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Physical properties of chlorinated liquid crystals

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Physical properties of chlorinated liquid crystals

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Dielectric, optical and electro-optical properties of four chlorinated nematic liquid crystal compounds and a eutectic mixture were characterized. Some chlorinated liquid crystals are found to exhibit a wide nematic range, modest dielectric and optical anisotropies, low viscosity and small UV absorption. Potential application of mixtures containing chlorinated liquid crystals for information displays in the visible spectral region is foreseeable.

1. Introduction

Nematic liquid crystals with a cyano terminal group [1, 2] have been used extensively for information displays. The main feature of liquid crystals with the cyano group is their large dielectric anisotropy ($\Delta\varepsilon$). A large dielectric anisotropy helps to lower the operation voltage of a liquid crystal device. However, liquid crystals with the cyano group are found to exhibit a relatively large viscosity which, in turn, results in slow response times of the liquid crystal device. Also, the photostability of these materials is generally unsatisfactory for long term application [3].

Fluorinated liquid crystals have been recently developed with the intention of replacing liquid crystals with a cyano group [4–7]. The dielectric and optical anisotropies of fluorinated liquid crystals are somewhat lower, but their viscosity and photostability are much better than the corresponding materials with a cyano group. However, the nematic temperature range of the fluorinated compounds is not ideal: many fluorinated liquid crystals show a wide smectic phase before nematic, and some are not even mesogenic [5].

In this paper, we report some important physical properties of four chlorinated nematic liquid crystals and a eutectic mixture. In section 2, the phase transition temperatures and heat diffusion enthalpy of these materials are given. Measurements and results of dielectric anisotropy, UV absorption, birefringence, splay elastic constant, order parameter, and rotational viscosity are described in section 3.

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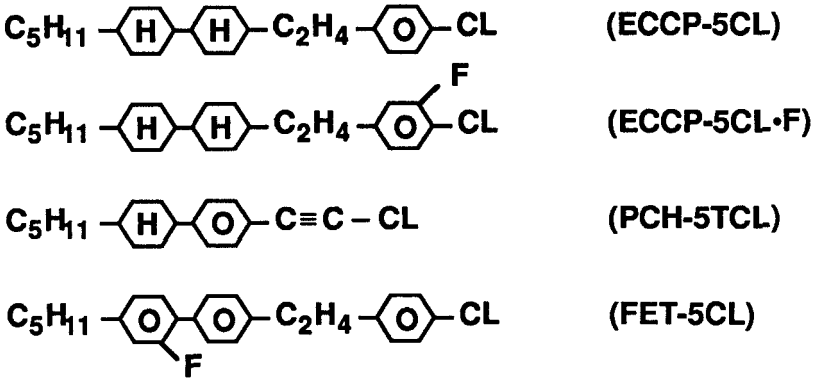
2. Meso phases

The structures and abbreviations for the four chlorinated liquid-crystalline compounds studied are shown below:



The synthetic methods for ECCP-5CL, ECCP-5CL.F and FET-5CL have been reported in [8] and for PCH-5TCL in [9]. The transition temperatures were identified using a Mettler hot plate under a polarizing microscope, and the heat diffusion enthalpies were measured by differential scanning calorimetry. Results are listed as follows:

$$\text{ECCP-5CL C} \xrightarrow[\Delta H = 4{\cdot}1\ \text{kcal mol}^{-1}]{57{\cdot}6^{\circ}\text{C}} \text{N} \xrightarrow{161{\cdot}3^{\circ}\text{C}} \text{I}$$

$$\text{ECCP-5CL.F C} \xrightarrow[\Delta H = 6{\cdot}3\ \text{kcal mol}^{-1}]{39{\cdot}9^{\circ}\text{C}} \text{N} \xrightarrow{142{\cdot}5^{\circ}\text{C}} \text{I}$$

$$\text{FET-5CL C} \xrightarrow[\Delta H = 5{\cdot}4\ \text{kcal mol}^{-1}]{50{\cdot}9^{\circ}\text{C}} \text{N} \xrightarrow{79{\cdot}5^{\circ}\text{C}} \text{I}$$

$$\text{PCH-5TCL C} \xrightarrow[\Delta H = 5{\cdot}3\ \text{kcal mol}^{-1}]{66^{\circ}\text{C}} \text{N} \xrightarrow{70^{\circ}\text{C}} \text{I}$$

