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Unusual mesomorphic behaviour of new bent-core mesogens derived from 4-cyanoresorcinol

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New five-ring bent-core mesogens derived from 4-cyanoresorcinol as the central core have been synthesized. The mesophases were assigned by polarizing microscopy, X-ray diffraction and electro-optical measurements. It was found that the mesophase behaviour clearly depends on the direction of the carboxyl linking groups between the aromatic rings. If the outer and inner ester linking groups of a wing are in the same direction then nematic phases and, for long chain members, SmC and $\mathrm{SmCP_A}$ phases are observed. If the outer and inner linking groups are in opposing directions a dimorphism, SmA–$\mathrm{SmAP_A}$, is observed and the clearing temperatures are increased by about 55 K. It is remarkable that in the $\mathrm{SmCP_A}$ phases of the first series, polar switching preferably takes place through a collective rotation of the molecules around their long axes which is accompanied by the inversion of the chirality of the smectic layers.

1. Introduction

Bent-core mesogens constitute a new sub-field of liquid crystal research which is attracting considerable research interest. The reason for this particular interest is not only the occurence of new mesophases, some of which possess unusual physical properties, for example ferroelectricity or antiferroelectricity [1–4], but also because some tilted mesophases are able to form chiral structures although the individual molecules are achiral [2, 5]. In order to obtain a deeper insight into the structure–property relationships of bent mesogens there has been much activity in the synthesis of such materials [1, 3, 6–21]. It was found that mesophase behaviour is strongly influenced by the number of rings, the type and direction of linking groups, the nature and position of lateral substituents and the type and length of the terminal chains. It is remarkable that the dependence of mesophasic behaviour on these structural features is much more pronounced than in calamitic compounds.

In this paper we present new five-ring bent-core mesogens which contain 4-cyanoresorcinol as the central core and only ester connecting groups between the aromatic rings. It will be shown that different directions of the outer ester linking group can lead to quite different mesophases having some unusual properties.

2. Experimental

Phase transitions were detected by polarizing optical microscopy (Leitz Laborlux) and by differential scanning calorimetry (DSC Pyris 1, Perkin-Elmer). The assignments of the mesophases were made by observation of the optical textures and by X-ray experiments. X-ray investigations on non-oriented samples were carried out using a Guinier goniometer or a Guinier film camera.

Investigations on oriented samples were performed using a 2D detector (HI-Star, Siemens AG). For compounds which form a nematic phase the homogeneous orientation of the nematic phase was achieved by a sufficiently high magnetic field ($\mathbf{B} \approx 1\,\mathrm{T}$). This orientation could be maintained by a very slow cooling of the nematic phase into the smectic phase in the presence of the magnetic field. In the absence of a nematic phase, well developed monodomains of the smectic phase were obtained by slow cooling a drop of the isotropic liquid below the clearing temperature on a thin and cleaned glass plate. The incident beam was nearly parallel to the glass plate and because of this geometry only the upper half of the reciprocal space could be observed, the lower part being shadowed by the glass.

3. Materials

The phase transition temperatures, transition enthalpies and phase sequences of the compounds studied are summarized in tables 1 and 2. The compounds **1a**–**g** and

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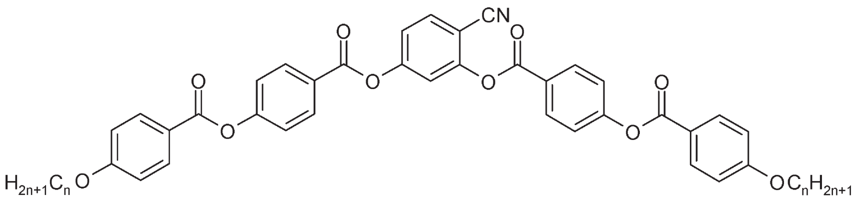
Table 1. Transition temperatures (°C) and associated enthalpy changes ($kJ\,mol^{-1}$, in square brackets) for the compounds **1a**–**1g**; brackets designate monotropic phases.



	n	Cr		B_1		$SmCP_A''$		$SmCP_A'$		$SmCP_A$		SmC		N		I
1a	6	•	105 [44.6]	—		—		—		—		—		•	140 [0.8]	•
1b	7	•	96 [37.7]	(•	68) [4.8]	—		—		—		—		•	132 [0.7]	•
1c	8	•	99 [52.0]	—		—		—		—		—		•	132 [0.8]	•
1d	9	•	92 [47.7]	—		—		—		—		—		•	128 [0.7]	•
1e	10	•	99 [66.1]	—		—		(•	66 [2.2]	•	77) [2.0]	—		•	128 [1.1]	•
1f	12	•	103 [65.7]	—		(•	68 [0.4]	•	75 [0.5]	•	94) [0.2]	•	109 [0.5]	•	129 [1.3]	•
1g	16	•	103 [65.7]	—		(•	74 [0.4]	•	79 [0.4]	•	99) [—][a]	•	133.5 [6.3]	—		•

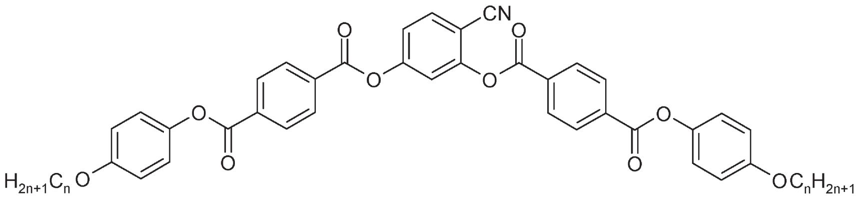
[a] This transition could not be detected by calorimetry.

