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## Liquid Crystals

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### **Dimesogenic liquid crystal consisting of cholesterol and Schiff base moieties: dependence of LC properties on the spacer length and fluorination of the alkoxy tails**

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# Dimesogenic liquid crystal consisting of cholesterol and Schiff base moieties: dependence of LC properties on the spacer length and fluorination of the alkoxy tails

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The liquid crystalline properties of two series of non-symmetric liquid crystal dimers consisting of cholesterol and Schiff base moieties interconnected by  $\omega$ -oxyalkanoyl spacers of varying length are compared: one series (**SBOC-*n***) carry the octyloxy tail on the Schiff base mesogen, and the other (**SBOF-*n***) a perfluoroheptylmethoxy tail. In general, compounds with the fluorinated alkoxy tail exhibited mesophases over a much wider temperature range than those with the alkoxy tail. The latter series favoured the formation of more diverse mesophases than the former. **SBOC-4**, **-5** and **-7**, and **SBOF-4**, **-5** and **-10** formed the chiral smectic C phase.

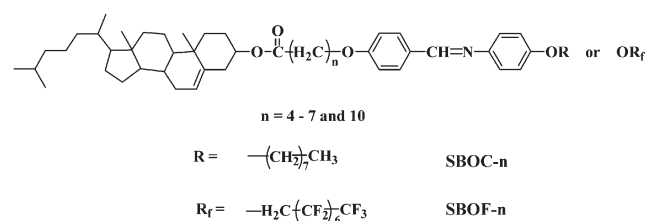
## 1. Introduction

We and others [1–16] reported earlier a great abundance of polymorphism exhibited by some non-symmetric dimers. Most of the non-symmetric dimers described by ourselves [3, 5, 6, 8, 10, 11–13] contain the cholesterol moiety as an integral structural component, which contains several chiral centres. Luckhurst *et al.* [7] reported LC properties of dimesogens consisting of a Schiff base mesogen bearing a chiral alkyl tail and a cyanobiphenyl mesogen. More scientific studies have been discussed by many research groups [1, 4, 7, 14] on a wide variety of non-symmetric and symmetric dimers without chiral centres. We were particularly interested in investigating (i) the dependence of liquid crystal (LC) behaviour on the length and parity of the spacers, (ii) the dependence of LC behaviour on the nature of the tails on the Schiff base mesogen and (iii) induction of the formation of the chiral smectic phase, especially of the SmC\* phase, by the chiral centres in the cholesterol moiety. The chiral smectic C phase may be induced in these systems, depending on the length of alkoxy and perfluorinated tails.

Meanwhile, the effect of fluorination of aromatic mesogenic cores and polyfluorinated alkyl or alkoxy tails on LC properties of monomesogenic compounds have been the subjects of many studies [17–21].

Recently, we compared LC properties including ferroelectric behaviour of a series of non-symmetric dimers in which the cholesterol moiety is interlinked through the 6-oxyhexanoyl spacer to the second azobenzene-based mesogen bearing the octyloxy or perfluoroheptylmethoxy tail [12, 13].

In this study, we compare the LC properties of two series of dimesogens on varying the length of the central spacer linking the two mesogenic units. The **SBOC-*n*** series have an octyloxy tail while the **SBOF-*n*** series possess the perfluoroheptylmethoxy tail, with '*n*' denoting the number of methylene groups in the central spacer.

