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S. Umadevi[a]; S. Radhika[a]; B. K. Sadashiva[a]
[a] Raman Research Institute, Bangalore-560080, India

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The SmCP$_A$ phase in five-ring bent-core compounds derived from 5-methoxyisophthalic acid

S. UMADEVI, S. RADHIKA and B.K. SADASHIVA*
Raman Research Institute, C.V. Raman Avenue, Sadashivanagar, Bangalore-560080, India

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The synthesis and mesophase characterization of a homologous series of five-ring bent-core compounds derived from 5-methoxyisophthalic acid are described. Most of the compounds exhibit a polar antiferroelectric smectic C phase. Replacement of the terminal *n*-alkoxy chains by *n*-alkyl carboxylate groups, not only destabilizes the formation of mesophases but induces a calamitic mesophase. However, extension of the arms of the bent-core molecule by a phenyl moiety stabilizes the switchable phase. The mesophases were investigated using a combination of polarizing optical microscopy, differential scanning calorimetry, X-ray diffraction and electro-optical methods.

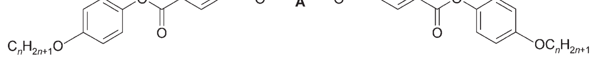
1. Introduction

The liquid crystalline phases formed by compounds composed of bent-core (BC) molecules are fascinating and intriguing. The general symbol B was assigned to the mesophases exhibited by such compounds to differentiate them from calamitics and mesophases formed by disc-like molecules. However, the letter B signifies the shape of the constituent molecules, viz. banana, bow, etc. Initially, seven different phases were identified which were called B_1, B_2, B_3,….B_7, and the structural as well as the mesomorphic properties of a large number of compounds have been reviewed [1]. In the last two years a number of new phases have been discovered and hence there is a need for a reclassification of the various liquid crystalline phases formed by bent-core compounds.

Although several hundred bent-core compounds have been synthesized and the mesophases exhibited by them investigated [1–12], the structure–property relationships are still not clear. A majority of these compounds contain five, six or seven aromatic rings in the core and, appended by *n*-alkyl or *n*-alkoxy chains in the two terminal positions. In addition, in some unsymmetrical systems, one of the terminal positions has been substituted by various other small groups [13]. A wide variety and combination of linking groups such as –COO–, –CH=N–, –N=N–, –CH=CH–, –CH$_2$O–, etc. have been used to connect the aromatic rings, and have provided interesting B mesophases.

It is well known that the mesomorphic properties of compounds exhibiting calamitic phases can be modified by using suitable lateral substituents. The influence of lateral substituents on the mesomorphic behaviour of bent-core compounds has been investigated quite extensively [8, 14, 15]. The results from these studies indicate that dipolar effects dominate over steric factors, which is quite contrary to the situation observed in calamitic liquid crystals [14]. The results obtained in the investigations carried out so far on a number of bent-core compounds indicate that the nature, size and position of the substituent has a strong bearing on the type of mesophase formed. For example, in a five-ring system with a central phenyl unit, a cyano substituent in position 2 [12] gives rise to a B_7 mesophase, while the same substituent at position 5 [16] does not favour the formation of a mesophase. Interestingly, a 4-cyano substituent gives rise to the formation of both B and calamitic mesophases [17]. It has also been shown from a number of studies on five-ring BC compounds that groups such as cyano, methyl or methoxy at the apex position (position 5), prevent the formation of a mesophase [16].

Here, we report the first observation of an electro-optically switchable phase in five-ring BC compounds in which a bulky substituent is at position 5. These are derived from 5-methoxyisophthalic acid and have the general structure **A**. The only other examples of a



A

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*Corresponding author. Email: sadashiv@rri.res.in

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five-ring BC compound containing a substituent at this position and exhibiting a switchable phase are derived from 5-fluororesorcinol [18], methyl 3,5-dihydroxybenzoate [19], and 5-methylresorcinol [20]. It is also appropriate to mention that several five-ring bent-core compounds derived from isophthalic acid and exhibiting interesting mesophases have also been reported [21].

2. Experimental

2.1. *Characterization*

Structural characterization of the compounds was carried out through a combination of infrared spectroscopy (Shimadzu FTIR-8400 spectrophotometer), ^{1}H NMR (Bruker AMX 400 spectrometer), and elemental analysis (Carlo-Erba 1106 analyser). The mesophase behaviour was studied using polarizing optical microscopy (POM) (Olympus BX50) using a heating stage and a central processor (Mettler FP82HT and Mettler FP90, respectively) and differential scanning calorimetry (DSC Perkin-Elmer, Model Pyris 1D). The reported transition temperatures and the associated enthalpy values were obtained from thermograms recorded by DSC. X-ray diffraction (XRD) measurements were performed on powder samples using Cu-K$_\alpha$ radiation from a rotating anode generator (Rigaku Ultrax-18) with graphite crystal as monochromator. The diffraction patterns of the samples were collected on an image plate (Marresearch). The electro-optical studies and the polarization measurements were carried out using a standard triangular-wave method (waveform generator WAVETEK model 39, amplifier TREK model 601B-3, oscilloscope Agilent 54621A) employing commercially available polyimide-coated ITO glass cells (EHC, Japan, cell gap 8 µm).