2a, **b** were prepared by esterification of the corresponding substituted 4-(4-*n*-alkyloxybenzoyloxy)benzoic acids or 4-(4-*n*-alkyloxyphenoxycarbonyl)benzoic acids with 4-cyanoresorcinol by means of dicyclohexylcarbodiimide (DCC) [21]. As an example, the following experimental procedure is given for compound **1c**.

A mixture of 0.28 g (2.0 mmol) of 2,4-dihydroxybenzonitrile, 1.50 g (4.0 mmol) of 4-(4-*n*-octyloxybenzoyloxy)benzoic acid, 0.86 g (4.1 mmol) DCC and dimethylaminopyridine as catalyst, in 60 ml dichloromethane, was stirred at room temperature for 24–48 h. The precipitate was filtered off and extracted again with 30 ml dichloromethane. The mixed solvent was evaporated, and the crude product recrystallized from a DMF/ethanol mixture. To remove traces of dicyclohexylurea the solid compound was digested by stirring in 50 ml of ethanol at 60°C for 2 h. It was then recrystallized twice from DMF/ethanol; yield 0.72 g (43%). Analytical data are given for the compounds **1c** and **2a**

1c: ^1H NMR (400 MHz, $CDCl_3$) δ: 0.87 (t, $^3J=6.7$ Hz, 6H, CH_3), 1.28–1.49 (m, 20H, CH_2), 1.78–1.85 (m, 4H, $OCH_2\underline{CH_2}$), 4.04 (t, $^3J=6.7$ Hz, 4H, OCH_2), 6.97 (d,

Table 2. Transition temperatures (°C) and associated enthalpy changes ($kJ\,mol^{-1}$, in square brackets) for the compounds **2a** and **2b**.



	n	Cr		$SmAP_A$		SmA		I
2a	8	•	113 [32.4]	•	144 [1.2]	•	187 [8.4]	•
2b	12	•	106 [16.7]	•	112 [0.9]	•	185 [9.8]	•

^3J=8.8 Hz, 4H, Ar–H), 7.31 (dd, ^3J=8.6 Hz, ^4J=2.2 Hz, 1H, Ar–H), 7.38 (d, ^3J=8.9 Hz, 4H, Ar–H), 7.52 (d, ^4J=2.1 Hz, 1H, Ar–H), 7.78 (d, ^3J=8.6 Hz, 1H, Ar–H), 8.14 (d, ^3J=8.8 Hz, 4H, Ar–H), 8.25 (d, ^3J=8.9 Hz, 2H, Ar–H), 8.32 (d, ^3J=8.9 Hz, 2H, Ar–H).

2a: ^{1}H-NMR (200 MHz, $CDCl_3$) δ: 0.84–0.88 (m, 6H, CH_3), 1.29–1.45 (m, 20H, CH_2), 1.71–1.81 (m, 4H, $OCH_2\underline{CH_2}$), 3.95 (t, ^3J=6.5 Hz, 4H, OCH_2), 6.93 (d, ^3J=9.1 Hz, 4H, Ar–H), 7.12 (d, ^3J=9.3 Hz, 4H, Ar–H), 7.36 (dd, ^3J=8.6 Hz, ^4J=2.0 Hz, 1H, Ar–H), 7.58 (d, ^4J=2.2 Hz, 1H, Ar–H), 7.82 (d, ^3J=8.4 Hz, 1H, Ar–H), 8.31–8.35 (8H, Ar–H).

4. Experimental results

4.1. *Compounds 1a–g*

With the exception of the hexadecyloxy compound **1g**, all the homologues listed in table 1 exhibit a nematic phase. Homologue **1b** forms, in addition, a monotropic phase which is highly viscous and displays a mosaic texture. X-ray investigations were impossible because this phase crystallizes immediately after formation. According to the texture and its nucleation, we assigned this phase as a B_1 phase. The homologues with longer terminal chains form two (**1e**) or four (**1f**, **1g**) smectic phases. Before we discuss the interesting structural features and physical properties of these phases, it should be noted that the nematic phase of these compounds shows unusual electro-optical behaviour as illustrated, for example, for compound **1c** on applying an electric field. The nematic phase of **1c** forms domains parallel to the original direction of the director above a voltage of 4 V, figure 1 (*a*). The period of these domains was found to be 10 µm which is clearly greater than the sample thickness. On increasing the voltage the domain period decreases, 8 µm at 6 V, see figure 1 (*b*). Above 10 V the domain pattern is completely changed. As seen in figure 1 (*c*) equidistant stripes within the domains form while their period decreases with increasing voltage from 12 to 17 V, figure 1 (*d*). On further increasing the voltage (26 and 32 V) the stripes disappear and an optical texture results which is reminiscent of a smectic fan-shaped texture, figures 1 (*e*) and 1 (*f*). If the field is switched off, the smectic-like texture disappears. Similar electro-optical behaviour has been reported for the nematic phase of other bent-core compounds [22, 23].