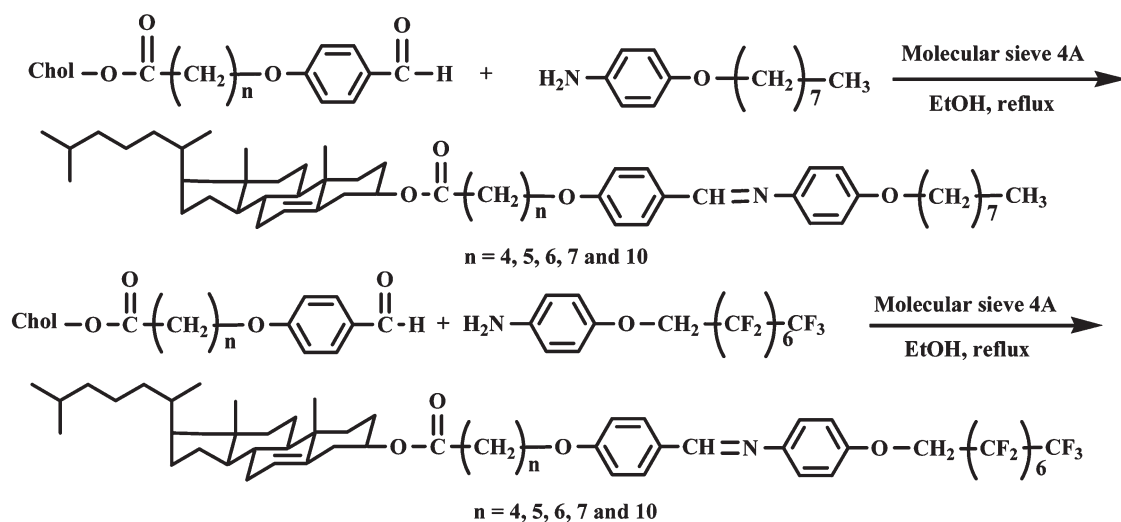


## 2. Experimental

### 2.1. Synthesis

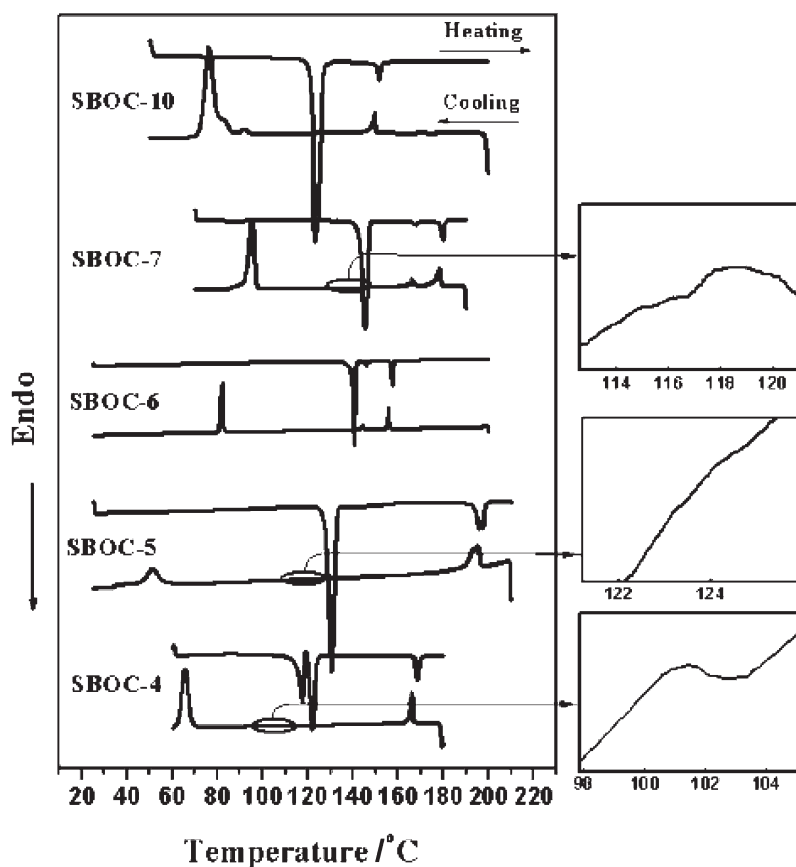
The compounds of the **SBOC-*n*** and **SBOF-*n*** series were prepared following the multi-synthetic routes shown in scheme 1. Since details of the synthetic procedure have

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Scheme 1. Synthetic route to compounds of the **SBOC-*n*** and **SBOF-*n*** series.

already been reported by us [12, 13] and others [22, 23] for similar compounds, we here present only spectral and elemental analysis data for representative examples of the newly prepared compounds. As shown in

scheme 1, the two series of **SBOC-*n*** and **SBOF-*n*** compounds were prepared by reacting cholesteryl-5-(4-formylphenoxy)alkanoate (1) with either 4-oxyloxyphenylamine (2) or 4-(1H,1H-perfluorooctyloxy)phenylamine

Figure 1. DSC thermograms of **SBOC-*n*** series.

