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## Fast response property of low-viscosity difluorooxymethylene-bridged liquid crystals

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In this study, three kinds of low-viscosity difluorooxymethylene-bridged (CF<sub>2</sub>O) liquid crystals (LCs) were synthesised. These materials exhibit low melting point and low viscosity at room temperature. The viscosity of the final LC mixture significantly decreased when LC compounds were mixed with high-birefringence ( $\Delta n$ ) isothiocyanato LCs. The tested curves with varied temperatures of LC mixtures showed that some CF<sub>2</sub>O LC compounds could enhance the response performance of high- $\Delta n$  LCs in a specific temperature range. Therefore, LC compounds with low viscosity could become a component of LC mixtures with fast response performance.

**Keywords:** low viscosity; fast response; liquid crystals

### 1. Introduction

Liquid crystal (LC) devices have great application potential when used as display, light valves, tuneable-focus lens, spatial light modulators and other photonic devices [1–5]. Photonic devices always need fast response speed. The rise time and decay time for homogeneous LC devices are described in the following equations [6]:

$$\tau_{rise} = \frac{\gamma_1 d^2}{K_{11} \pi^2 [(V/V_{th})^2 - 1]} \quad (1)$$

$$\tau_{decay} = \frac{\gamma_1 d^2}{K_{11} \pi^2} \quad (2)$$

where  $\gamma_1$  is the rotational viscosity,  $K_{11}$  is the elastic constant,  $V$  and  $V_{th}$  are turn-on driving and threshold voltages, respectively, and  $d$  is the cell gap. Generally, the rise time can be shortened by the overdriving method. However, the decay time depends on the intrinsic parameters of LC devices, which are the key factors for response performance. According to Equation (2), the low visco-elastic coefficient ( $\gamma_1/K_{11}$ ) and the thin cell gap could give a fast response for LC devices. However, these photonic devices require a definite retardation depth, such as a  $2\pi$  phase change for wavefront corrector. Hence, the cell gap cannot be arbitrarily decreased. Based on the same modulation depth, increasing the birefringence ( $\Delta n$ ) of LC materials allows a thinner cell gap to enhance response speed. Therefore, LC materials with high  $\Delta n$  and low viscosity are needed for these photonic devices.

A series of high- $\Delta n$  LC compounds with a linear shape and a long conjugated group was synthesised to obtain a fast-response LC material. Gauza *et al.* synthesised biphenyl, cyclohexyl-biphenyl isothiocyanato (NCS) LC compounds with an  $\Delta n$  value of 0.2 to 0.4 and a visco-elastic coefficient of approximately  $10 \text{ ms } \mu\text{m}^{-2}$  [7]. Figure-of-merit (*FoM*) [8] was defined to describe the response performance of the LC material in Wu's work (Equation (3)). An NCS LC material with unsaturated substituted groups was synthesised and investigated in 2006 [9]. In this study, the final two NCS LC mixtures showed  $\Delta n$  values of 0.25 and 0.35, a visco-elastic coefficient of approximately  $6 \text{ ms } \mu\text{m}^{-2}$ , and *FoM* values of 10.1 and  $18.7 \text{ } \mu\text{m}^2 \text{ s}^{-1}$ . The response speed of the LC material, which worked in a  $2 \text{ } \mu\text{m}$  device, was  $640 \text{ } \mu\text{s}$  at  $35^\circ\text{C}$ . In subsequent research, a series of fluoro-substituted phenyl-tolane NCS LC materials with an  $\Delta n$  value of up to 0.5 at room temperature was developed, some of which showed better response performances [10]:

$$FoM = K_{11} \Delta n^2 / \gamma_1 \quad (3)$$

The aforementioned studies hardly attempted to develop an LC compound with a small  $\Delta n$  and a low viscosity to formulate a fast-response LC mixture. We previously synthesised difluorooxymethylene-bridged (CF<sub>2</sub>O) LCs [11] with an ultra-low viscosity of approximately 90 cP. Within a certain concentration range, the response performance of the final LC mixture could be improved by mixing it with a high- $\Delta n$  NCS LC [12]. In the present work, three kinds of CF<sub>2</sub>O LC compounds were synthesised and mixed

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into a high- $\Delta n$  LC. The response performance of the final mixtures and the effects of the CF<sub>2</sub>O group on the visco-elastic coefficient of the LC compound were investigated.

