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## Liquid Crystals

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# TGB<sub>A</sub> phase in a series of swallowtailed chiral liquid crystals

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A homologous series of chiral materials, 1-butylpentyl (S)-2-{6-[4-(4'-alkoxyphenyl)benzoyloxy]-2-naphthyl}propionates, BP<sub>m</sub>PB<sub>m</sub>NP (*m* = 6-16), derived from a chiral moiety, (S)-2-(6-hydroxy-2-naphthyl)propionic acid, in conjunction with a swallowtail group attached to the external side of the chiral centre, has been synthesized and their mesomorphic phases have been studied. There were two frustrated phases: a blue (BP) phase and a twist-grain-boundary (TGB<sub>A</sub>) phase found enantiotropically in all materials. It is worth noting that, depending on the nature of the terminal chain length, two types of filament textures were observed in the TGB<sub>A</sub> phase: a *vermis* (filament) texture and a spiral filament texture. The formation of the TGB<sub>A</sub> phase was rationalized as due to the steric effect caused by the swallowtail of the materials. All materials exhibited a wide temperature range of the SmA phase, but only three materials (*m* = 12, 13 and 16) possessed a monotropic SmC\* phase over a narrow temperature range, indicating that the swallowtailed materials inhibit the formation of a tilted smectic phase.

## 1. Introduction

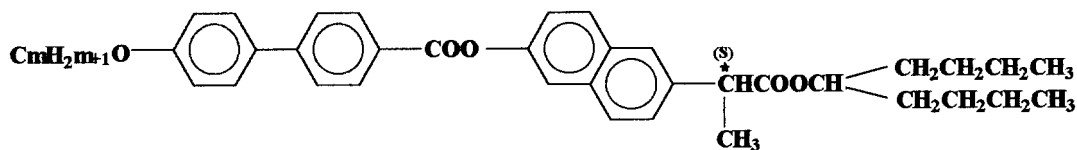
The theoretical predictions [1-3] and discoveries [4-7] of twist grain boundary phases in chiral liquid crystalline systems, have stimulated a great interest in the search for new types of these materials. In the past few years, many homologous compounds [4-14], based on the first TGB<sub>A</sub> series of chiral materials, 1-methylheptyl 4'-[(4-alkoxyphenyl)propionoyloxy]biphenyl-4-carboxylates [4], have been designed, synthesized and investigated. Accordingly, the correlation of molecular structure to the appearance of TGB phases has been primarily established and reviewed [15].

In this paper, we report a new series of chiral materials derived from a chiral moiety, (S)-2-(6-hydroxy-2-naphthyl)propionic acid, in conjunction with a swallowtail group attached to the external side of the chiral centre, which exhibit the TGB<sub>A</sub> phase. This series, 1-butylpentyl (S)-2-{6-[4-(4'-alkoxyphenyl)benzoyloxy]-2-naphthyl}-propionates, BP<sub>m</sub>PB<sub>m</sub>NP (*m* = 6-16), has the general structure:

## 2. Experimental

### 2.1. Characterization of materials

The chemical structures of the target materials were identified by proton nuclear magnetic resonance (<sup>1</sup>H NMR) spectroscopy using a JEOL EX-400 FT-NMR spectrometer. The purity of the final compounds was estimated by thin layer chromatography (TLC), and further confirmed by elemental analysis using a Perkin-Elmer 2400 instrument. Analytical data showed that errors in the measured carbon and hydrogen contents of the target materials were less than 1% as compared with calculated results. The magnitudes of specific rotations were measured in dichloromethane using a JASCO DIP-360 digital polarimeter. Transition temperatures and enthalpies of the transitions were determined by differential scanning calorimetry (DSC) using a Perkin-Elmer DSC 7 calorimeter. Mesophases were identified by observation of the textures using a Nikon Microphot-FAX optical microscope under crossed polarizers with a Mettler FP82-HT hot stage connected



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to a Mettler FP80-HT heat controller. Sample cells with homogeneously aligned polyimide film coated onto ITO-deposited glass plates were purchased from the E.H.C. Co., Japan. The optical purity of intermediates and products was not measured at each synthetic step. However, since esterification using *N,N'*-dicyclo-carbodiimide (DCC) and 4-dimethyl-aminopyridine (DMAP) [16, 17], and demethylation using tribromoborane ( $\text{BBr}_3$ ) [18] have been reported to be free from racemization, target materials possessing high optical purity could be expected.