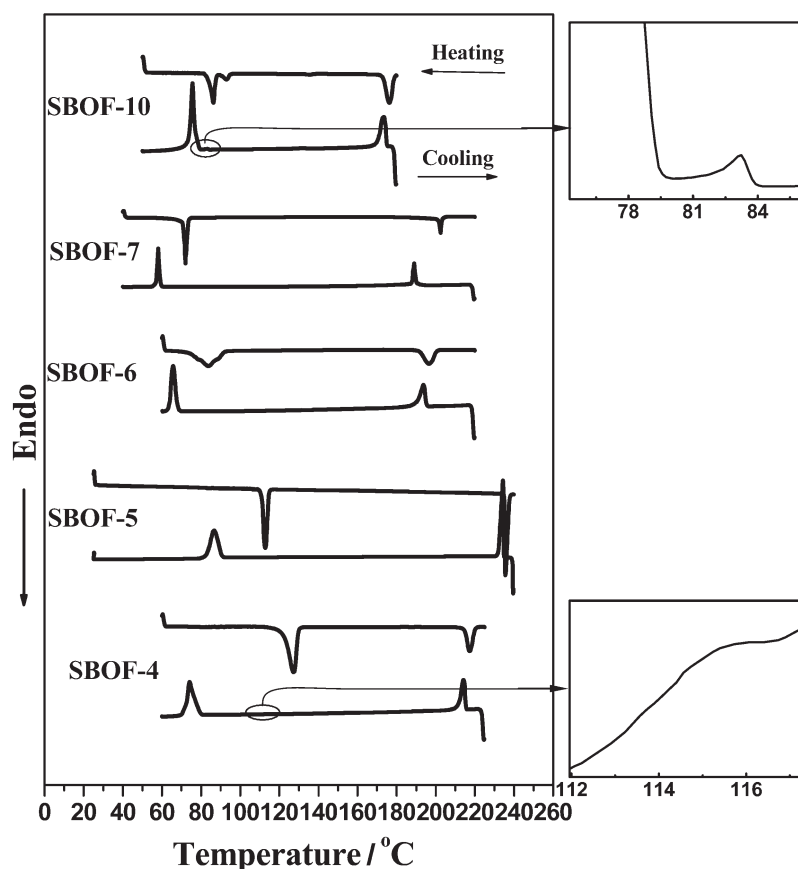


Figure 2. DSC thermograms of SBOF-*n* series.

(3) in ethanol in the presence of activated molecular sieve (4 Å). The synthetic details for the preparation and LC properties of SBOC-5 and SBOF-5 can be found in our previous report [12].

**2.1.1. N-[4-{5-(Cholesteryloxycarbonyl)butyloxy}benzylidene]-4-*n*-octyloxyaniline (SBOC-4).** <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, ppm): 0.5–1.9 (m, 60H, –CH–, –CH<sub>2</sub>–, –CH<sub>3</sub>–), 2.4 (m, 4H, –C=CH–CH<sub>2</sub>– and –OCO–CH<sub>2</sub>–), 4.0 (t, 4H, ArOCH<sub>2</sub>–), 4.6 (m, 1H, –OCH–), 5.4 (m, 1H, –CH=CH–) 6.9–7.8 (m, 8H, –Ar–), 8.4 (s, 1H, –CH=N–). FTIR (KBr, cm<sup>-1</sup>): 2943 and 2868 (C–H stretching), 1728 (C=O stretching), 1626 (C=N stretching), 1601, 1571, 1511, 1464 (C=C stretching), 1250 and 1172 (C–O stretching). Elemental analysis: calcd for C<sub>52</sub>H<sub>78</sub>N<sub>2</sub>O<sub>4</sub>, C 80.15, H 10.03, N 1.76; found, C 80.11, H 10.10, N 1.71%.

**2.1.2. N-[4-{5-(Cholesteryloxycarbonyl)butyloxy}benzylidene]-4-*n*-pentadecafluorooctyloxyaniline (SBOF-4).** <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, ppm): 0.5–1.9 (m, 45H, –CH–, –CH<sub>2</sub>–, –CH<sub>3</sub>–), 2.3 (m, 4H, –C=CH–CH<sub>2</sub>– and –OCO–CH<sub>2</sub>–), 4.0 (t, 2H, –ArOCH<sub>2</sub>–), 4.5 (t, 2H,

–ArOCH<sub>2</sub>CF<sub>2</sub>–), 4.6 (m, 1H, –OCH–), 5.4 (m, 1H, –CH=CH–) 6.9–7.8 (m, 8H, –Ar–), 8.4 (s, 1H, –CH=N–). FTIR (KBr, cm<sup>-1</sup>): 2932, 2865 (C–H stretching), 1725 (C=O stretching), 1624 (C=N stretching), 1604, 1572, 1511, 1468 (C=C stretching), 1250, 1171 (C–O stretching). Elemental analysis: calcd for C<sub>52</sub>H<sub>63</sub>F<sub>15</sub>N<sub>2</sub>O<sub>4</sub>, C 58.64, H 5.96, N 2.63; found, C 58.64, H 5.96, N 2.52%.

