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# Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information: http://www.tandfonline.com/loi/gmcl20

## Structure-Property Relationships in Non-Chiral Liquid Crystal Oligomers Stabilizing Blue Phases

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To cite this article: Hirotoshi Iwamochi & Atsushi Yoshizawa (2009) Structure-Property Relationships in Non-Chiral Liquid Crystal Oligomers Stabilizing Blue Phases, Molecular Crystals and Liquid Crystals, 509:1, 223/[965]-232/[974], DOI: 10.1080/15421400903065697

To link to this article: http://dx.doi.org/10.1080/15421400903065697

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#### Structure-Property Relationships in Non-Chiral Liquid Crystal Oligomers Stabilizing Blue Phases

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We prepared 4-(4-cyanophenyl)phenyl 4-octyloxybenzoate derivatives possessing a laterally attached mesogen and investigated their phase transition behavior. They showed a wide temperature range of a nematic phase in spite of being far from a rod-like structure. Each oligomer was found to induce blue phases in the mixture with a conventional chiral smectic liquid crystal. A binary mixture of 4-(4-cyanophenyl)phenyl 4-octyloxy-2-/11-/4-[2-(4-hexyloxyphenypyrimidin-5-yl)phenyloxy]decanoyloxy/benzoate and the chiral compound exhibited a blue phase III with the temperature range of 21 K on cooling.

Keywords: blue phase; chirality; liquid crystal; molecular biaxility; oligomer

#### 1. INTRODUCTION

Blue phases are of particular interest because they have a fluid lattice whose structure is stabilized by lattice defects. Appearance of blue phases results from the competition between the chiral twisting force and the desire for molecules to pack in ways such that they fill space uniformly. Blue phases are believed to consist of a double twist cylinder, so are classified into 3 categories depending on packing structure of double twist cylinders, i.e., blue phase I (BPI), blue phase II (BPII) and blue phase III (BPIII) [1–4]. The packing structure of BPI is body-centered cubic structure; that of BPII is simple cubic structure [5,6]. On the other hand, theoretical insights have revealed that BPIII and the isotropic phase have the same symmetry [7]. It can

This work was partly supported by a Grant-in-Aid for Scientific Research from the Japan Society for the Promotion of Science (No. 19550175).

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be expected that BPIII consists of double twist cylinders with arbitrary orientation [5,8,9]. Usually, blue phases are found in a very narrow temperature range ( $\sim 1$  K) between the isotropic liquid (Iso) and the chiral nematic (N<sup>\*</sup>) phase of sufficiently short pitch. Blue phases have the potential of applications as fast light modulators or tunable photonic crystals, but their narrow temperature range is a critical problem. Therefore, stabilizing the blue phases has attracted much attention [10–15].

We reported that T-shaped chiral oligomers possessing molecular biaxiality exhibit BPIII with a wide temperature range on cooling [16,17]. Then, we observed an electric-field induced phase transition between BPIII and N of a chiral T-shaped compound [18]. However, the temperature range of BPIII is still narrow. We have designed a host nematic liquid crystal for developing practical blue phase materials. Recently, we prepared a new liquid crystal oligomer in which a mesogenic moiety possessing a terminal cyano group and a mesogenic moiety possessing lateral fluorine atomes are connected via a flexible spacer as shown in Figure 1 [19]. The compound induced blue phases in the mixture with a chiral smectic liquid crystal.

For the present study, we prepared three modifications of the liquid crystal oligomer, and investigated phase transition behavior of a binary mixture of each liquid crystal oligomer and a chiral compound. We discuss the role of a liquid crystal oligomer in stabilizing blue phases.



FIGURE 1 Molecular structure of non-chiral liquid crystal oligomer I-6.

