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Liquid Crystals

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New ferroelectric liquid crystals containing 2(*S*)-[2(*S*)-methylbutyloxy]propanol moiety

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The alcohol, 2(*S*)-[2(*S*)-methylbutyloxy]propanol (**1**), containing two centres of chirality was used as a chiral building block to prepare two series of new FLCs. The first series, 4-{2(*S*)-[2(*S*)-methylbutyloxy]propoxy}phenyl 4-*n*-alkoxybenzoate (**7–10**), exhibits monotropic S_C^* and S_A phases at or below room temperature. The second series, 4,4′-{2(*S*)-[2(*S*)-methylbutylbutoxy]propoxy}biphenyl 4-*n*-alkoxybenzoate (**11–15**), possesses enantiotropic S_C^* and N* phases. The S_C^* phase temperature range is up to 37°C. The spontaneous polarization of 22–32 nC cm^{-2} was measured in the series of the FLCs **11–15**. The comparison experiments indicate that the dipoles of the two chiral centres in the alcohol **1** add in the S_C^* phase so that the *Ps* value of the new FLCs are enhanced.

1. Introduction

Because of their application potential in opto-electric devices, ferroelectric liquid crystals (FLCs) [1, 2] have attracted a great interest in both fundamental and applied research. The optimization of FLC materials to improve device performance is an important goal for FLC materials research. In a previous paper [3] we have synthesized a new alcohol containing two centres of chirality, 2(*S*)-[2(*S*)-methylbutyloxy]propanol (**1**), from natural ethyl lactate and (*S*)-1-iodo-2-methylbutane as well as prepared a series of new FLC materials from this alcohol. In addition to the S_A phase, a S_C^* phase with a temperature range up to 60°C was found in that series of FLCs. The results indicate that the chiral alcohol **1** is useful for preparing the FLCs. To study the structure–physical property relationship of FLCs containing **1** more deeply, we modify the structure of the core molecular part and prepare two series of new FLCs. Their syntheses and physical properties are discussed in this paper.

2. Experimental

Infrared and NMR spectra were recorded on a Nicolet FTIR and Varian Gemini 200 spectrometer respectively. Elemental analyses were made by a Heraeus CHN-O-RAPID combustion apparatus. Mass spectra were recorded on a JMS-D100 instrument. Specific optical rotation was measured with a Jasco DIP-710 polarimeter. Textures and transition temperatures of liquid crystal phases were determined by a SEIKO DSC200 differential scanning calorimeter, as well as with a Nikon Microphot-FXA polarizing microscope in conjunction with a Mettler FP 82 heating stage. Spontaneous polarization was measured by the triangular wave method [4].

2.1. *Synthesis of 2(S)-[2(S)-methylbutyloxy]propyl 4-toluenesulphonate* (**2**)

To a solution of 0·35 g (2·40 mmol) of 2(*S*)-[2(*S*)-methylbutyloxy]propanol (**1**) [3] in 8 ml of pyridine was added 0·73 g (3·80 mmol) of *p*-toluenesulfonyl chloride at 0°C. The solution was stored at 0°C for 10 h. The mixture was extracted with ether (3 × 50 ml), washed with 10 per cent HCl (3 × 50 ml), saturated $NaHCO_3$ (3 × 50 ml) and then with H_2O (3 × 50 ml). The ether phase was dried over Na_2SO_4. After concentration under vacuum 0·68 g of **2** was obtained as a colourless liquid, 95 per cent yield. $[\alpha]_D^{20} = -1{\cdot}54°$ ($c = 1{\cdot}04$, EtOH). IR (neat): 2963, 2929, 2876, 1458, 1363, 1181, 1099, 985, 816, 668 cm^{-1}. ^{1}H NMR ($CDCl_3$): 0·82 (6 H, m), 1·08 (4 H, m), 1·40 (2 H, m), 2·42 (3 H, s), 3·18 (2 H, m), 3·57 (1 H, m), 3·91 (2 H, m), 7·55 (4 H, dd, $J = 9{\cdot}2$, 8·2 Hz). ^{13}C NMR ($CDCl_3$): 11·77, 16·96, 17·18, 22·12, 26·56, 35·66, 73·22, 73·49, 75·27, 128·44 (2 C), 130·29 (2 C), 133·54, 145·23. MS: 300(M^+), 259, 115, 71. Elemental analysis for $C_{15}H_{24}O_4S$:calculate; C 60·00, H 8·00, S 10·67; found; C 59·93, H 8·03, S 10·67 per cent.