$$\text{Mixture C} \xrightarrow{8^{\circ}\text{C}} \text{N} \xrightarrow{109^{\circ}\text{C}} \text{I}$$

where C, N and I represent crystal, nematic and isotropic phases, respectively. The eutectic mixture listed above consists of 42 wt% ECCP-5CL, 38 wt% FET-5CL and 20 wt% PCH-5TCL. The refractive indices of this mixture were measured using an Abby refractometer at $\lambda = 589$ nm and $T = 22°C$ and were found to be $n_e = 1{\cdot}665$ and $n_0 = 1{\cdot}503$. From the transition temperatures it is seen that most chlorinated liquid-crystalline compounds exhibit a relatively wide nematic range except PCH-5TCL. Owing to the narrow nematic range of PCH-5TCL, its physical properties were not characterized in its normal nematic phase, rather, they were extrapolated from the results measured in a host, ZLI-1132. Some of the derived results are listed in table 1.

3. Physical properties

In this section, the dielectric constants, UV absorption spectra, birefringence, splay elastic constants and rotational viscosities of the compounds and the mixture studied are reported.

3.1. *Dielectric constants*

3.1.1. *Experimental*

The dielectric constants ($\varepsilon_{\parallel}$ and $\varepsilon_{\perp}$) of a liquid-crystalline compound can be measured by a commonly used two cell method [10]. In this method, two liquid crystal

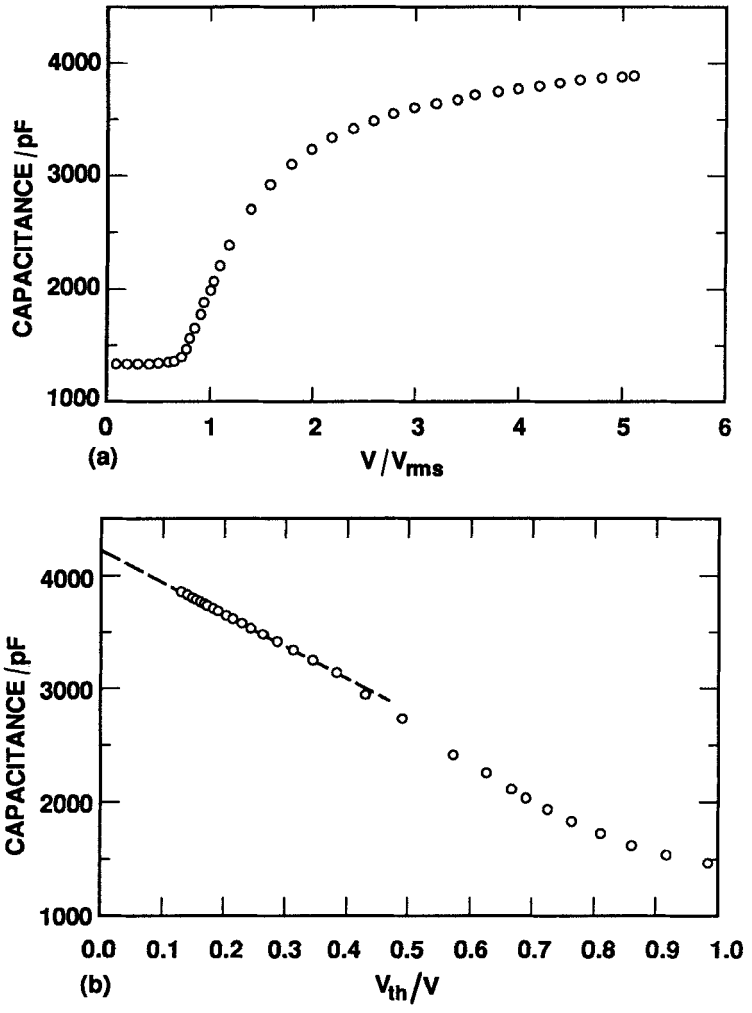


Figure 1. (*a*) Voltage dependent capacitance of a parallel aligned 5CB cell. The dimension of the patterned electrodes is 13 × 25 mm^2. The cell thickness is 13 μm. The empty cell capacitance was measured to be 220 pF at T = 22°C and 1 kHz frequency. From this figure, V_{th} is found to be 0·69 V_{rms}. (*b*) Data shown in (*a*) replotted as a function of V_{th}/V. A linear relationship below $V_{th}/V < 0{\cdot}35$ is observed. From the extrapolation, $C_{\parallel}$ is found to be 4237 pF.

