-$), 1.83–1.76 (quin, 3J 6.72 Hz, 4H, $2 \times \text{Ar}-\text{CO}-\text{O}-\text{CH}_2-\text{CH}_2-$), 1.46–1.26 (m, 44H, $2 \times (-\text{CH}_2-)_{11}$), 0.87 (t, 3J 6.56 Hz, 6H, $2 \times -\text{CH}_3$). ^{13}C NMR (100 MHz,

CDCl_3) δ (ppm): 166.0, 163.3, 161.0, 157.2, 153.5, 139.3, 133.5, 132.0, 130.0, 129.0, 125.7, 121.7, 121.1, 120.2, 65.6, 31.9, 29.7, 29.3, 28.8, 26.0, 22.7, 14.1. Elemental analysis: $\text{C}_{65}\text{H}_{79}\text{N}_3\text{O}_8$ requires C 75.77, H 7.72, N 4.07; found C 75.77, H 7.56, N 4.24%.

3. Results

3.1. Thermal analysis

All three series of compounds are symmetrical about the central phenyl unit and contain five rings in the core. The two terminal chains are incorporated as esters in contrast to the usual *n*-alkyl or *n*-alkoxy chains. In series I, the parent compounds, there is no lateral substituent, while in series II and III a strongly polar cyano or nitro group is substituted in the angular position. The transition temperatures and associated enthalpy changes for these three series of compounds

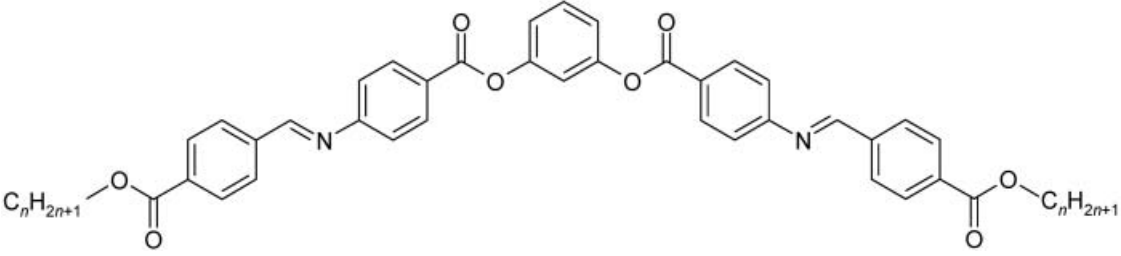
are summarized in tables 1, 2 and 3, respectively. In series **I**, only six compounds were prepared; the lower homologues were not prepared because compound **Ia** with *n*-decyl chains showed no mesophase. The other five homologues exhibit a metastable mesophase whose optical textures are similar. When a sample of compound **Id** was cooled from the isotropic phase, the mesophase appeared as chiral domains of opposite handedness. The two chiral regions could be easily distinguished by rotating the analyser from the crossed position by 7°. Photomicrographs showing these chiral domains are shown in figure 1. The observation of such textures were first reported by Thisayukta *et al.* [16, 17] and Heppke *et al.* [5]. We have also observed such textures [18], and by a combination of XRD studies and electro-optical investigations, characterized the mesophase as B₂ (SmCP_A). The remaining homologues, **Ib**, **Ic**, **Ie** and **If**, exhibit similar textures, which have all been characterized as SmCP_A phase (see §3.3). A plot of transition temperature as a function of terminal chain length is shown in figure 2. The clearing points fall on a smooth curve, whose slope is similar to those seen earlier in various other homologous series of compounds exhibiting the SmCP_A phase [18].

