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## Liquid Crystals

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# Mesomorphic properties of achiral halogen-substituted banana-shaped compounds

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Three banana-shaped achiral compounds, derivatives of 4,6-dichloro-1,3-phenylene bis-[4-(4-*n*-octyloxyphenyliminomethyl)benzoate], were synthesized by varying the substituent ( $X = \text{H, F and Cl}$ ). Their mesomorphic properties were investigated by differential scanning calorimetry, polarizing optical microscopy, and X-ray analysis. The compound with  $X = \text{H}$  exhibited an enantiomeric nematic phase. The compounds with  $X = \text{F and Cl}$  formed a nematic phase on heating, while on cooling they formed a nematic phase at high temperature and a smectic A phase at a lower temperature (monotropic). A schematic representation of the mesophase structures for the compound with  $X = \text{F}$  is also illustrated.

## 1. Introduction

Classical thermotropic liquid crystals are commonly composed of linear molecules, their structural linearities giving an advantage in the formation of mesophases. Recently, however, many unconventional liquid crystals with non-linear structures have been reported by a number of research groups [1–5]. Vorländer [6] was the first to synthesize mesogenic materials with a bent molecular shape; he noted that some compounds such as bis[4-(4-methoxyphenylazo)phenyl]isophthalate and 1,2-phenylene bis[4-(4-ethoxyphenylazo)benzoate] are liquid crystalline [6–8]. Later, mesogenic compounds containing acute-angled configurations, such as 1,2-phenylene and 2,3-naphthalene moieties, were shown

to exhibit mesomorphic properties. Their phases were classical nematic and/or smectic [1]. Recently, Pelzl *et al.* [9], investigated original samples of Vorländer's materials, and one of these compounds indeed forms a  $B_6$  phase, which is characteristic for banana-shaped molecules.

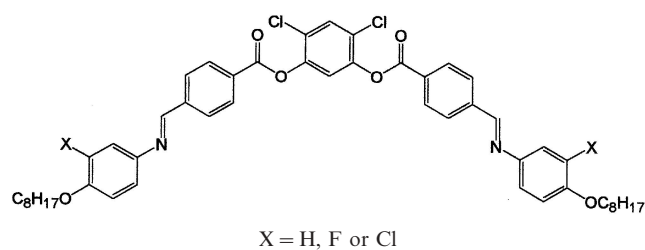
In 1996, Niori *et al.* [2] observed the first obvious example of ferroelectricity in a smectic phase formed by achiral banana-shaped molecules; these consist of a central angular unit and two linear mesogenic units attached to a terminal flexible chain. Subsequently, numerous attempts were made by several groups to understand clearly the structure–property relationships of banana-shaped molecules. However, up to now these relationships are not completely understood. From the structural point of view, molecules of this type reportedly have five, six and seven aromatic rings. Among the

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banana-shaped mesogens reported so far, the five-aromatic-ring system has been extensively studied; minor changes in the structure of these molecules can lead to great changes in mesomorphic behaviour. The mesomorphic properties of banana-shaped molecules are very sensitive to magnitude of the bend [10], position of substituents [11, 12], direction of linking group [13], and type of terminal chains [14, 15]. Because the structure of the mesophase formed by banana-shaped molecules is not fully understood, the mesophases are tentatively denoted B<sub>1</sub>–B<sub>7</sub> according to the order of discovery.

Link *et al.* [16] reported that achiral molecules with bent cores could form a chiral layer structure and exhibit antiferroelectric properties. Sekine *et al.* [17, 18] reported that achiral banana-shaped molecules could form helical domains in the smectic phases. We reported work on the mesomorphic and electro-optical properties of 1,3-phenylene bis[4-(4-*n*-alkoxyphenyliminomethyl)benzoate] having a lateral halogen substituent such as Cl or F in the 3-position of the *p*-alkoxyaniline Schiff's base moiety. Their mesomorphic and electro-optical properties were greatly influenced by the introduction of the Cl or F substituent. The molecule with a Cl substituent showed ferroelectricity while the molecule with a F substituent revealed antiferroelectricity [19, 20].