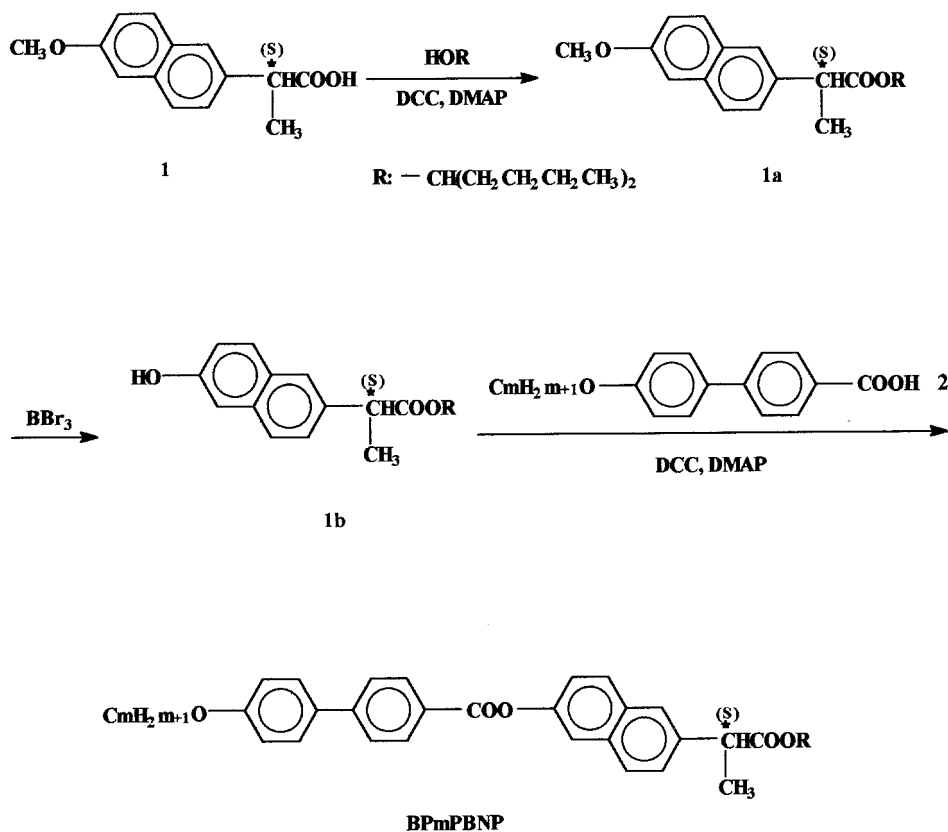
## 2.2. Preparation of materials

The chiral starting material for the synthesis of the compounds BPmPBNP was (*S*)-2-(6-methoxy-2-naphthyl)-propionic acid, purchased from Tokyo Chemical Industry (TCI) Co. Ltd, Japan, with optical purity greater than 99% enantiomeric excess. The synthetic procedures were carried out in the same manner as described before [19] and are shown in the scheme. The acid **1** was esterified with 5-nonanol in the presence of DCC and DMAP to produce the ester, 1-butylpentyl (*S*)-2-(6-methoxy-2-naphthyl)propionate **1a**. The methoxy group of this ester was demethylated by treatment with  $\text{BBr}_3$ , and the resulting hydroxy group of 1-butylpentyl

(*S*)-2-(6-hydroxy-2-naphthyl)propionate **1b** was subsequently esterified with a variety of 4-(4-alkoxyphenyl)-benzoic acids **2** ( $m=6-16$ ) using DCC and DMAP, to produce the target compounds BPmPBNP. The synthetic details for these materials are described below.

### 2.2.1. 1-Butylpentyl (*S*)-2-(6-methoxy-2-naphthyl)-propionate, **1a**

The acid **1** (25 mmol) and 5-nonanol (27.5 mmol) were dissolved in dry dichloromethane (100 ml). After the addition of DCC (27.5 mmol) and DMAP (2.5 mmol) the solution was stirred at room temperature for five days. The precipitates were filtered off and washed with dichloromethane. The filtrate was successively washed with 5% acetic acid, 5% aqueous sodium hydroxide and water, and then dried over anhydrous magnesium sulphate and concentrated under vacuum. The residue was purified by column chromatography on silica gel (70–230 mesh) using dichloromethane as eluent. The isolated ester in 89–97% yield was identified by its  $^1\text{H}$  NMR spectrum and used directly for the ensuing reaction without further purification.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 0.6–1.6 (m, 18H), 1.55 (d, 3H), 3.8–3.9 (q, 1H), 3.9 (s, 3H), 4.8–4.9 (m, 1H), 7.1–7.7 (m, 6H).