## 2. Experimental details

### 2.1 Synthesis

Scheme 1 illustrates the procedure used to synthesise 4'-*n*-propyl-biphenyl- $\alpha$ ,  $\alpha$ -difluoromethyl 3,4,5-trifluorophenyl ether (LC2) [11].

#### 2.1.1 Synthesis of intermediate compound 1

Compounds 4'-*n*-propylbiphenyl-4-carboxylic acid (17.0 g, 0.10 mol) and 1, 3-propanedithiol (14.07 g, 0.13 mol) were dissolved in a mixed solvent of toluene (30 mL) and isooctane (30 mL). Trifluoromethanesulfonic acid (15.59 g, 0.13 mol) was added dropwise in 30 min, and the suspension was stirred at 50°C. The reaction mixture was heated to 100°C at reflux and was stirred for 4 h. The suspension was cooled to room temperature and then filtered. The obtained yellowish solid product is **compound 1**. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>,  $\delta$ /ppm): 0.978 (t, 3H,  $J$  = 14.4 Hz), 1.648–1.716 (m, 2H), 2.625–2.669 (m, 2H), 7.272–7.300 (m, 2H), 7.518–7.575 (m, 2H), 7.634–7.702 (m, 2H), 8.012–8.033 (d, 2H,  $J$  = 8.4 Hz), 8.154–8.175 (d, 2H,  $J$  = 8.4 Hz).

#### 2.1.2 Synthesis of 4'-*n*-propyl-biphenyl- $\alpha$ , $\alpha$ -difluoromethyl 3,4,5-trifluorophenyl ether (LC2)

The compound 3,4,5-trifluorophenol (8.10 g, 55 mmol) was dissolved in a mixture of triethylamine (5.8 g, 58 mmol) and CH<sub>2</sub>Cl<sub>2</sub> (90 mL). The solution was cooled to -70°C. Subsequently, a solution of compound **1** (50 mmol, 23.0 g) dissolved in

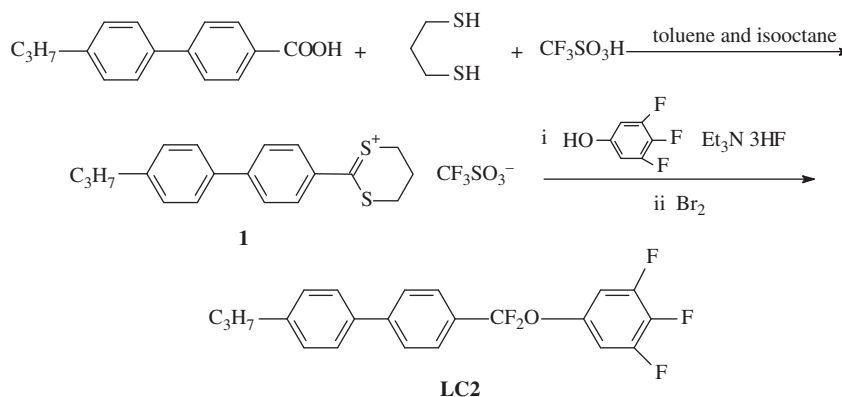
CH<sub>2</sub>Cl<sub>2</sub> (90 mL) was added at the same temperature. The mixture was stirred for 1 h, and Et<sub>3</sub>N 3HF (20 mL, 250 mmol) was added by a syringe. A solution of bromine (40.0 g, 250 mmol) in CH<sub>2</sub>Cl<sub>2</sub> was added dropwise in 30 min at -70°C. The solution was stirred for one more hour at -70°C. The solution was then warmed to room temperature. The solution was poured into an aqueous solution of NaOH (0.8 mol) with ice. The organic layer was collected, and the aqueous layer was washed twice with CH<sub>2</sub>Cl<sub>2</sub>. The organic solution was dried over anhydrous MgSO<sub>4</sub>. After the solvent was removed, the crude product was purified by column chromatography (silica gel/petroleum ether) to give a white solid with a 50% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>,  $\delta$ /ppm): 0.985 (t, 3H,  $J$  = 14.4 Hz), 1.649–1.742 (m, 2H), 2.651 (t, 2H,  $J$  = 15.2 Hz), 6.958–6.993 (m, 2H), 7.278–7.298 (d, 2H,  $J$  = 8.0 Hz), 7.523–7.543 (d, 2H,  $J$  = 8.0 Hz), 7.675–7.747 (m, 4H).