2.2. *Synthesis*

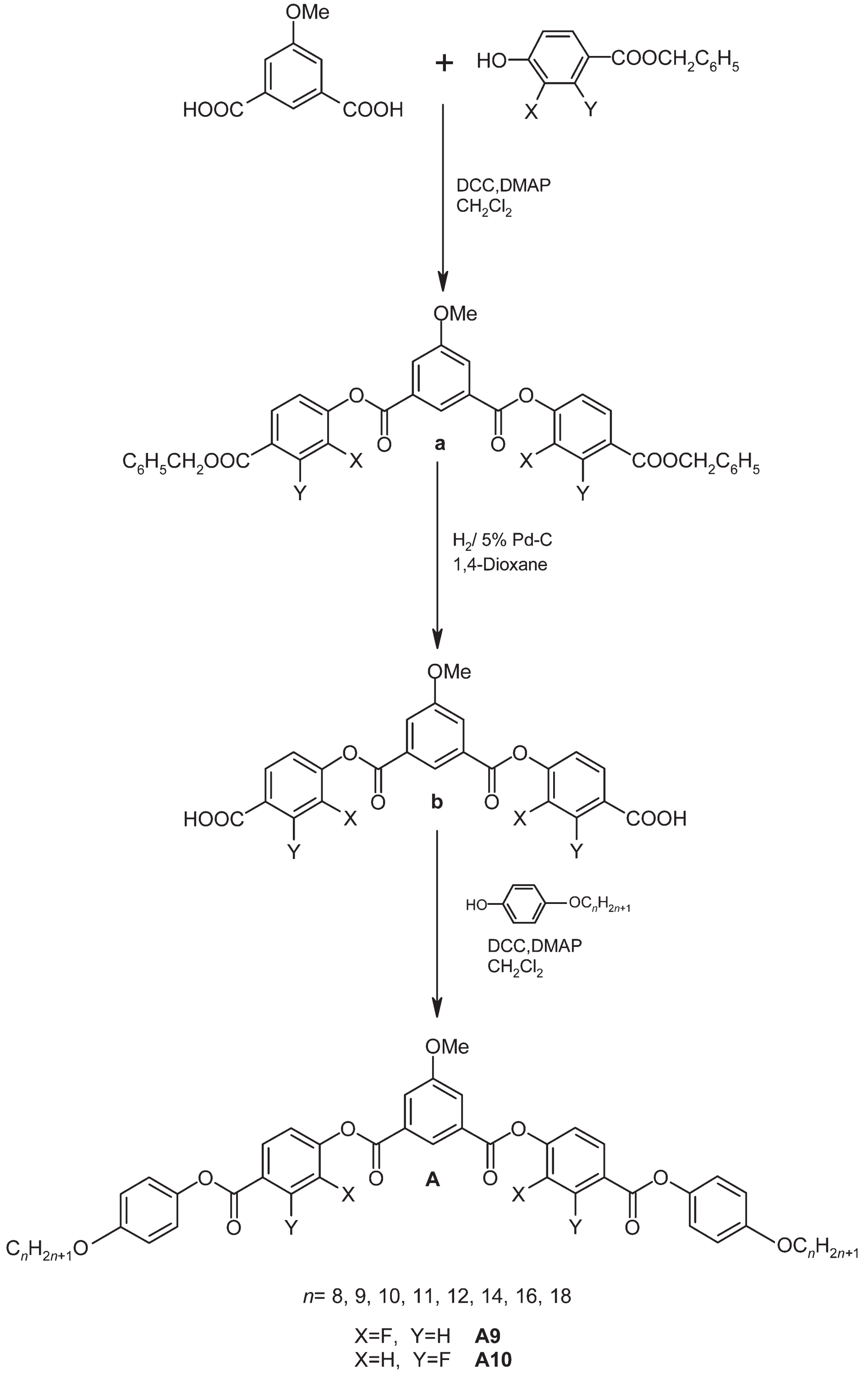
The symmetrical five-ring bent-core compounds were prepared following the pathway shown in the scheme. 5-Methoxyisophthalic acid was prepared from commercially available 5-hydroxyisophthalic acid and esterified with benzyl 4-hydroxybenzoate using *N*, *N*′-dicyclohexylcarbodiimide (DCC) and a catalytic amount of 4-(*N*, *N*-dimethylamino)pyridine (DMAP) to obtain compound **a**. The benzyl group in compound **a** was cleaved using H_2 and 5%Pd-C catalyst to get the bis-carboxylic acid **b**. The bent-core compounds **A** were prepared by condensing the bis-carboxylic acid **b** with the appropriate *n*-alkoxyphenols and compound **B** was obtained by condensing the acid **b** with 4-*n*-hexadecyloxy-4′-hydroxybiphenyl. Similarly, compounds **C** and **D** were prepared by treating the bis-carboxylic acid **b** with two equivalents of an appropriate 4-*n*-alkyl 4′-hydroxybenzoate and *n*-hexadecyl 4-hydroxybiphenyl-4′-carboxylate, respectively. All the compounds were purified by column chromatography on silica gel (60–120 mesh) and repeated crystallization using analytical grade solvents. A detailed procedure for the synthesis of the compound **A6**, with its spectral and analytical data, is given below.

