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Synthesis and Thermal Properties of Antiferroelectric Liquid Crystals Having Phenylalkanoate and Phenylalkenoate Moieties

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Novel antiferroelectric liquid crystals having phenylalkanoate and phenylalkenoate moieties were synthesized and their mesomorphic properties were studied. 4-[2-{1-(Trifluoromethyl)heptyloxycarbonyl}ethyl]phenyl 4′-decyloxybiphenyl-4-carboxylate **1b** exhibited the chiral smectic C_A phase at a temperature below the chiral smectic C phase. Biphenyl-cinnamate derivatives of the three-ring system exhibited a stable mesomorphic phase and enantiotropic smectic C_A phase, with the carbon-carbon double bond acting as a mesogenic core.

Keywords: antiferroelectricity, ferroelectricity, synthesis, thermal property

INTRODUCTION

Since the discovery of antiferroelectric liquid crystals (AFLCs),[1–3] a great deal of attention has been focused on their potential application in display devices.[4] Up to now, several AFLC compounds have been synthesized. Most of these have benzoate moieties, e.g., 1-methylheptylbenzoate[1] and 1-(trifluoromethyl)heptyl benzoate.[5]

We have prepared a series of new antiferroelectric liquid crystal compounds containing phenylalkanoate and phenylalkenoate moieties. In this paper, we report

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on the synthesis and thermal properties of antiferroelectric liquid crystals with phenylalkanoate and phenylalkenoate as shown in Scheme I.

RESULTS AND DISCUSSION

Synthesis of Liquid Crystals

The synthesis of liquid crystals having phenylalkanoate moiety **1b** was carried out as outlined in Scheme II. (*R*)-1,1,1-Trifluoro-2-octanol was reacted with 3-(4-hydroxyphenyl)propanoic acid **4** in the presence of sulfuric acid and boric acid to give (*R*)-1-(trifluoromethyl)heptyl 3-(4-hydroxyphenyl)propanoate **5**. Esterification was performed with 4′-decyloxybiphenyl-4-carboxylic acid using dicyclohexylcarbodiimide (DCC) to give (*R*)-4-[2-{1-(trifluoromethyl)heptyloxycarbonyl}ethyl]phenyl 4′-decyloxybiphenyl-4-carboxylate **1b**. Use of 4-hydroxyphenylacetic acid produced **1a** (Scheme II).

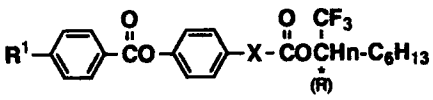
The phenylalkenoate containing liquid crystal **1c** was prepared according to the route shown in Scheme III. (*E*)-4-Hydroxycinnamic acid **6** was treated with acetic anhydride and pyridine to give (*E*)-4-acetoxycinnamic acid **7**. Esterification was performed with (*R*)-1,1,1-trifluoro-2-octanol using DCC to yield (*R*)-1-trifluoromethylheptyl (*E*)-4-acetoxycinnamate **8**. Deacetylation of **8** with butylamine afforded hydroxycinnamate **9**, which was esterified with 4′-decyloxybiphenyl-4-carboxylic acid to give (*R*)-(*E*)-4-[2-{1-(trifluoromethyl)heptyloxycarbonyl}ethenyl]phenyl 4′-decyloxybiphenyl-4-carboxylate **1c**. In a similar manner, cinnamates **1d**–**1g** and **2a**–**2c** were prepared (Scheme III).

6-Benzyloxy-2-naphthoic acid **12**, obtained through the reaction of 6-hydroxy-2-naphthoic acid **10** and benzyl bromide followed by hydrolysis, was esterified with (*R*)-1,1,1-trifluoro-2-octanol to yield (*R*)-1-(trifluoromethyl)heptyl 6-benzyloxy-2-naphthoate **13**. Deprotection of **13** with a H_2/Pd-C produced (*R*)-1-(trifluoromethyl)heptyl 6-hydroxy-2-naphthoate **14**, which was esterified with (*E*)-4-nonyloxycinnamic acid to give (*R*)-6-[1-(trifluoromethyl)heptyloxycarbonyl]-2-naphthyl (*E*)-4-nonyloxycinnamate **3a**. In a similar manner, cinnamates **3b**–**3e** were obtained (Scheme IV).