The other CF<sub>2</sub>O compounds, LC1 and LC3, were synthesised through the similar reaction process. The NMR results are listed as follows:

**4'-*n*-propyl-cyclohexyl- $\alpha$ ,  $\alpha$ -difluoromethyl 3,4-difluorophenyl ether (LC1):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>,  $\delta$ /ppm): 7.45–7.43 (d, 1H), 7.19–7.15 (m, 2H), 2.64–2.59 (m, 1H), 1.69–1.55 (m, 1H), 1.35–0.81 (m, 15H).

**4'-*n*-butyl-tolan- $\alpha$ ,  $\alpha$ -difluoromethyl 4-fluorophenyl ether (LC3):** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>,  $\delta$ /ppm): 0.935 (t, 3H,  $J$  = 14.4 Hz), 1.334–1.390 (m, 2H), 1.591–1.610 (m, 2H), 2.633 (t, 2H,  $J$  = 15.6 Hz), 7.031–7.074 (m, 2H), 7.170–7.190 (d, 2H,  $J$  = 8.0 Hz), 7.213–7.247 (m, 2H), 7.450–7.470 (d, 2H,  $J$  = 8.0 Hz), 7.597–7.618 (d, 2H,  $J$  = 8.4 Hz), 7.675–7.696 (d, 2H,  $J$  = 8.4 Hz).

For comparison, the LC compounds without the CF<sub>2</sub>O group were synthesised through the Suzuki couple reaction of substituted boronic acids and substituted bromobenzene, respectively.



Scheme 1. Synthesis of CF<sub>2</sub>O compound.

**4'-*n*-propyl-cyclohexyl-3,4-difluorobenzene (LC4):**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ): 0.80–1.45 (m, 15H), 1.60–1.75 (m, 1H), 2.62–2.66 (m, 1H), 7.20–7.28 (m, 2H), 7.38–7.48 (d, 1H,  $J = 8.0$  Hz).

**4''-*n*-propyl-3,4,5-trifluoroterphenyl (LC5) [13]:**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ,  $\delta/\text{ppm}$ ): 0.983 (t, 3H,  $J = 14.8$  Hz), 1.664–1.720 (m, 2H), 2.644 (t, 2H,  $J = 15.2$  Hz), 7.212–7.288 (m, 4H), 7.535–7.569 (m, 4H), 7.658–7.679 (d, 2H,  $J = 8.4$  Hz).

## 2.2 Characterisation techniques of an LC compound

The chemical structure was confirmed by  $^1\text{H}$  NMR spectra using a Bruker 400 MHz spectrometer (av400), with  $\text{CDCl}_3$  as the solvent and TMS as the internal standard. The phase transition temperature was detected on a differential scanning calorimetry (DSC) instrument (CDR-4P) at a heating rate of  $10^\circ\text{C min}^{-1}$ .