2.2. *Synthesis of 4-benzyloxy-1-{2(S)-[2(S)-methylbutyloxy]propoxy}benzene* (**3**)

To a solution of 0·46 g (2·30 mmol) of hydroquinone monobenzyl ether and 0·2 g of KOH in 60 ml of ethanol

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was added 0·68 g (2·3 mmol) of **2** dropwise. The mixture was refluxed for 24 h. After the mixture was cooled to room temperature, it was extracted with CH_2Cl_2 (3 x 50 ml). The organic phase was dried over Na_2SO_4 and concentrated. After flash chromatography (SiO_2, *n*-hexane/ethyl acetate = 1/1), 0·52 g of **3** was obtained as a colourless oil, 70 per cent yield. $[\alpha]_D^{22}$ = −2·0° (c = 1·0, EtOH). IR (neat): 2964, 2926, 2873, 1507, 1459, 1231, 1107, 1042, 825 cm^{-1}. ^{1}H NMR ($CDCl_3$): 0·87 (6 H, m), 1·12 (1 H, m), 1·22 (3 H, d, *J* = 6·1 HZ), 1·50 (2 H, m), 3·34 (2 H, m), 3·80 (3 H, m), 4·99 (2 H, s), 6·85 (4 H, m). 7·38 (5 H, m). ^{13}C NMR ($CDCl_3$): 11·92, 17·22, 17·98, 26·84, 35·87, 71·32, 72·98, 74·67, 75·46, 116·17 (2 C), 116·43 (2 C), 128·07 (2 C), 128·46, 129·14 (2 C), 137·96, 153·66, 154·02. MS: 328 (M$^+$), 270, 200, 129. Elemental analysis for $C_{21}H_{28}$ O_3: calculate; C 76·83, H 8·54; found; C 76·63, H 8·58 per cent.

2.3. *Synthesis of 4-benzyloxy-4'-{2(S)[-2(S)-methylbutyloxy]propoxy}biphenyl* (**4**)

A similarly preparative procedure to that for synthesizing **3** was used. The product was obtained as a colourless solid, 70 per cent yield. $[\alpha]_D^{23}$ = −16° (*c* = 1·0, $CHCl_3$). IR (KBr): 2967, 1608, 1504, 1461, 1380, 1272, 1248, 1158, 1048, 820, 735, 694 cm^{-1}. ^{1}H NMR ($CDCl_3$): 0·90 (6 H, m), 1·26 (4 H, m), 1·55 (2 H, m), 3·38 (2 H, m), 3·85 (2 H, m), 4·1 (1 H, m), 5·10 (2 H, s), 6·99 (4 H, m), 7·44 (9 H, m). ^{13}C NMR ($CDCl_3$): 12·12, 17·39, 18·19, 27·01, 36·03, 70·87, 72·51, 74·77, 75·69, 115·65 (2 C), 115·88 (2 C), 128·28 (2 C), 128·47 (2 C), 128·52 (2 C), 128·76, 129·39 (2 C), 134·23, 134·53, 137·82, 158·68, 158·90. MS: 404 (M$^+$), 213, 185, 91. Elemental analysis for $C_{27}H_{32}O_3$: calculated; C 80·20, H 7·92; found; C 80·14, H 8·02 per cent.

2.4. *Synthesis of 4-{2(S)-[2(S)-methylbutyloxy]propoxyl}phenol* (**5**)