## 2.2. Experimental techniques

The IR and <sup>1</sup>H NMR spectra of the intermediates and final compounds were recorded on a Bomem MB FTIR spectrophotometer (Canada) and a Bruker AM 300 spectrometer (Germany), respectively. Thermal transitions of the LC compounds were studied under a nitrogen atmosphere using a Mettler Toledo DSC821e (Switzerland) differential scanning calorimeter. The heating and cooling rates in general were kept at 2 °C min<sup>-1</sup>. Indium was employed as a reference material for the calibration of temperature and enthalpy. The peak maximum or minimum point was taken as the transition temperature. Thermodynamic

Table 1. Transition temperatures ( $^{\circ}\text{C}$ ), enthalpy changes ( $\text{kJ mol}^{-1}$ ) in parentheses, and mesophase range ( $^{\circ}\text{C}$ ), for the phase transitions of **SBOC-*n*** series compounds.

<i>n</i>	Transition		$\Delta T_{i-\text{Cr}}$
4	Cr (35.3) 118.4 SmA SmC* $\xleftarrow{(0.02)}$   101.5	(7.2) 168.4 I	50
5 <sup>a</sup>	Cr (53.5) 130.5 SmA SmC* $\xleftarrow{(0.04)}$   124.1	(1.0) (1.9) 195.5 N* 197.6 I	67.1
6	Cr (59.3) 140.4 SmA	(1.1) (1.8) 145.5 N* 156.9 I	16.5
7	Cr (43.8) 144.8 SmA SmC* $\xleftarrow{(0.01)}$   117.9	(0.9) (6.5) 167.2 N* 179.6 I	34.8
10	Cr (61.7) 122.8 SmA SmX $\xleftarrow{(1.1)}$   125.9	(0.32) (3.0) 127.6 N* 151.5 I	28.7

<sup>a</sup>Data taken from [12].

parameters for the transitions were obtained from the DSC thermograms.

The optical textures of the mesophase formed by the compounds obtained were observed using a polarizing microscope (Olympus BH-2, Japan) equipped with a hot stage (FP-82HT) and an automatic temperature controller (Mettler FP-90, Switzerland). When the

sample formed a homeotropic texture on a regular slide glass, a rubbed polyimide substrate was used to observe the optical texture.

X-ray diffractograms of the compounds were obtained at varying temperatures using the synchrotron radiation ( $1.542 \text{ \AA}$ ) of the 3C2 beam line at the Pohang Synchrotron Laboratory, Pohang, Korea.

Table 2. Transition temperatures ( $^{\circ}\text{C}$ ), enthalpy changes ( $\text{kJ mol}^{-1}$ ) in parentheses, and mesophase range ( $^{\circ}\text{C}$ ) for the phase transitions of **SBOF-*n*** series compounds.

<i>n</i>	Transition		$\Delta T_{i-\text{Cr}}$
4	Cr (22.7) 127.1 SmA SmC* $\xleftarrow{(0.01)}$   112.2	(11.4) 217.0 I	89.9
5 <sup>a</sup>	Cr (21.8) 111.9 SmC*	(0.05) (16.6) 143.7 SmA 235.3 I	123.4
6	Cr (21.1) 83.4 SmA	(11.3) 196.5 I	113.1
7	Cr (13.5) 72.0 SmA	(9.5) 200.3 I	128.3
10	Cr (22.0) 86.4 SmA SmC* $\xleftarrow{(0.05)}$   132.6	(11.9) 176.4 I	90

<sup>a</sup>Data taken from [12].

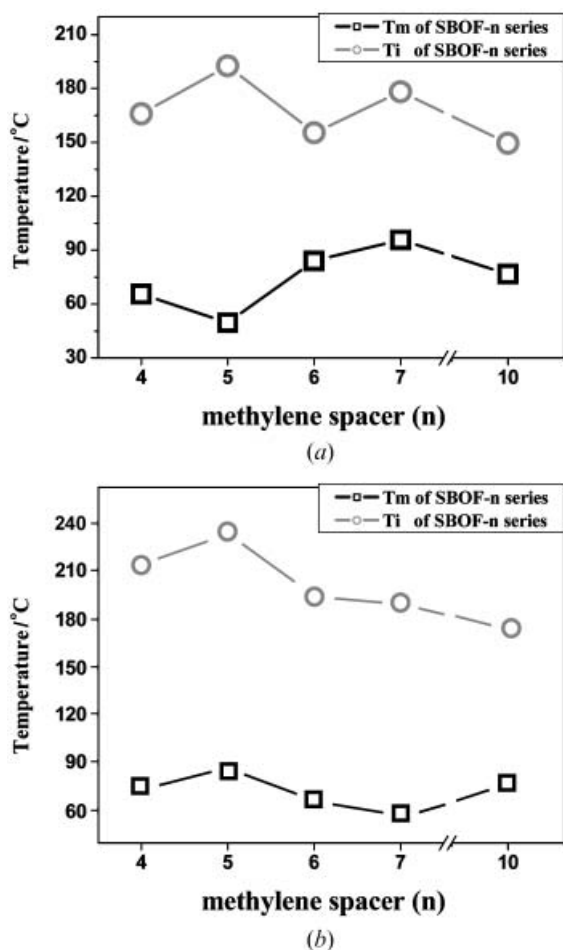


Figure 3. The dependence of  $T_m$  and  $T_i$  on the number of the methylene unit ( $n$ ) in SBOC- $n$  and SBOF- $n$ .