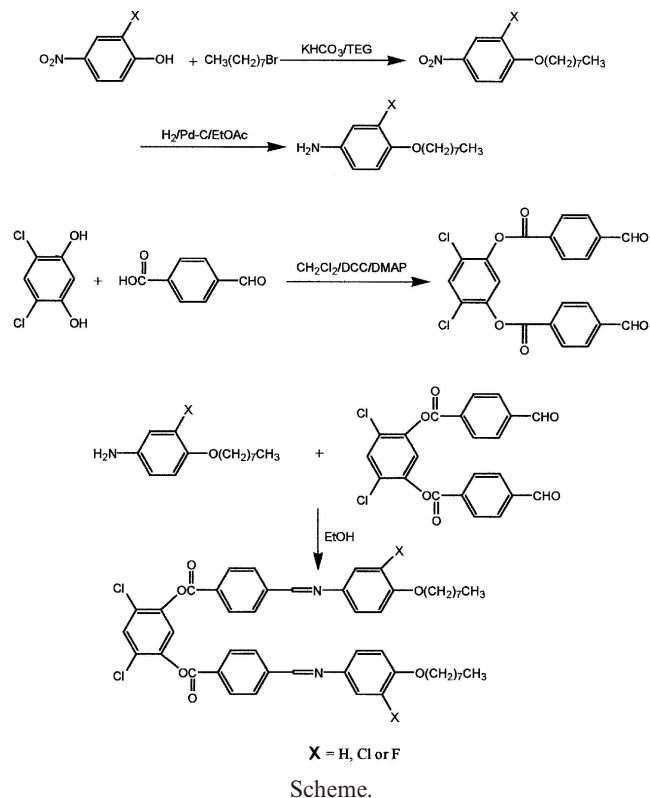
In this paper, we report the crystalline and mesomorphic properties of banana-shaped molecules with variations  $X = \text{H, F}$  and Cl and the introduction of dichloro-substitution at the 4,6-positions of the central phenylene unit.



## 2. Experimental

### 2.1. Synthesis

The synthetic route followed to obtain the banana-shaped molecules used a modified literature procedure [21, 22] and is shown in the scheme. Since the synthetic procedures for the three compounds were essentially the same, we show one representative synthesis for 4,6-dichloro-1,3-phenylene bis[4-(3-chloro-4-*n*-octyloxyphenyliminomethyl)benzoate] (DPBCOB). First, 3-chloro-4-*n*-octyloxyaniline was prepared from 3-chloro-4-nitrophenol and 1-bromooctane, followed by hydrogenation with H<sub>2</sub> gas in the presence of palladium on activated carbon. Second, 4,6-dichloro-1,3-phenylene bis(4-formylbenzoate) was obtained by the esterification reaction between 4,6-dichlororesorcinol and 4-carboxy-



benzaldehyde. This reaction was performed in methylene chloride with dicyclohexylcarbodiimide and a catalytic amount of dimethylaminopyridine. The DPBCOB was finally obtained by the reaction between 3-chloro-4-*n*-octyloxyaniline and 4,6-dichloro-1,3-phenylene bis(4-formylbenzoate). The crude product was purified by chromatography on silica gel, and recrystallized several times from ethanol/dimethylformamide. Yield after purification was 20 ~ 30%. Spectroscopic data: <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz);  $\delta = 0.6\text{--}1.9$  (30H, m), 4.0 (4H, t), 6.9–8.3 (16H, m), 8.5 (2H, s).

### 2.2. Characterization

IR and NMR spectra were obtained with Hitachi 270-50 IR and Bruker DRX NMR spectrometers, respectively. The phase transition temperatures were determined by differential scanning calorimetry (Perkin-Elmer DSC7) and polarizing optical microscopy (POM) (Nikon Eclipse E400 POL). DSC measurements were performed in a N<sub>2</sub> atmosphere, with heating and cooling rates of 10 and 0.5°C min<sup>-1</sup>, respectively; indium was used for calibration. Optical textures were observed using a polarizing microscope equipped with a digital camera and temperature controller.

SAXS measurements were performed in transmission mode with synchrotron radiation at the beamline 4C1. Two-dimensional scattering patterns were collected on a CCD (charge coupled device) detector (Roper Scientific)

with a wavelength of 1.608 Å. The observed intensity was corrected for background scatter and absorption by the sample. Since the samples exhibited typical isotropic scattering patterns in the absence of an external field, the intensity data were normalized to one-dimensional scattering patterns by integration through circular averaging to improve the data resolution, and therefore to reduce the measurement time. WAXS measurements were conducted with an apparatus consisting of an 18 kW rotating anode X-ray generator (Rigaku Co.) operated at 50 kV  $\times$  20 mA, mirror optics with point focusing, and a one-dimensional position sensitive detector (M. Braun Co.). The  $\text{CuK}_\alpha$  radiation ( $= 1.542 \text{ \AA}$ ) from a  $0.1 \times 1 \text{ mm}^2$  microfocus cathode was used. A beam path was maintained under vacuum to reduce air scattering, and tungsten foil ( $50 \text{ }\mu\text{m}$ ) was used for primary beam protection. The data are presented as a function of  $q = 4\pi \sin(2\theta)/\lambda$ , where  $2\theta$  is the scattering angle and  $\lambda$  is the X-ray wavelength. In order to investigate structural changes on heating and cooling, the samples were held in an aluminium sample holder, sealed with a window of  $7 \text{ }\mu\text{m}$  thick Kapton film on both sides. The sample was heated with two cartridge heaters and its temperature monitored by a thermocouple placed close to the sample.