THERMAL PROPERTIES

The thermal properties of **1** are given in Table I. The phase behavior is influenced by the spacer structure X. The phase transition temperature and phase stability depend upon the tail part and mesogenic core.

The compounds **1a** and **1b** have a phenylalkanoate moiety with a mesomorphic phase. Phenylacetate **1a** exhibits a stable crystalline phase, and a chiral smectic C (SmC*) phase appears in a super cooled state. On the other hand, phenylpropanate **1b** exhibits enantiotropic smectic A (SmA) and SmC* phases in a broad temperature range above the chiral smectic C_A phase (SmC^*_A). The SmA temperature range is 48°C and the SmC* temperature range is 20°C.



1

a: $R^1 = n\text{-}C_{10}H_{21}O$ X = CH_2

b: 〃 CH_2CH_2

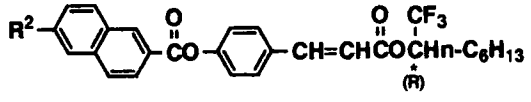
c: 〃 CH=CH (*trans*)

d: $n\text{-}C_{16}H_{33}O$ 〃

e: $n\text{-}C_{11}H_{23}$ 〃

f: $n\text{-}C_{12}H_{25}$ 〃

g: $n\text{-}C_8H_{17}$ 〃

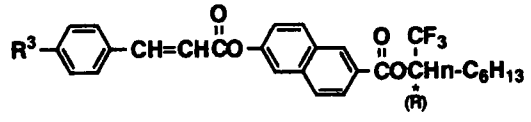


2

a: $R^2 = n\text{-}C_9H_{19}O$

b: $n\text{-}C_{10}H_{21}O$

c: $n\text{-}C_{11}H_{23}O$



3