## 2.3 Response parameter measurement of LC mixture

The melting points of the three kinds of  $\text{CF}_2\text{O}$  LCs were very different. Each compound was mixed into a high- $\Delta n$  LCs at 10.0 wt% for measurements of  $\Delta n$ ,  $\gamma_1/K_{11}$ , and  $FoM$ . The high  $\Delta n$  LC is the mixture of isothiocyanato LCs, SG2, which consists of 4'-alkyl-cyclohexyltolaneisothiocyanate, 4'-alkyl-4-isothiocyanatetolanes, and 4'-alkyl-terphenyl-4-isothiocyanate [14]. Each LC mixture was injected to a homogeneous cell of  $7.78\ \mu\text{m}$  (pretilt angle of alignment film:  $\sim 1.0^\circ$ ). At a specific temperature, the  $\Delta n$  and visco-elastic coefficient were determined using an optical method. The LC cells were sandwiched between two crossed polarisers. The LC directors without electric field were located  $45^\circ$  to the polarising direction of the polariser, whereas the incident light ( $\lambda = 589\ \text{nm}$ ) direction was perpendicular to the substrate. When the applied voltage ( $\pm 10\ \text{V}$ , square wave, 1000 Hz) was released, transient light intensity was recorded using a photomultiplier and an oscillograph. Thus, the phase retardation of decay response was also obtained. The  $\Delta n$  value of the LC mixture at 589 nm was obtained by measuring the phase retardation of the LC cell. The value of  $\tau_{\text{decay}}$  was calculated according to the following equation through the fitting method. The visco-elastic coefficient was calculated through Equation (4) [15]:

$$\delta(t) = \delta_0 \exp(-2t/\tau_{\text{decay}}) \quad (4)$$

where  $\delta_0$  is the initial relative phase retardation.

## 3. Results and discussion

### 3.1 Mesomorphic properties

Table 1 lists the phase transition temperatures of the five LC compounds that were investigated. The

melting points of  $\text{CF}_2\text{O}$  LCs (LC1 and LC2) are lower compared with those of their corresponding biphenyl LCs (LC4 and LC5):

$$\text{LC1} < \text{LC4} \quad \text{LC2} < \text{LC5}$$

The  $\text{CF}_2\text{O}$  compounds show weak intermolecular force, which decreases the melting point of the LCs. When a practical LC mixture is formed, the component with a low melting point is useful. The  $\text{CF}_2\text{O}$  compound will decrease the melting point of the LC mixture for applications at low air temperature.

### 3.2 Birefringence

The guest–host method determines the  $\Delta n$  of the LC compound. The  $\text{CF}_2\text{O}$  compounds were dissolved in the host LC mixture, SG2, at 10 wt%. The  $\Delta n$  of the  $\text{CF}_2\text{O}$  LC compound at  $25^\circ\text{C}$  can be calculated as follows:

$$(\Delta n)_{gh} = x(\Delta n)_g + (1 - x)(\Delta n)_h \quad (5)$$

where  $gh$ ,  $g$ , and  $h$  denote the guest–host system, guest LC, and host LC mixture, respectively, and  $x$  is the concentration of the guest LC. Table 1 shows the extrapolated  $\Delta n$  values of the LC compounds. The sequence of the  $\Delta n$  value is as follows:

$$\text{LC3} > \text{LC2} > \text{LC1}$$

The tolan group in LCs has richer  $\pi$ -electrons than the biphenyl and cyclohexyl groups. Therefore, LC3 has the highest  $\Delta n$  value among the  $\text{CF}_2\text{O}$  LC compounds. This phenomenon is similar to the results for common LC compounds.

The  $\Delta n$  value of the terphenyl LC compound was evidently higher than that of the corresponding  $\text{CF}_2\text{O}$  LC compound:

$$\text{LC5} > \text{LC2}$$

The effective  $\pi$ -electron conjugation length of  $\text{CF}_2\text{O}$  LC2 is shorter than that of terphenyl LCs, because the  $\text{CF}_2\text{O}$  linking group breaks the molecular conjugation. Hence, the  $\Delta n$  value of the SG2+LC2 mixture is lower than that of SG2+LC5 throughout the whole temperature range (Figure 1(b)).