The bulk sample, about 1 mm thick and 6 mm in diameter, was sealed between two thin Mylar sheets spaced with a Teflon O-ring. The O-ring was held between two aluminium plates, pressing the Mylar sheets tightly against the O-ring surface. The sample cell was placed inside a two-stage oven to eliminate any temperature gradients. The sample was aligned in the nematic phase with the help of a  $6 \sim 7 \text{ kG}$  magnetic field produced by a pair of cobalt-samarium permanent magnets, and an iron yoke and tapered pole pieces with  $\sim 109^\circ$  cone angle to concentrate the field on to a small area [23]. The sample was heated to the isotropic phase and cooled slowly, in the presence of the field, to the smectic phase. The field was applied perpendicular to the incident X-ray beam in the scattering plane. The adapter with the sample sat inside the two-stage oven connected to a precision temperature controller having long term stability of 1 mK.

### 3. Results and discussion

#### 3.1. Thermal property and optical texture

Figure 1 displays the DSC thermograms of the achiral banana-shaped liquid crystalline (LC) compounds. On the DSC heating scan, the compound with  $X = \text{H}$  shows three endothermic peaks, corresponding to solid–solid, melting, and isotropic transitions. As its isotropic liquid was cooled, the compound clearly showed two exothermic peaks corresponding to isotropic–mesophase and

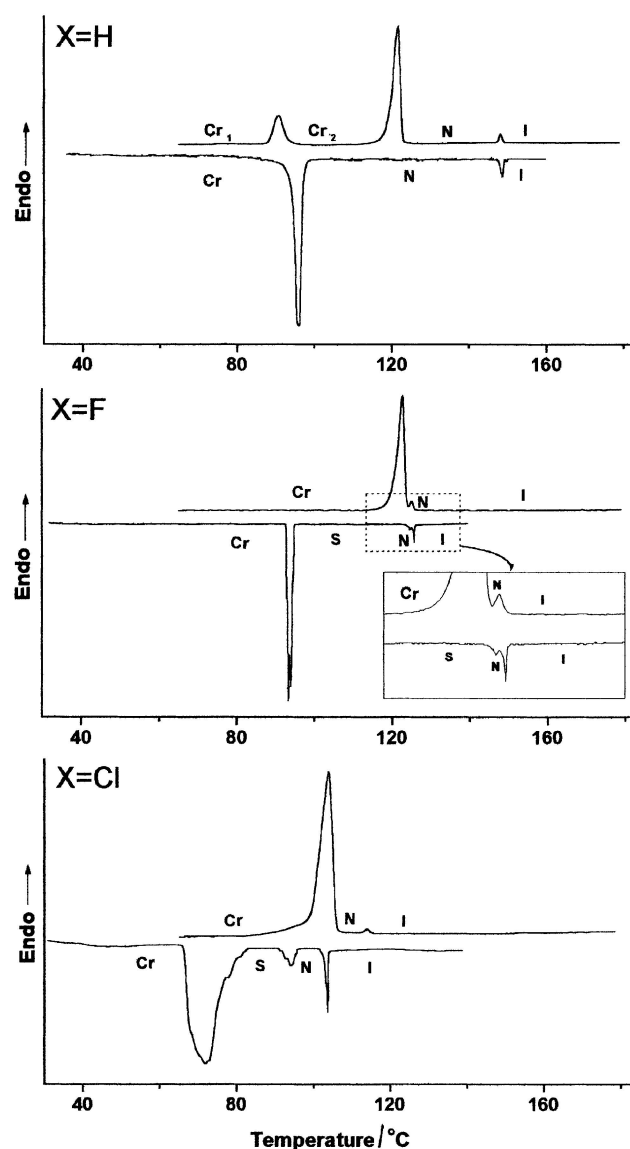


Figure 1. DSC thermograms of the three achiral banana-shaped liquid crystalline compounds on heating and cooling (heating rate  $10^\circ\text{C min}^{-1}$ ; cooling rate  $0.5^\circ\text{C min}^{-1}$ ).

mesophase–solid transitions. On heating, the compounds with  $X = \text{F}$  and  $\text{Cl}$  showed two peaks for melting and isotropic transitions; while on cooling, three peaks correspond to isotropic–nematic, nematic–mesophase 2 and mesophase 2–solid transitions were seen.

