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Liquid Crystals

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2-Hydroxy-5,5-dialkyl- δ -valerolactone derivatives as chiral dopants for practical ferroelectric liquid crystals

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To cite this Article Sakashita, Keiichi , Ikemoto, Tetsuya , Nakaoka, Yuriko , Terada, Fumiko , Sako, Yoshihiro , Kageyama, Yoshitaka and Mori, Kenji(1993) '2-Hydroxy-5,5-dialkyl- δ -valerolactone derivatives as chiral dopants for practical ferroelectric liquid crystals', *Liquid Crystals*, 13: 1, 71 – 81

To link to this Article: DOI: 10.1080/02678299308029054

URL: <http://dx.doi.org/10.1080/02678299308029054>

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2-Hydroxy-5,5-dialkyl- δ -valerolactone derivatives as chiral dopants for practical ferroelectric liquid crystals

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(Received 20 May 1992; accepted 17 August 1992)

2-Hydroxy-5,5-dialkyl- δ -valerolactone derivatives have shown interesting properties as a chiral dopant for ferroelectric liquid crystals. In small amounts these compounds induce high magnitudes of spontaneous polarization and long helical pitches in the chiral nematic phase (N*). Alkyl chains attached at the C-5 position play an important role in these properties. Here we discuss the stereochemistry and ferroelectric properties of these lactones and compare them with those of 2-hydroxy-5-alkyl- δ -valerolactone derivatives reported previously. The ring structure of 5,5-dialkyl- δ -valerolactone is found to be a rigid 'pseudo-chair' conformation by ^1H NMR studies.

1. Introduction

Ferroelectric liquid crystals have been actively studied as potential materials for future new optical devices. Numerous compounds have been synthesized since the discovery of DOBAMBC [1]. However, no compound was discovered which had a practical chiral smectic C (S_C^*) phase range with a reasonable magnitude of spontaneous polarization and desirable phase sequence. Therefore, non-chiral smectic C liquid crystals were usually doped with a chiral compound (chiral dopant) to obtain a practical ferroelectric liquid crystal with a quick response.

Since the response time is said to be directly proportional to the rotational viscosity and inversely proportional to the magnitude of spontaneous polarization and the strength of applied electric field [2] it would be effective to decrease the rotational viscosity and to increase the magnitude of spontaneous polarization in order to obtain a quick responding ferroelectric liquid crystal. However, since it would not be wise to raise the magnitude of spontaneous polarization as high as possible, the normal response would not be obtained due to a high internal electric field [3]. As general chiral compounds have a high viscosity, a small amount of addition would be better to lower the viscosity of the ferroelectric liquid crystal. Therefore, it is meaningful to design and synthesize a chiral compound capable of inducing a high magnitude of spontaneous polarization in order to obtain a quick responding ferroelectric liquid crystal.

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Since the relationship between chemical structure of a chiral compound and induced ferroelectric properties, such as the magnitude of spontaneous polarization, the helical pitch of S_C^* and N^* phases, and the tilt angle are complicated, there is no general rule for designing a new chiral dopant.

We have studied δ -valerolactone derivatives to be used as chiral dopants in order to induce a high magnitude of spontaneous polarization, since the δ -valerolactone ring has a large lateral dipole moment (4.1 D) and restricted internal rotation around the molecular long axis due to steric hindrance of the ring structure [4]. We have reported the synthesis, ferroelectric properties and stereochemistry of 2,5-disubstituted- δ -valerolactones [5,6]. Recently, we have found that chiral 2-(4'-octyloxybiphenyl-4-carboxy)-5,5-dialkyl- δ -valerolactones are attractive and practical chiral dopants for ferroelectric liquid crystals [7].

In this paper, we shall describe some new experimental results obtained by the addition of a series of chiral 2-(4'-octyloxybiphenyl-4-carboxy)-5,5-dialkyl- δ -valerolactones to a non-chiral S_C mixture and by the NMR studies of these compounds, and we shall discuss the relationship between stereochemistry and ferroelectric properties.

2. Experimental

2.1. Synthesis of chiral compounds

The melting points are uncorrected. The NMR spectra were recorded on Hitachi R-90H and JEOL JNM-EX 400 instruments. TMS was used as the internal standard.