The decyloxy homologue **1e** forms two smectic phases in addition to the nematic phase. The high temperature smectic phase exhibits a texture with small fan-like domains if formed on cooling the planar oriented nematic phase. On cooling the homeotropically oriented nematic phase, this smectic phase adopts a schlieren-like texture in which irregular parallel stripes (defects) appear, figure 2 (*a*). It is remarkable that in the schlieren texture domains of opposite handedness spontaneously arise, which could be visualized by a slight decrossing of the polarizers. At the transition into the low temperature smectic phase (at 66°C) the fan-like texture becomes grainy, while in the schlieren texture the stripes disappear, figure 2 (*b*).

The X-ray patterns of powder-like samples show strong Bragg reflections in the small angle region and a diffuse scattering maximum in the wide angle region for both smectic phases.

We obtained well oriented nematic samples by applying a magnetic field of 1 T. The X-ray pattern shows a diffuse dumb-bell-like scattering maximum in the small angle region, and a diffuse wide angle scattering located around the equator, figures 3 (*a*) and 3 (*b*). This suggests the existence of cybotactic groups (a smectic-like packing of short range order) in which the molecules are tilted with respect to the layer normal. The splitting-off of the diffuse maxima is increasingly pronounced with decreasing temperature, and the maxima condense to Bragg-spots with the same period at the transition into the smectic phase indicating the layer structure, figure 3 (*c*). At the transition into the smectic phases, the diffuse wide angle scattering remains unchanged. From the χ-scan, the tilt angle was found to be 34° in the high temperature smectic phase and 37° in the low temperature phase. From the small angle reflections a layer spacing d of 37.6 Å was determined, which is clearly smaller than the molecular length L (51 Å). From the experimentally determined tilt angle θ and layer spacing d the effective length L_{eff} can be calculated ($L_{eff}=d/\cos\theta=37.6$ Å$/\cos 34°=45$ Å), which leads to a difference of about 6 Å with respect to the molecular length. Such a large difference is explained by an intercalation of molecules in adjacent layers.

The fan-shaped texture of the high temperature smectic phase can be switched by an electric field (threshold$\approx$5 V µm^{-1}) and the textures of the switched state are independent of the field polarity. At the transition into the low temperature phase, the threshold of the switching is clearly increased to about 15 V µm^{-1}; the optical appearance of the switching is also changed. As in the high temperature smectic phase, the texture of the switched state is independent of the polarity of the applied field, but in contrast to the high temperature smectic phase, the textures of the field-off and field-on states are identical, only during switching is a slight change of the birefringence observed. Furthermore, in both smectic phases the current response shows two