2.2.1. Compound a (*X*=H, *Y*=H). 5-Methoxyisophthalic acid (3 g, 15.3 mmol) was reacted with benzyl 4-hydroxybenzoate (7 g, 30.6 mmol) in dry dichloromethane in the presence of a catalytic amount of DMAP and the mixture was stirred for 10 min. To this stirred mixture, DCC (6.9 g, 34 mmol) was added and stirring continued for 24 h at room temperature. The precipitated urea was filtered off and washed with an excess of chloroform. Evaporation of the solvent from the filtrate gave a white product which was passed through a column of silica gel using chloroform as eluant. The product thus obtained was further purified by repeated crystallization from ethanol. Yield 9 g (95%), m.p. 117.5–119°C. IR (nujol) ν_{max}: 2923, 2854, 1749, 1724, 1604, 1460 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.59 (t, 1H, Ar–H), 8.17 (d, ^3J 8.68 Hz, 4H, Ar–H), 7.96 (d, ^4J 1.24 Hz, 2H, Ar–H), 7.46–7.35 (m, 10H, Ar–H), 7.32 (d, ^3J 8.68 Hz, 4H, Ar–H), 5.38 (s, 4H, Ar–COO–CH_2–), 3.96 (s, 3H, Ar–O–CH_3). Elemental analysis: $C_{37}H_{28}O_9$ requires C 72.09, H 4.64; found C 71.61, H 4.66%.

2.2.2. Compound b, (*X*=H, *Y*=H). To compound **a** (5 g, 8.1 mmol) in 1,4-dioxane (40 ml), 5% Pd-C catalyst (1 g) was added. The mixture was stirred in an atmosphere of hydrogen at 60°C until the absorption of hydrogen ceased. The solution was filtered hot and the filtrate allowed to cool slowly; the crystallized product was filtered and dried. Yield 3 g (85%), m.p. 261–269°C. IR (nujol) ν_{max}: 3078, 2923, 2852, 2673, 2551, 1741, 1749, 1693, 1697, 1602, 1456 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.42 (t, 1H, Ar–H), 8.07 (d, ^3J 8.64 Hz, 4H, Ar–H), 7.97 (d, ^4J 1.24 Hz, 2H, Ar–H), 7.49 (d, ^3J 8.64 Hz, 4H, Ar–H), 3.99 (s, 3H, Ar–OCH_3). Elemental analysis: $C_{23}H_{16}O_9$ requires C 63.31, H 3.69; found C 63.12, H 3.88%.

2.2.3. Compound A6, (*X*=H, *Y*=H). This compound was obtained following a similar procedure to that for compound **a.** Quantities: compound **b** (0.2 g, 4.5×10^{-4} mol), 4-*n*-tetradecyloxyphenol (0.27 g, 9×10^{-4} mol), DCC (0.2 g, 9.9×10^{-4} mol), DMAP (cat.amount), dry dichloromethane (10 ml); solvent for crystallization: a mixture of chloroform and acetonitrile; yield 0.35 g(76%), m.p. 105°C. IR (KBr)

ν_{max}: 2920, 2852, 2665, 1737, 1735, 1728, 1465 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.64 (t, 1H, Ar–H), 8.30 (d, ^3J 8.68 Hz, 4H, Ar–H), 7.99 (d, ^4J 1.2 Hz, 2H, Ar–H), 7.40 (d, ^3J 8.68 Hz, 4H, Ar–H), 7.12 (d, ^3J 8.96 Hz, 4H, Ar–H), 6.93 (d, ^3J 8.96 Hz, 4H, Ar–H), 3.98 (s, 3H, Ar–OCH_3), 3.96 (t, ^3J 6.48 Hz, 4H, Ar–OCH_2–), 1.8–1.75 (quin, ^3J 6.64 Hz, 4H, Ar–OCH_2–$\underline{CH_2}$–), 1.46–1.26 (m, 2 × 22H), 0.88 (t, ^3J 6.44 Hz, 6H).



Scheme. Synthetic pathway used for the preparation of the bent-core compounds.

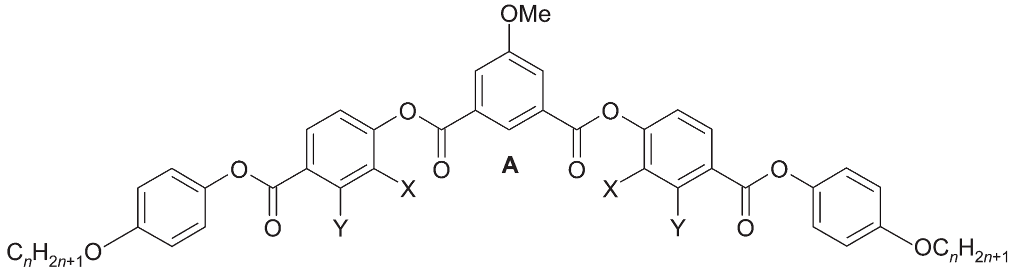
Elemental analysis: $C_{63}H_{80}O_{11}$ requires C 74.68, H 7.95; found C 74.91, H 7.99%.