a: $R^3 = n\text{-}C_9H_{19}O$

b: $n\text{-}C_{10}H_{21}O$

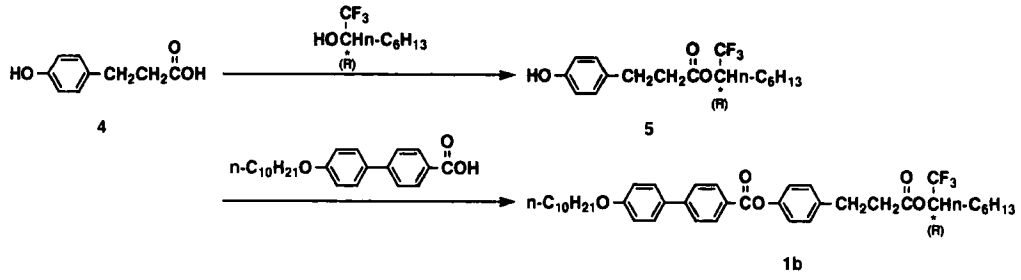
c: $n\text{-}C_7H_{15}COO$

d: $n\text{-}C_8H_{17}COO$

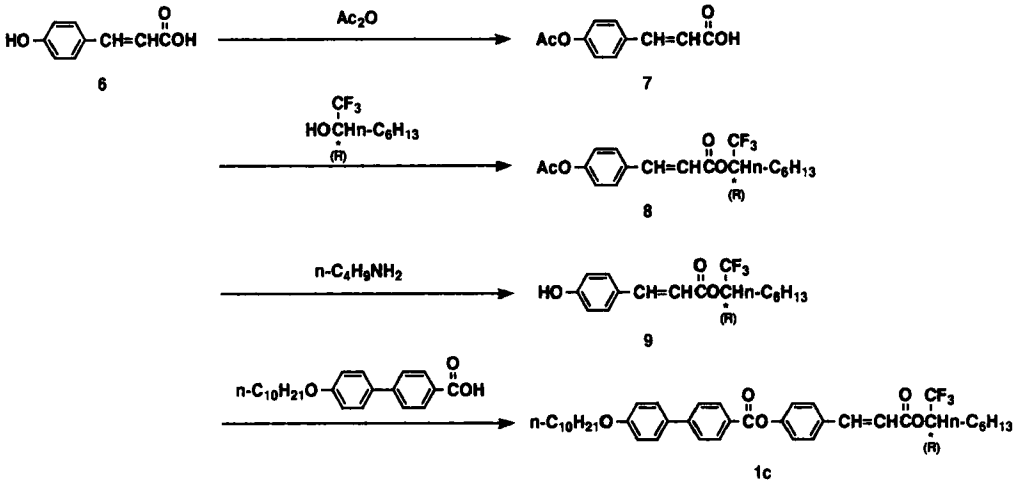
e: $n\text{-}C_9H_{19}COO$

SCHEME I

The compounds with a phenylalkenoate framework **1c–1g** exhibit a stable mesomorphic phase, and have enantiotropic SmA and SmC^*_A. By comparing the phase behavior of **1c** with that of 4-[1-(trifluoromethyl)heptyloxycarbonyl]phenyl 4′-decyloxybiphenyl-4-carboxylate,[5] it was found that **1c** showed higher phase transition temperatures. Since the carbon-carbon double bond acts as a mesogenic core, the



SCHEME II

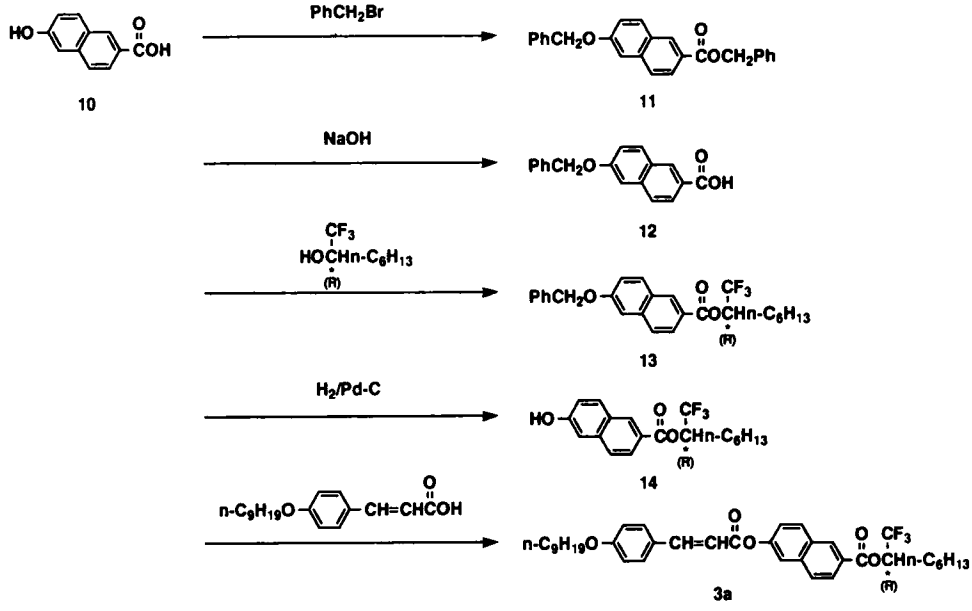


SCHEME III

crystalline and mesomorphic phases are thermodynamically stabilized. Comparing the phase behavior of **1c** and **1e**, it was found that the tail alkyl group diminished the stability of the crystalline and mesomorphic phases as a result of the low phase transition temperature. The mesogenic core with a *trans*-cyclohexane ring framework caused the diminished stability of the mesomorphic phase.