Figure 2 displays the optical textures of these compounds. The samples, under observation by POM, were cooled from isotropic melts slowly to room temperature. As shown in figure 2(a), the H-compound showed a threaded nematic texture on cooling the isotropic liquid; on further cooling it crystallized. In figure 2(b), the F-compound formed a nematic schlieren texture on cooling from the isotropic liquid; further cooling led to

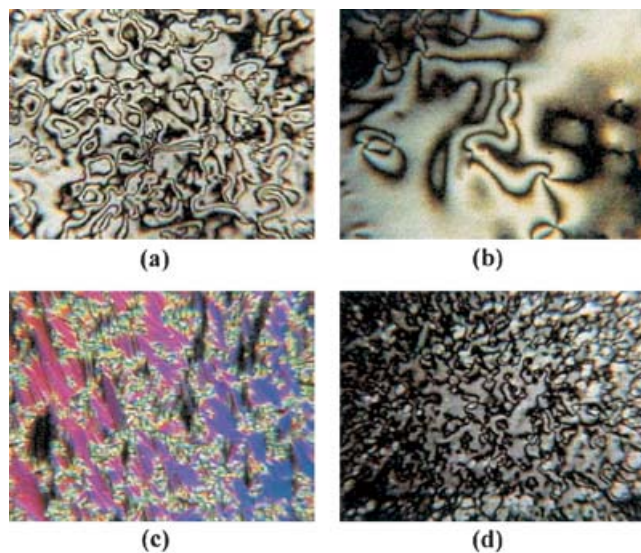


Figure 2. Optical textures of the achiral banana-shaped liquid crystalline compounds. (a) Nematic texture for H-compound taken at 127°C on cooling from the isotropic liquid. (b) Nematic texture for F-compound taken at 124°C on cooling from the isotropic liquid. (c) Smectic texture for F-compound taken at 105°C. (d) Nematic texture for Cl-compound taken at 98°C on cooling from the isotropic liquid.

the formation of a smectic texture as shown in figure 2(c). The Cl-compound, figure 2(d), showed a threaded texture for the nematic phase, while its smectic region showed a fan-like texture which was further identified as a smectic A phase by X-rays. The table summarizes transition temperatures and corresponding enthalpy changes obtained from DSC thermograms. The phase sequences shown in the table were corroborated by microscopic observation and X-ray analysis.

Table. Phase sequences, transition temperatures (°C) and corresponding enthalpy changes (in parenthesis, J g<sup>-1</sup>) for the banana-shaped compounds<sup>a</sup>. Cr = crystalline phase; N = nematic phase; SmA = smectic A phase; I = isotropic phase.

X	Heating <sup>a</sup>			Cooling <sup>b</sup>		
	Cr–Cr	Cr–N	N–I	I–N	N–SmA	SmA–Cr
H	90.8 (12.3)	121.8 (38.9)	148.2 (1.2)	148.8 (1.7)	—	96.1 (42.7)
F	—	122.8 (36.9)	125.1 (1.1)	125.8 (1.2)	124.8 (0.3)	93.5 (54.1)
Cl	—	104.0 (51.8)	114.1 (0.4)	103.9 (1.5)	93.9 (1.2)	71.6 (35.4)

<sup>a</sup> DSC heating rate: 10°C min<sup>-1</sup>.

<sup>b</sup> DSC cooling rate: 0.5°C min<sup>-1</sup>.

### 3.2. X-ray study

Figures 3–5 display the X-ray diffraction (XRD) patterns obtained at a given temperature. In figure 3(a), the XRD pattern of the H-compound at room temperature shows several strong diffraction peaks, indicating the presence of a crystalline phase. As the sample was heated to 130°C, the nematic characteristics of a broad wide angle peak and a rather narrow small angle peak were obtained, see figure 3(b). In the isotropic phase, the small angle peak became broader as shown in figure 3(c). Subsequently, as the isotropic liquid of the same sample was cooled, a nematic broad diffraction pattern reversibly appeared at 140°C, figure 3(d), and a crystalline diffraction pattern appeared at room temperature, see figure 3(e). The lattice spacing was calculated to be 42 Å from the relative sharp peak at  $q = 1.49 \text{ nm}^{-1}$ , corresponding to the length of a fully extended molecule.