3. Results and discussion

3.1. *Thermal studies*

A total of ten symmetrical five-ring bent-core compounds having the general structure **A** with a bulky methoxy substituent at position 5 were investigated for mesomorphic properties. The transition temperatures together with the associated enthalpy changes of these compounds are summarized in table 1. Compound **A1** is non-mesomorphic, while the remaining showed a mesophase. When a sample of compound **A6** was cooled slowly from the isotropic phase, schlieren and focal-conic textures developed, which is typical for a SmCP phase. A photomicrograph of the texture exhibited by compound **A6** is shown in figure 1. In fact, all the remaining homologues (**A2**–**A8**) exhibited similar patterns, suggesting that the mesophase is of the same type. A plot of the transition temperatures as a function of the number of carbon atoms in the terminal *n*-alkoxy chain for these compounds is shown in figure 2. It can be seen that the clearing temperature curve is smooth; it rises initially and levels off on ascending the homologous series. This is quite typical for the SmCP phase in general and has been observed in a number of different homologous series of compounds [6, 8]. Compound **A8** is enantiotropic with a thermal range of 5°C for the mesophase. When a lateral fluorine substituent is introduced on the middle phenyl ring of the arms of the bent-core compound, either at *meta* or *ortho* position with respect to the carbonyl group (compounds **A9** and **A10** respectively), the mesophase is destabilized

Table 1. Transition temperatures (°C) and associated enthalpy values (J g^{-1}) for the compounds **A**. Cr=crystalline phase; SmC$_S$P$_A$=polar smectic C phase, S and A refers to synclinic and antiferroelectric; SmA=uniaxial smectic A phase; I=isotropic phase.



Compound	*n*	*X*	*Y*	Cr		SmC$_S$P$_A$		I
A1	8	H	H	•	104.0	—		•
					52.0			
A2	9	H	H	•	107.0	(•	100.5)	•
					60.5		*14.0*	
A3	10	H	H	•	108.0	(•	107.0)	•
					51.5		*16.0*	
A4	11	H	H	•	108.5	•	111.0	•
					65.5		*16.0*	
A5	12	H	H	•	102.5	•	113.5	•
					49.5		*15.5*	
A6	14	H	H	•	105.0	•	115.0	•
					63.0		*16.5*	
A7	16	H	H	•	107.5	•	116.0	•
					67.5		*16.0*	
A8	18	H	H	•	111.0	•	116.0	•
					77.5		*16.0*	
A9	18	F	H	•	116.0	(•	103.0)[a]	•
					179.5			
A10	18	H	F	•	117.0	(•	100.7)[a]	•
					103.5			

[a] Enthalpy could not be measured from DSC as the sample crystallizes immediately.

Figure 1. Photomicrograph of the SmCP$_A$ phase of compound **A6** obtained on cooling the isotropic phase.

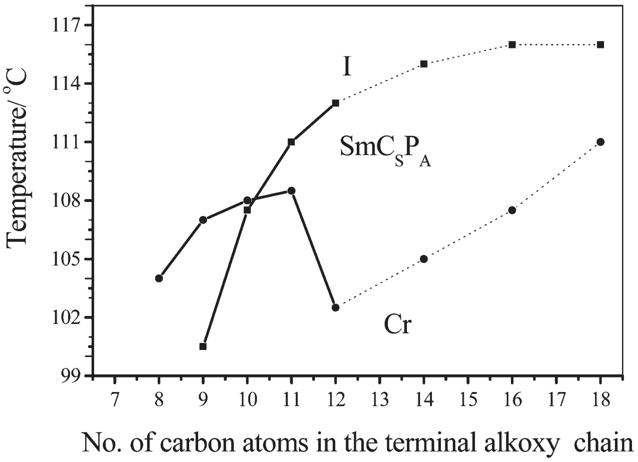
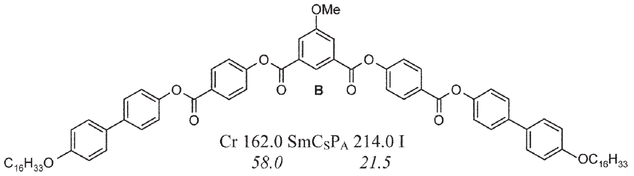


Figure 2. A plot of transition temperature as a function of the number of carbon atoms in the terminal chain for compounds **A1**–**A8**.