The compounds of series **II** were derived from 2-cyanoresorcinol. As can be seen in table 2, all the six

homologues are enantiotropically monomorphic with compound **IId** having the largest thermal range of about 50°C. These compounds exhibit a variety of textures which are typical for a B₇ phase [2], one whose characteristic textures is the formation of helical patterns. For example, when a sample of compound **IId** is cooled slowly from the isotropic phase, helical textures form, as depicted in figure 3. In addition, various fascinating patterns are also formed, three of which are shown in figure 4. We have reported [11, 12] several compounds derived from 2-cyanoresorcinol and exhibiting the B₇ phase which showed several beautiful textures. On the basis of these textural observations, XRD studies and electro-optical investigations (the phase does not respond to an applied electric field), we have characterized the mesophase exhibited by compounds **IIa–f** as B₇. A plot of the transition temperatures as a function of the number of carbon atoms in the terminal chain is shown in figure 5. The B₇→I transition points fall on a smooth curve which is again typical for this phase [12–14].

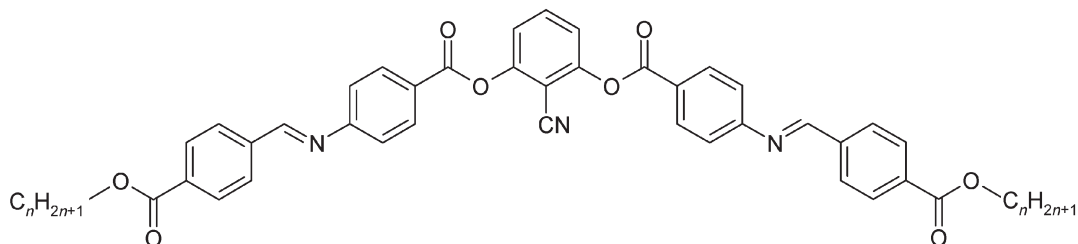
The compounds of series **III** were obtained using 2-nitroresorcinol. As in series **II**, the six homologues are all enantiotropically mesomorphic. Compound **IIIe** has a fairly wide thermal range of 54°C for the mesophase. Surprisingly, compounds **IIIa–c** exhibit chiral domains of opposite handedness as seen for the unsubstituted

Table 1. Transition temperatures (°C) and associated enthalpy changes (in italics) (kJ mol⁻¹) for the compounds of series **I**.

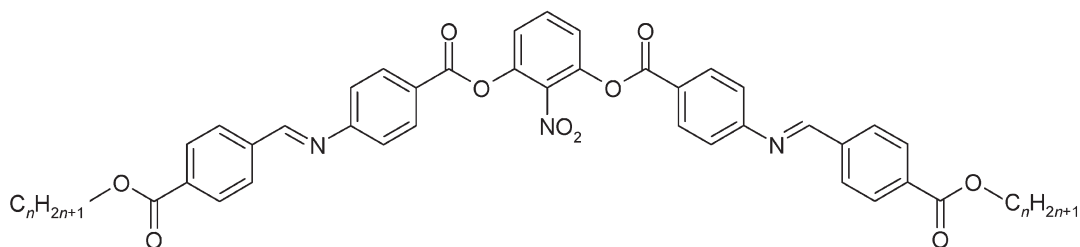


Compound	<i>n</i>	Cr		SmCP _A	I
Ia	10	•	139.6 ^a <i>73.0</i>	—	•
Ib	11	•	137.5 <i>69.0</i>	(• 120.0) <i>16.5</i>	•
Ic	12	•	16.0 ^a <i>72.0</i>	(• 120.5) <i>15.0</i>	•
Id	14	•	133.2 ^a <i>120.4</i>	(• 121.0) <i>18.3</i>	•
Ie	16	•	132.5 <i>131.5</i>	(• 121.0) <i>18.5</i>	•
If	18	•	130.7 ^a <i>134.2</i>	(• 119.0) <i>16.6</i>	•

^aThis compound has a crystal–crystal transition; the enthalpy denoted is the sum of all such transitions.

Table 2. Transition temperatures (°C) and associated enthalpy changes (in italics) (kJ mol⁻¹) for the compounds of series **II**.