In figure 4(a), the XRD pattern of the F-compound at room temperature displays several strong diffraction peaks indicating a crystalline phase. On heating to 125°C, above the transition temperature of 122.8°C (by DSC), most of the diffraction peaks disappeared except for the peak at  $q = 1.49 \text{ nm}^{-1}$  in the small angle region, figure 4(b). Upon further heating to 128°C, the peak vanished as shown in figure 4(c), indicating a transition to the isotropic phase. On cooling the isotropic melt to 125°C, a peak at  $q = 1.49 \text{ nm}^{-1}$  appeared reversibly,

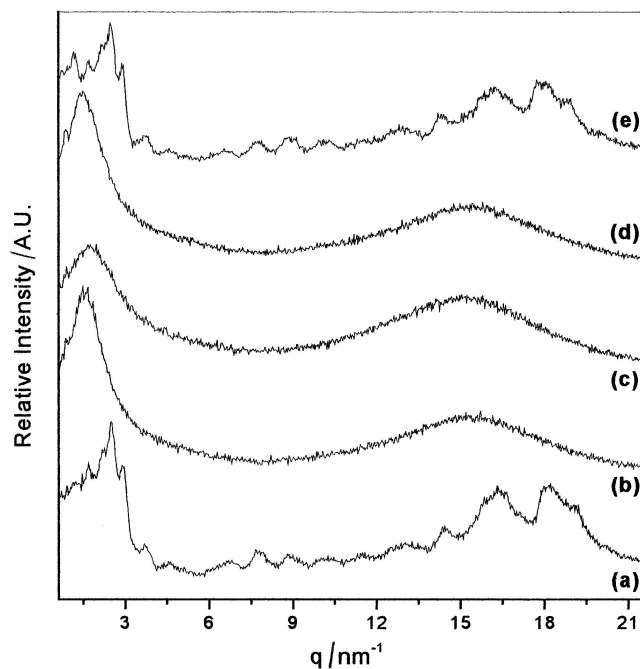


Figure 3. XRD patterns of the H-compound: upon heating as-prepared sample, measured at (a) room temperature, (b) 130°C and (c) 160°C; upon cooling the isotropic liquid of the same sample, measured at (d) 140°C and (e) room temperature.

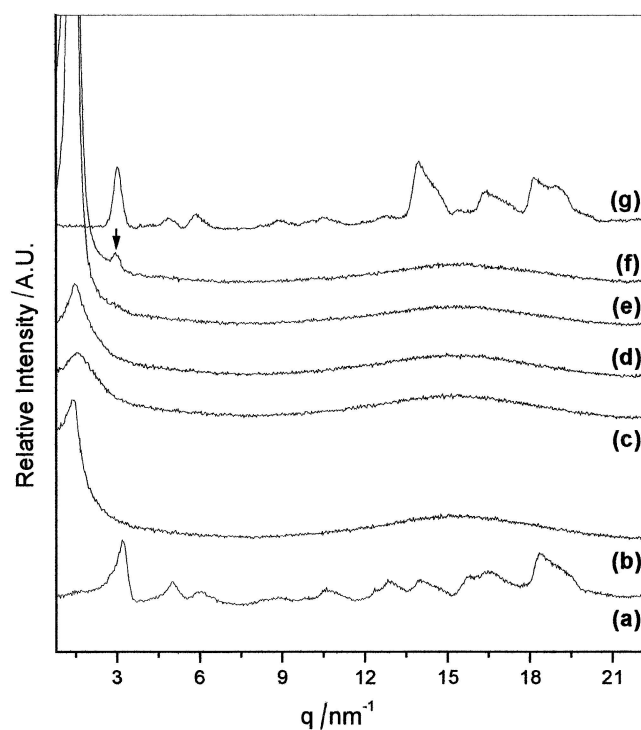


Figure 4. XRD patterns of the Cl-compound: upon heating as-prepared sample, measured at (a) room temperature, (b) 125°C and (c) 128°C; upon cooling the isotropic liquid of the same sample, measured at (d) 125°C, (e) 120°C, (f) 100°C and (g) room temperature.

figure 4(d), representing the nematic phase. On further cooling the sample to 120°C, a very strong peak begins to appear at  $q = 1.46 \text{ nm}^{-1}$ , figure 4(e). A second peak (indicated by an arrow) was well developed at 100°C as shown in figure 4(f). Since no crystalline peaks are found at the wide angle region, this XRD pattern is indicative of a smectic phase having a layer spacing of 43 Å with a 1:2 ratio of peak scattering vectors. The XRD pattern confirmed the crystalline structure at temperatures below 93.5°C, with many sharp peaks in the diffraction pattern, figure 4(g).