### 3. Results and discussion

#### 3.1. Phase transitions and mesophases

The thermal transitions and mesophases of the compounds were studied by differential scanning calorimetry (DSC) and their optical textures were observed on a polarizing microscope. The nature of the mesophases was also confirmed by small angle X-ray diffractometry (SAXD). Figures 1 and 2 compare the DSC thermograms of the alkoxy tail compounds with those of the perfluoroalkoxy tail compounds. Tables 1 and 2 summarize the data relating to the thermal transitions and the nature of the mesophases of the compounds; the enthalpy changes of the phase transitions are also included. The DSC thermograms show that all the transitions involving the SmA and cholesteric ( $N^*$ ) phases are enantiotropic whereas those involving the SmC\* phase are monotropic. However, we earlier showed that the SBOF-5 compound forms the SmC\* phase enantiotropically [12]. Because the peak intensity for the SmA–SmC\* transitions are so weak, expanded peaks for the transitions are shown in the insets of figures 1 and 2. As for the melting transition temperatures ( $T_m$ ) of the two series, as shown in figures 3(a) and 3(b), the melting points (118–145°C) of the SBOC- $n$  series are in general much higher than those (72–127°C) of the SBOF- $n$  series. No regular dependence of  $T_m$  on the length ( $n$ ) and parity of the spacer is apparent. However, the isotropic temperatures ( $T_i$ ) show a distinctive, so-called, odd–even dependence on the number of methylene groups in the central spacer, although data points for  $n=8$  and  $9$  are missing. In both

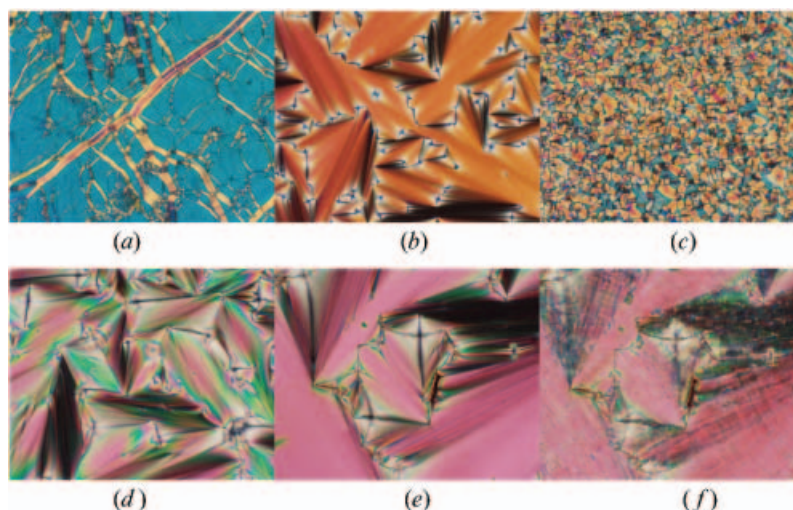


Figure 4. Optical textures of (a) SBOC-10 taken at 148.7°C on heating, (b) SBOC-7 taken at 163.2°C on cooling, (c) SBOC-10 taken at 92.4°C on cooling, (d) SBOF-10 taken at 171.3°C on heating, (e) SBOF-4 taken at 207.8°C on cooling, and (f) SBOF-4 taken at 113.5°C on cooling [magnification 400X].