Table 1. Some important physical properties of PCH-5TCL. Results are extrapolated from a guest–host system: 10 per cent of PCH-5TCL mixed in ZLI-1132. v represents the flow viscosity.

Liquid crystal	$\varepsilon_{\parallel}$ (20°C, 1 kHz)	$\varepsilon_{\perp}$ (20°C, 1 kHz)	n_e (589 nm, 20°C)	n_0 (589 nm, 20°C)	v (20°C)/ mm^2 S^{-1}	v (0°C)/ mm^2 S^{-1}
PCH-5TCL	8·7	2·0	1·70	1·49	13	58

cells with patterned electrodes but different alignments (one parallel and one perpendicular) were prepared. By measuring the capacitance of each cell before and after being filled with a liquid crystal, both $\varepsilon_{\parallel}$ and $\varepsilon_{\perp}$ can be obtained from the ratio of the measured capacitance of each cell. Although this technique is simple, its accuracy depends on how well the liquid crystal is aligned on the substrates. For the liquid crystals listed previously, we found all of them to align well on substrates with parallel alignment treatment, but only FET-5CL aligns on substrates with perpendicular alignment treatment. We tried several different surface alignment agents, including lecithin and long chain alcohols, but none of them works for ECCP-5CL, ECCP-5CL.F and the mixture. Therefore, an alternative technique for measuring $\varepsilon_{\parallel}$ is needed.

The second method we employed, called the single cell method, has been used previously [11] and later analysed theoretically by Welford and Sambles [12]. In this single cell method, only a parallel aligned cell with patterned electrodes is required. For the cell we used, the size of the patterned electrodes was $13 \times 25\,\text{mm}^2$. The cell gap was measured by the interferometric method. Typically, the cell thickness is $13\,\mu\text{m}$. The capacitance (C_0) of the empty cell was measured to be 220 pF, which is insensitive to temperature within the range of our study (from 20 to 95°C). The cell was then filled with liquid crystal by the capillary method. After equilibrium, the voltage dependent capacitance was recorded. The instrument we used was a HP-4274A multifrequency LCR meter. Results of 5CB (4-pentyl-4′-cyano-biphenyl) at $T = 22$°C are shown in figure 1(*a*) as an example.

At voltages below the threshold (V_{th}), the measured capacitance represents $C_{\perp}$ because the electric field is normal to the liquid crystal director. The ratio of $C_{\perp}/C_0$ gives $\varepsilon_{\perp}$ as 6·17. Also, from figure 1(*a*), we obtain V_{th} equal to $0{\cdot}69\,V_{\text{rms}}$ at 1 kHz. This value agrees well with that obtained from the voltage dependent optical transmission method.

To obtain $C_{\parallel}$, we cannot simply extrapolate the capacitance from figure 1(*a*) to $V \to \infty$ because C is a non-linear function of V. Rather, we plot the capacitance data as a function of V_{th}/V, as shown in figure 1(*b*). A linear dependence between the capacitance and V_{th}/V, as indicated by dashed lines, is observed at $V_{\text{th}}/V < 0{\cdot}35$ or at $V > 3\,V_{\text{th}}$. This linear dependence has been found experimentally [11] and predicted theoretically [12]. From the intersection of the dashed lines and the vertical axis (i.e. $V_{\text{th}}/V = 0$), $C_{\parallel}$ is found to be 4237 pF or $\varepsilon_{\parallel}$ is 19·26. From the two cell method, we obtained $\varepsilon_{\parallel}$ to be 18·89; the agreement is within 2 per cent.