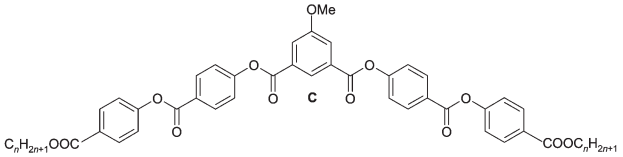
and becomes metastable. This is in complete contrast to what has been observed in five-ring compounds derived from resorcinol [8]. This once again emphasizes the fact that in bent-core compounds, dipolar effects are dominant over steric factors [14].

When the terminal phenyl group is replaced by a biphenyl moiety (compound **B**), both the melting and clearing temperatures are enhanced, as expected. For example, the thermal range of the mesophase increased from 8.5°C for compound **A7** to 52°C for compound **B** with no change in the nature of the mesophase. Interestingly, when the terminal *n*-alkoxy chains are replaced by *n*-alkyl carboxylate groups (compound **C**), the occurrence of the mesophase is suppressed and only a metastable SmA phase is obtained (see table 2). The nature of this phase was confirmed by observing the texture of a sample of this compound in separate cells treated for homogeneous and homeotropic alignment of the molecules, as shown in figures 3(*a*) and 3(*b*), respectively. When the terminal phenyl ring in compound **C** was replaced by a biphenyl moiety (compound **D**), a switchable SmCP phase was obtained. The occurrence of a calamitic phase can be attributed to an increase (>140°) in the bend angle between the two arms of the bent-core molecule. However, the possible explanation in the present case could be that the rotational freedom of the molecules along their long axis is facilitated by the flexible terminal *n*-alkyl carboxylate groups, which in turn will inhibit a steric packing along the bend direction resulting in a calamitic phase. It is interesting to point out here that in a bent-core system containing terminal perfluorinated chains with a bend angle of 125°, a SmA phase has been observed [19]. In addition, it has been reported [9] that in the case of a Schiff's base ester with a terminal *n*-tetradecyloxy chain, and derived from isophthalic acid, the formation of a mesophase is prevented. However, switchable phases are obtained when the terminal *n*-alkoxy chain is replaced by an *n*-alkyl carboxylate group. The behaviour of all the four different types of BC compounds investigated here suggests that it is difficult to relate the chemical structure to the type of mesophase exhibited by them.

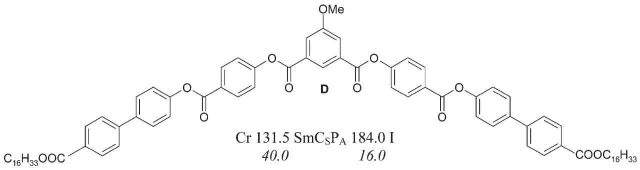


B

Cr 162.0 SmC$_S$P$_A$ 214.0 I
58.0 *21.5*



C



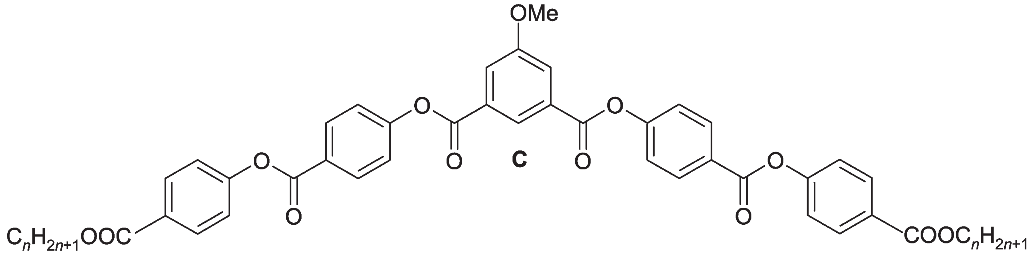
D

Cr 131.5 SmC$_S$P$_A$ 184.0 I
40.0 *16.0*

3.2. *XRD measurements*

XRD studies were carried out on powder samples of compounds **A4** and **A6**. Each sample was taken in a

Table 2. Transition temperatures (°C) and associated enthalpy changes (J g^{-1}) (in italics) for the compounds **C**. Cr=crystalline phase; SmC$_S$P$_A$=polar smectic C phase, S and A refers to synclinic and antiferroelectric; SmA=uniaxial smectic A phase; I=isotropic phase.



Compound	n	Cr		SmA		I
C1	14	•	100.5 *155.5*	(•	68.5) *7.0*	•
C2	18	•	92.5 *111.0*	(•	78.0) *5.5*	•

sealed Lindemann capillary (diameter 0.7 mm) and cooled slowly from the isotropic phase to the mesophase. An unoriented sample of compound **A4** showed two reflections in the small angle region with *d*-spacings of 45.8 and 22.9 Å in the ratio 1:1/2, indicating a lamellar ordering of the molecules in the mesophase. Similarly, compound **A6** showed two reflections in the small angle region with the *d*-spacings 50.6 and 25.3 Å, which again indicated a lamellar ordering. The first order layer spacing was found to be smaller than the measured molecular length assuming an all-*trans* conformation of the terminal *n*-alkoxy chains (for example $L \approx 63.0$ Å for compound **A6**). This suggests tilting of the molecules in the mesophase with a tilt angle of about 36.5°.