Scheme. Procedures for the synthesis of compounds 1-butylpentyl (*S*)-2-[6-[4-(4'-alkoxyphenyl)benzoyloxy]-2-naphthyl]propionates.

### 2.2.2. 1-Butylpentyl (S)-2-(6-hydroxy-2-naphthyl)-propionate, **1b**

The ester **1a** (22.5 mmol) dissolved in dry dichloromethane (82.4 ml) was mixed with tribromoborane (4.12 ml) at  $-20^{\circ}\text{C}$ . The mixture was stirred at  $-20^{\circ}\text{C}$  for 5 min, and at  $0^{\circ}\text{C}$  for 50 min. After diluting with dichloromethane (164.8 ml), the solution was poured into a mixture of saturated aqueous ammonium chloride (82.4 ml) and ice chips (82.4 g). The organic layer was separated and washed with brine ice, dried over anhydrous sodium sulphate, and concentrated under vacuum. The residue was purified by silica gel (70–230 mesh) column chromatography using dichloromethane as eluent. The alcohol **1b** in 55.14% yield was collected after recrystallization from hexane. Elemental analysis: (calculated) C 77.16, H 8.83; (found) C 77.22, H 8.78%.  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 0.6–1.7 (m, 18H), 1.6 (d, 3H), 3.8–3.9 (q, 1H), 4.8–4.9 (m, 1H), 7.0–7.7 (m, 6H). Specific rotation,  $[\alpha]_{\text{D}}^{26} = +10.32$  (c 0.601 g 100 ml $^{-1}$ ).

### 2.2.3. 1-Butylpentyl (S)-2-{6-[4-(4'-alkoxyphenyl)-benzyloxy]-2-naphthyl}propionates, BPmPBNP

The esters BPmPBNP ( $m = 6-16$ ) were synthesized in the same manner as described for esters **1a**. A mixture of 4-(4'-alkoxyphenyl)benzoic acids (1.15 mmol), alcohol **1b** (1.05 mmol), DCC (1.26 ml), DMAP (0.105 mmol) and dry tetrahydrofuran (3 ml) was stirred at room temperature for five days. After purification, 80–90% yields of products were obtained. All materials were analysed and identified satisfactorily. A typical example of analytical data from BP10PBNP is given as follows. Elemental analysis: (calculated) C 79.61, H 8.61; (found) C 79.62, H 8.67%;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  (ppm) 0.6–1.8 (m, 37H), 1.6 (d, 3H), 3.8–3.9 (q, 1H), 4.0–4.1 (t, 2H), 4.8–4.9 (m, 1H), 7.0–8.3 (m, 14H). Specific rotation,  $[\alpha]_{\text{D}}^{26} = +6.62$  (c 0.601 g 100 ml $^{-1}$ ).

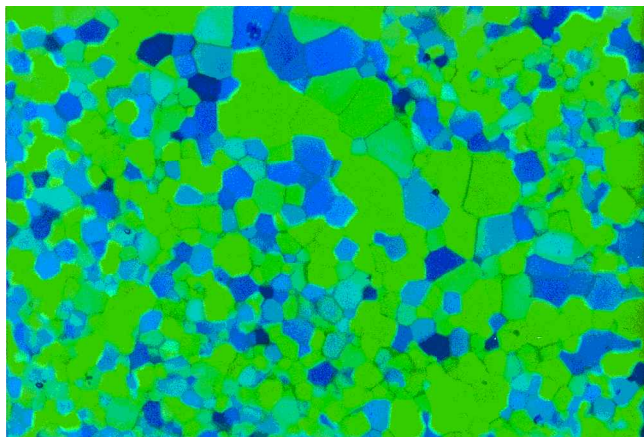


Figure 1. The iridescent platelet texture of the blue phase obtained from BP13PBNP under the crossed polarizing microscope at  $131.3^{\circ}\text{C}$  (magnification  $\times 400$ ).