(S)- γ -Butyrolactone- γ -carboxylic acid tert-butyl ester. *(S)- γ -Butyrolactone- γ -carboxylic acid* was synthesized from L-glutamic acid by the method described by Okabe [8].

(S)- γ -Butyrolactone- γ -carboxylic acid (13 g, 0.1 mol.) was treated with thionyl chloride (10 ml) to give the corresponding acid chloride after distillation of excess reagent. This material was used for the next step without further purification. Both *tert*-butyl alcohol (10 g) and *N,N*-dimethylaniline (13.3 g, 1.11 mol) were dissolved in dry benzene (90 ml) and *(S)- γ -butyrolactone- γ -carboxylic acid chloride* (14.9 g) in dry benzene (60 ml) was added over a one hour period with ice cooling. The stirring was continued at room temperature overnight. The reaction mixture was then poured into cold 20 per cent sulphuric acid and the organic layer was separated. The aqueous layer was extracted with diethyl ether several times. The combined organic layers were dried over anhydrous magnesium sulphate and concentrated to give a crude esterified product. This was recrystallized from hexane ether: mp 52–54°C; $^1\text{H NMR}$ (CDCl_3) δ , 1.50 (s, 9 H), 2.1–2.7 (m, 4 H) and 4.78 ppm (dd, 1H, $J = 4$ Hz, 7 Hz).

(S)-2,5-Dihydroxy-5-methylcapronic acid tert-butyl ester. *(S)- γ -Butyrolactone- γ -carboxylic acid tert-butyl ester* (5 g, 26.8 mmol) was dissolved in 80 ml of dry THF and the solution was cooled to -78°C under an argon atmosphere. Then, 42.8 ml of 0.94 M methylmagnesium bromide in THF (1.5 equiv.) was added dropwise and stirring was continued for an additional 6 h. The temperature was raised to 0°C and the reaction mixture was treated by adding a saturated ammonium chloride solution and water successively. The products were extracted with ethyl acetate. The organic layer was washed with a saturated sodium bicarbonate solution, dried over sodium sulphate and concentrated. The residue was chromatographed on silica gel. Elution with 20 per cent ethyl acetate in hexane gave 5 g of *(S)-2,5-dihydroxy-5-methylcapronic acid tert-butyl ester* as a colourless oil: $^1\text{H NMR}$ (CDCl_3) δ , 1.20 (s, 6 H), 1.48 (s, 1 H), 1.6–2.1 (m, 4 H),

3.00 (d, 1 H, $J = 6$ Hz), 3.5–3.8 (m, 1 H) and 3.9–4.2 ppm (m, 1 H); IR (neat), 3350, 2950, 1720 cm^{-1} .

(S)-2-Hydroxy-5,5-dimethyl- δ -valerolactone. A mixture of (*S*)-2,5-dihydroxy-5-methylcapronic acid *tert*-butyl ester (2.18 g, 10 mmol) and *p*-toluenesulphonic acid (17 mg, 0.1 mmol) was refluxed in 100 ml of benzene for 6 h. After cooling the mixture, a saturated sodium bicarbonate solution was poured into it. The organic layer was separated, washed with water, dried over magnesium sulphate and concentrated. The residue was chromatographed on silica gel. Elution with 1 per cent ethanol in benzene gave 1.0 g of (*S*)-2-hydroxy-5,5-dimethyl- δ -valerolactone as a colourless oil: ^1H NMR (CDCl_3) δ , 1.50 (s, 6 H), 1.9–2.5 (m, 4 H), 3.25 (s, 1 H) and 4.08 ppm (m, 1 H); IR (KBr), 3450, 2970, 1730 cm^{-1} .