XRD patterns obtained for the mesophase of compounds **B** and **D** also showed equally spaced reflections in the small angle region indicating a layered arrangement in the mesophase. The *d*-spacings obtained for all these four compounds are shown in table 3. In all cases a diffuse reflection was observed in the wide angle region at about 4.4 Å indicating the fluidity of the phase. XRD results coupled with electro-optical studies (described below) suggest that the phase exhibited by compounds **A1**–**A6**, **B** and **D** is SmCP$_A$. An X-ray diffractogram obtained for the mesophase of compound **A6** is shown in figure 4 and is typical for the other homologues.

3.3. *Electro-optical studies*

The electro-optical switching behaviour of the mesophase exhibited by these compounds was studied using ITO-coated cells treated with polyimide for homogeneous alignment of the mesophase. The switching polarization measurements were carried out employing triangular-wave and modified triangular-wave electric field methods. For example, a sample of compound **A6** in its isotropic state was filled into a cell of thickness 8 μm through capillary action. The sample was cooled slowly from the isotropic phase to the mesophase and a triangular-wave voltage at a frequency of 100 Hz was applied and increased gradually. Two current peaks per half-period of the applied voltage were observed at a threshold of 8.5 V μm^{-1}, indicating an antiferroelectric ground state for the mesophase. The current response trace obtained for compound **A6** at 110°C is shown in figure 5. The switching polarization value obtained from the current response trace was 280 nC cm^{-2}.

Experiments were also carried out on the same sample using a d.c. field. On slow cooling the isotropic liquid under a d.c. voltage of 5 V μm^{-1} aligned domains were obtained in which extinction brushes were oriented parallel to the crossed polarizers. The orientation of the brushes was same either on reversing the sign of the applied voltage or on switching off the field. However, a colour change and stripes could be observed on the smooth domains on terminating the field. This indicates that the molecular organization in the ground state is possibly synclinic in adjacent layers, and there is a change in the clinicity after a few layers. This alternation of clinicity after few layers is repeated so that there exists a number of domains with opposite clinicity. The observed stripes are indicative of the domain walls where the tilt direction changes. Similar observations were made earlier [2, 22] which suggest a racemic

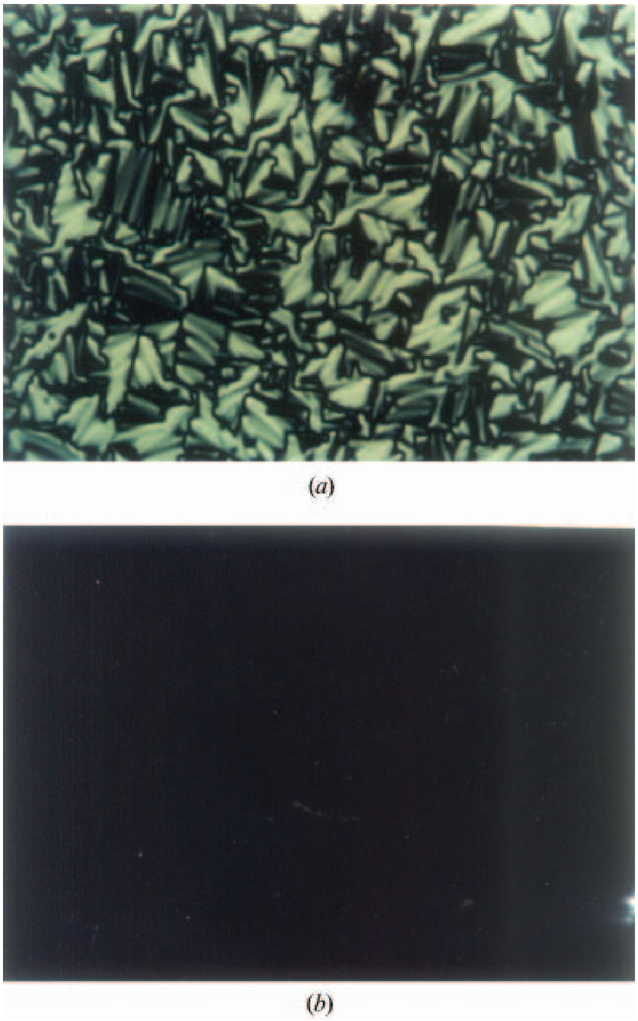


Figure 3. Photomicrographs of the SmA phase of compound **C1**: (*a*) homogeneously aligned sample; (*b*) homeotropically aligned sample.

antiferroelectric ground state for the mesophase. Photomicrographs illustrating the switching behaviour of the mesophase under a d.c. field of +5, 0 and $-5\,\mathrm{V\,\mu m^{-1}}$ are shown in figures 6 (*a–c*) respectively.