The thermal properties of **2** are given in Table II. All of the **2** compounds have the mesomorphic phase, and exhibit enantiotropic SmC^*_A. By comparing the phase behavior of **1c–1d** with that of **2a–2c**, it was found that the thermodynamically stable mesomorphic phase preferred the naphthalene framework to the biphenyl one as a mesogenic core group in the AFLC compound with a phenylalkenoate moiety. Moreover, the phase transition temperatures and phase stability depend on the length of the tails, so, the longer the tail, the lower the phase transition temperature.

The thermal properties of **3** are given in Table III. When the liquid crystalline tail part has an ether group **3a** and **3b**, each compound exhibits monotropic SmC^*_A. It seems that the position of the naphthalene ring and cinnamate framework



SCHEME IV □

TABLE I

Phase behavior of compounds **1a–1g**

Compound	Cry		SmC_A^*		SmC*		SmA		Iso
1a	•	94			(•	80) •	114	•	
1b	•	34	•	54	•	74 •	122	•	
1c	•	84	•	139			• 149	•	
1d	•	79	•	112			• 120	•	
1e	•	42	•	99			• 106	•	
1f	•	34	•	95			• 101	•	
1g	•	35	•	50			• 69	•	

Cry : Crystal, SmC_A^* : Chiral smectic C_A phase, SmC* : Chiral smectic C phase
SmA : Smectic A phase, Iso : Isotropic liquid
Temperature in degrees Celsius
( ) : Monotropic transition

has a subtle influence on the stability of the mesomorphic phase. When the liquid crystalline tail part has an ester group **3c–3e**, all these compounds exhibit monotropic or enantiotropic SmC_A^*. By comparing the ether group with the ester group in the tail part, it seems that the thermodynamically stable mesomorphic phase prefers an ether group to an ester group, as the tail part in the AFLC compound with a cinnamate framework.

TABLE II

Phase behavior of compounds **2a–2c**

Compound	Cry	SmC_A*	SmA	Iso
2a	• 51	• 63	• 69	•
2b	• 50	• 56	• 66	•
2c	• 45	• 52	• 61	•

Cry : Crystal, SmC_A* : Chiral smectic C_A phase,
SmA : Smectic A phase, Iso : Isotropic liquid
Temperature in degrees Celsius

TABLE III

Phase behavior of compounds **3a–3e**

Compound	Cry	SmC_A*	SmA	Iso
3a	• 75	(• 57)	(• 67)	•
3b	• 53	(• 47)	• 58	•
3c	• 71	(• 66)	• 87	•
3d	• 64	• 67	• 84	•
3e	• 50	• 67	• 82	•

Cry : Crystal, SmC_A* : Chiral smectic C_A phase,
SmA : Smectic A phase, Iso : Isotropic liquid
Temperature in degrees Celsius
( ) : Monotropic transition

EXPERIMENTAL

The structure of the synthesized compounds were determined by spectroscopic methods, ^{1}H-NMR (JEOL PMX60si), IR (JASCO IRA-3), mass spectrum (JMS SX-102), elemental analysis (Perkin-Elmer 240C). The specific optical rotation was measured by using JASCO DIP-370 polarimeter. The identification of the phases for the compounds were carried out by microscopic observation using a polarizing microscope with a hot stage having a temperature control unit. The phase transition temperatures were determined by DSC (MAC Science DSC3100).

Preparation of (*R*)-4-[2-{1-(Trifluoromethyl)heptyloxycarbonyl}ethyl]phenyl 4′-decyloxybiphenyl-4-carboxylate (1b)