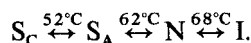
4'-Octyloxybiphenyl-4-carboxylic acid. Both 4'-cyano-4-hydroxybiphenyl (19.5 g, 0.1 mol) and 1-bromooctane (23.3 g, 0.12 mol) were dissolved in 300 ml of acetone with powdered potassium carbonate (20 g) as alkylating catalyst. The mixture was refluxed for 5 h. After filtering off insoluble salts, the reaction mixture was concentrated and the residue was chromatographed on silica gel. Elution with 20 per cent hexane benzene gave 4'-octyloxy-4-cyanobiphenyl (25 g). Then, it was dissolved in 1 N potassium hydroxide in ethylene glycol (100 ml) and the mixture was refluxed for 2 h. It was poured into an ice cold 1 N hydrochloric acid (300 ml). The precipitate was collected by filtration, washed with water and recrystallized from ethanol. 4'-Octyloxybiphenyl-4-carboxylic acid (21 g) was obtained as colourless crystals: mp 181–183°C; ^1H NMR (CDCl_3) δ 0.87 (t, 3 H, $J = 5$ Hz), 1.05–2.0 (m, 12 H), 4.0 (t, 2 H, $J = 7$ Hz), 7.02 (d, 2 H, $J = 9$ Hz), 7.58 (d, 2 H, $J = 9$ Hz), 7.64 (d, 2 H, $J = 9$ Hz) and 7.98 ppm (d, 2 H, $J = 9$ Hz); IR (KBr), 2970, 1695 cm^{-1} .

(S)-2-(4'-Octyloxybiphenyl-4-carboxy)-5,5-dimethyl- δ -valerolactone. To a solution of (*S*)-2-hydroxy-5,5-dimethyl- δ -valerolactone (144 mg, 1 mmol) and 4'-octyloxybiphenyl-4-carboxylic acid (359 mg, 1.1 mmol) in dichloromethane (2.5 ml), dicyclohexyl carbodiimide (240 mg, 1.2 mmol) and 4-(*N,N*-dimethylamino)-pyridine (125 mg) were added at 5°C. The stirring was continued at room temperature overnight. After filtration of the reaction mixture, the filtrate was concentrated and the residue was chromatographed on silica gel. Elution with benzene gave 320 mg of (*S*)-2-(4'-octyloxybiphenyl-4-carboxy)-5,5-dimethyl- δ -valerolactone, followed by recrystallization from diethyl ether in hexane: mp 126°C; ^1H NMR (CDCl_3) δ 0.89 (t, 3 H, $J = 5$ Hz), 1.1–1.6 (m, 10 H), 1.50 (s, 3 H), 1.6–1.9 (m, 2 H), 1.95–2.45 (m, 2 H), 4.02 (t, 2 H, $J = 7$ Hz), 5.48 (t, 1 H, $J = 9$ Hz), 6.98 (d, 2 H, $J = 9$ Hz), 7.58 (d, 2 H, $J = 9$ Hz), 7.64 (d, 2 H, $J = 9$ Hz) and 8.13 ppm (d, 2 H, $J = 9$ Hz); IR (KBr), 2920, 2860, 1710 cm^{-1} .

Other series of derivatives. A series of (*S*)-2-hydroxy-5,5-dialkyl- δ -valerolactones with different alkyl chain lengths were synthesized by the method described above using alkylmagnesium bromide instead of methylmagnesium bromide.

2.2. Preparation of ferroelectric liquid crystals

A non-chiral S_C liquid-crystalline mixture was composed of several kinds of 2-(4-alkoxyphenyl)-5-alkylpyrimidines and had the phase transition temperatures as follows [9]:



The ferroelectric liquid crystals were prepared by adding 2 mol% of the synthesized chiral compound to this S_C mixture.

2.3. Measurements of ferroelectric properties

Measurements of the phase sequences, the transition temperatures, the magnitude and the polarity of spontaneous polarization and the tilt angle were carried out according to the procedures reported previously [2].

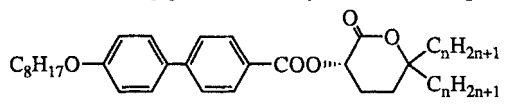
The specimen was placed between two indium–tin oxide (ITO) glass plates to form a cell. The glass surface was coated with a polyimide. The polyimide film was rubbed in one direction. The cell thickness was $2\ \mu\text{m}$ and the applied squarewave voltage was $\pm 10\ \text{V}$. The response time used in this paper was defined as the time required for the optical transmittance through the cell to change from 0 per cent to 50 per cent at 25°C when the voltage was applied to the cell. The helical pitch of the N^* phase was measured at 62°C , just above the S_A to N^* , transition temperature, using the Cano Wedge method.

3. Results and discussion

The melting points of the synthesized compounds are listed in table 1. They do not show any mesophase by themselves.