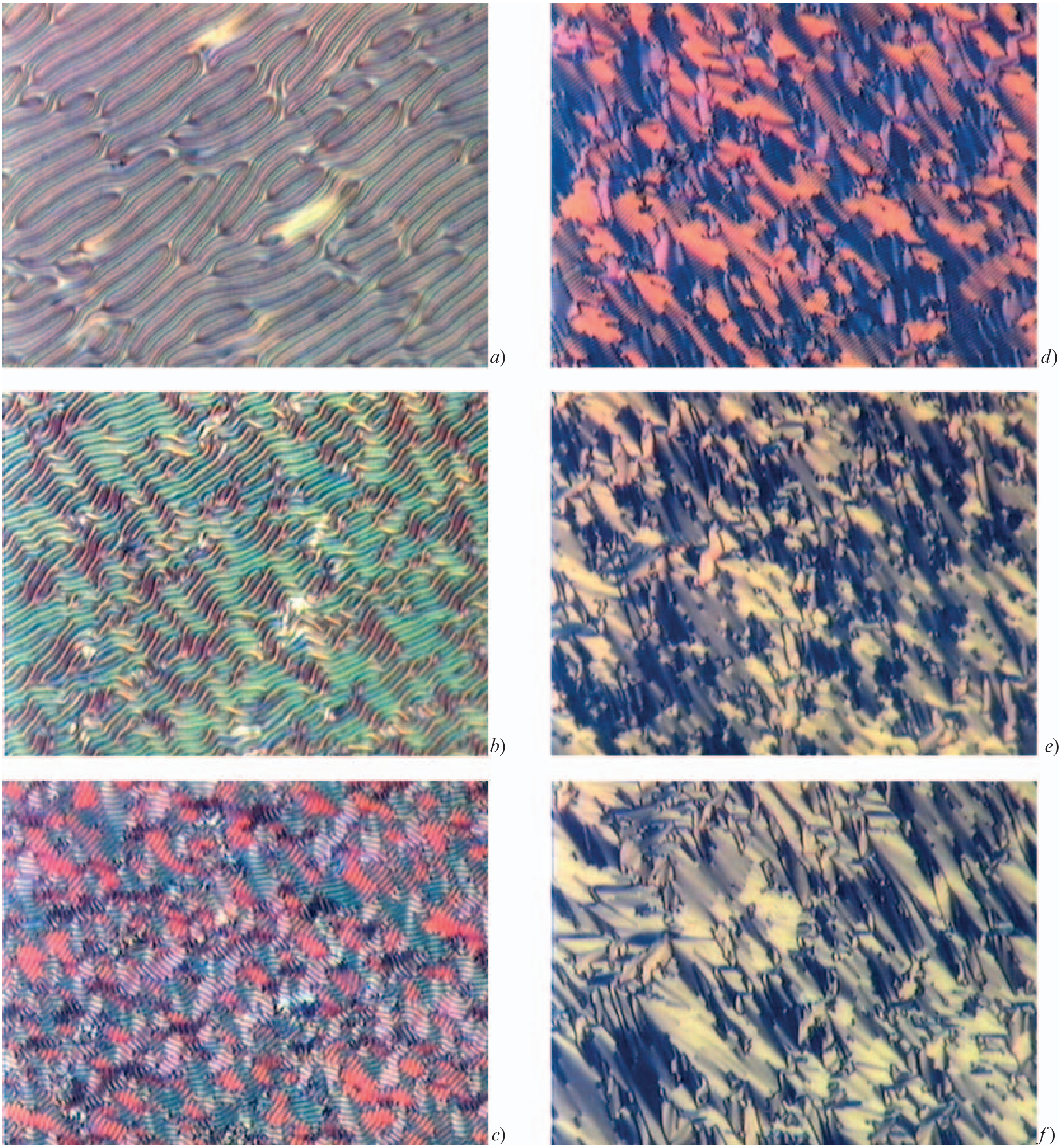


Figure 1. Field-induced texture of the nematic phase of compound **1c** (sample thickness 6 μm, temperature 121°C): (*a*) 4 V, (*b*) 6 V, (*c*) 10 V, (*d*) 17 V, (*e*) 26 V and (*f*) 32 V.

peaks per half-period of a triangular voltage, indicating a switching from an antiferroelectric ground state into ferroelectric states. In the high temperature smectic phase, the switching polarization is relatively small (≈150 nC cm^{-2}); it increases strongly in the low temperature phase (770 nC cm^{-2} at 65°C and

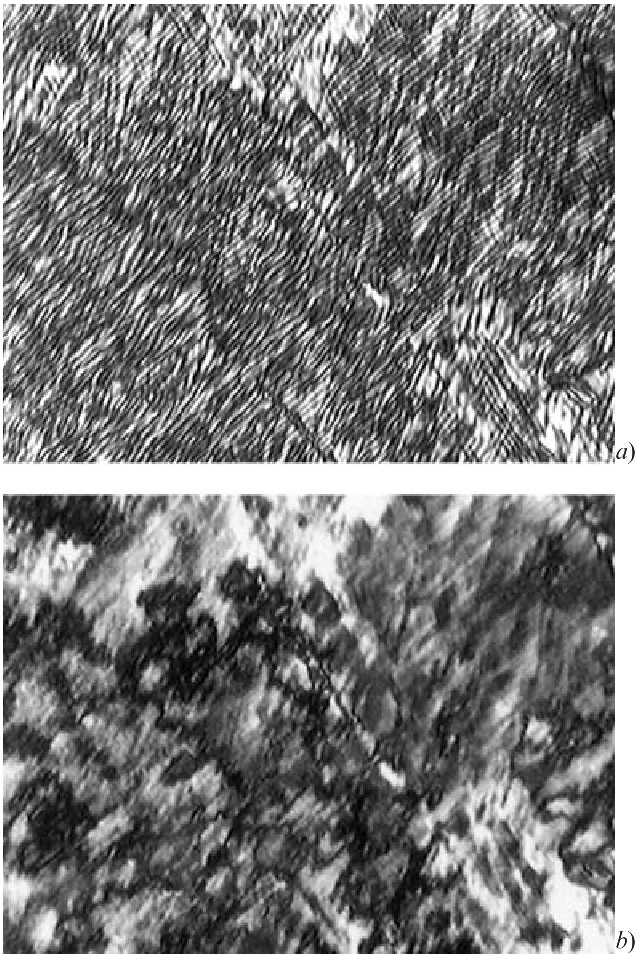


Figure 2. (*a*) Texture of the SmCP$_A$ (72°C) phase, and (*b*) texture of the SmCP$_A$′ phase (62°C) of compound **1e**.