## 3. Results

Mesophases and their corresponding phase transition temperatures were principally determined by the use of thermal optical microscopy in conjunction with DSC calorimetry. Compounds BPmPBNP ( $m = 6-16$ ), layered between two untreated glass plates, were investigated photomicrographically at a heating and cooling rate of  $2^{\circ}\text{C min}^{-1}$ . On cooling from the isotropic liquid (I), the blue phase (BP) was characterized by the formation of an iridescent platelet defect texture, as shown in figure 1. The formation of a scale-like or fan-like texture demonstrated the existence of a chiral nematic ( $\text{N}^*$ ) phase. The TGB<sub>A</sub> phase situated between  $\text{N}^*$  and  $\text{S}_\text{A}$  phases was identified by the appearance of filaments. It is worth noting that, depending on the nature of the terminal chain length  $m$ , two different types of filament textures were observed. A *vermis* (filament) texture [4, 7, 8] detected for  $m = 6-9, 12$  and  $14-16$  is shown in figure 2, and a spiral filament texture [20, 21] for  $m = 10, 11$  and

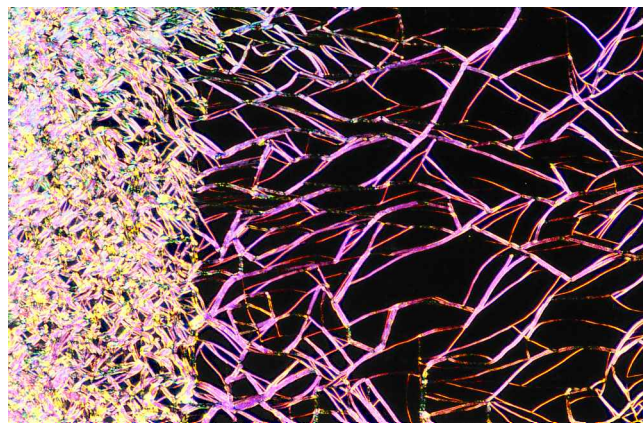


Figure 2. The *vermis* (filament) texture of the TGB<sub>A</sub> phase obtained from BP9PBNP under the crossed polarizing microscope at  $121.7^{\circ}\text{C}$  (magnification  $\times 400$ ).

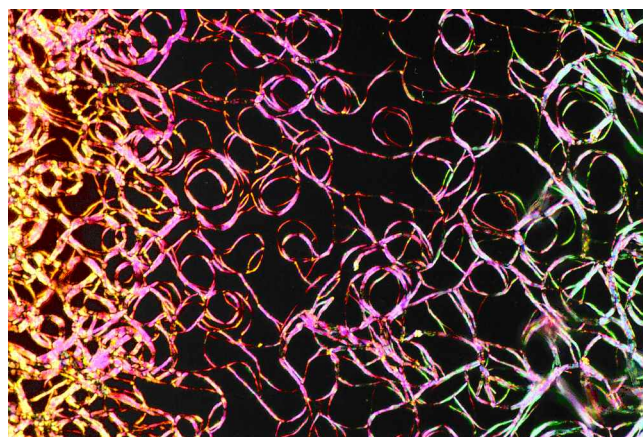


Figure 3. The spiral filament texture of the TGB<sub>A</sub> phase obtained from the BP13PBNP under the crossed polarizing microscope at  $120.2^{\circ}\text{C}$  (magnification  $\times 400$ ).

13 in figure 3. The SmA phase formed as a homeotropic texture in conjunction with the focal-conic fan texture. On cooling from the SmA phase, the ensuing broken focal-conic and schlieren texture displayed the formation of the SmC\* phase. The results indicated that all compounds exhibit enantiotropic BP, N\*, TGB<sub>A</sub> and SmA phases. Three compounds, having the longer terminal chain length ( $m = 12, 13$  and  $16$ ), also exhibited the monotropic SmC\* phase. The resulting mesophases and their corresponding phase transition temperatures measured by microscopic observations are summarized in the table. A phase diagram as a function of terminal chain length is plotted in figure 4. It can be seen that the thermal stability of the N\* phase is greater at  $m = 6-9$ , and gradually decreases as  $m$  ascends. The thermal stability of the TGB<sub>A</sub> phase, however, appears to be wider at  $m = 10$  and  $11$ , and gradually decreases as  $m$  descends from  $m = 10$ , or ascends from  $m = 11$ , indicating that a moderate terminal chain length favours formation of the TGB<sub>A</sub> phase. The thermal stability of the BP phase displays no significant trend to the variation of  $m$ . It also has no clear correlation of thermal stability between BP and TGB<sub>A</sub> phases. The SmA phase possesses a wide temperature range, but the SmC\* phase appears only in a narrow temperature range, suggesting

that swallowtailed materials suppress the formation of a tilted smectic phase.