The ferroelectric properties, such as the phase transition temperatures, the magnitude and the polarity of spontaneous polarization, the tilt angle, the helical pitch of the N^* phase and the response time, are tabulated in table 2. The phase transition temperatures of these ferroelectric liquid crystals are not so different from those of non-chiral S_C mixtures containing chiral 2-(4'-octyloxybiphenyl-4-carboxy)-5-alkyl- δ -valerolactones [6].

Table 1. Melting point of the synthesized compounds.



Compound No.	Chain length (n)	MP $^\circ\text{C}$
1	1	126
2	2	82
3	3	66
4	4	78
5	6	83
6	10	66

Table 2. Ferroelectric properties of the mixtures containing the synthesized compounds.

Compound No.	Transition temperatures/ $^\circ\text{C}$				$P_s/n\text{C}$ $\text{cm}^{-2}\dagger$	Pitch of $\text{N}^*/\mu\text{m}\ddagger$	$\tau/\mu\text{s}\S$	$\theta/^\circ\ddagger$			
	S_C^*	S_A	N^*	I							
1	●	51	●	60	●	67	●	+5.5	17	103	22
2	●	51	●	59	●	66	●	+7.2	39	86	22
3	●	52	●	60	●	66	●	+9.6	> 150	75	22
4	●	52	●	60	●	66	●	+9.4	> 150	71	22
5	●	52	●	59	●	66	●	+11.3	—	70	22
6	●	50	●	57	●	65	●	+11.0	—	76	22

\dagger Measured at 25°C .

\ddagger Measured just above the S_A to N^* transition temperature.

\S Transmittance change from 0 to 50 per cent at 25°C , $V_{\text{p-p}} = 10\ \text{V}\ \mu\text{m}^{-1}$, cell thickness = $2\ \mu\text{m}$.

Figure 1 shows the relationship between the alkyl chain length (n) of three kinds of chiral δ -valerolactone derivatives and the magnitude of the spontaneous polarization of the ferroelectric liquid crystals. The magnitude of the spontaneous polarization of ferroelectric liquid crystals containing 5,5-dialkyl- δ -valerolactone, (A), are much higher than those of ferroelectric liquid crystals with *cis* and *trans* 5-alkyl- δ -valerolactone derivatives, (B) and (C), respectively, for any n . The magnitude of the spontaneous polarization of ferroelectric liquid crystals with 5,5-dialkyl- δ -valerolactone derivatives strongly depends on the alkyl chain length. Moreover, it seems that the magnitude of the spontaneous polarization is nearly proportional to the mole fraction of compounds 2 and 3 in the solvent liquid crystal as shown in figure 2.

The magnitude of the spontaneous polarization of the mixture with 8 mol% of 2-(4'-octyloxybiphenyl-4-carboxy)-5,5-dipropyl- δ -valerolactone is 45 nC cm^{-2} at 25°C .

The polarity of the spontaneous polarization is identical to that of ferroelectric liquid crystals containing 5-alkyl- δ -valerolactone derivatives with the same configuration at the C-2 position.

The response time, which is one of the most important properties for obtaining ferroelectric liquid crystal devices, becomes shorter on lengthening the alkyl chain at the C-5 position (see table 2). In other words, the response time is probably proportional to the magnitude of the spontaneous polarization. However, it seems that the viscosity of these ferroelectric liquid crystals is increased slightly by the addition of these compounds, because the tilt angle of ferroelectric liquid crystals is almost the same (see table 2).