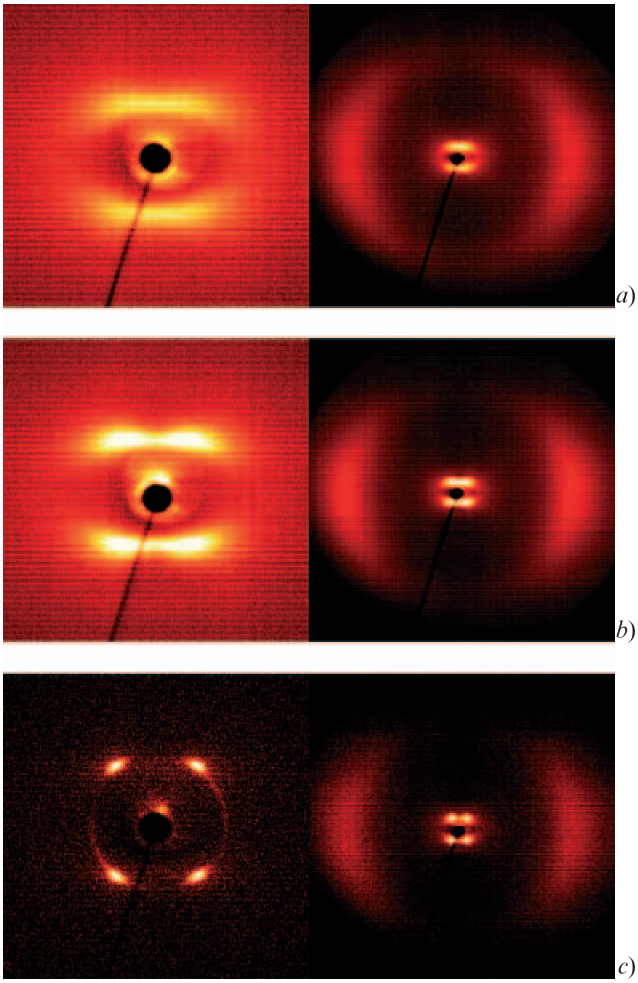


Figure 3. X-ray patterns of oriented samples in the mesophases of compound **1e**: (*a*) nematic phase (122°C), (*b*) nematic phase (87°C) and (*c*) SmCP$_A$ phase (76°C).

1000 nC cm^{-2} at 62°C). On the basis of the structural and electro-optical investigations both smectic phases can be assigned as SmCP$_A$ phases.

In the dodecyloxy homologue **1f**, four smectic phases may be distinguished by calorimetry in addition to a nematic phase. If the planar oriented nematic phase is cooled, the smectic high temperature phase appears with a texture which contains small fan-like domains. At 94°C the number of defects decreases and the birefringence increases. The phase transitions at 75 and 68°C (see table 1) could not be clearly identified by polarizing microscopy because the paramorphotic textures change essentially continuously. On cooling the homeotropically oriented nematic phase, the smectic high temperature phase adopts a schlieren texture which transforms into paramorphotic schlieren textures on cooling into the low temperature phases. Unfortunately, X-ray studies could only be performed on the nematic and the two high temperature smectic phases because of crystallization of the sample. The layer spacing *d* in the high temperature smectic phase was found to be 42.2 Å at 100°C; it decreases slightly in the phase below (d=40.1 Å at 85°C). As with compound **1e**, we were able to obtain well oriented monodomains by cooling the nematic sample in the presence of a magnetic field of **B**≈1 T. The X-ray patterns of the nematic and smectic phases are reasonably similar to those of compound **1e**. Thus, cybotactic groups are seen in the nematic phase with a tilt angle of the molecules within the groups of 28°. In the high temperature smectic phase this tilt angle remains unchanged, but in the smectic phase below 95°C the tilt angle clearly increases to 35°.

The high temperature smectic phase shows no current response. This is a clear indication of a non-polar SmC phase. This SmC phase may be switched by application of a sufficiently high d.c. or a.c. field, corresponding to a Fréedericksz transition. This switching is not accompanied, however, by a noticable change of the texture;

the domain walls and birefringence remain unchanged. In all the smectic phases below the SmC phase, we found two current peaks per half-period of a triangular voltage, indicating an antiferroelectric groundstate, figures 4 (*a*) and 4 (*b*). Therefore we designated these phases by the coded $SmCP_A$, $SmCP_A'$ and $SmCP_A''$. The switching polarization was found to be rather high ($650\,nC\,cm^{-2}$ at 80°C and $970\,nC\,cm^{-2}$ at 66°C). It is remarkable that the optical appearance of the switching is similar to that for the switching in the SmC phase, i.e. in the $SmCP_A$ and $SmCP_A'$ phases the textures of the switched state are independent of the polarity of the field. On the other hand, the field-off and the field-on states were also found to be identical provided that the field was applied or removed relatively slowly. In the case of the $SmCP_A''$ phase, the textures with field and without field are also identical for fast switching (see figure 5). Such switching behaviour may be understood if the polar switching occurs through a collective rotation of the molecules around their long axes and not, in the usual way, by rotation around the tilt cone (see the schematic representation in figure 6). This switching mechanism has been observed for $SmAP_A$ phases [24, 25], for some columnar B_1 phases [26] and also for SmCP phases [27–30].