As shown in figure 5, the XRD patterns of the Cl-compound showed the same aspect as those of the F-compound. The nematic phase occurred reversibly as shown in figures 5(b) and 5(d), whereas the smectic phase appeared only monotonically on cooling, as figure 5(e).

On the basis of the X-ray results, schematic representations of the molecular structure of the smectic and nematic phases of the F-compound can be constructed as shown in figure 6. Although the presence of a bent moiety and the fluorine substituent prevents regular stacking of the molecules, they can rotate and glide and form a two-dimensional structure. Thus, a stable smectic A mesophase with a layer spacing of 43 Å can possibly

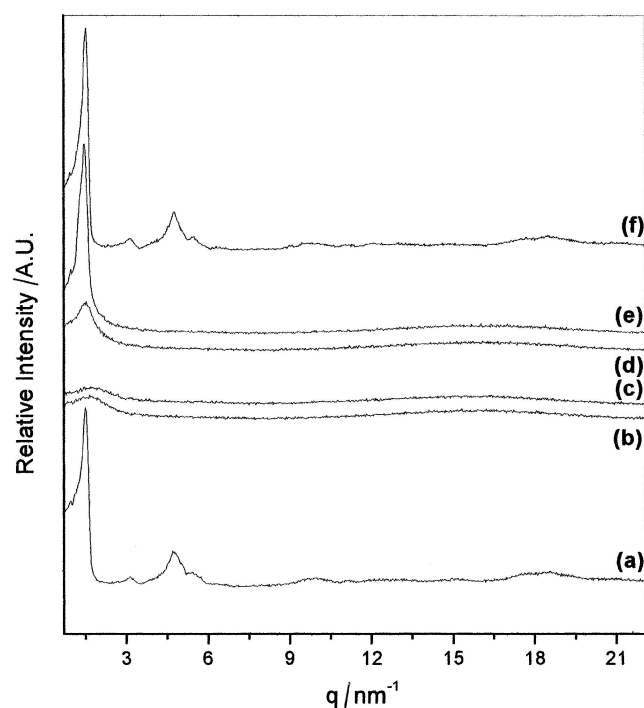


Figure 5. XRD patterns of the F-compound: upon heating as-prepared sample, measured at (a) room temperature, (b) 110°C and (c) 140°C; upon cooling the isotropic liquid of same sample, measured at (d) 103°C, (e) 93°C and (f) room temperature.

be formed. In the case of the nematic phase, the molecules can self-assemble into a rod-like cylinder, which behaves like a nematic mesogen. The larger distance of 43 Å in the nematic mesophase may originate from interference among these self-assembled cylinders.

Figure 7 shows a representative longitudinal scan and a transverse in-plane scan for a sample of the F-compound in  $\sim 6 \text{ kG}$  magnetic field at temperature  $T = 110^\circ\text{C}$ . The wave vector at Bragg peak,  $q_{\parallel}^0 = 2\pi/d$ , that corresponds to the layer spacing  $d$  is almost constant in the temperature range of the smectic region, as seen by microscopic observation. This result confirms that there is only one phase, and the  $q_{\parallel}^0$  is equal to  $0.140 \text{ \AA}^{-1}$  ( $d = 44.6 \text{ \AA}$ ). The full width at half maximum of the wave vector is  $\sim 0.00085 \text{ \AA}^{-1}$  which is a typical value obtained in a very well aligned sample in the smectic A phase. The transverse in-plane scan at  $q_{\perp} = q_{\parallel}^0$ , shown in figure 7(b), shows a typical peak obtained in the smectic A phase at  $q_{\perp} = 0$ . The peak intensities at representative longitudinal and transverse in-plane scans are the same. A  $5 \mu\text{m}$  test cell rubbed anti-parallel on the substrate cannot be switched at the above temperature range, confirming that no smectic C phase exists. (The diffraction pattern at higher temperature (125°C) looks like a pattern obtained from a nematic phase, although the results are not given in this paper).