The  $\Delta n$  values of LC compounds were also calculated using a theoretical method, as presented in Table 2. The values of polarisability ( $\Delta\alpha$ ,  $\alpha$ ) were calculated from  $\alpha_{xx}$ ,  $\alpha_{yy}$ , and  $\alpha_{zz}$  that come from the semi-empirical AM1 method acting on the relevant chemical structure. The  $\Delta n$  values were enumerated

Table 1. Chemical structures and properties for LC compounds.

LC No.	Structure	Phase transition temperature (°C)	$\Delta n^*$	$T_c^{**} (°C)$
1		< -60	0.03	94.2
2		Cr 73.6 I	0.14	98.6
3		Cr 47.5 N 73.6 I	0.21	117.0
4		Cr 23.0 I	0.01	90.3
5		Cr 103.7 I	0.22	107.4

\*Host mixture: SG2,  $T_c = 112.6^\circ\text{C}$ ; test temperature:  $25^\circ\text{C}$ . \*\*Clear point of SG2 mixed with LCx ( $x = 1-5$ ) at 10.0 wt%.

Table 2. Calculated polarisability and  $\Delta n$  of the  $\text{CF}_2\text{O}$  compounds.

	$\Delta\alpha$	$\alpha$	$\alpha_{xx}$	$\alpha_{yy}$	$\alpha_{zz}$	$\Delta n$ (cal)
LC1	83.581	145.960	201.684	146.091	90.114	0.085
LC2	220.234	223.125	369.947	157.850	141.577	0.16
LC3	329.883	267.317	487.239	186.283	128.430	0.24
LC4	65.269	128.265	171.778	133.056	79.961	0.084
LC5	236.052	212.396	369.764	197.109	70.315	0.21

using the Vuks formula [16], while the density and the order parameter  $S$  were assumed to be  $1.0 \text{ g cm}^{-3}$  and 0.6, respectively. The calculated  $\Delta n$  values of LC2, LC3 and LC5 are close to their experimental data

in Table 1. However, the experimental  $\Delta n$  values of LC1 and LC4 are much smaller than the calculated values, because the clear point of the host mixture mixed LC1 (or LC4) was obviously decreased, as shown in Table 1, and the order parameter of the LC1 (or LC4) mixture at  $25^\circ\text{C}$  is lower than that of host mixture. Thus, the guest-host birefringence of LC1 and LC4 is quite small.

Figure 1(a) shows the temperature-dependent  $\Delta n$  value of SG2 that was mixed with different  $\text{CF}_2\text{O}$  LCs. The  $\Delta n$  value of the LC mixture with  $\text{CF}_2\text{O}$  LCs became smaller than the pure SG2 at most of the test temperatures because the  $\Delta n$  value of  $\text{CF}_2\text{O}$  LCs is lower. The experimental data of the LC

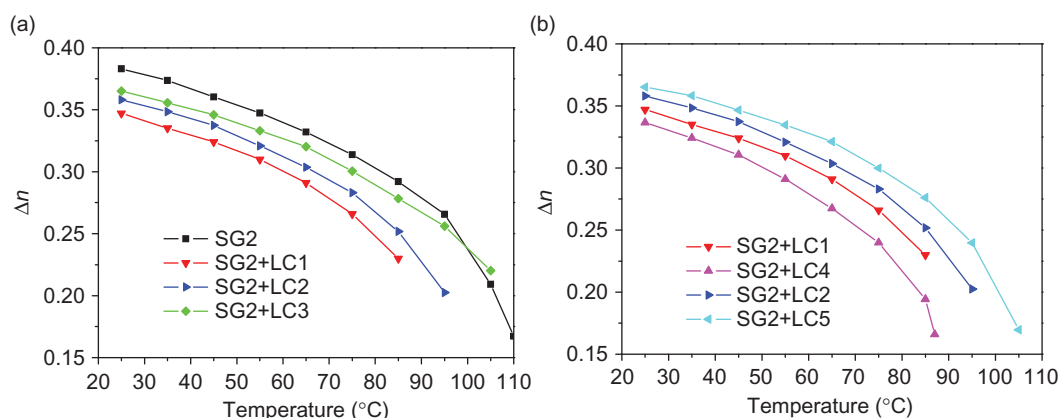


Figure 1. Temperature-dependent  $\Delta n$  of different LC mixtures. (a) SG2 host and  $\text{CF}_2\text{O}$  LC compounds dissolved in the host, (b) comparison of guest LCs with and without the  $\text{CF}_2\text{O}$  group dissolved in SG2. The dots are experimental data, and the lines are connecting lines.