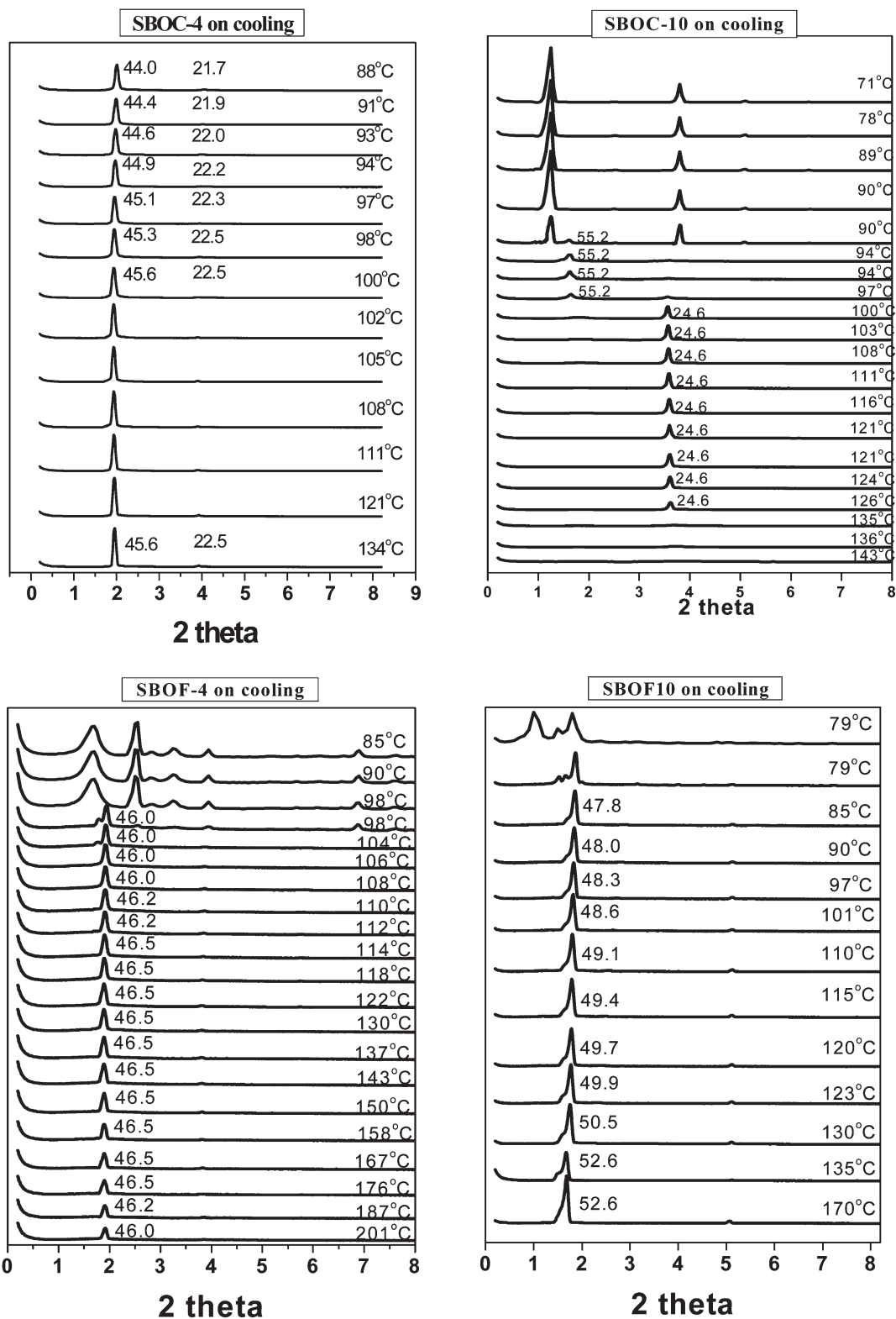


Figure 5. Small angle X-ray diffractograms of SBOC-4, SBOC-10, SBOF-4 and SBOF-10.

Table 3. Layer spacing in the SmA and SmC\* phases of the **SBOC-*n*** and **SBOF-*n*** series.

Compound	Layer spacing/Å in SmA phase; (half layer spacing)	Layer spacing/Å in SmC or SmC* phase (tilt angle/°)	Estimated molecular length/Å
<b>SBOC-4</b>	45.6	44.0 (15.2)	46.9
<b>SBOC-5<sup>a</sup></b>	46.9	42.5 (14)	47.5
<b>SBOC-6</b>	48.3	—	49.0
<b>SBOC-7</b>	50.2	48.0 (17.0)	50.1
<b>SBOC-10</b>	55.2 (24.6)	—	53.8
<b>SBOF-4</b>	46.5	46.0 (8.4)	47.7
<b>SBOF-5</b>	49.3	46.8 (17)	47.9
<b>SBOF-6</b>	49.1 (24.5)	—	49.1
<b>SBOF-7</b>	50.5 (25.2)	—	51.1
<b>SBOF-10</b>	52.6	47.8 (24.7)	54.1

<sup>a</sup>Data taken from [12].

series the  $T_i$  values for compounds with the same spacer parity decreased with increasing  $n$ . Similar behaviour has been observed in other series earlier [13]. Various molecular theories [24] have been proposed to explain the alternation of the transition properties with the number of the methylene groups in the central spacers of liquid crystalline dimers.