DSC studies were carried out for further investigation of mesophase transitions at a heating and cooling rate of  $1^\circ\text{C min}^{-1}$ . Representative DSC thermograms obtained for compound BP10PBNP are depicted in figure 5. It is seen in figure 5(a) that, as the temperature is increased, the phase transition of SmA–TGB<sub>A</sub> occurs at  $116.5^\circ\text{C}$  and that of TGB<sub>A</sub>–N\* at  $120.8^\circ\text{C}$ . The peak of the TGB<sub>A</sub>–N\* transition is broad and overlaps that of the SmA–TGB<sub>A</sub> transition. The N\*–BP transition occurs at  $137.5^\circ\text{C}$  and BP–I transition at  $140.2^\circ\text{C}$ . The N\*–BP transition peak is rather small and situated in the shoulder of the N\*–I transition peak. A similar trend of the phase transitions is also seen in the cooling trace of the DSC thermogram, as shown in figure 5(b), indicating again that the N\*, TGB<sub>A</sub>, SmA and SmC\* phases are enantiotropic.

The enthalpies of the transitions measured by DSC calorimetry are also summarized in the table. For the overlapped peaks, the enthalpies are summed and listed in the table. For example, the enthalpies of I–BP and BP–N\* transitions are added together and placed under BP phase in the table. The enthalpy of SmA–SmC\* transitions was too small to be measured.

Table. Transition temperatures and enthalpies  $\Delta H$  (in italics) of chiral compounds BP<sub>m</sub>PBNP. Cr = crystal, m.p. = melting point, [ ] denotes a monotropic phase transition.

m	T / °C and $\Delta H$ / kJ mol <sup>-1</sup> (in italics)													
	I	BP		N*	TGB <sub>A</sub>		SmA	SmC*	Cr	m.p.				
6	•	156.1	•	153.2	•	135.8	•	135.7	•	—	83.2	•	105.5	
			<i>0.07<sup>a</sup></i>				<i>0.68<sup>b</sup></i>				<i>17.19</i>	•	<i>21.91</i>	
7	•	150.2	•	146.6	•	128.9	•	128.8	•	—	81.2	•	103.5	
			<i>0.36<sup>a</sup></i>				<i>0.43<sup>b</sup></i>				<i>27.26</i>	•	<i>30.97</i>	
8	•	145.5	•	144.5	•	124.9	•	124.2	•	—	76.7	•	90.8	
			<i>0.33<sup>a</sup></i>				<i>0.25<sup>b</sup></i>				<i>17.25</i>	•	<i>19.81</i>	
9	•	140.1	•	139.8	•	121.0	•	120.1	•	—	73.9	•	88.7	
			<i>0.35<sup>a</sup></i>				<i>0.62<sup>b</sup></i>				<i>25.60</i>	•	<i>27.84</i>	
10	•	140.0	•	136.8	•	121.3	•	117.5	•	—	76.5	•	87.7	
			<i>0.84<sup>a</sup></i>				<i>0.63<sup>b</sup></i>				<i>19.58</i>	•	<i>20.14</i>	
11	•	136.8	•	134.3	•	123.8	•	120.7	•	—	72.6	•	86.5	
			<i>0.72<sup>a</sup></i>				<i>0.69<sup>b</sup></i>				<i>33.12</i>	•	<i>35.38</i>	
12	•	132.2	•	128.7	•	117.5	•	115.4	•	[75.0] <sub>c</sub>	•	69.5	•	85.7
			<i>0.54<sup>a</sup></i>				<i>0.51<sup>b</sup></i>				<i>39.66</i>	•	<i>43.46</i>	
13	•	130.6	•	126.3	•	121.4	•	120.1	•	[76.5] <sub>c</sub>	•	68.4	•	77.5
			<i>1.25<sup>a</sup></i>				<i>0.48<sup>b</sup></i>				<i>35.26</i>	•	<i>36.40</i>	
14	•	113.6	•	110.5	•	107.6	•	107.0	•	—	67.4	•	73.4	
			<i>0.68<sup>a</sup></i>				<i>0.09<sup>b</sup></i>				<i>34.25</i>	•	<i>35.10</i>	
15	•	113.5	•	109.4	•	106.3	•	106.1	•	—	67.1	•	73.0	
			<i>0.52<sup>a</sup></i>				<i>0.32<sup>b</sup></i>				<i>34.68</i>	•	<i>35.30</i>	
16	•	116.2	•	115.4	•	111.9	•	111.8	•	[73.4] <sub>c</sub>	•	67.6	•	78.7
			<i>0.31<sup>a</sup></i>				<i>0.25<sup>b</sup></i>				<i>35.94</i>	•	<i>38.58</i>	