#### 2. EXPERIMENTAL

#### 2.1 Preparation of Materials

4-(4-Hexylphenyl)-1-(4-hydroxyphenyl)-2,3-difluorobenzene, 5-(4-hydroxy)phenyl-2-(2-fluoro-6-hexyloxyphenyl)pyrimidine and 5-(4-hydroxy)phenyl-2-(6-hexylphenyl)pyrim-idine were obtained from Midori Kagaku Co., Ltd. A chiral smectic liquid crystal, (S)-4-(1-methyl)heptyloxycarbonylphenyl 4-hexyloxybenzoate (**S811**), was obtained from Merck Co., Ltd.

Purification of final products was carried out using column chromatography over silica gel (61–210  $\mu$ m), (KANTO CHEMICAL Co., INC.) using a toluene-ethyl acetate mixture as the eluent, followed by recrystalization from ethanol. The purities of all final compounds were checked using elementary analysis (EA. 1110; CE Instruments Ltd). The structures of the final products were elucidated by infrared (IR) spectroscopy (BIO RAD FTS-30) and proton nuclear magnetic resonance (<sup>1</sup>H NMR) spectroscopy (JEOL JNM-ECA 500).

#### 2.1.1 4-(4-Cyanophenyl)phenyl 4-octyloxy-2-{11-{4-[4-(4-hexylphenyl)-2,3-difluorophenyl]phenyloxy} decanoyloxy}benzoate, I-10

Potassium carbonate (2.7 g, 20 mmol) was added to a solution of methyl 2,4-dihydroxybenzoate (3.4 g, 20 mmol) and 1-bromooctane (3.9 g, 20 mmol) in cyclohexanone (18 ml). The reaction mixture was stirred under reflux for 5 h. After filtration of the precipitate, the solvent was removed by evaporation. The residue was purified by column chromatography on silica gel with toluene to give methyl 2-hydroxy-4-octyloxybenzoate; yield 2.4 g (43%).

Methyl 2-hydroxy-4-octyloxybenzoate (2.2 g, 8.0 mmol) was added to a solution of KOH (1.3 g, 24 mmol) in an ethanol/water (3/1) mixture. The resulting solution was stirred under reflux for 4 h. After removal of the ethanol by evaporation, the residue was acidified with aq. HCl. The solution was extracted with dichloromethane. The organic layers were combined, dried over anhydrous magnesium sulfate, filtered and evaporated. 2-Hydoroxy-4-octyloxybenzoic acid was obtained; yield 2.0 g (95%).

To a solution of 2-hydroxy-4-octyloxybenzoic acid (1.3 g, 5.0 mmol)and 4-(4-cyanophenyl)phenol (0.98 g, 5.0 mmol) in dichloromethane (30 ml), N,N'-dicyclohexylcarbodiimide (1.0 g, 5.0 mmol) and 4-(N,Ndimethylamino)pyridine (0.061 g, 0.05 mmol) were added. The resulting solution was stirred at room temperature for 24 h. Precipitated materials were removed by filtration. After removal of the solvent by evaporation, the residue was purified by recrystallization from ethanol to give 4-(4-cyanophenyl)phenyl 2-hydroxy-4-octyloxybenzoate; yield 0.84 g (38%).

Pottassium carbonate (0.83 g, 6.0 mmol) was added to a solution of 4-(4-hexylphenyl)-1-(4-hydroxyphenyl)-2,3-difluorobenzene (2.2 g, 6.0 mmol) and ethyl 11-bromoundecanoate (1.8 g, 6.0 mmol) in cyclohexanone (14 ml). The reaction mixture was stirred under reflux for 9 h. After filtration of the precipitate, the solvent was removed by evaporation. The residue was purified by column chromatography on silica gel with toluene to give ethyl 11-{4-[4-(4-hexylphenyl)-2,3-difluorophenyl]phenyloxy}decanoate; yield 2.3 g (67%).

Ethyl 11-{4-[4-(4-hexylphenyl)-2,3-difluorophenyl]phenyloxy}decanoate (1.7 g, 3.0 mmol) was added to a solution of KOH (0.36 g, 9.0 mmol) in an ethanol/water (3/1) mixture. The resulting solution was stirred under reflux for 4 h. After removal of the ethanol by evaporation, the residue was acidified with aq. HCl. The solution was extracted with dichloromethane. The organic layers were combined, dried over anhydrous magnesium sulfate, filtered and evaporated. 11-{4-[4-(4-hexylphenyl)-2,3-difluorophenyl]phenyloxy}decanoic acid was obtained; yield 1.35 g (82%).