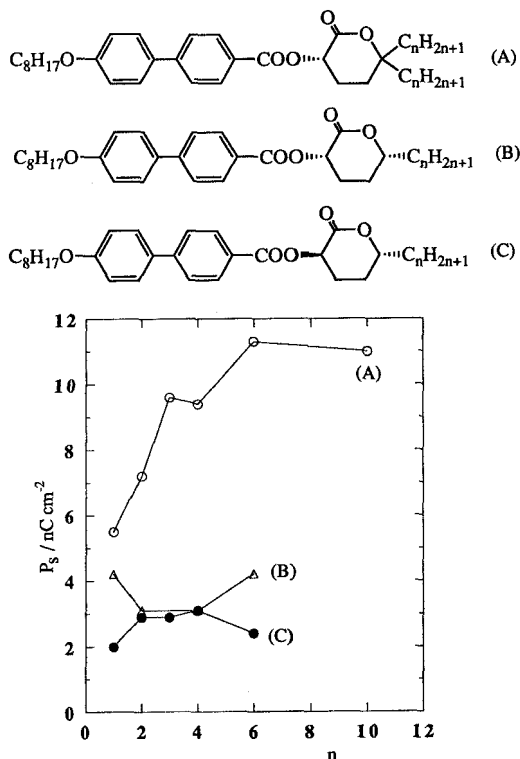


Figure 1. A comparison of P_s versus alkyl chain length of three kinds of δ -valerolactone derivatives.

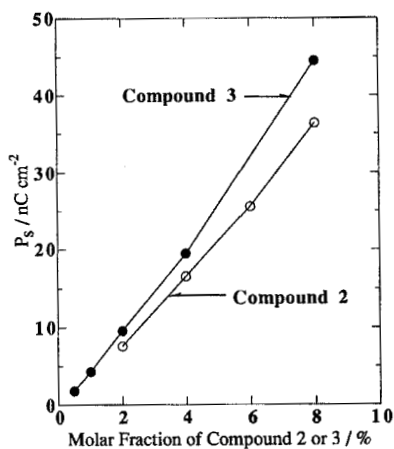


Figure 2. A plot of P_s versus molar fraction of compound 2 or 3.

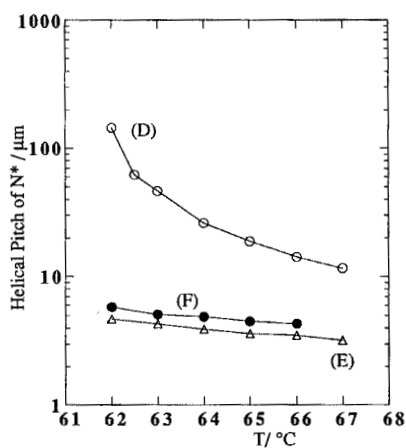
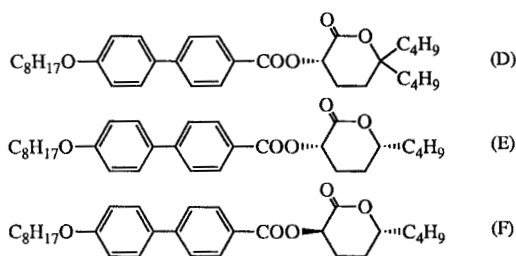


Figure 3. The temperature dependence of the helical pitch of the N^* phase.

The N^* phase of these ferroelectric liquid crystals has an extremely long helical pitch. In figure 3, we compare the temperature dependence of the helical pitch of the N^* phase of the mixtures containing three kinds of δ -valerolactone derivatives with the butyl group at the C-5 position. The helical pitch of the N^* phase of the mixture with compound (D) was over $150 \mu\text{m}$ at 62°C and over 30 times as long as that with compounds (E) and (F).

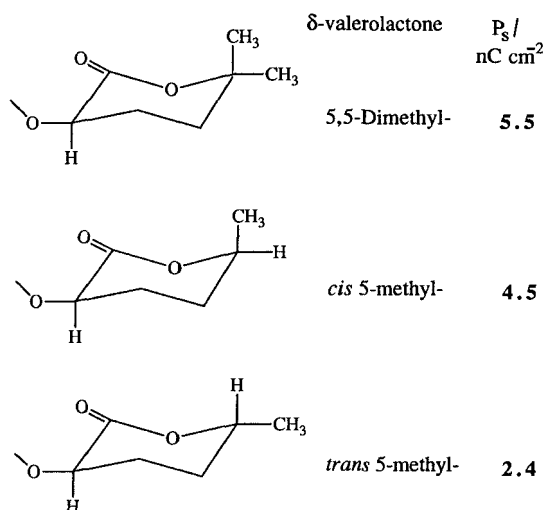


Figure 4. A simple model for the relationship between P_s and the stereochemistry of δ -valerolactones.