mixture as a function of temperature can be fitted with Equation (6) [17], where the  $\beta$  values of SG2, SG2+LC1, SG2+LC2, and SG2+LC3 were calculated as 0.239, 0.205, 0.210, 0.252, respectively:

$$\Delta n \cong \Delta n_0(1 - T/T_c)^\beta \quad (6)$$

### 3.3 Visco-elastic coefficient

Figure 2(a) shows the visco-elastic coefficient value of the SG2 host and SG2 mixed with CF<sub>2</sub>O LCs. When the CF<sub>2</sub>O LCs were mixed with SG2, the visco-elastic coefficient of the LC mixture decreased obviously. As a result, the visco-elastic coefficient of SG2+LC1 is 8.19 ms  $\mu\text{m}^{-2}$ , whereas the value of SG2 is 9.85 ms  $\mu\text{m}^{-2}$  at 25°C. The sequence of  $\gamma_1/K_{11}$  is:

$$\text{SG2} > \text{SG2} + \text{LC2} >> \text{SG2} + \text{LC3} > \text{SG2} + \text{LC1}$$

The rotational viscosity of the LC mixture can be approximately expressed as the arithmetic average of the components in low concentration [18]. The sequence above indicates that LC1 has a lower viscosity than LC3, LC2, where  $K_{11}$  is assumed as a constant. Anyway, LC1 can decrease the viscosity of host LCs more sharply. The  $\gamma_1/K_{11}$  values of all the LC mixtures became close as the temperature increased to 55°C.

In the comparison of the  $\gamma_1/K_{11}$  values of the LC compound with and without the CF<sub>2</sub>O group, the experimental data indicate that the LCs with the CF<sub>2</sub>O group have a lower visco-elastic coefficient (Figure 2(b)).

### 3.4 FoM

The FoM value can be used to compare the response performance of LC materials which is calculated

according to Equation (3) by  $\Delta n$  and  $\gamma_1/K_{11}$ . Figure 3(a) shows the temperature-dependent FoM value of different CF<sub>2</sub>O LC compounds that were dissolved in SG2. At 25°C, the values of SG2, SG2+LC3, SG2+LC1, and SG2+LC2 were 14.8, 15.9, 14.7, and 13.5  $\mu\text{m}^2 \text{s}^{-1}$ , respectively. Among these LCs, the mixture of SG2+LC3 has the highest FoM value because LC3 has a lower visco-elastic coefficient and a moderated  $\Delta n$  value. Thus, LC3 could improve the response performance of SG2. However, LC1 maintained and LC2 degraded the response performance of SG2 at 25°C. At test temperatures greater than 45°C, the FoM values of SG2 mixed with the CF<sub>2</sub>O LC compounds are all less than for pure SG2 because the visco-elastic coefficients of the four LC mixtures are similar; furthermore, the  $\Delta n$  value of pure SG2 is the largest.

Figure 3(b) shows the temperature-dependent FoM value of the SG2-mixed LC compound with and without the CF<sub>2</sub>O group. The CF<sub>2</sub>O LC mixtures SG2+LC2 and SG2+LC1 have larger FoM values in the low temperature region because their visco-elastic coefficients are lower:

$$\text{LC2} > \text{LC5} \quad \text{LC1} > \text{LC4}$$

The FoM value of SG2+LC5 is larger than that of SG2+LC2 when the temperature is higher than 65°C because the  $\Delta n$  value of SG2+LC5 is larger than that of SG2+LC2 and the visco-elastic coefficient of SG2+LC5 is as low as that of SG2+LC2.