To a solution of 11-[4-[4-(4-hexylphenyl)-2,3-difluorophenyl]phenyloxy}decanoic acid (0.18 g, 0.4 mmol) and 4-(4-cyanophenyl)phenyl 2-hydroxy-4-octyloxybenzoate (0.22 g, 0.4 mmol) in dichloromethane (8 ml), N,N'-dicyclohexylcarbodiimide (0.083 g, 0.4 mmol) and 4-(N,N-dimethylamino)pyridine (0.005 g, 0.04 mmol) were added. The resulting solution was stirred at room temperature for 24 h. Precipitated materials were removed by filtration. After removal of the solvent by evaporation, the residue was purified by column chromatography on silica gel with a toluene/ethyl acetate (19/1) mixture. Recrystallization from ethanol gave the desired product; yield 0.14 g (36%).

<sup>1</sup>H NMR (500 MHz, solvent CDCl<sub>3</sub>, standard TMS)  $\delta_{\rm H}/\rm{ppm}$ : 8.18 (d, 1H, Ar–H,  $J = 9.2 \,\rm{Hz}$ ), 7.73 (d, 2H, Ar–H,  $J = 8.5 \,\rm{Hz}$ ), 7.67 (d, 2H, Ar–,  $J = 8.1 \,\rm{Hz}$ ), 7.62 (d, 2H, Ar–H,  $J = 9.2 \,\rm{Hz}$ ), 7.51 (d, 2H, Ar–H,  $J = 7.5 \,\rm{Hz}$ ), 7.50 (d, 2H, Ar–H,  $J = 6.9 \,\rm{Hz}$ ), 7.29–7.27 (m, 4H, Ar–H), 7.25–7.20 (m, 2H, Ar–H), 6.98 (d, 2H, Ar–H,  $J = 9.2 \,\rm{Hz}$ ), 6.88 (dd, 1H, Ar–H,  $J = 2.3 \,\rm{Hz}$ , 8.6 Hz), 6.66 (d, 1H, Ar–H,  $J = 2.3 \,\rm{Hz}$ ), 4.03 (t, 2H, Ar–OCH<sub>2</sub>–,  $J = 6.6 \,\rm{Hz}$ ), 3.99 (t, 2H, Ar–OCH<sub>2</sub>–,  $J = 6.3 \,\rm{Hz}$ ), 2.66 (t, 2H, Ar–CH<sub>2</sub>–,  $J = 7.7 \,\rm{Hz}$ ), 2.60 (t, 2H,  $-\rm{OCOCH_2}$ –,  $J = 7.7 \,\rm{Hz}$ ), 1.83–1.29 (m, 36H, aliphatic), 0.90 (t, 6H,  $-\rm{CH}_3$ ,  $J = 6.9 \,\rm{Hz}$ ). IR(KBr)  $\nu_{\rm max}/\rm{cm}^{-1}$ : 2925, 2855, 2225, 1763, 1730, 1610, 1248, 1120. Anal. Calcd for C<sub>63</sub>H<sub>71</sub>NO<sub>6</sub>F<sub>2</sub>: C, 77.51; H, 7.33; N, 1.43. Found: C, 77.27; H, 7.86; N, 1.46.

The other compounds presented in this paper were obtained by a similar method to that for **I-10**. Analytical data for the other compounds are given.