Generally speaking, ferroelectric liquid crystals with a high magnitude of spontaneous polarization have a short N^* phase with a helical pitch. However, these 5,5-dialkyl- δ -valerolactone derivatives are the only compounds which induced an N^* phase with a long helical pitch in ferroelectric liquid crystals with a high magnitude of spontaneous polarization. In fact, these compounds are good chiral dopants since they do not need to be compensated by other chiral dopants to obtain good alignment.

It was mentioned previously that the magnitude of spontaneous polarization has a close relationship to the stereochemistry of the chiral dopant. In a recent paper, we have shown that *cis*-5-alkyl- δ -valerolactone derivatives induce a higher magnitude of spontaneous polarization than *trans* ones [6]. Moreover, we have also demonstrated that 5,5-dialkyl- δ -valerolactones induce much higher magnitudes of spontaneous polarization than *cis*-5-alkyl lactones [7]. From these observations, we can show a simple relationship between the stereochemistry of δ -valerolactones and the magnitude of induced spontaneous polarization as follows; suppose the δ -valerolactone ring has the 'chair' conformation with an equatorial substituent at the C-2 position, then configuration of the substituents at the C-5 position are as shown in figure 4.

All chiral dopants inducing a high magnitude of spontaneous polarization have an axial substituent of the C-5 position. It seems that an axial alkyl substituent at the C-5 position plays an important role in inducing spontaneous polarization. However, if this is the case the phenomena would be more complicated. These lactones are probably not in the 'chair' conformation as reported by Philip in the case of the non-substituted δ -valerolactone [10]. Therefore, we studied the stereochemistry of 5,5-dialkyl- δ -valerolactones in solution by means of ^1H NMR measurements in order to discover the real conformation. The chemical shifts and the signal splittings for the C-2 hydrogen of various δ -valerolactones are shown in figure 5.

In a previous paper, we reported the ^1H NMR study of 5-alkyl- δ -valerolactone derivatives with the *cis* and *trans* configuration [6]. In comparison to those results the chemical shifts for the C-2 hydrogen of 5,5-dialkyl- δ -valerolactones with different alkyl