3.1.2. *Temperature and frequency effects*

The temperature dependence of the dielectric constants of FET-5CL, the mixture, ECCP-5CL and ECCP-5CL.F are shown in figure 2. From figure 2, the value for $\Delta\varepsilon$ for ECCP-5CL, ECCP-5CL.F, and FET-5CL is found to be 2·7, 5·6 and 6·4, respectively at $T_r = 0{\cdot}8$; T_r is defined as T/T_C where T_C is the clearing point. Comparing ECCP-5CL.F and ECCP-5CL, the lateral fluorine substitution enhances the dielectric anisotropy by about two times. Furthermore, the melting point of ECCP-5CL.F is lower than that of ECCP-5CL by nearly 20°C. However, this lateral fluorine also affects the absorption spectrum and increases the rotational viscosity, as discussed later.

The frequency dependent dielectric constants for each compound were measured from $f = 120$ Hz to 100 kHz. Below 40 kHz, both $\varepsilon_{\parallel}$ and $\varepsilon_{\perp}$ are independent of frequency, whereas above 40 kHz they gradually decrease. The crossover frequency (where $\Delta\varepsilon$ changes sign) of these compounds is extrapolated to be in the 5–10 MHz region.

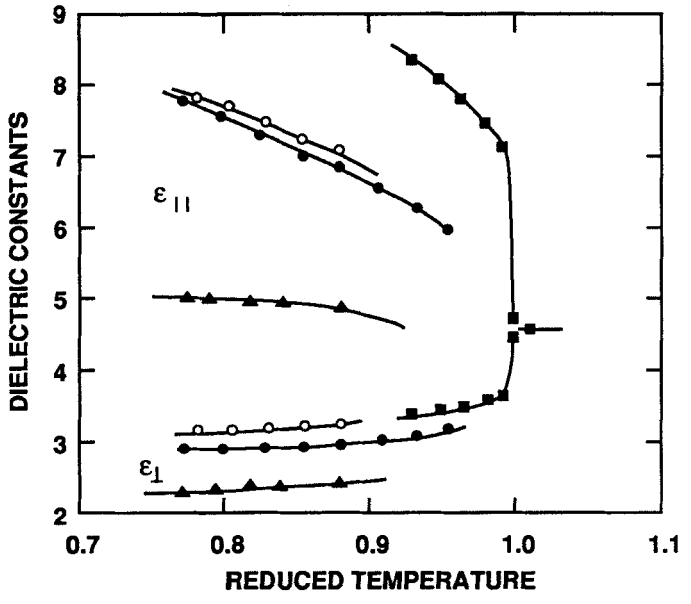


Figure 2. The temperature dependent dielectric constants of the liquid crystals studied. ■, FET-5CL; ○, ECCP-5CL.F; ●, Mixture; ▲, ECCP-5CL.

3.2. *UV absorption spectra*

The UV absorption spectra of the previously mentioned four chlorinated liquid-crystalline compounds are shown in figure 3. The measurement technique has been reported in [13]. Here, we focus on the comparison of two groups:

(1) [ECCP-5CL, ECCP-5CL.F];
(2) [FET-5CL, PCH-5TCL].

The similar UV absorption spectra for ECCP-5CL and ECCP-5CL.F is a consequence of their similar structures. A careful examination reveals that the lateral fluorine substitution slightly modifies the absorption spectrum: it reduces the oscillator strength of the λ_1 band ($\lambda_1 \cong 194$ nm) but increases the absorption in the 250–280 nm region. A small decrease in the oscillator strength of the λ_1 band has little effect on birefringence [14], but the higher absorption in the longer UV region certainly deteriorates the photostability of the liquid crystal if it is exposed to UV radiation.

It should be mentioned here that the electronegativity of a chlorine atom is not so strong as that of a fluorine. Therefore, the blue shift [15] as observed in a terminal chlorinated liquid crystal is less than that of a corresponding fluorinated material. That is to say, the resonance absorption wavelengths of the chlorinated liquid crystal are longer than those of the corresponding fluorinated compound. As a result, the birefringence of a chlorinated liquid crystal is expected to be higher than that of the corresponding fluorinated material.