#### 2.1.2 4-(4-Cyanophenyl)phenyl 4-octyloxy-2-{11-{4-[2-(4-hexyloxyphenyl)pyrimidin-5-yl]phenyloxy} decanoyloxy}benzoate, II-10

<sup>1</sup>H NMR (500 MHz, solvent CDCl<sub>3</sub>, standard TMS)  $\delta_{\rm H}/\rm{ppm}$ : 8.96 (s, 2H, Ar–H), 8.37 (d, 2H, Ar–H,  $J = 8.6\,\rm{Hz}$ ), 8.18 (d, 1H, Ar–H,  $J = 9.2\,\rm{Hz}$ ), 7.73 (d, 2H, Ar–H,  $J = 8.6\,\rm{Hz}$ ), 7.67 (d, 2H, Ar–H,  $J = 9.2\,\rm{Hz}$ ), 7.62 (d, 2H, Ar–H,  $J = 9.2\,\rm{Hz}$ ), 7.55 (d, 2H, Ar–H,  $J = 9.2\,\rm{Hz}$ ), 7.32 (d, 2H, Ar–H,  $J = 9.2\,\rm{Hz}$ ), 7.28 (d, 2H, Ar–H,  $J = 9.2\,\rm{Hz}$ ), 7.03 (d, 2H, Ar–H,  $J = 8.6\,\rm{Hz}$ ), 6.88 (dd, 1H, Ar–H,  $J = 2.9\,\rm{Hz}$ , 8.9 Hz), 6.65 (d, 1H, Ar–H,  $J = 2.9\,\rm{Hz}$ ), 4.03 (t, 2H, Ar–GCH<sub>2</sub>–,  $J = 6.6\,\rm{Hz}$ ), 4.00 (t, 2H, Ar–OCH<sub>2</sub>–,  $J = 6.3\,\rm{Hz}$ ), 2.69 (t, 2H, Ar–OCH<sub>2</sub>–,  $J = 7.7\,\rm{Hz}$ ), 2.60 (t, 2H, OCOCH<sub>2</sub>–,  $J = 7.4\,\rm{Hz}$ ), 1.83–1.29 (m, 36H, aliphatic), 0.90 (t, 3H, -CH<sub>3</sub>,  $J = 6.9\,\rm{Hz}$ ), 0.89 (t, 3H, -CH<sub>3</sub>,  $J = 6.9\,\rm{Hz}$ ). IR(KBr)  $\nu_{\rm{max}}/\rm{cm}^{-1}$ : 2926, 2855, 2225, 1763, 1729, 1610, 1248, 1119. Anal. Calcd for C<sub>63</sub>H<sub>71</sub>N<sub>3</sub>O<sub>6</sub>: C, 77.76; H, 7.60; N, 4.46. Found: C, 77.36; H, 7.86; N, 4.47.

#### 2.1.3 4-(4-Cyanophenyl)phenyl 4-octyloxy-2-{11-{4-[2-{2-fluoro-4-hexyloxyphenyl)pyrimidin-5-yl] phenyloxy}decanoyloxy}benzoate, III-10

<sup>1</sup>H NMR (500 MHz, solvent CDCl<sub>3</sub>, standard TMS)  $\delta_{\rm H}$ /ppm: 8.99 (s, 2H, Ar–H), 8.18 (d, 1H, Ar–H, J=8.6 Hz), 8.10 (t, 1H, Ar–H, J=8.9 Hz), 7.73 (d, 2H, Ar–H, J=8.6 Hz), 7.67 (d, 2H, Ar–H, J=8.6 Hz), 7.67 (d, 2H, Ar–H, J=8.6 Hz), 7.28 (d, 2H, Ar–H, J=8.6 Hz), 7.03 (d, 2H, Ar–H, J=8.6 Hz), 7.28 (d, 2H, Ar–H, J=8.6 Hz), 7.03 (d, 2H, Ar–H, J=8.6 Hz), 6.87 (dd, 1H, Ar–H, J=2.3 Hz, 8.9 Hz), 6.82 (dd, 1H, Ar–H, J=2.3 Hz, 13.2 Hz), 6.65 (d, 1H, Ar–H, J=2.9 Hz), 4.03 (t, 2H, Ar–OCH<sub>2</sub>–, J=6.6 Hz), 2.60 (t, 2H, Ar–OCH<sub>2</sub>–, J=6.9 Hz), 4.00 (t, 2H, Ar–OCH<sub>2</sub>–, J=6.6 Hz), 2.60 (t, 2H, OCOCH<sub>2</sub>–, J=7.4 Hz), 1.84–1.29 (m, 36H, aliphatic), 0.92 (t, 3H, –CH<sub>3</sub>, J=7.2 Hz), 0.90 (t, 3H, –CH<sub>3</sub>, J=6.9 Hz). IR(KBr)  $\nu_{\rm max}/{\rm cm}^{-1}$ : 2927, 2854, 2225, 1763, 1727, 1610, 1248, 1119. Anal. Calcd for C<sub>63</sub>H<sub>71</sub>N<sub>3</sub>O<sub>7</sub>F: C, 75.05; H, 7.23; N, 4.30. Found: C, 74.84; H, 7.86; N, 4.24.