To a solution of 1·21 g (3·70 mmol) of **3** was added 5 ml of 1M trimethylsilyl iodide(TMSI) in CH_2Cl_2 via a syringe under N_2. After stirring for 30 min at room temperature, the solution was treated with 4 ml of MeOH. The mixture was stirred for 10 min, concentrated, and extracted with ether (3 x 25 ml). The organic phase was dried over Na_2SO_4 and concentrated. After chromatography (SiO_2, *n*-hexane/ethyl acetate = 1/5) 0·62 g of **5** was obtained as a colourless liquid, 70 per cent yield. $[\alpha]_D^{25}$ = −2·2° (*c* = 0·98, EtOH). IR:3330, 2963, 2927, 2875, 1510, 1452, 1229, 1098, 826 cm^{-1}. ^{1}H NMR ($CDCl_3$): 0·86 (6 H, m), 1·12 (1 H, m), 1·23 (3 H, d, *J* = 6·1 Hz), 1·50 (2 H, m), 3·35 (2 H, m), 3·80 (3 H, m), 4·43 (1 H, s), 6·76 (4 H, m). ^{13}C NMR ($CDCl_3$): 11·92, 17·20, 17·93, 26·83, 35·79, 72·97, 74·76, 75·56, 116·34 (2 C), 116·63 (2 C), 150·22, 153·70. MS: 238 (M$^+$), 110, 71, 59. Elemental analysis for $C_{14}H_{22}O_3$: calculated; C 70·59, H 9·24; found; C 70·49, H 9·35 per cent.

2.5. *Synthesis of 4-hydroxy-4'-{2(S)-[2(S)-methylbutyloxy]propoxy}biphenyl* (**6**)

A similarly preparative procedure to that for synthesizing **5** was used. The product was obtained as a colourless solid, 75 per cent yield. $[\alpha]_D^{23}$ = −13·4° (*c* = 1·19, $CHCl_3$). IR (KBr): 3297, 2964, 1615, 1504, 1380, 1270, 1247, 1175, 1089, 1065, 813 cm^{-1}. ^{1}H NMR ($CDCl_3$: 0·87 (6 H, m), 1·24 (4 H, m), 1·50 (2 H, m), 3·37 (2 H, m), 3·80 (2 H, m), 4·0 (1 H, m), 4·89 (1 H, s), 6·90 (4 H, m), 7·42 (4 H, m). ^{13}C NMR ($CDCl_3$): 11·96, 17·25, 17·97, 26·86, 35·82, 72·32, 74·82, 75·68, 115·49 (2 C), 116·27 (2 C), 128·30 (2 C), 128·57 (2 C), 134·17 (2 C), 155·38, 158·64. MS: 314 (M$^+$), 186, 71. Elemental analysis for $C_{20}H_{26}O_3$: calculated; C 76·43, H 8·28; found; C 76·58, H 8·38 per cent.

2.6. *Synthesis of 4-{2(S)-[2(S)-methylbutyloxy]propoxy}phenyl 4-n-dodecanyloxybenzoate* (**10**)

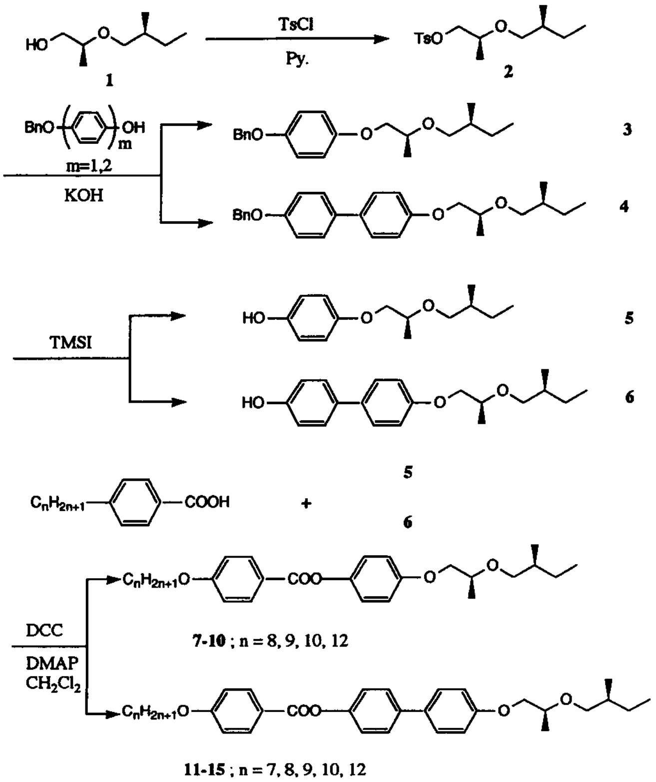
A solution of 0·03 g (0·10 mmol) of 4-*n*-dodecanyloxybenzoic acid, 0·026 g (0·11 mmol) of **5**, 0·04 g (0·20 mmol) of *N*,*N'*-dicyclohexylcarbodiimide (DCC), and 0·01 g (0·01 mmol) of dimethylaminopyridine (DMAP) in 5·0 ml of CH_2Cl_2, was stirred at room temperature until the reaction was complete (monitored by TLC). Filtration and removal of the solvent gave the crude product which was then purified by flash column chromatography (SiO_2, CH_2Cl_2/*n*-hexane = 1/1). The pure product **10** was obtained as a colourless solid, 73 per cent yield. $[\alpha]_D^{20}$ = −2·18° (*c* = 1·01, EtOH). IR: 2958, 2927, 1732, 1606, 1508, 1254, 1195, 1165, 1071, 848 cm^{-1}. ^{1}H NMR ($CDCl_3$): 0·89 (9 H, m), 1·26 (24 H, m), 1·83 (2 H, m), 3·37 (2 H, m), 3·81 (2 H, m), 4·02 (3 H, m), 6·95 (4 H, m), 7·10 (2 H, d, *J* = 9·1 Hz), 8·12 (2 H, d, *J* = 8·9 Hz). ^{13}C NMR ($CDCl_3$): 12·09, 14·90, 17·37, 18·13, 23·48, 26·77, 27·00, 29·89, 30·14 (2 C), 30·35 (2 C), 30·42 (2 C), 32·70, 36·04, 69·10, 72·89, 74·73, 75·67, 115·03, (2 C), 116·01 (2 C), 122·46, 123·26 (2 C), 132·99 (2 C), 145·38, 157·38, 164·24, 165·00. MS: 526 (M$^+$), 289, 121. Elemental analysis for $C_{33}H_{50}O_5$: calculated; C 75·29, H 9·51; found; C 75·59, H 9·54 per cent.