## 4. Conclusions

Three difluorooxymethylene-bridged LCs with different  $\Delta n$  values were synthesised. The visco-elastic coefficient of host LC mixtures decreased at different degrees when the CF<sub>2</sub>O LC compounds were mixed

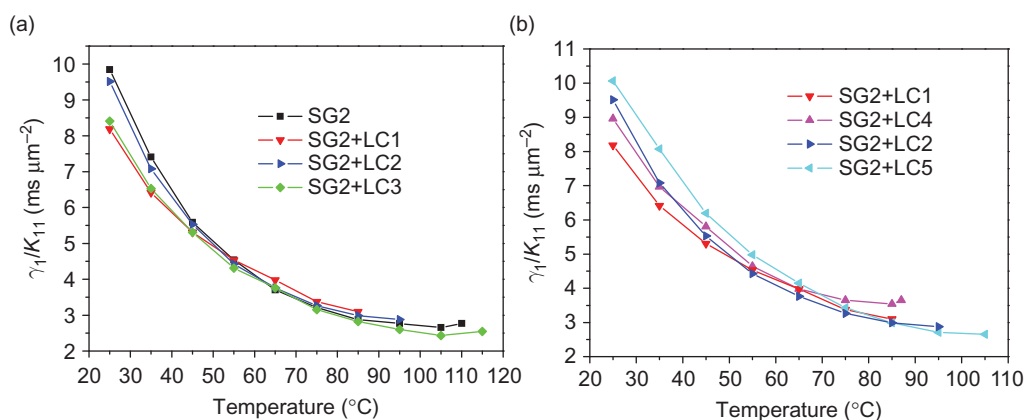


Figure 2. Temperature-dependent visco-elastic coefficient of different LC mixtures. (a) SG2 host and CF<sub>2</sub>O LC compounds dissolved in the host, (b) comparison of guest LCs with and without the CF<sub>2</sub>O group dissolved in SG2. The dots are experimental data, and the lines are connecting lines.

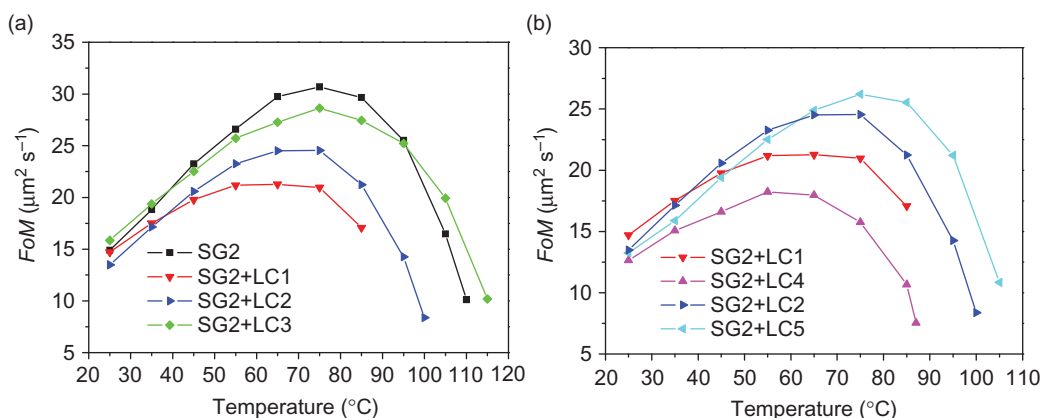


Figure 3. Temperature-dependent  $FoM$  values of the different LC mixtures. (a) SG2 host and CF<sub>2</sub>O LC compounds dissolved in the host, (b) comparison of guest LCs with and without the CF<sub>2</sub>O group dissolved in SG2. The dots are experimental data, and the lines are connecting lines.

at high  $\Delta n$  isothiocyanato LCs. The CF<sub>2</sub>O LC, 4'-*n*-butyl-tolan- $\alpha$ ,  $\alpha$ -difluoromethyl 4-fluorophenyl ether, could enhance the  $FoM$  value of the NCS LCs at room temperature. Therefore, some CF<sub>2</sub>O LC materials can be used in fast-response LC photonic devices.

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