Compound	<i>n</i>	Cr		B ₇		I
IIa	10	•	123.7 ^a <i>45.6</i>	•	144.0 <i>26.3</i>	•
IIb	11	•	128.5 ^a <i>34.0</i>	•	145.5 <i>26.0</i>	•
IIc	12	•	114.0 ^a <i>39.5</i>	•	144.5 <i>21.0</i>	•
IId	14	•	96.7 ^a <i>56.4</i>	•	147.0 <i>24.1</i>	•
IIe	16	•	103.0 ^a <i>64.0</i>	•	147.0 <i>23.9</i>	•
IIf	18	•	107.5 <i>72.7</i>	•	147.5 <i>22.0</i>	•

^aSee table 1.Table 3. Transition temperatures (°C) and associated enthalpy changes (in italics) (kJ mol⁻¹) for the compounds of series **III**.

Compound	<i>n</i>	Cr		B ₇		I
IIIa	10	•	106.7 ^a <i>18.0</i>	•	143.0 <i>32.0</i>	•
IIIb	11	•	101.0 <i>43.5</i>	•	143.5 ^a <i>33.0</i>	•
IIIc	12	•	91.0 <i>4.5</i>	•	143.5 <i>28.5</i>	•
IIId	14	•	93.0 ^a <i>55.2</i>	•	143.5 <i>29.0</i>	•
IIIe	16	•	89.5 ^a <i>34.8</i>	•	143.5 <i>27.4</i>	•
IIIf	18	•	92.0 ^a <i>70.9</i>	•	143.0 <i>26.8</i>	•

^aSee table 1.

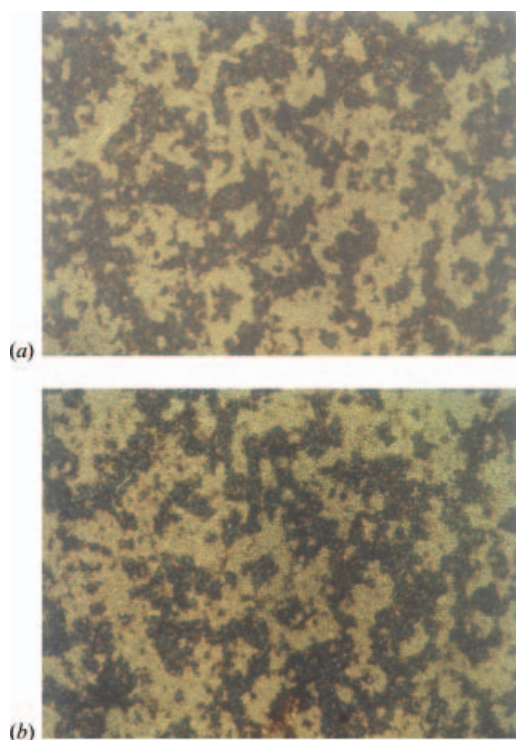


Figure 1. Chiral domains of opposite handedness obtained in the mesophase of compound **Id** ($T=119^{\circ}\text{C}$) by rotating the analyser from crossed position by (a) $+7^{\circ}$ and (b) -7° .

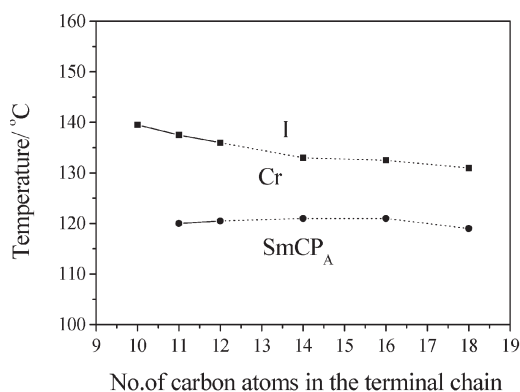


Figure 2. A plot of transition temperature as a function of the number of carbon atoms in the terminal chain for compounds of series **I**.

compounds described earlier, but in contrast the domains are fairly large. Photomicrographs showing the chiral domains of opposite handedness obtained for the B_7 mesophase of compound **IIIb** are shown in figure 6(a) and 6(b). To the best of our knowledge, such chiral domains have not previously been seen for the B_7 phase exhibited by compounds derived from 2-nitroresorcinol. However, the formation of these chiral