2.7. *Synthesis of 4,4'-{2(S)-[2(S)-methylbutyloxy]propoxy}biphenyl 4-n-dodecanyloxybenzoate* (**15**)

A similarly preparative procedure to that for synthesizing **10** was used. The pure product was obtained as a colourless solid, 70 per cent yield. $[\alpha]_D^{25}$ = −12·62° (*c* = 1·03, $CHCl_3$). IR (KBr): 2919, 2849, 1726, 1611, 1497, 1272, 1172, 1082, 847 cm^{-1}. ^{1}H NMR ($CDCl_3$): 0·90 (9 H, m), 1·20 (1 H, m), 1·29 (3 H, d, *J* = 5·9 Hz), 1·45 (18 H, m), 1·85 (4 H, m), 3·40 (2 H, m), 3·85 (2 H, m), 4·04 (3 H, m), 6·98 (4 H, d, *J* = 8·8 Hz), 7·22 (2 H, d, *J* = 6·1 Hz), 7·54 (4 H, dd, *J* = 8·6, 8·8 Hz), 8·15 (2 H, d, *J* = 8·9 Hz). ^{13}C NMR ($CDCl_3$): 11·99, 14·81, 17·26, 18·05, 23·37, 26·66, 26·89, 29·77, 30·03 (2 C), 30·24 (2 C), 30·91 (2 C), 32·59, 35·59, 35·92, 69·00, 72·40, 74·70, 75·59, 114·95 (2 C), 115·59 (2 C), 122·20, 122·67 (2 C), 128·38 (2 C), 128·77 (2 C), 132·96 (2 C), 139·15, 150·69, 159·22, 164·21, 165·55. Elemental analysis for $C_{39}H_{54}O_5$: calculated; C 77·74, H 8·97; found; C 77·46, H 9·07.

3. Synthesis

The synthesis of new chiral compounds and FLCs is shown in the scheme. The detailed synthetic procedure of the alcohol **1** has been published previously [3, 5]. The tosylation of **1** gave the toluenesulphonate **2** in high yield. The substitution reaction of hydroquinone monobenzyl ether and 4,4′-biphenol monobenzyl ether with **2** gave the compounds **3** and **4** respectively. The deprotection of the benzyl group in **3** and **4** was realized in the presence of trimethylsilyl iodide [6]. The new FLCs **7–15** were synthesized from **5** or **6** and the corresponding 4-*n*-alkoxybenzoic acid, following a standard reaction procedure [7]. There was no preparative difficulty during the whole synthetic sequence.