A mixture of 3-(4-hydroxyphenyl)propanonic acid (0.83 g, 5.0 mmol), (*R*)-1,1,1-trifluoro-2-octanol (Morita Kagaku Kogyo Co., Ltd., 93.0% e.e.) (0.91 g, 5.0 mmol), sulfuric acid (0.02 g, 0.25 mmol), boric acid (0.02 g, 0.25 mmol) and 20 ml of toluene was refluxed for 3 h. The reaction mixture was poured into water and extracted with ether. The organic layer was washed with water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue

was purified by column chromatography on silica gel using a mixture of hexane and ethyl acetate (1:1) as eluent. (*R*)-1-(Trifluoromethyl)heptyl 3-(4-hydroxyphenyl)propanoate was obtained 1.33 g (4.0 mmol, 80%). A mixture of 4′-decyloxybiphenyl-4-carboxylic acid (0.27 g, 0.75 mmol), (*R*)-1-(trifluoromethyl)heptyl 3-(4-hydroxyphenyl)propanoate (0.25 g, 0.75 mmol), 4-dimethylaminopyridine (0.05 g, 0.41 mmol) and 10 ml of dichloromethane was stirred for 5 min. To the mixture, was added DCC (0.20 g, 0.98 mmol), and stirring was continued for 6 h at room temperature. The reaction mixture was filtered, and the filtrate was diluted with dichloromethane. The solution was washed with 5% aqueous acetic acid and water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel using dichloromethane as eluent. (*R*)-4-[2-{1-(Trifluoromethyl)heptyloxycarbonyl}ethyl]phenyl 4′-decyloxybiphenyl-4-carboxylate was obtained 0.39 g (0.59 mmol, 78%). ^{1}H-NMR ($CDCl_3$) δ 0.89 (m, 6H), 1.28–1.50 (m, 22H), 1.70–1.74 (m, 2H), 1.78–1.83 (m, 2H), 2.73–3.30 (m, 4H), 4.02 (t, 2H, J = 5 Hz), 5.31 (m, 1H), 7.05 (d, 2H, J = 8 Hz), 7.16 (d, 2H, J = 8 Hz), 7.27 (d, 2H, J = 8 Hz), 7.62 (d, 2H, J = 8 Hz), 7.75 (d, 2H, J = 8 Hz), 8.29 (d, 2H, J = 8 Hz), IR (KBr) 2950, 2860, 1760, 1610, 1510, 1300, 1270, 1180, 1080, 1020, 830, 770, 690 cm^{-1}, Mass spectrum m/e (rel. %) 669 (16, MH^+), 668 (8, M^+), 338 (100), Elemental analysis calcd. for $C_{40}H_{51}O_5F_3$, C, 71.86, H, 7.63, found C, 71.82, H, 7.62 $[\alpha]_D$ +22.4° (c 1.38, CH_2Cl_2).

Preparation of (*R*)-(*E*)-4-[2-{1-(trifluoromethyl)heptyloxycarbonyl}ethenyl]phenyl 4′-decyloxybiphenyl-4-carboxylate (1c)

A mixture of (*E*)-4-hydroxycinnamic acid (4.0 g, 24.3 mmol), pyridine (3.9 g, 48.7 mmol), and 100 ml of tetrahydrofuran was heated at 60°C under nitrogen. To the solution, was added dropwise acetic anhydride (5.0 g, 48.7 mmol), and the mixture was refluxed for 10 h. The reaction mixture was quenched with 2M hydrochloric acid and extracted with ethyl acetate. The extract was washed with water and dried over anhydrous magnesium sulfate. The crude product, obtained by concentration, was used for the next esterification.

A mixture of (*E*)-4-acetoxycinnamic acid (0.38 g, 1.84 mmol), (*R*)-1,1,1-trifluoro-2-octanol (0.28 g, 1.54 mmol), 4-dimethylaminopyridine (0.11 g, 0.92 mmol) and 10 ml of dichloromethane was stirred for 5 min. To the mixture, was added DCC (0.48 g, 1.54 mmol), and stirring was continued for 6 h at room temperature. The reaction mixture was filtered, and the filtrate was diluted with dichloromethane. The solution was washed with 5% aqueous acetic acid and water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel using a mixture of hexane and ethyl acetate (4:1) as eluent. (*R*)-1-(Trifluoromethyl)heptyl (*E*)-4-acetoxycinnamate was obtained 0.47 g (1.24 mmol, 80%).