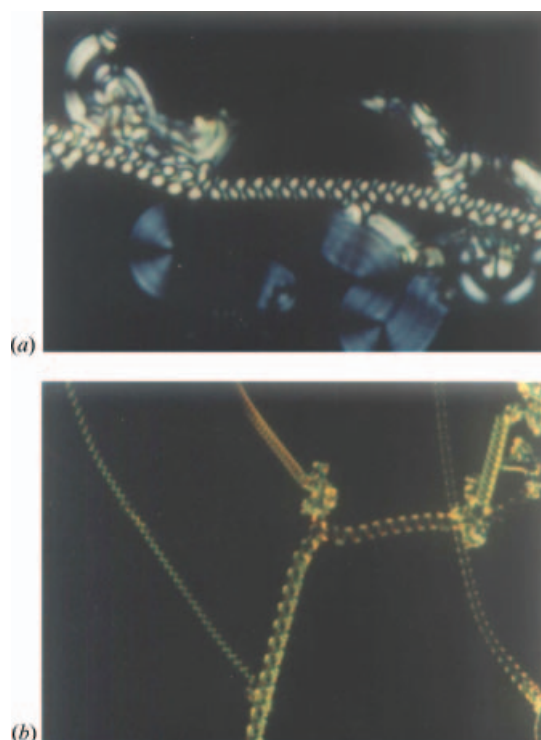


Figure 3. (a, b) Photomicrographs of the helical patterns forming on cooling the isotropic phase of compound **III** ($T=145.5^{\circ}\text{C}$).

domains depends on the rate of cooling. In addition, compounds **III**d–**f** show filamentary texture when their isotropic liquids are cooled slowly. A typical texture obtained on cooling the isotropic liquid of compound **III**d is shown in figure 7. This again is a rather unusual observation. We previously observed such filamentary growth patterns at the isotropic to SmCP_A phase transition in a number of unsymmetrical bent-core compounds derived from 3-hydroxybenzoic acid [19]. It should be noted that helical filaments and other two-dimensional patterns, normally seen for the B_7 phase, could also be observed on very slow cooling of the isotropic liquid. However, the mesophases of all the six compounds show the same XRD pattern and also do not switch electro-optically. A plot of the transition temperatures versus the number of carbon atoms in the terminal chain for this series is shown in figure 8. The clearing temperatures follow a smooth curve, as seen for the compounds of series **II**.

3.2. X-ray diffraction studies

The compounds of series **I** show metastable mesophases which also crystallize quite rapidly. XRD measurements could not be made on these compounds. However, we have performed XRD experiments on non-oriented