The mesophases exhibited by compounds **B** and **D** were also found to be switchable by applying a triangular-wave voltage. The mesophase of compound **B** showed two current peaks per half-cycle of the applied voltage at a threshold field of about $7\,\mathrm{V\,\mu m^{-1}}$, indicating that the ground state is antiferroelectric. The polarization value was found to be $148\,\mathrm{nC\,cm^{-2}}$ at 180°C by integrating the area under the current response peaks; the current response trace obtained is shown in figure 7.

Table 3. X-ray diffraction data for four of the compounds investigated.

Compound	*d*-spacings/Å	Temperature/°C
A4	45.8 (01), 22.9 (02)	103
A6	50.6 (01), 25.3 (02)	108
B	55.6 (01), 27.8 (02)	175
D	57.4 (01), 28.8 (02)	165

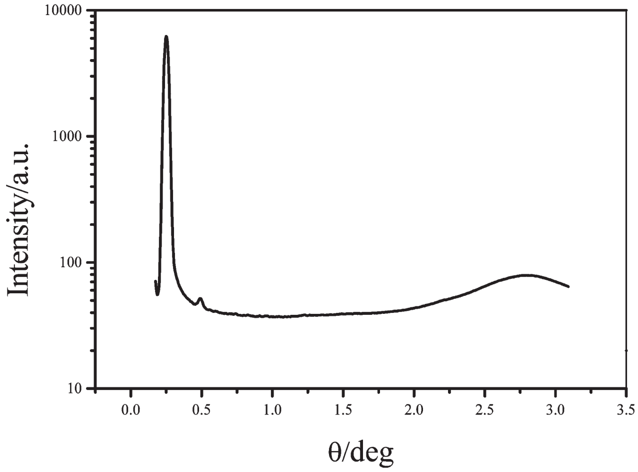


Figure 4. X-ray angular intensity profile obtained for the mesophase of compound **A6** at 108°C.

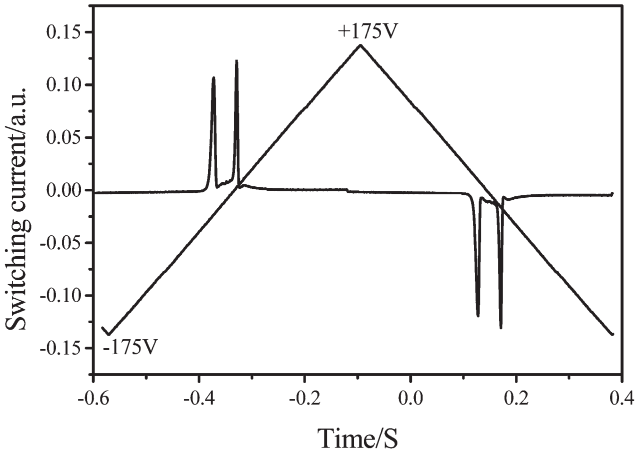
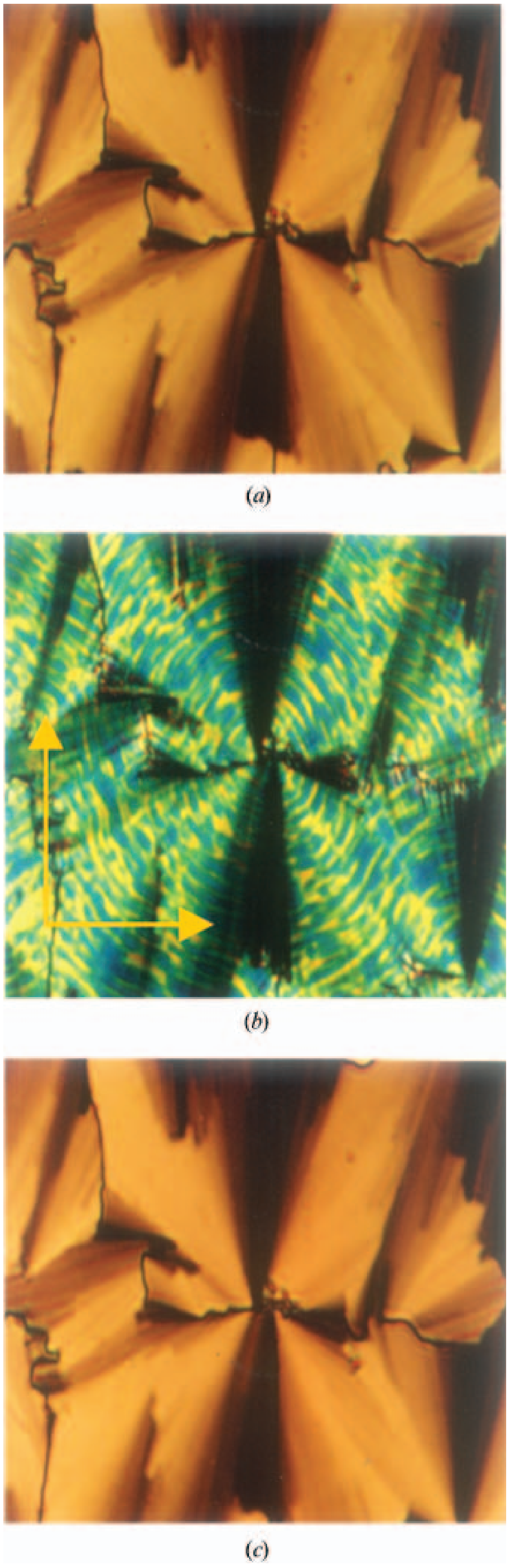


Figure 5. Switching current response trace obtained for the mesophase of compound **A6** by applying a triangular voltage: cell gap=8 μm; T=110°C; 1 Hz; 350 $\mathrm{V_{pp}}$; $\mathbf{P}_s \approx 280\,\mathrm{nC\,cm^{-2}}$.