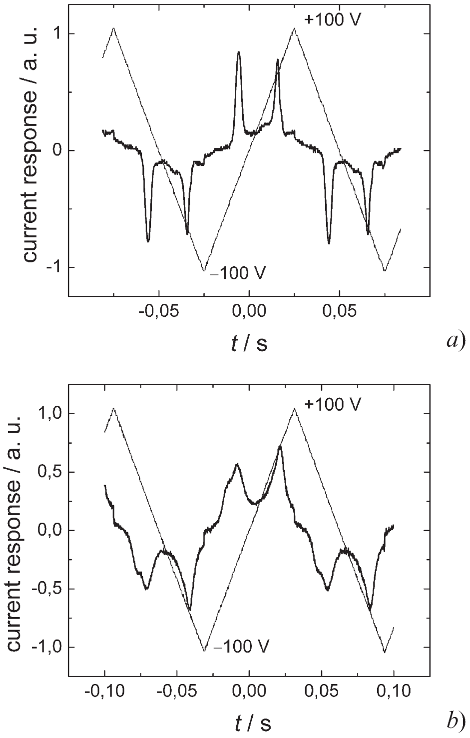


Figure 4. Switching current response on applying a triangular voltage ($210\,V_{PP}$, 20 Hz, cell thickness 6 μm): (*a*) in the $SmCP_A$ phase (80°C) and (*b*) in the S_mCP_A'' phase (66°C) of compound **1f**.

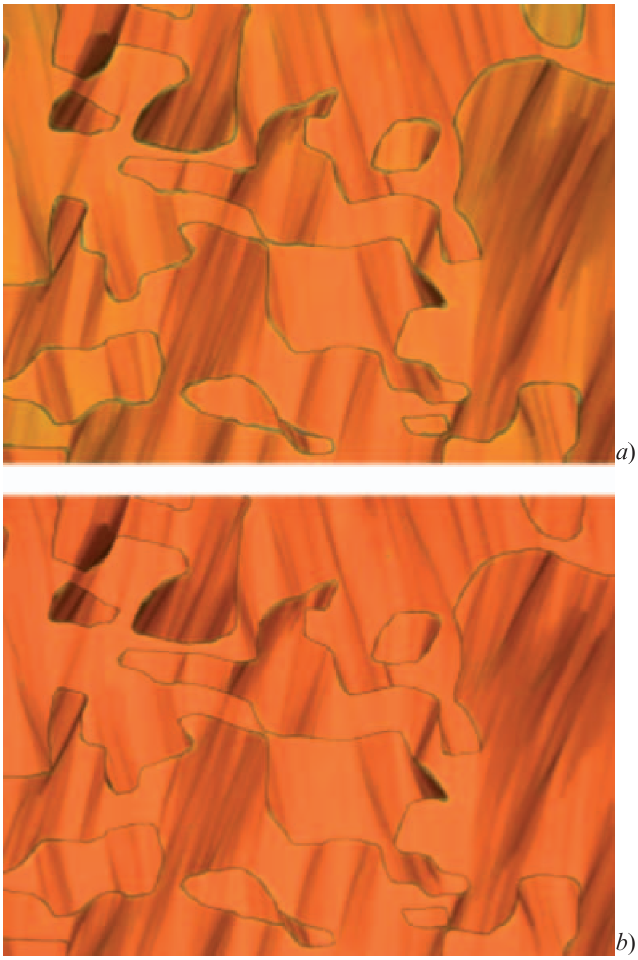


Figure 5. Texture of the $SmCP_A$ phase of compound **1f** (cell thickness 6 μm): (*a*) 0 V and (*b*) ±105 V (80°C).

Apart from the absent nematic phase, the mesophase behaviour of the hexadecyloxy homologue **1g** is relatively similar to that of compound **1f**. In this case, also, the transition from the SmC to the high

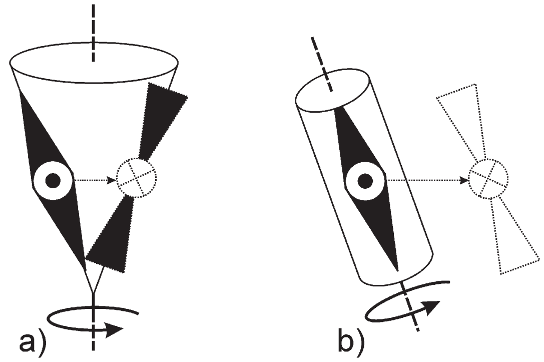


Figure 6. Mechanisms of polar switching in SmCP phases: (*a*) switching by a collective rotation of the molecules around the tilt cone, and (*b*) switching by a collective rotation of the molecules around their long axes.