#### 2.2 Liquid-Crystalline and Physical Properties

The initial assignments and corresponding transition temperatures for the final products were determined by thermal optical microscopy using a polarizing microscope (Olympus BX-51) equipped with a temperature control unit (Japan High Tech LK-600PM). The heating and cooling rates were  $5^{\circ}$ C min<sup>-1</sup>. Temperatures and enthalpies of transition for the final products and the intermediate compounds were investigated by differential scanning calorimetry (DSC) using a Seiko DSC 6200 calorimeter, with the heating and cooling rates of  $5^{\circ}\mathrm{C\,min^{-1}},$  for both heating and cooling cycles, after being encapsulated in aluminum pans.

#### 3. RESULTS AND DISCUSSION

#### 3.1. Phase Transition Properties

Molecular structures of novel liquid crystal oligomers **I-10**, **II-10**, and **III-10** are shown in Figure 2. Temperatures and enthalpies of the transition for the compounds determined by optical microscopy and differential scanning calorimetry are listed in Table 1.

All liquid crystal oligomers were found to show a phase sequence of Iso-N-glass on cooling, and they exhibited a wide temperature range of the nematic phase in spite of being far from a rod-like structure. The Iso-N transition temperature of **I-10** is lower than that of **I-6**, indicating that introduction of the longer spacer length destabilizes the N phase.





III-10

FIGURE 2 Molecular structures of non-chiral liquid crystal oligomers I-10, II-10, and III-10.

	glass		Ν		Iso	mp
I-6 [19]	•	-12.6	•	150(2.1) 129(2.3)	•	71 76
II-10 II-10	•	-15.0 -6.4	•	129(2.3) 143(1.3)	•	70 91
III-10	•	-5.6	•	145 (1.2)	•	99

**TABLE 1** Transition temperatures ( $^{\circ}$ C) on cooling and enthalpies (kJ mol<sup>-1</sup>) of transition (in brackets) for **I-6**, **I-10**, **II-10**, and **III-10** 

#### 3.2. Miscibility Studies

We investigated transition behavior of binary mixtures of each liquid crystal oligomer and a chiral smectic liquid crystal (S811). The molecular structure and transition temperatures of S811 are shown in Figure 3.

Figure 4a shows a binary phase diagram between S811 and compound **I-10** on cooling. For mixtures containing 40-80 wt% of I-10, blue phases were found to be induced. In a mixture of S811 (40 wt%) and compound **I-10** (60 wt%), a platelet texture with various colours appeared at 63.1°C, and then it changed to a typical N\* texture at 53.6°C. The platelet texture is characteristic of cubic BP. However, we do not have further information to determine the phase structure. On heating, the N<sup>\*</sup> phase changed to the cubic BP reversibly. The melting temperature was 62.6°C. In a mixture of S811 (60 wt%) and compound I-10 (40 wt%), isotropic liquid changed to BP at 31.9°C. The blue phase showed fluidity and did not appear as platelets. These observations indicate that the phase is amorphous BPIII. On further cooling, the BPIII changed to an N<sup>\*</sup> phase at 18.1°C. The temperature range of the BPIII was over 13K in this binary mixture. The melting temperature was 60.4°C. With respect to a binary system of **S811** and compound I-6, blue phases were observed in mixtures containing 40-70 wt% of I-6 [19]. The BPIII were shown in those containing 40–55 wt% of I-6. The Iso-BPIII transition temperature of the mixture containing 55 wt% of I-6 was 65.7°C. Therefore, as increasing the



S811: Cry 41 [SmA 22] Iso

FIGURE 3 Molecular structure and transition temeperatures (°C) of S811.