A mixture of (*R*)-1-(trifluoromethyl)heptyl (*E*)-4-acetoxycinnamate (0.47 g, 1.24 mmol), butylamine (0.27 g, 3.73 mmol) and 20 ml of diisopropyl ether was stirred for 5 h at room temperature. The reaction mixture was washed with dilute aqueous hydrochloric acid and water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chroma-

tography on silica gel using a mixture of hexane and ethyl acetate (4:1) as eluent. (*R*)-1-(Trifluoromethyl)heptyl (*E*)-4-hydroxycinnamate was obtained 0.40 g (1.20 mmol, 96%).

A mixture of 4′-decyloxybiphenyl-4-carboxylic acid (0.40 g, 1.12 mmol), (*R*)-1-(trifluoromethyl)heptyl (*E*)-4-hydroxycinnamate (0.25 g, 0.75 mmol), 4-dimethylaminopyridine (0.07 g, 0.60 mmol) and 10 ml of dichloromethane was stirred for 5 min. To the mixture, was added DCC (0.31 g, 1.50 mmol), and stirring was continued for 7 h at room temperature. The reaction mixture was filtered, and the filtrate was diluted with dichloromethane. The solution was washed with 5% aqueous acetic acid and water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel using a mixture of hexane and ethyl acetate (5:1) as eluent. (*R*)-(*E*)-4-[2-{1-(Trifluoromethyl)heptyloxycarbonyl}ethenyl]phenyl 4′-decyloxybiphenyl-4-carboxylate was obtained 0.41 g (0.62 mmol, 83%). ^{1}H-NMR ($CDCl_3$) δ 0.89 (m, 6H), 1.26–1.50 (m, 22H), 1.78–1.85 (m, 4H), 4.02 (t, 2H, J = 5 Hz), 5.43 (m, 1H), 6.47 (d, 1H, J = 15 Hz), 7.00 (d, 2H, J = 8 Hz), 7.30 (d, 2H, J = 8 Hz), 7.60 (d, 2H, J = 8 Hz), 7.63 (d, 2H, J = 8 Hz), 7.70 (d, 2H, J = 8 Hz), 7.78 (d, 1H, J = 15 Hz), 8.23 (d, 2H, J = 8 Hz), IR (KBr) 2920, 2850, 1740, 1720, 1640, 1600, 1510, 1400, 1300, 1270, 1180, 1070, 980, 860, 830, 770 cm^{-1}, Mass spectrum m/e (rel.%) 667 (25, MH^+), 666 (12, M^+), 338 (100), Elemental analysis calcd. for $C_{40}H_{49}O_5F_3$, C, 72.07, H, 7.36, found C, 72.02, H, 7.34, $[\alpha]_D$ + 34.6° (c 1.62, CH_2Cl_2).

Preparation of (*R*)-(*E*)-6-[1-(trifluoromethyl)heptyloxycarbonyl]-2-naphthyl 4-nonyloxycinnamate (3a)

6-Hydroxy-2-naphthoic acid (2.0 g, 10.6 mmol) was dissolved in 40 ml of *N*,*N*-dimethylformamide. To this solution, was added sodium hydride (60% dispersion in oil, washed several times with hexane) (0.61 g, 25.4 mmol), and the reaction mixture was heated at 110°C and allowed to react for 2 h. The reaction mixture was poured into water and extracted with ethyl acetate. The extract was washed with water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel using dichloromethane as eluent. Benzyl 6-benzyloxy-2-naphthoate was obtained 2.22 g (6.03 mmol, 57%).

A mixture of benzyl 6-benzyloxy-2-naphthoate (2.0 g, 5.42 mmol), sodium hydroxide (0.26 g, 6.50 mmol), 30 ml of acetone and 30 ml of water was refluxed for 14 h with stirring. After the reaction mixture was acidified by 6M aqueous hydrochloric acid, the precipitated crystal was filtered off. The collected crystal was washed with water and dried in vacuo. 6-Benzyloxy-2-naphthoic acid was obtained 0.55 g (1.97 mmol, 36%).