temperature $SmCP_A$ phase at 99°C (see table 1) can be identified by a change of the schlieren texture into a texture with additional defects. This texture adopts irregular parallel stripes which disappear at the transition at 79°C. Interestingly, the phase transition at 99°C could not be detected by calorimetry or by a significant change of the X-ray pattern, which is an indication of a second order phase transition. This phase transition is accompanied, however, by the appearance of a polar electro-optical response. Using the triangular wave voltage method, we recorded two peaks per half-cycle which were also found in the other low temperature smectic phases. Thus, all these phases possess an antiferroelectric ground state and were therefore designated as $SmCP_A$, $SmCP_A'$ and $SmCP_A''$. Figure 7 shows the temperature dependence of the switching polarization $\mathbf{P}_{SW}$. It can be seen that $\mathbf{P}_{SW}$ shows a strong change in the $SmCP_A$ phase and remains nearly constant in the low temperature $SmCP_A'$ and $SmCP_A''$ phases.

4.2. *Compounds 2a,b*

It is remarkable that in comparison with the analogous compounds of series **1**, the clearing temperatures of series **2** are enhanced by about 55°C, although the isomeric compounds (**1c**, **1f** and **2a**, **2b**; see tables 1 and 2) are distinguished only by the direction of the outer ester linking groups. Another key difference is the absence of nematic phases in compounds **2a** and **2b** and the predominance of smectic phases. Both homologues show smectic dimorphism. The high temperature smectic phase is a SmA phase which is characterized by a homeotropic or fan-shaped texture. If the homeotropic texture is cooled, the low temperature smectic phase forms a weakly birefringent and strongly fluctuating schlieren texture which becomes more strongly birefringent on further cooling. If the SmA fan-shaped texture is cooled, the texture of the low temperature phase remains almost unchanged. Only some coloured irregular stripes perpendicular to the fans form (figure 8), the number of which increases with decreasing temperature. By application of an electric field above $4\,V\,\mu m^{-1}$, the stripes disappear and the colour (birefringence) of the fan-shaped texture is changed. This field-induced texture is identical for the opposite polarity of the applied field. The current response shows two peaks per half-period of the applied triangular field, which points to an antiferroelectric ground state (figure 9). The switching polarization was found to be 640 and $320\,nC\,cm^{-2}$ for compounds **2a** and **2b**, respectively.

It follows from X-ray investigations on non-oriented samples that the layer spacings *d* in both smectic phases are almost equal and temperature-independent. For compound **2a** the *d*-value varies from 41.9 Å at 180°C to 43.0 Å at 110°C. For compound **2b** the *d*-value increases from 49.0 Å at 160°C to 50.4 Å at 80°C. In both compounds the layer spacing of the smectic phases is

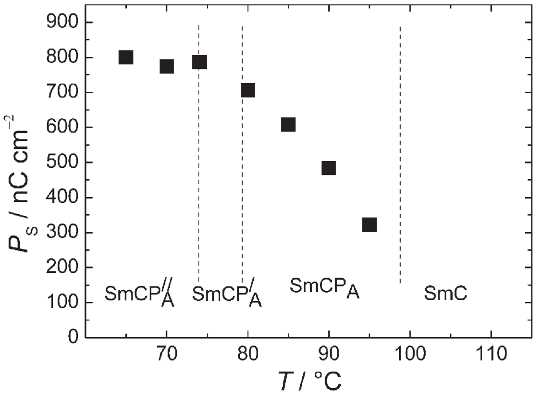


Figure 7. Temperature dependence of the switching polarization of compound **1g** ($380\,V_{PP}$, 100 Hz).