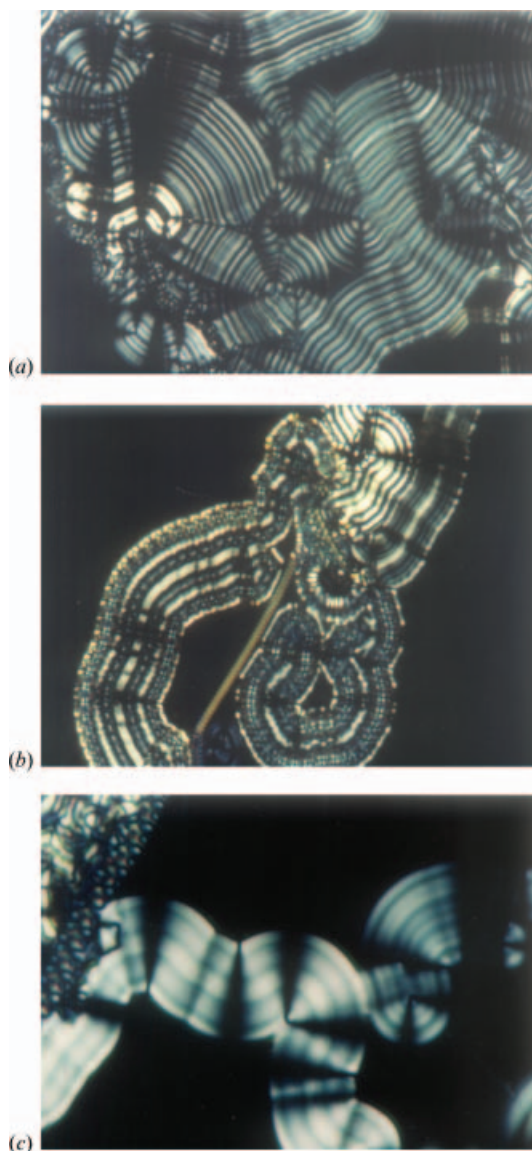


Figure 4. (a–c) Various textural patterns of the B_7 phase of compound **III d** ($T=145.5^\circ\text{C}$) obtained on slow cooling of the isotropic phase.

mesophases of compounds belonging to series **II** and **III**. The data obtained are summarized in table 4, and the X-ray diffractograms of all these compounds are quite similar. For example, the XRD pattern of compound **III d** showed several reflections in the small angle region with d spacings of 43.5, 32.2, 11.3, 9.5, 7.5 Å, ruling out the possibility of a simple layer structure. The X-ray angular intensity profile obtained for the mesophase of this compound is shown in figure 9. The pattern has a very close similarity to that of the B_7 phase reported earlier [12, 13]. Similar patterns were obtained for all the other compounds of the two

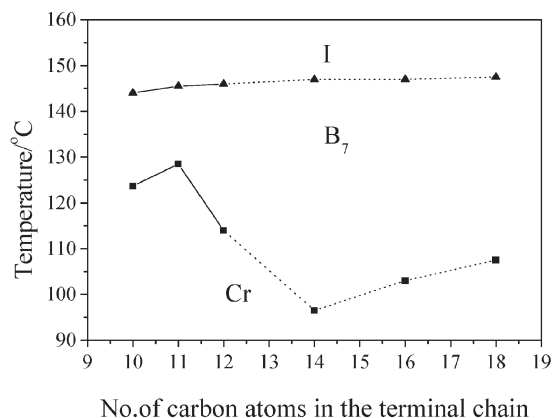


Figure 5. A plot of transition temperature as a function of the number of carbon atoms in the terminal chain for compounds of series **II**.

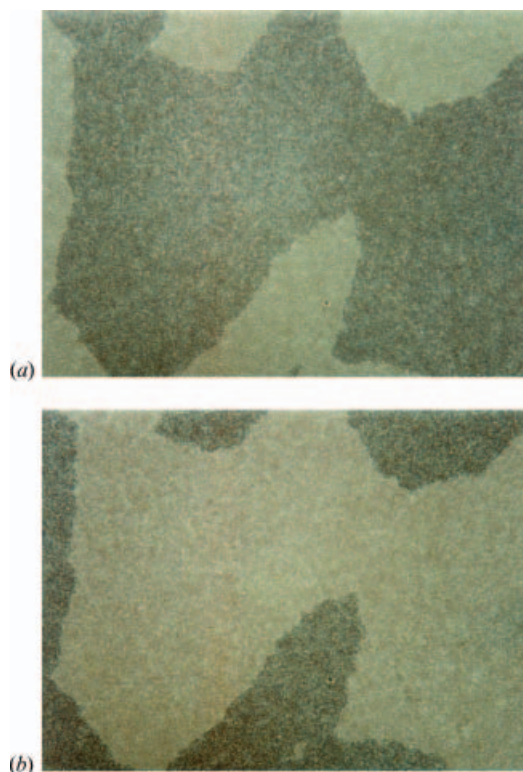


Figure 6. Microscopic texture of the B_7 phase of compound **III b** on rotating the polarizer by (a) $+5^\circ$ and (b) -5° from its crossed position ($T=140^\circ\text{C}$).

series. A reflection centred around 7.5 Å was observed in all of these, and appears to be a characteristic reflection seen in the B_7 phase of all compounds derived from 2-cyano- and 2-nitro-resorcinol. A diffuse wide angle reflection was observed around 4.2 Å indicating a liquid-like in-plane order.