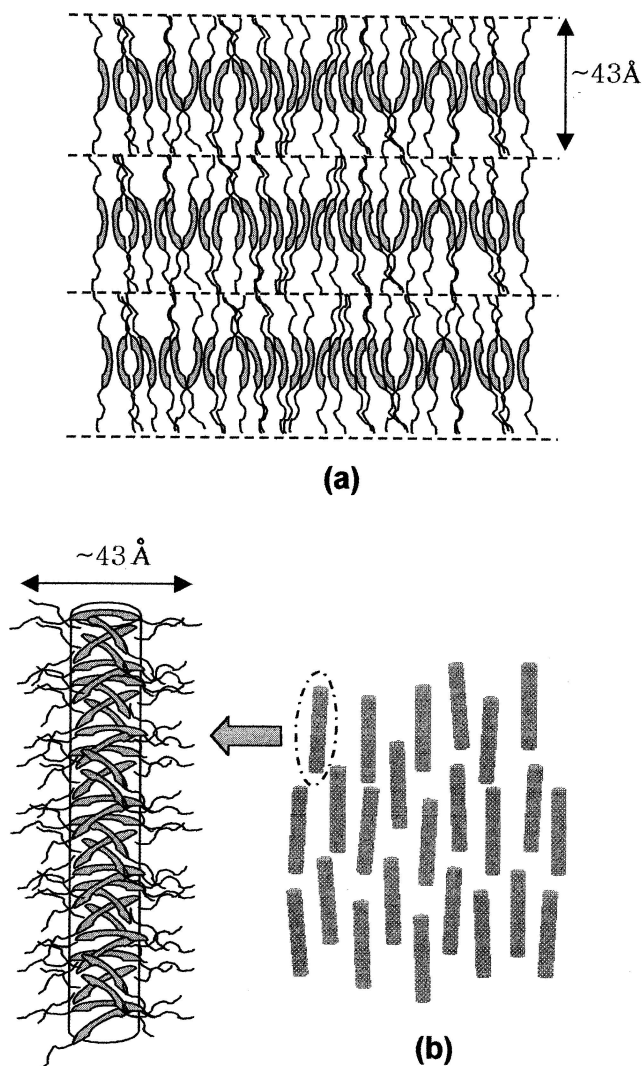


Figure 6. Schematic representation of the mesophase structures of the F-compound. (a) Smectic A phase where the bent molecules in each layer act as a smectic mesogen. (b) The nematic phase is composed of cylindrical rod columns derived from the layer structure of the smectic A phase.

Figure 8 shows a representative longitudinal scan and a transverse in-plane scan for a sample of the X-compound in  $\sim 6$  kG magnetic field at temperature  $T = 90^\circ\text{C}$ . With the same analysis, the wave vector monotonically decreases without any jump; this indicates that there is no phase transition from smectic A to variant smectic in the temperature range of the smectic region seen by DSC cooling scan; its range is from  $0.148$  to  $0.149 \text{ \AA}^{-1}$  ( $d = 42.3 \sim 42.2 \text{ \AA}$ ). The full width at half maximum of the wave vector is  $\sim 0.0013 \text{ \AA}^{-1}$ , a slightly higher value than previously obtained from the F-compound. Thus the alignment of the F-compound is better than that of the Cl-compound. The transverse in-plane scan at  $q_{\parallel} = q_{\parallel}^0$  shown in figure 8 has a typical