The structure of FET-5CL seems complicated at first glance. But actually it is not too difficult to understand. The ethylene linking group in FET-5CL breaks the molecular conjugation into two parts: the fluorinated-biphenyl and the chlorinated-phenyl. Experimental results [13] shows that the absorption of a biphenyl is similar to that of a phenyl ring plus a triple bond, for example as that of PCH-5TCL. The

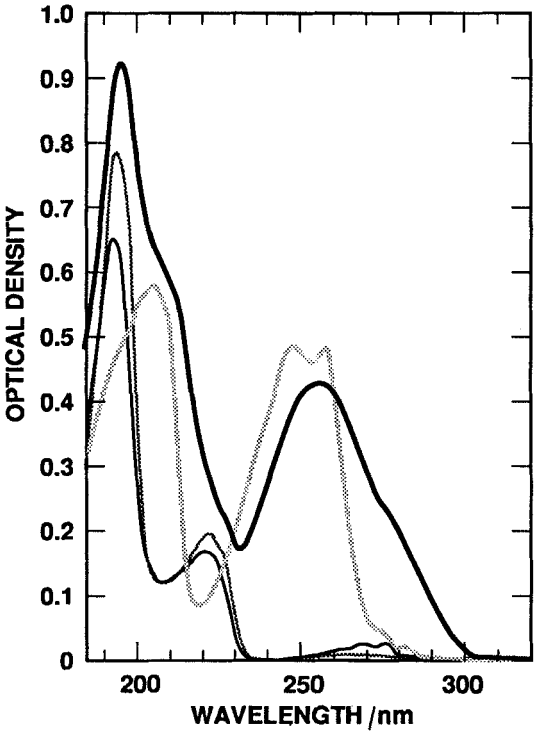


Figure 3. The UV absorption spectra of the liquid crystals studied. One per cent of the liquid crystal was mixed in a host (ZLI-2359). The cell thickness is 6 μm. T_r = 0·865. An identical cell containing only host liquid crystal was used as a reference in all measurements. Thick line, FET-5CL; dotted line, PCH-5TCL; wavy line, ECCP-5CL; thin line, ECCP-5CL.F.

chlorinated phenyl ring (its π electron conjugation length is identical to that of ECCP-5CL) shows a strong absorption band centred at λ_1 approximately equal to 194 nm and a weak band at λ_2 equal to 220 nm. Thus, the absorption spectrum of FET-5CL should be nearly the same as the sum of PCH-5CL and ECCP-5CL. This expectation is confirmed by the spectra shown in figure 3. The dichroic ratio of the λ_2 band (centred at about 250 nm) of FET-5CL and PCH-5TCL was measured and found to be about six for T_r equal to 0·865. As the temperature increases, the dichroic ratio decreases and eventually vanishes at the clearing point.

3.3. *Birefringence*

A comparison of the birefringence for the different liquid-crystalline materials is meaningful only when it is made at the same reduced temperature and the same wavelength. The temperature and wavelength dependent birefringence were measured for the liquid crystals studied except for PCH-5TCL due to its narrow temperature range.

3.3.1. *Temperature effect*

The temperature dependent birefringence of four samples was measured at λ = 633 nm and the results are shown in figure 4 (*a*). The solid lines in figure 4 (*a*) were fitted to the data using a semi-empirical formula [16]