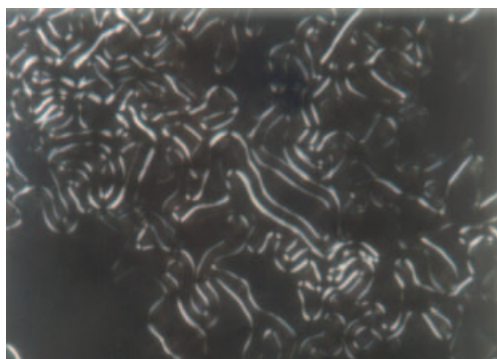


Figure 7. Photomicrograph showing the filamentary texture for the B_7 phase of compound **III d** ($T=141.5^\circ\text{C}$).

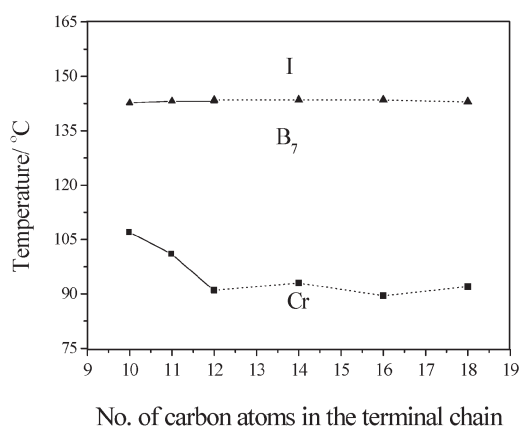


Figure 8. A plot of transition temperature as a function of the number of carbon atoms in the terminal chain for compounds of series **III**.

3.3. Electro-optical experiments

Electric field experiments were carried out on representative compounds of all three homologous series. ITO-coated transparent cells treated for planar alignment (EHC, Japan) with a cell gap of $8\ \mu\text{m}$ were used for the experiments. The cells were filled with the samples in their isotropic phase by capillary action. As described

Table 4. Layer spacings from XRD studies of various compounds under investigation.

Compound	d spacings/ \AA	Temperature/ $^\circ\text{C}$
II a	37.4, 19.1, 7.7	135
II d	43.0, 21.8, 7.6	125
II f	46.8, 16.1, 7.7	125
III a	39.0, 7.7	130
III d	43.5, 32.2, 11.3, 9.5, 7.5	135
III f	48.8, 24.7, 7.5	125

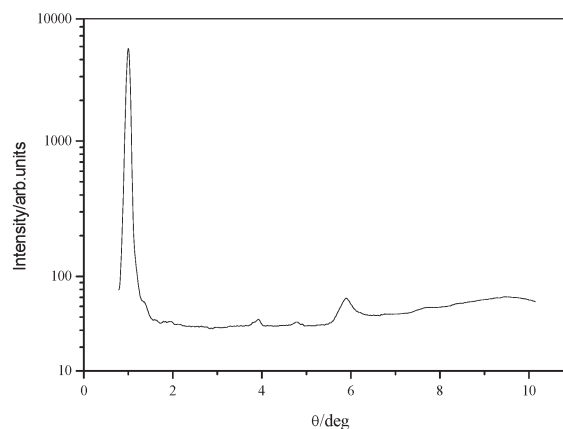


Figure 9. X-ray angular intensity profile obtained for the B_7 mesophase of compound **III d** ($T=135^\circ\text{C}$).

earlier, all the compounds of series **I** show a metastable mesophase and crystallize quite quickly. Hence the measurements were carried out close to the clearing temperature. For example, a sample of compound **Id** was cooled from the isotropic phase under a triangular wave electric field ($60\ \text{V}_{\text{pp}}$, $100\ \text{Hz}$); at 119°C a single peak per half period was observed. However, this single peak split into two on reducing the frequency to $5\ \text{Hz}$, indicating an antiferroelectric switching behaviour. The switching current response obtained at $150\ \text{V}_{\text{pp}}$ and $5\ \text{Hz}$ is shown in figure 10. The calculated polarization value was found to be $30\ \text{nC cm}^{-2}$, which is quite low for a SmCP_A phase. This is probably because measurement was made close to the clearing temperature.