<sup>a</sup> The sum of two transition enthalpies: I–BP and BP–N\*.

<sup>b</sup> The sum of two transition enthalpies: N\*–TGB<sub>A</sub> and TGB<sub>A</sub>–SmA.

<sup>c</sup> The enthalpy was too small to be measured.

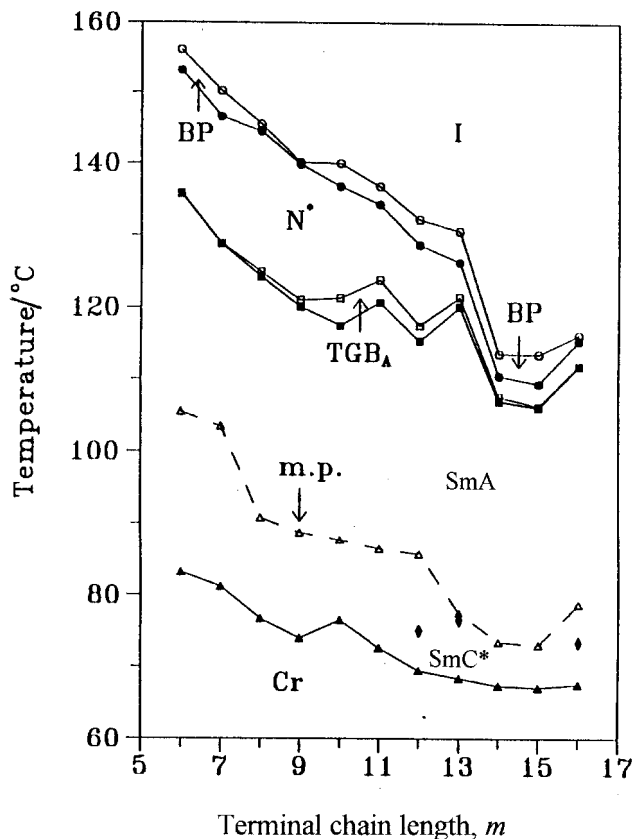


Figure 4. Transition temperature versus terminal chain length for compounds BP<sub>m</sub>PBNP. ○, I-BP; ●, BP-N\*; □, N\*-TGB<sub>A</sub>; ■, TGB<sub>A</sub>-SmA; ◆, SmA-SmC\*; ▲, SmA-Cr or SmC\*-Cr, △; m.p.

#### 4. Discussion

It has been pointed out that the chirality of a molecule has a profound effect on the formation of mesophases in chiral liquid crystals, especially the frustrated phases [15]. This series of materials exhibit BP and TGB<sub>A</sub> phases indicating that they possess high chirality. This can be presumably expected from the molecular structure, in that the chiral centre is attached at the nearest vicinity to the rigid core. However, further investigation in a series of homologous compounds, 1-pentyl (S)-2-{6-[4-(4'-alkoxyphenyl)benzoyloxy]-2-naphthyl}propionates, P<sub>m</sub>PBNP, where a straight terminal chain, *n*-pentyl group, is attached to the external side of the chiral centre, showed that neither BP nor TGB<sub>A</sub> phases may exist.