The heats of melting of the **SBOC-*n*** series are significantly higher than those of the other series. Although there is no information on the crystal structures of the compounds, it is conjectured that a better molecular packing is achieved in the former than in the latter. All the compounds revealed a much higher degree of supercooling prior to crystallization, that is, in the transition from the LC phase to crystalline solid state, than in other phase transitions involving LC phases. This phenomenon is very common in the phase transitions of LC compounds, and is due to the fact that crystallization requires more extensive molecular reorganization than other phase transitions involving only mesophases and the isotropic phase.

The alkoxy tail compounds are able to form SmC\*, SmA, and cholesteric (N\*) phases. In contrast, the number of mesophases formed by the fluoroalkoxy tail compounds is reduced. They exhibited focal-conic fan textures, figures 4(b), (d) and (e), for SmA phases and the oily streak texture, Figure 4(a), for the cholesteric phase. The focal-conic fan textures of the SmC\* phase clearly showed equally spaced retardation lines, figure 4(f). The nature of the SmX phase formed by **SBOC-10** should be clarified further. The optical texture of the SmX phase is given in figure 4(c).

The examination of data given in tables 1 and 2 reveals in part the influence of the perfluoroalkoxy tail. (1) The perfluoroalkoxy tail expands the mesophase temperature range much more efficiently than the alkoxy tail. For example, the total mesophase

temperature range of **SBOF-4** is 90°C, which is much greater than that of **SBOC-4** (50°C). This is caused by the significant increase in the  $T_i$  values of the fluorinated series. Lowered melting temperature, with the sole exception of **SBOF-4**, also contributes to the widening of the mesophase temperature range. (2) The perfluoroalkoxy tails effectively suppress the formation of the cholesteric phase by the **SBOF-*n*** series. It is probable that, due to their incompatibility with the hydrogenated part of the molecules and strong affinity among themselves, the perfluoroalkyl tail favours microscopically segregated morphology that promotes the formation of the layered smectic (SmA) phase. The perfluoroalkoxy tail also tends to prevent the formation of the SmC\* phase as we earlier observed for another series of compounds [12, 13]. However, it is intriguing to note that the **SBOF** compounds with  $n=4, 5$  and 10 form the SmC\* phase and **SBOF-5** is unique in that it forms the SmC\* phase enantiotropically. Thus, the introduction of fluorine atoms into the terminal alkoxy chain increases the thermal stability of the mesophases of the resulting compounds. This observation may be attributed to the perfluorinated alkyl chain having a higher tendency to reside in the all-*trans*-conformation [13, 17] when compared with hydrogenated alkyl groups.

### 3.2. Mesophase structures by X-ray diffraction

The nature of the mesophases formed by the present compounds was determined by polarizing microscopy and X-ray diffraction. In addition, the LC behaviour of **SBOC-5** and **SBOF-5** reported earlier in detail by us provides a reliable basis for the identification of mesophases exhibited by other members of the present series [12].

The smectic phase of the present compounds all exhibited Bragg diffraction peaks (figure 5) in the small