The mesophase of compound **D** showed a single current peak when a triangular-wave field was applied at a very low threshold field of $1.5\,\mathrm{V\,\mu m^{-1}}$. The single current peak persisted even at a low frequency of 5 Hz. However, the single peak could be resolved into two by applying a modified triangular-wave with a plateau at 0 V, indicating an antiferroelectric ground state for the mesophase. The current response trace obtained at 165°C is shown in figure 8. A very low polarization value of $37\,\mathrm{nC\,cm^{-2}}$ was calculated from the current response trace.


(*a*)

(*b*)

(*c*)

Figure 6. Photomicrographs of the SmCP$_A$ phase of compound **A6** obtained under a d.c. field of (*a*) +5.0, (*b*) 0, (*c*) $-5.0\,V\,\mu m^{-1}$.

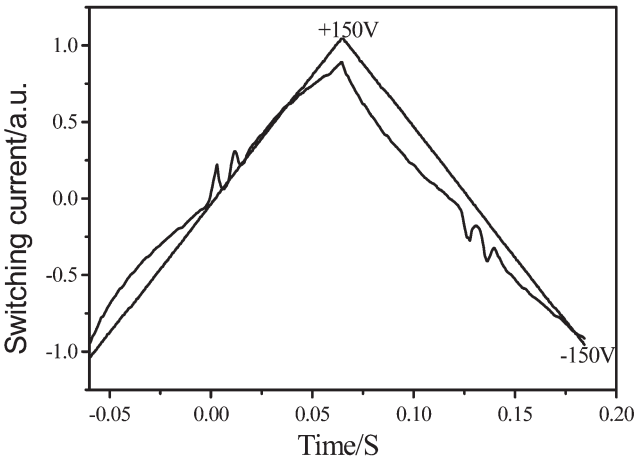


Figure 7. Switching current response trace obtained for the mesophase of compound **B** by applying a triangular voltage: cell gap=8 μm; $T=180°C$; 4 Hz; 300 V_{pp}; $\mathbf{P}_s \approx 148\,nC\,cm^{-2}$.

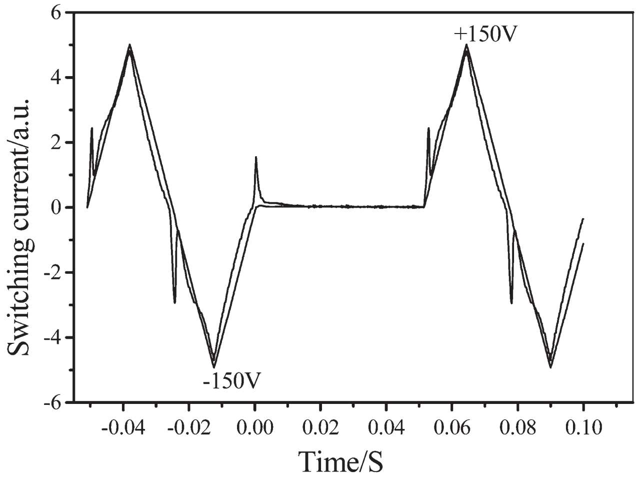


Figure 8. Switching current response trace obtained for the mesophase of compound **D** by applying a modified triangular voltage: cell gap=8 μm; $T=165°C$; 10 Hz; 300 V_{pp}; $\mathbf{P}_s \approx 37\,nC\,cm^{-2}$.

The low threshold field required for switching, as well as the low polarization value obtained in the case of compound **D**, is probably due to a decreased rotational barrier for the molecules because of the flexible terminal ester linking groups. The electro-optical switching behaviour for the mesophases of compounds **B** and **D** under a d.c. field is similar to that of compound **A6**, suggesting that the ground state is

SmC$_S$P$_A$. Electro-optical studies and XRD measurements could not be carried out for the mesophases of compounds **A9** and **A10**, since they are highly metastable.

4. Conclusions

In the case of bent-core compounds, various types of substituents in different positions along the core have a strong influence on the type of mesophase formed. In a five-ring BC system, the influence of a substituent situated on the central phenyl unit is profound. As mentioned earlier, studies on compounds derived from isophthalic acid are very limited, but show some interesting phases and phase sequences. Surprisingly, five-ring BC compounds derived from 5-methoxyisophthalic acid exhibit a electro-optically switchable mesophase. Hence, there is a need to investigate such compounds more intensively.

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