All new compounds were characterized by IR, ^{1}H NMR, ^{13}C NMR and MS. They also gave satisfactory elemental analyses.

4. Physical properties

The liquid crystal phases and phase transition temperatures of two series of new FLCs **7–15** were determined by the texture identification and DSC data. While the series of the compounds **7–10** exhibits monotropic S_C^* and S_A phases, the series of the compounds **11–15** possesses enantiotropic S_C^* and N* phases. The monotropic S_C^* phase transition temperatures of the FLCs **7–10** are at or below room temperature. But the enantiotropic S_C^* phase transition temperatures of the FLCs **11–15** are much higher, between 68°C and 82°C. The modification of the core molecular part has a profound influence on the liquid crystal phase formation and the phase stability. The phenomenon is well known in the liquid crystals field. It is apparently due to the difference of the length to width ratio when hydroquinone moiety in **7–10** is replaced by 4,4′-biphenol molecule to form **11–15**. The length of the alkoxy side chain has also a profound effect on the liquid crystal phase stability. The longer is the alkoxy side chain, the higher is the S_c^* and S_A phase stability of the FLCs **7–10**. This effect is different for the FLCs **11–15**. The S_c^* phase stability is increased and then decreased slightly as the alkoxy side chain is increased. But the stability of the N* phase is lower, the longer is the alkoxy side chain. The data including the specific optical rotation are summarized in tables 1 and 2 for the FLCs **7–10** and **11–15**, respectively.

FLCs at or below room temperature are scarce [8, 9]. FLCs containing 2(S)-alkoxy-1-propanol unit [8] exhibit an enantiotropic S_A phase and a monotropic S_C^* phase, while both the S_A and S_C^* phases of **7–10** are monotropic. The formation of the monotropic S_A phase in the FLCs **7–10** seems due to the presence of the second chiral

Table 1. Physical properties of the FLCs **7–10**.

Compound	Phase transition/°C	$[\alpha]_D^{25}$/° (EtOH)
7	$\begin{tikzcd} C \arrow[r, "29"] & I \arrow[d, "21"] \\ S_C^* \arrow[u, "-15"] & S_A \arrow[l, "-8"] \end{tikzcd}$	−2·0 (*c* = 1·00)
8	$\begin{tikzcd} C \arrow[r, "29"] & I \arrow[d, "21"] \\ S_C^* \arrow[u, "-9"] & S_A \arrow[l, "-4"] \end{tikzcd}$	−3·0 (*c* = 1·17)
9	$\begin{tikzcd} C \arrow[r, "28"] & I \arrow[d, "26"] \\ S_C^* \arrow[u, "4"] & S_A \arrow[l, "17"] \end{tikzcd}$	−2·5 (*c* = 0·48)
10	$\begin{tikzcd} C \arrow[r, "33"] & I \arrow[d, "28"] \\ S_C^* \arrow[u, "12"] & S_A \arrow[l, "19"] \end{tikzcd}$	−2·2 (*c* = 1·01)

Table 2. Physical properties of the FLCs **11–15** and **17**.

Compound	Phase transition/°C							$[\alpha]_D^{25}/°$ (CHCl$_3$)	P_s/nC cm^{-2}
	C		S$_C^*$		N*		I		
11	●	68	●	105	●	128	●	−13.4 (c=0·97)	32
12	●	80	●	110	●	128	●	−8·4 (c=0·95)	22
13	●	82	●	110	●	123	●	−9·7 (c=1·10)	32
14	●	82	●	110	●	121	●	−13·2 (c=1·06)	24
15	●	79	●	109	●	116	●	−12·6 (c=1·03)	22
17	●	77	●	92	●	105	●	−11·1 (c=0·99)	12

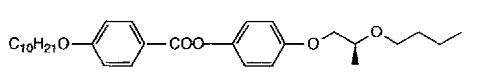
Table 3. Physical properties of the eutectic mixtures.