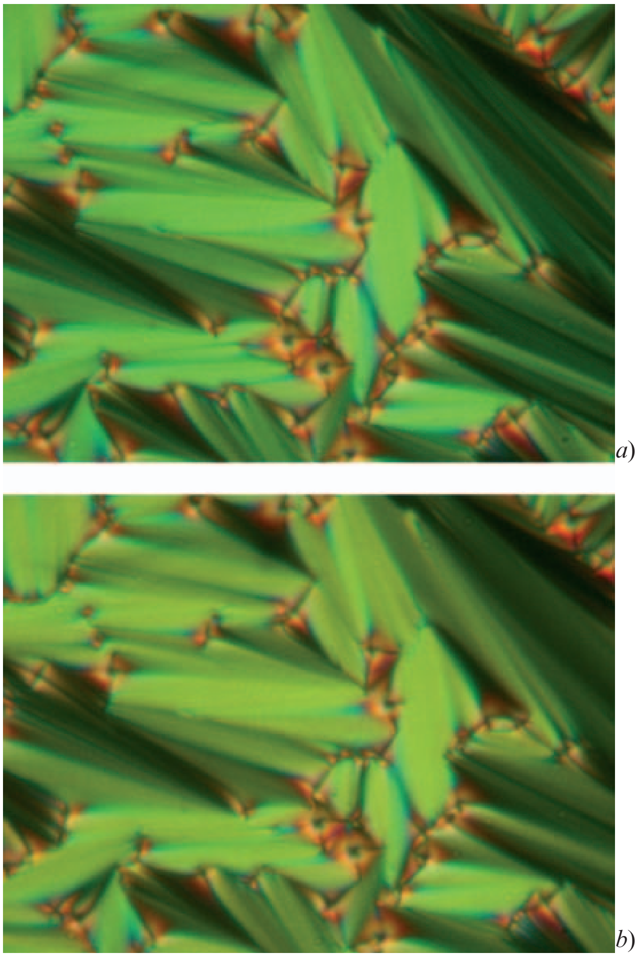


Figure 8. Fan-shaped texture of (*a*) the SmA phase (146°C) and (*b*) the $SmAP_A$ phase (140°C) of compound **2a**.

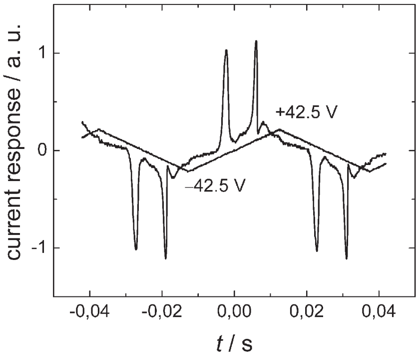


Figure 9. Switching current response on applying a triangular voltage (85 V_{PP}, 20 Hz) in the $SmAP_A$ phase of compound **2a** (cell thickness 5 μm; temperature 120°C).

clearly smaller than the molecular length ($L\approx48$ Å for compound **2a** and $L\approx55$ Å for compound **2b**).

The X-ray pattern of the SmA phase obtained from an oriented sample shows Bragg reflections on the meridian and a diffuse outer scattering, the maxima of which are, as expected, located on the equator. The corresponding X-ray pattern of the low temperature smectic phase is essentially identical; specifically in this phase the maximum of the diffuse scattering is also located (within the limits of error) on the equator, indicating that the molecular long axes are, on average, perpendicular to the layer planes. These experimental findings (X-ray patterns and electro-optical behaviour) provide evidence that the low temperature smectic phase is an antiferroelectric polar biaxial SmA phase ($SmAP_A$), which was theoretically predicted by Brand *et al.* [5] and experimentally reported by several authors [24, 25, 31].

The difference between the d-values of both homologues ($d_{2b}-d_{2a}\approx7$ Å) is the same as the difference of the estimated lengths of the molecules ($L_{2b}=55$ Å; $L_{2a}=48$ Å). This suggests that the assumed molecular shape, at least the direction of the aliphatic chains, is quite reasonable. On this basis it may be concluded that the difference between the d- and L-values in the orthogonal phases, SmA and SmAP, requires the intercalation of the alkyl chains in adjacent layers by about 6–7 Å.

5. Discussion

It is known that five-ring bent-core mesogens with 4-cyanoresorcinol as the central core and azomethine linking groups at the terminal rings are able to form both $SmCP_A$ and conventional smectic phases (SmA, SmC) and nematic phases [21]. The main reason for such behaviour is the relatively large bending angle between the two wings of the molecules which decreases with decreasing temperature. Similar behaviour can also be invoked to explain the behaviour of the compounds under discussion here.

A comparison with studies of laterally non-substituted resorcinol derivatives for which only banana phases have been observed [27] may help to further our understanding of our compounds. The tilt angle in the SmCP phase was experimentally found to be about 45°, yielding an 'orthoconic' structure. In this case the relationship between d and L corresponds to a value as expected for this tilt angle. The investigations reported here point to smaller tilt angles, although the difference between d and L is almost the same as in the preceding paper. The lateral substituent, however, prevents a similar dense lateral packing in the layers as observed for the laterally non-substituted analogues. There are two consequences arising from this structure: first, the weaker 'polar' packing may explain the occurrence of banana phases as well as nematic and conventional smectic phases; second, it provides the possibility for intercalation. In accordance with the literature [21, 32, 33] the homologues with shorter terminal chains preferably form nematic phases (typical for calamitic compounds) whereas in homologues with longer terminal chains the tendency to form banana phases is more pronounced.

The relatively large differences of the clearing temperatures between the corresponding members of series **1** and **2** are quite similar to those between the analogous compounds without the lateral cyano group [27, 30, 34]. It is remarkable that in series **2** SmA–$SmAP_A$ phase sequences occur which have only been observed for a few compounds.

Another remarkable experimental result is the unusual mechanism of the polar switching in the $SmCP_A$ phases of compounds **1**. We found that the switching between the antiferroelectric ground state and the switched ferroelectric states preferably takes place through the collective rotation of the molecules around their long molecular axes. To date, in SmCP phases such a switching mechanism has been found only for a limited number of materials [27–30].

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