$$\Delta n = \Delta n_0 S \qquad (1\,a)$$

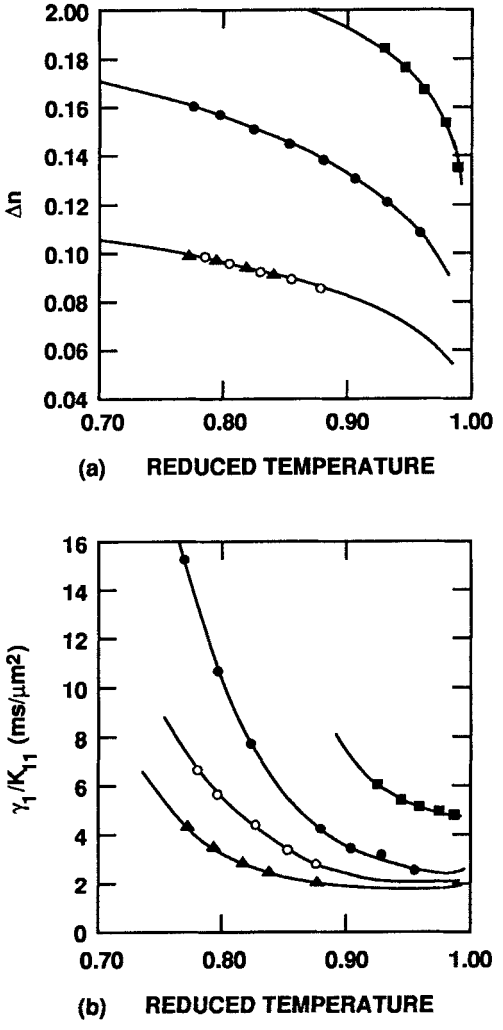


Figure 4. The temperature dependent (*a*) birefringence and (*b*) viscoelastic coefficients of the liquid crystals studied. The solid lines in (*a*) represent the data fitting using equations (1) with Δn_0 and β listed in table 2. $\lambda = 633$ nm. ■, FET-5CL; ●, Mixture; ○, ECCP-5CL.F; ▲, ECCP-5CL.

and the order parameter S is expressed as

$$S = [1 - T_r]^{\beta}, \tag{1 b}$$

where Δn_0 and β are the fitting parameters. Although equation (1 *b*) is not a rigorous expression for S, it works well in a wide nematic range except in the vicinity of the nematic–isotropic transition. Near the onset of the transition, S should be a discontinuous function, rather than one which is continuous as described by equation (1 *b*). Numerical results of Δn_0 and β for the samples studied are listed in table 2. Once β is known, the order parameter can be calculated at the temperature of interest using equation (1 *b*).

Table 2. Numerical values of Δn_0 and β used in equations 1.

Liquid crystal	Δn_0	β
FET-5CL	0·267	0·143
Mixture	0·225	0·232
ECCP-5CL	0·138	0·235
ECCP-5CL.F	0·137	0·235

3.3.2. *Wavelength effect*

The wavelength dependent birefringence of each sample was measured at a constant temperature. The wavelengths used for this study were obtained from tunable Ar^+ and HeNe lasers. The results are shown in figure 5. The dots are experimental data and the solid lines were fitted using the single band model [17]

$$\Delta n(\lambda, T) = G(T)[\lambda^2 \lambda^{*2}/(\lambda^2 - \lambda^{*2})], \quad (2)$$

where G is a parameter correlating the molecular packing density, differential oscillator strength, and order parameter, $G(T)$ describes the temperature dependence, and λ^* is the mean resonance wavelength. The G and λ^* values used for calculations for the liquid crystals studied are listed in table 3. Once G and λ^* are known, the wavelength dependent birefringence can be calculated through equation (2). Also included in table 3 are the threshold voltages of each sample at the specified temperature.

The birefringence results of the mixture, ECCP-5CL and ECCP-5CL.F, as shown in figure 5, were measured at nearly equal reduced temperature ($T_r \cong 0{\cdot}78$). However, for FET-5CL the measurement was made a few degrees above the melting point, but it corresponds to a high reduced temperature ($T_r = 0{\cdot}95$) because the nematic range of FET-5CL is not very wide. When T_r is reduced to 0·78, λ^* remains basically unchanged, but G increases in proportion to S. Thus, G increases from 3·40 to 4·24 when T_r drops from 0·95 to 0·78. The birefringence of FET-5CL at $T_r = 0{\cdot}78$ can be calculated by equation (2) using a value for G of 4·24 and λ^* of 212·1 nm. The results are shown as dashed lines in figure 5 for the purpose of comparison.

3.4. *Splay elastic constant*

From capacitance or birefringence measurements, the temperature dependent threshold voltage of each sample was determined. The threshold voltage determined by these two methods agrees well. From the measured threshold voltage and dielectric anisotropy (see figure 2), the splay elastic constant (K_{11}) of each sample was obtained through the following equation

$$V_{th} = \pi[K_{11}/\varepsilon_0 \Delta\varepsilon]^{1/2}, \quad (3)$$

Table 3. G and λ^* used in equation (2) for calculating birefringence dispersions. V_{th} is the threshold voltage at the given temperature.