**FIGURE 4** (a) Phase diagram between **S811** and compound **I-10**, (b) that between **S811** and compound **II-10**, and (c) that between **S811** and compound **III-10**.

methylene number in the spacer, stability of the induced BPIII decreases.

Figure 4b shows a binary phase diagram of S811 and compound **II-10** on cooling. For mixtures containing 40–70 wt% of **II-10**, blue phases were found to be induced. In a mixture of **S811** (30 wt%) and compound **II-10** (70 wt%), isotropic liquid changed to cubic BP at  $93.5^{\circ}C$  and then it changed to an N<sup>\*</sup> phase at  $89.7^{\circ}C$ . On heating, the N\* phase changed to cubic BP reversibly. The melting temperature was  $82.5^{\circ}$ C. In a mixture of **S811** (40 wt%) and compound **II-10** (60 wt%), isotropic liquid changed to BPIII at 72.9°C and then it changed to cubic BP at 68.2°C. On further cooling, the cubic BP changed to an N<sup>\*</sup> phase at 61.5°C. On heating, the N<sup>\*</sup> phase changed to the BPIII at 63.6°C. The cubic BP was not observed on heating. The melting temperature was 76.0°C. In a mixture of S811 (50 wt%) and compound II-10 (50 wt%), isotropic liquid changed to BPIII at 54.3°C and then it changed to an N<sup>\*</sup> phase at 33.0°C. The temperature range of BPIII is 21.3 K. On heating the N<sup>\*</sup> phase changed to BPIII at 47.6°C ant then it changed to isotropic liquid at 54.2°C. The melting temperature was 76.5°C.

Figure 4c shows a binary phase diagram **S811** and compound **III-10**. For mixtures containing 40–70 wt% of **III-10**, blue phases were found to be induced. In a mixture of **S811** (60 wt%) and compound **III-10** (40 wt%) isotropic liquid changed to BPIII at 33.7°C. On further cooling, the BPIII changed to an N\* phase at 28.2°C. The melting temperature was 79.1°C. In a mixture of **S811** (50 wt%) and compound **III-10** (50 wt%), isotropic liquid changed to cubic BP at 59.4°C and then it changed to an N\* phase at 50.8°C. The temperature range of cubic BP is 8.6 K. On heating, the N\* phase changed to the cubic BP at 52.6°C and then it changed to isotropic liquid at 60.0°C. The melting temperature was 82.9°C.

Compound **II-10** possessing a diphenylpyrimidine moiety was found to induce BPIII with the temperature range of 21.3 K in the one to one mixture with **S811**, whereas compound **III-10** possessing a fluorosubstituted diphenylpyrimidine moiety induced enantiotropic cubic BP with the temperature range of 8.6 K in the one to one mixture with **S811**.

#### 4. CONCLUSIONS

We prepared three non-chiral liquid crystal oligomers and found that they show a wide temperature range of a nematic phase. The cubic BP and amorphous BPIII were found to be induced in binary mixtures of each liquid crystal oligomer and a chiral smectic liquid crystal. Compound **II-10** possessing a diphenylpyrimidine stabilizes BPIII. On the other hand, compounds **I-10** possessing a difluorosubstituted mesogenic moiety and compound **III-10** possessing a monofluorosubstituted mesogenic moiety stabilize cubic BP. Introduction of a fluorine atom to the mesogenic moiety plays an important role in organizing a cubic structure in blue phases.

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