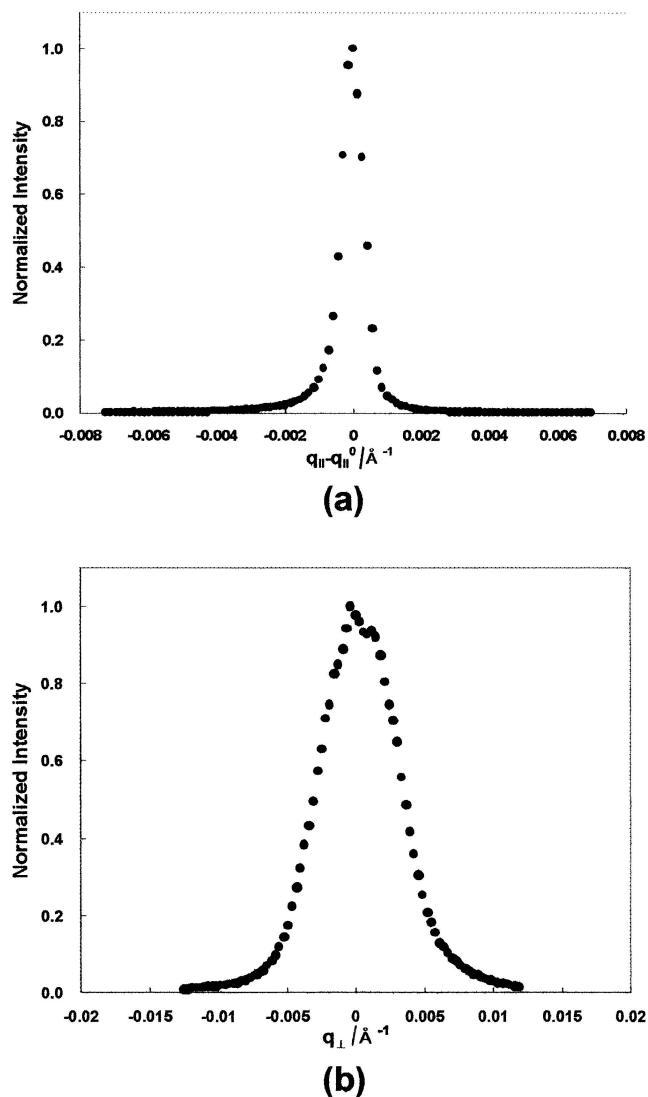


Figure 7. Representative (a) longitudinal scan ( $q_{\parallel}$ ) and (b) transverse in-plane scan ( $q_{\perp}$ ) plotted on normalized intensity for the F-compound at  $110^\circ\text{C}$ .

peak obtained in the smectic A phase at  $q_{\perp} = 0$ . Peak intensities at representative longitudinal and transverse in-plane scans are also identical. By analysing the transmittance of a twisted nematic cell we also find that there is a nematic phase at  $103^\circ\text{C}$ .

#### 4. Conclusion

We report three achiral halogen-substituted banana-shaped liquid crystal materials exhibiting an enantiomeric nematic phase. The banana-shaped mesogens with  $X = \text{F}$  and  $\text{Cl}$  exhibit a monotropic smectic A phase confirmed by various physical characterization techniques. Although this series of banana-shaped mesogens lack a switchable

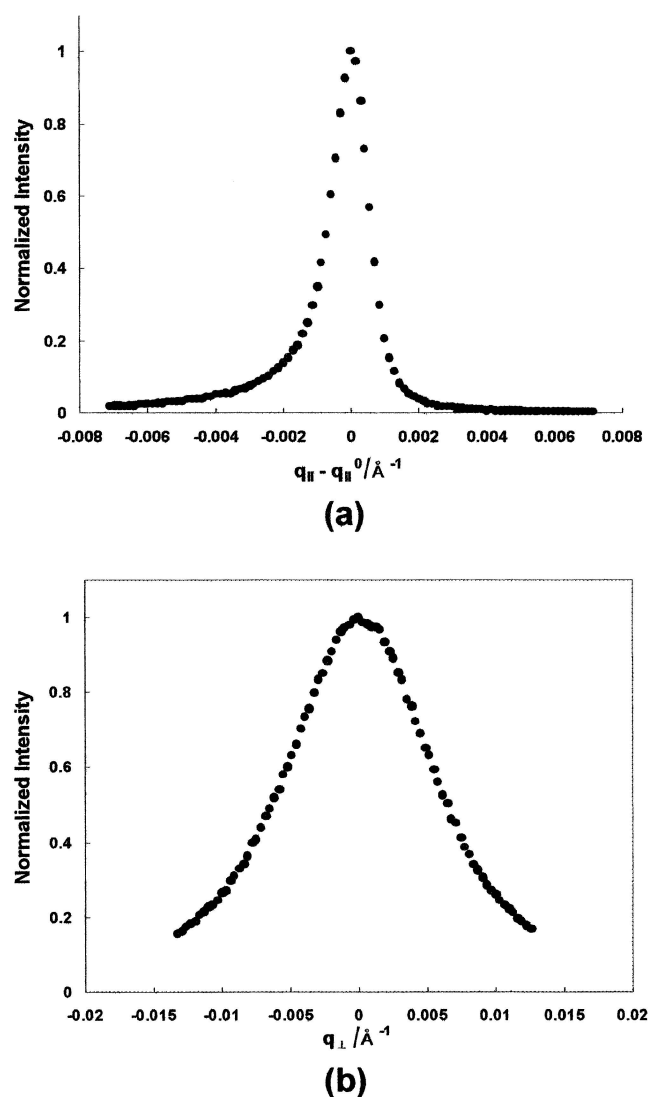


Figure 8. Representative (a) longitudinal scan ( $q_{\parallel}$ ) and (b) transverse in-plane scan ( $q_{\perp}$ ) plotted on normalized intensity for the Cl-compound at 90°C.

smectic (banana) phase, yet their ferroelectric properties may in future be studied by forming mixtures with an electrically switchable banana-shaped molecule.

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Laboratory (Beamline 4C1), Korea, and by Prof. Satyendra Kumar at Kent State University, USA.

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