Liquid crystal	T_r	$G/10^{-6}$ nm^{-2}	λ^*/nm	V_{th} (1 kHz)
FET-5CL	0·95	3·40	212·1	1·80
FET-5CL	0·78	4·24	212·1	—
Mixture	0·77	3·77	195·0	2·30
ECCP-5CL	0·77	4·0	151·5	3·06
ECCP-5CL.F	0·78	4·0	150·5	2·28

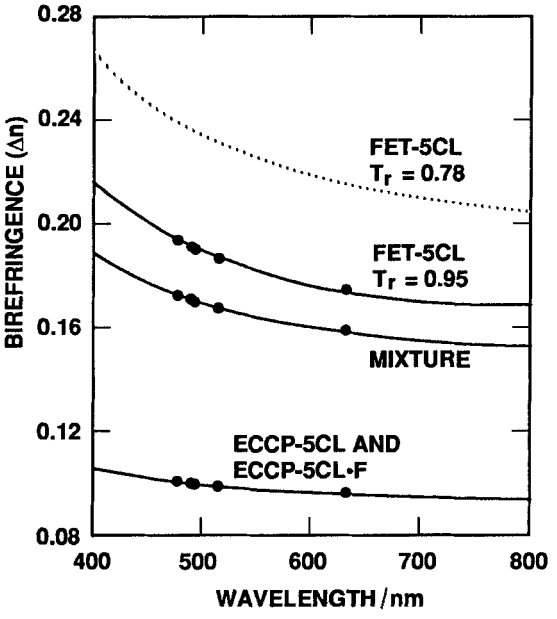


Figure 5. The wavelength dependent birefringence of the liquid crystals studied. The solid lines represent the data fitting using equation (2) with parameters and measurement temperatures listed in table 3. The dashed lines represent results calculated for FET-5CL at $T_r = 0{\cdot}78$ using equations (1) and (2). The results for ECCP-5CL and ECCP-5CL.F are nearly identical.

where ε_0 is the permittivity of free space. Figure 6 shows the elastic constant as a function of the order parameter (S). Results agree well with the Maier–Saupe mean field theory [18]

$$K_{11} = A_0 S^2, \tag{4}$$

where $A_0 = 45 \times 10^{-12}$ N for ECCP-5CL, ECCP-5CL.F and the mixture, and $A_0 = 30 \times 10^{-12}$ N for FET-5CL. A liquid crystal with a large elastic constant has two important effects on its electro-optical properties:

(1) it could lead to a high threshold voltage (see equation 3) depending on the associated $\Delta\varepsilon$;
(2) it may lower the viscoelastic coefficient depending on the detailed rotational viscosity.

Both the elastic constant, dielectric anisotropy and rotational viscosity are closely related to the molecular constituents and structures. Thus, they are interdependent.

3.5. *Rotational viscosity*

To obtain rotational viscosity, we first measured the temperature dependent viscoelastic coefficient. From the measured K_{11}, γ_1 is calculated. The phase decay time method [10, 19] has been demonstrated to be a convenient technique for determining γ_1/K_{11}. The temperature dependent viscoelastic coefficients of the samples studied are shown in figure 4(*b*). Combining the data shown in figure 4(*b*) and figure (6) yields γ_1 which is plotted in figure 7.

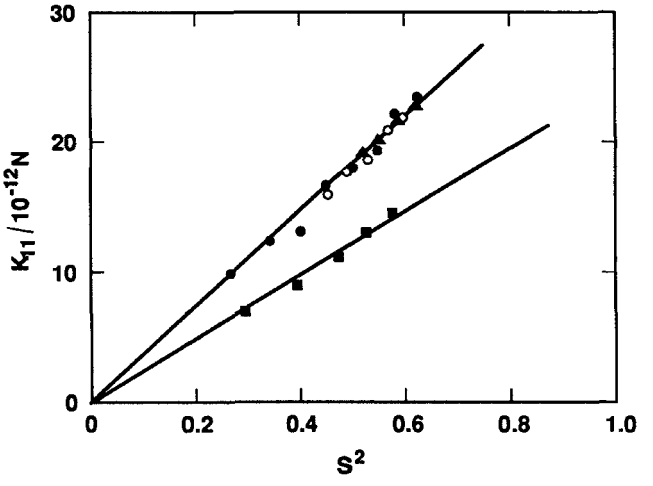


Figure 6. K_{11} as a function of S^2 as predicted in equation (4). A_0 is equal to 45×10^{-12} N for ECCP-5CL, ECCP-5CL.F and the mixture, and to 30×10^{-12} N for FET-5CL. ■, FET-5CL; ●, Mixture; ○, ECCP-5CL.F; ▲, ECCP-5CL.