For compounds belonging to series **II** and **III** which exhibit a B_7 phase, no electro-optical response was observed up to a triangular voltage of $50\ \text{V}\ \mu\text{m}^{-1}$. This is not surprising and is in conformity with the general

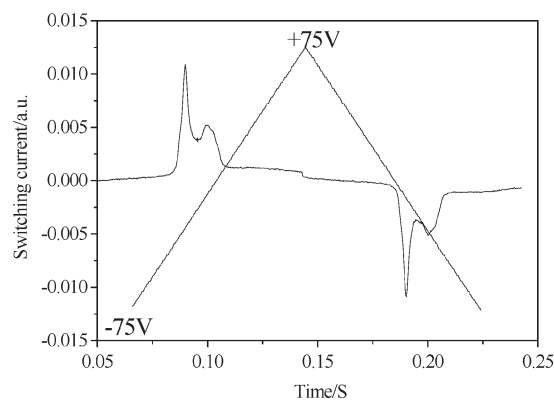


Figure 10. Switching current response obtained by the application of a triangular wave electric field for a sample of compound **Id** at $150\ \text{V}_{\text{pp}}$, $5\ \text{Hz}$, $T=119^\circ\text{C}$; cell thickness $8\ \mu\text{m}$.

behaviour of bent-core compounds containing a strongly polar (CN/NO₂) group in the angular position.

4. Discussion

An important liquid crystals feature is their optical texture. Among all the different types of liquid crystal, perhaps the B₇ phase exhibits the most beautiful and fascinating textures. One such texture is the helical nuclei that appear on cooling the isotropic liquid, resembling that of telephone wires. This texture of the B₇ phase is seen in practically all compounds containing a 2-nitro- or 2-cyano-resorcinol central unit. As pointed out earlier [11, 12], there have been a number of other reports in which mesophases have been designated as B₇ on the basis of the occurrence of spiral germs on cooling the isotropic liquids [5, 7, 8, 20]. Interestingly these compounds do not contain the polar cyano or nitro group in the angular position. However, some of these compounds do exhibit other variants of the B₇ texture shown by the original compound [2]. In addition, many of these B₇ phases switch electro-optically and their X-ray diffractograms are distinctly different. It is important to point out here that the XRD patterns of the B₇ phase exhibited by all compounds derived from 2-nitro- or 2-cyano-resorcinol are the same, although a variety of contrastingly different and beautiful textures are observed.

Compounds of series II show textural variants as already seen [12]. Remarkably, the compounds of series III that exhibit the B₇ phase show textures not seen earlier: chiral domains of opposite handedness for lower homologues and filamentary textures for compounds with longer chains. But, on the basis of similarity of the X-ray diffractogram with that of the original B₇ phase [2], all these can be classified as the same phase and assigned the same symbol. Indeed, a sketch of the X-ray scattering diagrams of the B₇ phase reported for different systems has recently been published [9]. This emphasizes the importance of characterization of a mesophase by XRD studies in addition to various other investigations. For example, Coleman *et al.* [10] have shown by synchrotron based XRD studies, freeze fracture electron microscopy and optical investigations, that the homochiral ferroelectric mesophase exhibited by MHOBOW [7] is a B₇ phase which is characterized by a polarization-modulated layer stripe structure. All these results suggest that there is more than one type of B₇ phase and a proper assignment of the symbols for these variants is necessary.

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