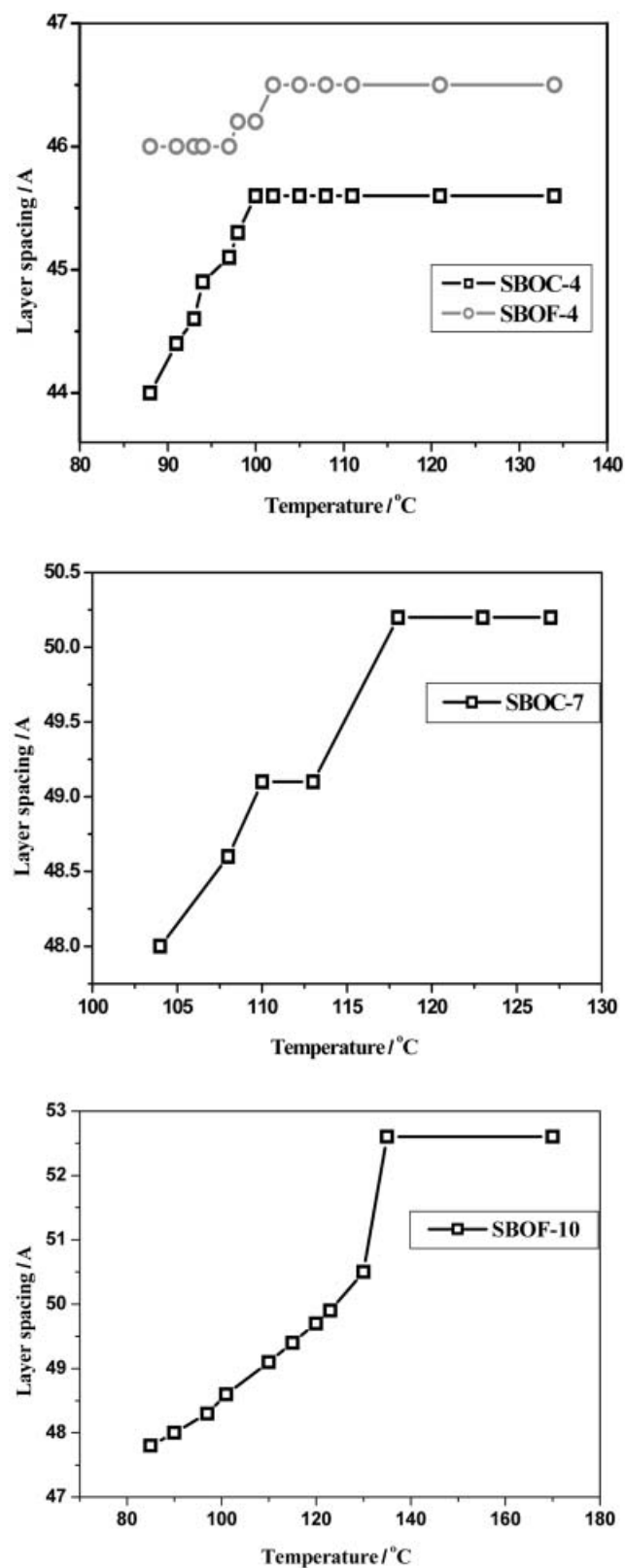


Figure 6. Temperature dependence of layer spacing of SBOC-4, SBOC-7, SBOF-4, and SBOF-10.

angle region, corresponding to the long spacing or layer reflections. Figure 6 shows the dependence of layer spacing on temperature for some of the present compounds in the smectic phases. Table 3 lists the layer spacing of the smectic phases estimated from the small angle X-ray diffractograms. Their molecular lengths were calculated using CS Chem 3D Pro (Cambridge Soft Co., USA) assuming an all-*trans*-conformation for all the CH<sub>2</sub> and CF<sub>2</sub> carbons in the central spacer and in the terminal chain.

According to the data given in table 3 the experimentally determined layer spacing values are very similar to or only slightly different from the calculated values, which indicates that the conformation around the carbon atoms in the spacers and tails is basically all-*trans*. Figure 6 shows how abruptly the layer spacing of the SmA phases of SBOC-4, SBOC-7, SBOF-4 and SBOF-10 decreases from 45.6 to 44.0 Å, from 50.2 to 48.0 Å, from 46.5 to 46.0 Å, and from 52.6 to 47.8 Å at the transition points, respectively. All of them undergo a phase transition from the SmA to SmC\*. The tilt angles of the SmC\* phases are estimated to be about 15.2°, 17.0°, 8.4° and 24.7°, respectively. Although the available data are limited, it appears that the longer spacer length increases the tilt angle in the SmC\* phase.

We earlier reported the LC properties of a similar series of compounds where the second mesogenic group was azobenzene-based in place of the Schiff base in the present series [13]. The main difference between the two series to be noted is that the present Schiff base series have a greater tendency to form the SmC\* phase. It is generally known [25, 26] that the Schiff base LC compounds display more abundant polymorphism than the azobenzene-based LC compounds. Another characteristic behaviour of the present and earlier series [13] bearing long fluorinated alkyl tails is that they both form only the SmA phase. This strongly indicates that strong interactions among the perfluoroalkyl group overwhelm the influence of linking groups, azo vs. imino group, in the mesogenic units.

#### 4. Conclusion

We have prepared two new series of non-symmetric dimesogens compounds containing cholesterol and Schiff base-type mesogens interconnected by  $\omega$ -oxyalkanoyl spacers of varying length. The first series (SBOC-*n*) contain the octyloxy tail on the Schiff base mesogen, while the other series (SBOF-*n*) have the perfluoro-octyloxy tail. The two series show differing mesomorphic behaviour. The SBOC-*n* series show SmA, SmC\*, and N\* phases depending on the length of central spacer, with the one exception of SBOC-4. In contrast, the SBOF-*n* series exhibit only the SmA and SmC\* phases,

suppressing the formation of the cholesteric (N\*) phase, with the exception of **SBOF-6** and **SBOF-7**. The perfluoroalkoxy tail also prevents the formation of the SmC\* phase by compounds having the medium length of central spacer. These observations are attributed to a strong incompatibility of the perfluorinated tails with hydrogenated chains. The perfluoroalkyl tail also tends to improve the thermal stability of the mesophase, increasing the mesophase temperature range.

#### Acknowledgements

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