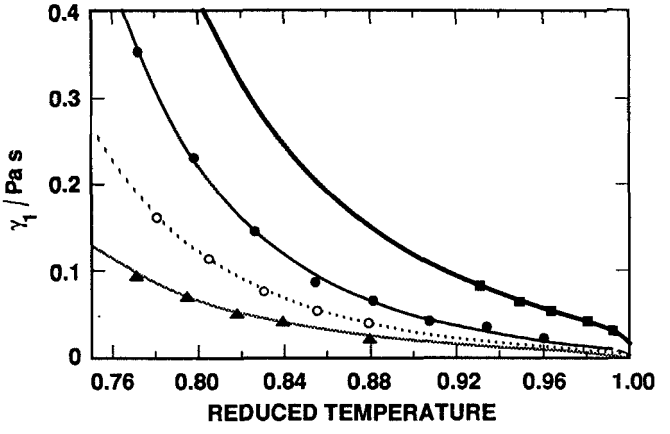


Figure 7. The temperature dependent rotational viscosity of the liquid crystals studied. Lines represent the data fitting using equation (5) with parameters listed in table 4. ■, FET-5CL; ●, Mixture; ○, ECCP-5CL.F; ▲, ECCP-5CL.

The lines in figure 7 are the fitted experimental data to the Osipov–Terenjev theory [10, 20]

$$\gamma_1 = g_0(T/T_C)(E_1/kT_C)^{-3}(J_0S/kT_C)^{1/2}\exp[(E_1 + J_0S)/kT], \quad (5)$$

where g_0 is the proportionality constant, E_1 is the activation energy, k is the Boltzmann constant, and $J_0 = 1{\cdot}8kT_C$ [10]. Equation (5) involves only two parameters: g_0 and E_1; their values are listed in table 4.

The lower γ_1 values observed for ECCP-5CL as compared to ECCP-5CL.F is attributed to its more linear molecular conformation. The molecular length-to-width ratio and moment of inertia (which are merged in g_0) all play important roles in the rotational viscosity.

Table 4. g_0 and E_1 used in equation (5) for fitting the experimental data to the rotational viscosity. In equation (5) $J_0 = 1{\cdot}8kT_C$.

Liquid crystal	$g_0/10^{-4}$ Pas	E_1/meV	J_0/meV
FET-5CL	8·85	290·7	55·5
Mixture	1·84	354·3	59·7
ECCP-5CL	2·43	353·3	64·9
ECCP-5CL.F	4·71	318·7	68·0

4. Discussion

A useful liquid-crystalline compound or eutectic mixture for information displays should meet the following basic requirements:

(1) low melting and high clearing points;
(2) low threshold voltage ($V_{th} < 3$ V);
(3) modest birefringence;
(4) low viscosity;
(5) excellent chemical and photostability.

The low melting point is important for application under various ambient conditions. On the other hand, a high clearing point makes the order parameter (and, therefore, birefringence, dielectric constants and elastic constants) rather less sensitive to temperature fluctuation originating from the possible heating effect of the liquid crystal device. A low operation voltage helps to reduce the cost of the associated driving electronics. A high birefringence may or may not be important depending on the wavelength of interest; for IR application, such as an IR spatial light modulator [21], high Δn is highly desirable. However, for displaying visible images low Δn improves the viewing angle [22, 23]. A low viscosity plays an important role in improving the response times of the device. Finally, excellent chemical- and photostability is an absolutely necessary requirement for achieving a long term device.

Although the chlorinated liquid-crystalline compounds reported here exhibit a wide nematic range, their melting points are not low enough. Thus, their usefulness is anticipated in the form of mixtures. The dielectric anisotropy of these compounds is in an acceptable range. The Δn and UV absorption are determined by the conjugation length of the molecule. Also, the molecular absorption makes a significant impact on its photostability. The viscosity of these compounds are generally low. Thus, potential application of chlorinated liquid-crystalline compounds for visible displays is foreseeable.

5. Conclusion

We have investigated the dielectric, optical and electro-optical properties of four chlorinated liquid-crystalline compounds and a eutectic mixture. Their properties are quite attractive for information display in the visible spectral region. Although the melting points of the chlorinated compounds are still somewhat too high, the formulation of eutectic mixtures will lower the melting point significantly and widen the nematic range.

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