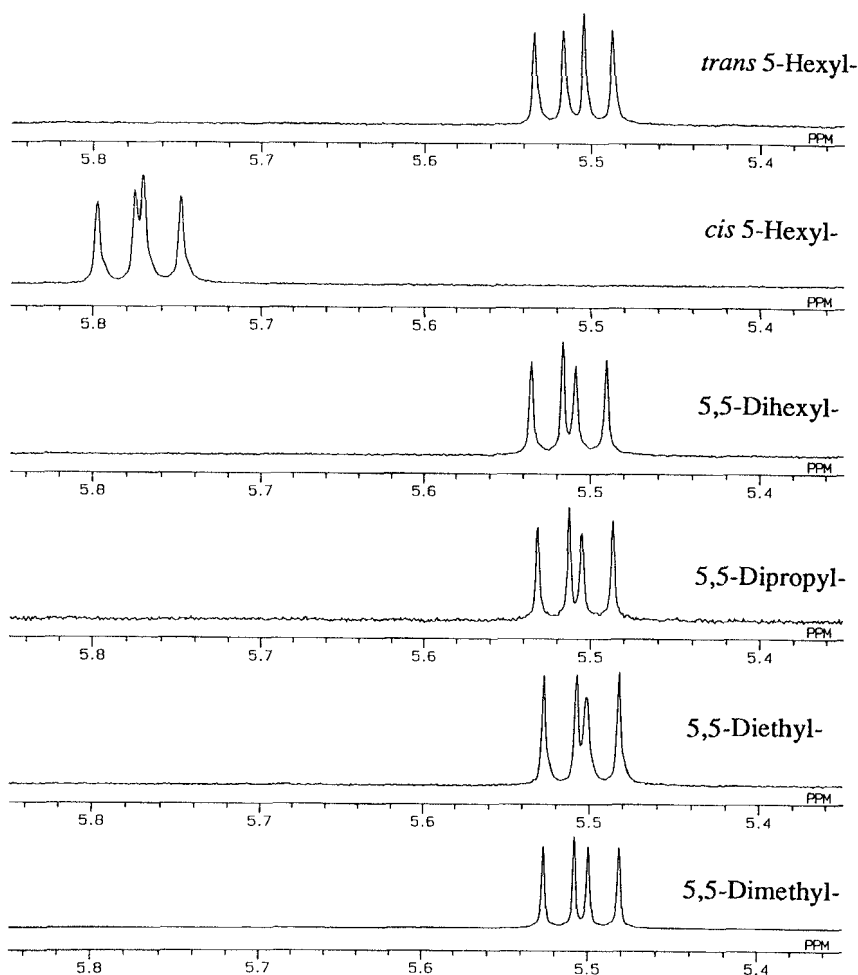


Figure 5. A comparison of the ^1H NMR signal of the C-2 proton of various δ -valerolactones in acetone- d_6 at room temperature.

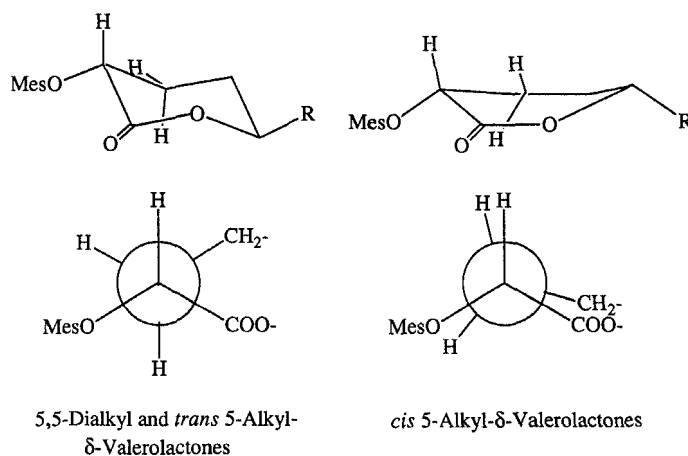


Figure 6. The conformation change of the C-2-C-3 bond of various δ -valerolactones.

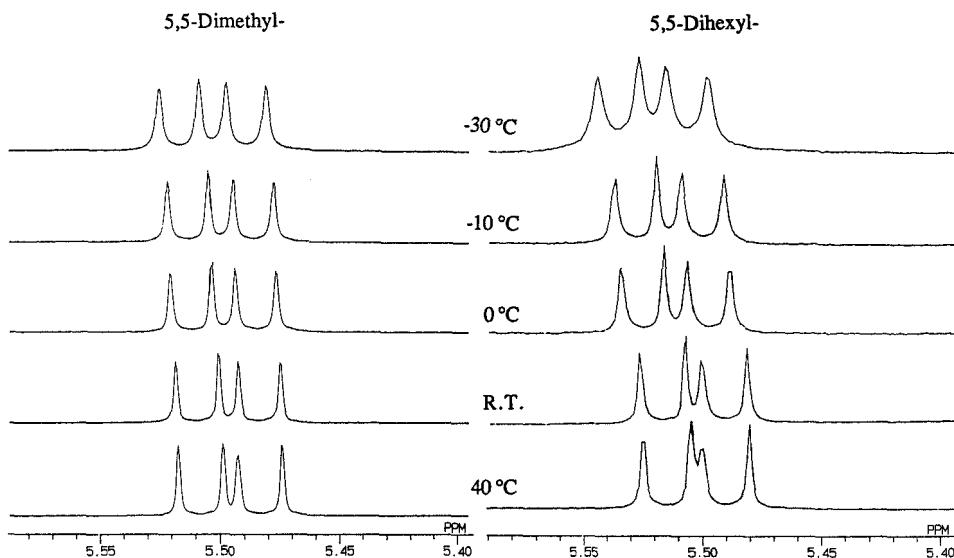


Figure 7. The temperature dependence of the ^1H NMR signal for the C-2 hydrogen of 5,5-dialkyl- δ -valerolactone derivatives in acetone- d_6 .

chains are around 5.50–5.51 ppm and were almost the same as those for *trans*-5-alkyl- δ -valerolactones. The signal splittings for the C-2 hydrogen of both 5,5-dialkyl- δ -valerolactones and *trans*-5-alkyl- δ -valerolactones are double doublets ($J = 4$ Hz, 11 Hz). However, the signal splittings for the C-2 hydrogen of *cis*-5-alkyl- δ -valerolactones are triplet-like double doublets ($J = 3$ Hz, $J = 11$ Hz). Alkyl groups at the C-5 position have different chemical shifts. From these observations, the C-2, C-1, O and C-5 positions of the 5,5-dialkyl- δ -valerolactones appear to form a plane making the magnetic field of the C-2 hydrogen constant. When the conformation of the C-2 hydrogen around the C-2–C-3 bond was changed from ‘staggered’ to ‘eclipsed’ as shown in figure 6, the signal splittings may be changed from triplet-like double doublets to double doublets.