Mixture	Phase transition/°C									P_s/nC cm^{-2}
9/W82	C	8	S$_C^*$	39	S$_A$	54	I			5
16/W82	C	15	S$_C^*$	34	S$_A$	53	I			3
14/18	C	−4	S$_C^*$	79	S$_A$	85	N*	95	I	9
17/18	C	−2	S$_C^*$	81	S$_A$	83	N*	96	I	4

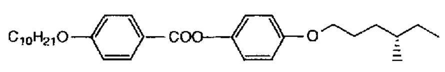
centre in the chiral moiety **1**. The introduction of the second chiral centre from (*S*)-1-iodo-2-methylbutane to the original one of ethyl lactate gives the alcohol **1** some novel properties. Although it may reduce the thermal stability of the liquid crystal phases, it may not change the phase sequence. It is also expected that the spontaneous polarization of the FLCs **7–15** can be increased by the chiral moiety **1**. This prediction is based on the intuitional consideration of the stereochemistry of both methyl groups at **1**. They are on the same side of a tilt plane, if a zigzag conformation of the FLCs **7–15** is assumed. Therefore, the component of the dipole normal to the tilt plane is in the same direction for the two methyl groups. To justify the assumption, a comparison experiment was carried out by mixing the FLCs **9** or **16** with 4-[(*S*)-4-methylhexyloxy]phenyl 4-decyloxybenzoate (W82) in a 1:1 molar ratio. The spontaneous polarization was measured at $T_c-T=10$°C by the triangular wave method [4], using a cell with a 2 μm gap and 0·16 cm^2 active area. An AC field of 20 V and 50 Hz was applied to the system. The eutectic mixture of **9** and **W82** has a larger P_s value than that of the mixture of **16** and **W82**. The P_s value is increased substantially. This seems to be due to the presence of the second chiral centre in **9**. The physical property data of the eutectic mixtures are summarized in table 3. The structures of compounds **16–18** and **W82** are shown opposite.

To study further the influence of the second chiral centre on the spontaneous polarization of the FLCs **11–15**, the FLC **17** containing only one chiral centre was synthesized and its P_s value was measured for comparison. The condition for the electrical property measurement is identical to that for the eutectic mixtures. The FLC **17** has the P_s value of 12 nC cm^{-2} while the FLCs **11–15** have the P_s value of 22–32 nC cm^{-2}. This result not only supports the stereochemical assumption of the similar orientation of the two methyl groups of **1** in the S$_C^*$ phase but indicates the utility of **1** as a chiral moiety for preparing FLC materials. The P_s values of the FLCs **11–15** and **17** as well as the phase transition temperatures of **17** are summarized in table 2.

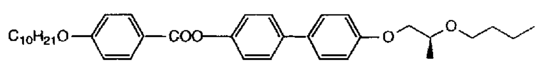
The physical property difference between the eutectic mixtures containing the FLCs **14** or **17** is an important indication of the practical use of the FLCs **11–15**. Therefore a mixture of phenyl pyrimidines **18** was prepared and used as an achiral host. It consists of 22·98 mol % of 2-*n*-octyloxyphenyl-5-*n*-octyl pyrimidine, 12·70 mol % of 2-*n*-decyloxyphenyl-5-*n*-octyl pyrimi-



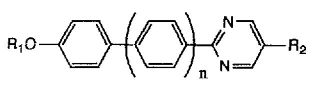
16



W82



17



18 ; n = 0, 1

dine, 56·00 mol % of 2-*n*-octyloxyphenyl-5-*n*-nonyl pyrimidine, and 8·32 mol % of 2-*n*-pentyloxybiphenyl-5-*n*-octyl pyrimidine. The phase and phase transition temperature is C 3 S_C 64 S_A 74 N 78 I. Two eutectic mixtures were prepared by mixing FLCs **14** or **17** with the host in a 1 : 1 molar ratio. The phases and phase transition temperatures of these two eutectic mixtures are almost indentical to each other, but the mixture containing **14** has a P_s value twice as large as that containing **17**. The result shows that the FLCs **11–15** could be useful as the FLC dopants. The physical property data of these two mixtures are summarized in table 3.

5. Conclusion

FLCs at or below room temperature were prepared. The comparison study of the FLCs containing 2-(*S*)-alkoxy-1-propanol or 2-(*S*)-[2(*S*)-methylbutyloxy]-propanol (**1**) shows that the chiral alcohol **1** could give these FLCs some advantages: (i) the liquid crystal phase sequence for the known core structure may not be changed; (ii) the phase transition temperature would be lowered; (iii) the spontaneous polarization could be increased substantially. The results indicate that the alcohol **1** will be useful for the formulation of new S_C^* materials.

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