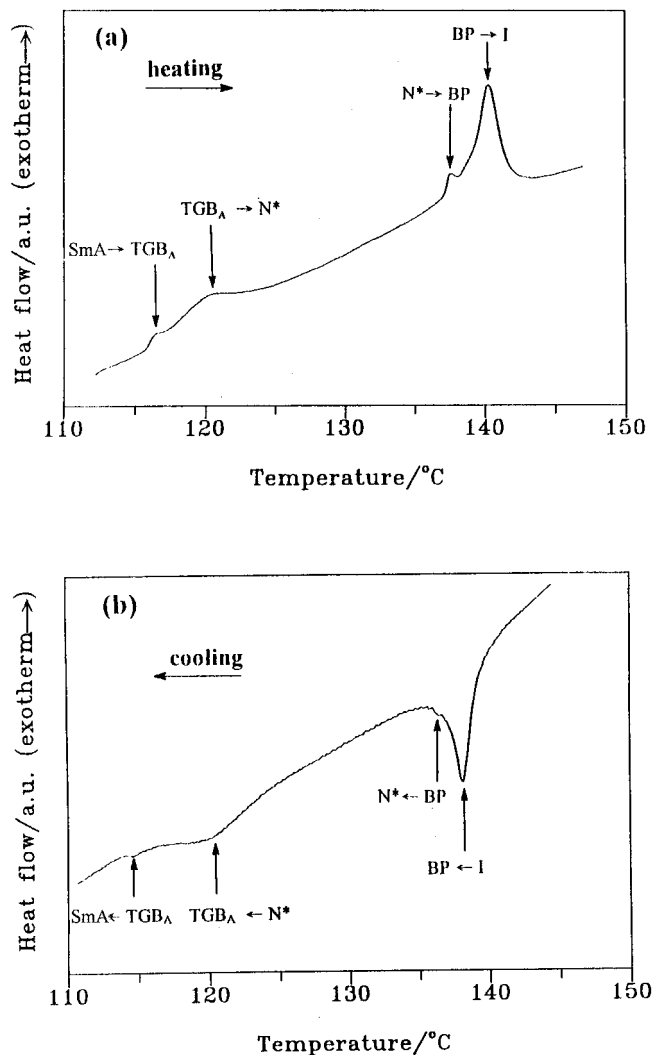
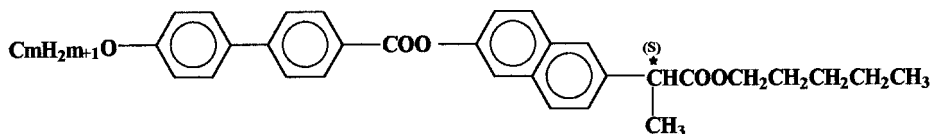


Figure 5. DSC thermogram for BP10PBNP: (a) on heating, (b) on cooling.

For example, compound P<sub>m</sub>PBNP ( $m = 10$ ) has the mesophases and corresponding phase transition temperatures on cooling as follows:

$$I \ 139.8 \ N^* \ 138.8 \ SmA \ 114.8 \ SmC^* \ 77.7 \ Cr \ (^{\circ}C)$$

This phenomenon strongly suggests that the chirality might not be the major cause of generating frustrated phases in swallowtailed materials. An alternative explanation has been proposed [8, 13, 15] that the formation of the TGB<sub>A</sub> phase requires a weakening of layer ordering in the smectic A phase. It is seen that the



bulky swallowtail in a molecule may affect the ease of smectic A layer packing. This behaviour can be confirmed to some extent by the disappearance of tilted SmC\* phase for the compound BP10PBNP as compared with the compound P10PBNP. Thus, the incidence and stability of TGB<sub>A</sub> phases in BP<sub>m</sub>PBNP materials can be rationalized as due to the steric repulsive interactions of the molecules arising from the swallowtail, so as to hinder the smectic A\* layer packing.

Recent studies show that certain achiral swallowtailed materials may display an 'antiferroelectric-like' smectic phase, the so called SmC<sub>alt</sub> phase [22, 23]. Therefore, compounds with  $m = 12, 13$  and  $16$ , which possess SmC\* phases have been studied for the possibility of an existing antiferroelectric SmC<sub>A</sub>\* phase. These compounds were filled separately into antiparallel liquid crystal cells with  $2\mu\text{m}$  cell thickness and investigated photomicrographically. It was unfortunate that the liquid crystals crystallized in the cell as the temperature cooled toward the SmC\* phase temperature.

### 5. Conclusions

A new series of chiral swallowtailed materials, BP<sub>m</sub>PBNP ( $m = 6-16$ ), has been demonstrated as possessing two frustrated phases: BP and TGB<sub>A</sub> phases. The appearance of the TGB<sub>A</sub> phase was rationalized to be the contribution of a steric effect by the bulk swallowtail attached to the external side of the chiral centre. Moreover, these swallowtailed materials favour the formation of SmA phases but suppress the formation of SmC\* phases.

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