A mixture of 6-benzyloxy-2-naphthoic acid (0.55 g, 1.97 mmol), (*R*)-1,1,1-trifluoro-2-octanol (0.36 g, 1.97 mmol), 4-dimethylaminopyridine (0.14 g, 1.18 mmol) and 10 ml of dichloromethane was stirred for 5 min. To the mixture, was added DCC (0.61 g, 3.00 mmol), and stirring was continued for 6 h at room temperature. The reaction mixture was filtered, and the filtrate was diluted with dichloromethane. The solution was washed with 5% aqueous acetic acid and water and

dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel using a mixture of hexane and dichloromethane (1:1) as eluent. (*R*)-1-(Trifluoromethyl)heptyl 6-benzyloxy-2-naphthoate was obtained 0.76 g (1.71 mmol, 87%).

A mixture of (*R*)-1-(trifluoromethyl)heptyl 6-benzyloxy-2-naphthoate (0.76 g, 1.71 mmol), 5% palladium on carbon (50% dispersion in water) (0.22 g) and 20 ml of acetic acid was stirred at room temperature for 10 h under atmospheric hydrogen. The catalyst was filtered off, the filtrate was diluted with ethyl acetate. The solution was washed with water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purified by column chromatography on silica gel using a mixture of hexane and ethyl acetate (1:1) as eluent. (*R*)-1-(Trifluoromethyl)heptyl 6-hydroxy-2-naphthoate was obtained 0.76 g (1.71 mmol, 87%).

A mixture of (*E*)-4-nonyloxycinnamic acid (0.17 g, 0.60 mmol), (*R*)-1-(trifluoromethyl)heptyl 6-hydroxy-2-naphthoate (0.18 g, 0.50 mmol), 4-dimethylaminopyridine (0.04 g, 0.30 mmol) and 10 ml of dichloromethane was stirred for 5 min. To the mixture, was added DCC (0.15 g, 0.75 mmol), and stirring was continued for 2 h at room temperature. The reaction mixture was filtered, and the filtrate was diluted with dichloromethane. The solution was washed with 5% aqueous acetic acid and water and dried over anhydrous magnesium sulfate. After the solvent was removed in vacuo, the residue was purifed by column chromatography on silica gel using a mixture of hexane and ethyl acetate (5:1) as eluent. (*R*)-(*E*)-6-[1-(Trifluoromethyl)heptyloxycarbonyl]-2-naphthyl 4′-nonyloxycinnamate was obtained 0.27 g (0.43 mmol, 86%). ^{1}H-NMR ($CDCl_3$) δ 0.89 (m, 6H), 1.28–1.51 (m, 20H), 1.68–1.90 (m, 4H), 4.11 (t, 2H, J = 5 Hz), 5.53 (m, 1H), 6.55 (d, 1H, J = 15 Hz), 6.94 (d, 2H, J = 8 Hz), 7.43 (d, 1H, J = 8 Hz), 7.56 (d, 2H, J = 8 Hz), 7.71 (s, 1H), 7.85 (d, 1H, J = 15 Hz), 7.93 (d, 1H, J = 8 Hz), 8.03 (d, 2H, J = 8 Hz), 8.09 (d, 2H, J = 8 Hz), 8.67 (s, 1H), IR (KBr) 2920, 2850, 1730, 1630, 1600, 1510, 1470, 1190, 1140, 970, 830 cm^{-1}, Mass spectrum m/e (rel.%) 627 (10, MH^+), 626 (3, M^+), 274 (100), Elemental analysis calcd. for $C_{37}H_{45}O_5F_3$, C, 70.93, H, 7.19, found C, 70.89, H, 7.19, $[\alpha]_D$ + 45.3° (c 1.61, CH_2Cl_2).

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