Thus the conformation of 5,5-dialkyl- δ -valerolactones would seem to be ‘pseudo-chair’ in which two alkyl groups are stereochemically non-equivalent. Figure 7 shows the temperature dependence of the chemical shifts and the signal splittings for the C-2 hydrogen of 5,5-dialkyl- δ -valerolactone derivatives.

The chemical shifts and the signal splittings are the same even at -55°C . It appears that the ring structure of the 5,5-dialkyl- δ -valerolactones is more rigid than that of 5-alkyl- δ -valerolactones [6]. Of course, these observations might be true for 5,5-dialkyl- δ -valerolactone derivatives in solution, but may not always be true in liquid crystals.

Since the influence of alkyl chain length of the 5,5-dialkyl- δ -valerolactones on the magnitude of spontaneous polarization is greater than that of 5-alkyl- δ -valerolactones, it seems that the alkyl chain at the C-5 position plays an important role in inducing a high magnitude of spontaneous polarization as mentioned previously. Alkyl chains might exist in the all-*trans* conformation to lower the sterically hindered intrachain mobility resulting from the 5,5-dialkyl- δ -valerolactones. Although we cannot guess the real conformation of these lactones in a liquid crystal, the asymmetric molecular shape of the 5,5-dialkyl- δ -valerolactones might restrict the internal rotation of the lactone



Figure 8. A microphotograph showing the alignment of the ferroelectric liquid crystal cell containing compound 2. The photograph was taken under dark-field illumination and using crossed polarizers (magnification $\times 40$).

Table 3. An example of a practical ferroelectric liquid crystal containing compound 2.

Phase transition temperatures	$C \xleftrightarrow{2^\circ\text{C}} S_C^* \xleftrightarrow{78^\circ\text{C}} S_A \xleftrightarrow{86^\circ\text{C}} N^* \xleftrightarrow{88^\circ\text{C}} I$
$P_s/nC\text{cm}^{-2}$ at 25°C	13.2
$\tau_{0-90}/\mu\text{s}$ at 25°C †	63
$\theta/^\circ$ at 25°C	25
Helical pitch of $N^*/\mu\text{m}$ ‡	> 100

† Thickness of the cell = $2\ \mu\text{m}$, applied voltage = $\pm 8\ \text{V}\ \mu\text{m}^{-1}$.

‡ Measured just above the S_A to N^* transition temperature.

ring around the molecular long axis and heighten the order parameter of the lactone ring dipole. Consequently, more ordered packing of molecules should result in a high magnitude of spontaneous polarization.

There has been no general relationship between the chemical structure of chiral dopants and the helical pitch of the N^* phase. We have shown that the helical pitch induced by *cis*- and *trans*-5-alkyl lactones is as short as for other chiral dopants inducing a high magnitude of spontaneous polarization reported in a previous paper [5]. The helical pitch induced by 5,5-dialkyl lactones is much longer. Although a short helical pitch is a result of strong, namely long distance, chiral–chiral interactions in the case of 5-alkyl lactones, the lack of long distance chiral–chiral interactions results in a longer helical pitch for 5,5-dialkyl lactones. In other words, 5,5-dialkyl lactones do not induce long range chiral–chiral interactions but a strong restriction of the internal rotation of the lactone ring results in a long helical pitch and a high magnitude of spontaneous polarization.

In conclusion, we have made a practical ferroelectric liquid crystal containing a chiral dopant as shown in table 3. It had a short response time and a wide S_C^* range and showed good alignment as shown in figure 8.

The authors would like to thank Dr Mizuo Shindo for his hearty encouragement throughout this work and Dr Koji Takahashi for his helpful suggestions. The authors also wish to thank Mr Noriyuki Komatsu for his measurement of the ^1H NMR spectra.

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