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## Liquid Crystals

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Online publication date: 06 August 2010

**To cite this Article** Shiota, Atsushi , Korner, Hilmar and Ober, Christopher K.(1998) 'Twin nematic phenylbenzoates in a.c. electric fields', *Liquid Crystals*, 25: 2, 199 – 206

**To link to this Article:** DOI: 10.1080/026782998206344

**URL:** <http://dx.doi.org/10.1080/026782998206344>

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# Twin nematic phenylbenzoates in a.c. electric fields

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(Received 21 October 1996; in final form 27 January 1998; accepted 13 February 1998)

The orientational behaviour of nematic compounds having twin phenylbenzoate mesogens was examined under a wide range of a.c. electric fields ( $0\text{--}2\text{ V }\mu\text{m}^{-1}$  and  $10\text{ Hz--}50\text{ kHz}$ ). For this study, crossed polarizing optical microscopy (POM) and real-time X-ray diffraction (RTXRD) measurements were employed to investigate optical and orientational response. These nematic compounds have a positive dielectric anisotropy and a relatively low  $\epsilon_{//}$  relaxation frequency which allowed study in both homeotropic and planar orientations over a controllable frequency range. The optical behaviour and X-ray results corresponded well, providing a tool for understanding the orientational behaviour of these liquid crystals. For homeotropic alignment, an electric field of over  $1\text{ V }\mu\text{m}^{-1}$  was required in order to obtain good orientation. However, homeotropic orientation depended on a delicate balance between thermal fluctuations and dielectric torque imposed by the electric field, which are both strongly related to the elasticity of the LC domains. Due to this effect, the highest orientation parameter achieved for homeotropic orientation was only 0.48, which indicated that this state was still non-equilibrium. On the other hand, for planar orientation, a uniform texture with orientation parameter of 0.65 was easily obtained even at electric fields as low as  $0.2\text{ V }\mu\text{m}^{-1}$ . The application of an electric field stronger than  $1\text{ V }\mu\text{m}^{-1}$  induced a distortion in the texture, and reduced the orientation parameter to 0.45 for planar alignment.

## 1. Introduction

The orientational behaviour of liquid crystalline (LC) molecules in external fields has been extensively investigated over the last three decades because of the widespread use of LCs in display and optical devices. [1] In particular, under a.c. electric fields, nematic compounds with either negative or weakly positive dielectric anisotropy offer a wide range of behaviour and are still the source of much research [2]. In a low frequency electric field ( $<1\text{ kHz}$ ), charge accumulation and anisotropic electrical conductivity (in many cases caused by a small amount of ionic impurities) play important roles in domain formation [3]. The Williams [4] and chevron domains [5] are examples of structures mediated by a.c. electric fields and are due to the periodic flow of LC molecules. Under conditions of higher applied electric fields, such regular patterns give way to turbulent flow accompanied by dynamic scattering, known as electrohydrodynamic instability [6].

Until now, while pattern formation under a.c. electric fields has been much studied, behaviour in the instability regime is not yet well understood. Independently, recent

investigations on nematic oligomers [7–9] and nematic thermoset materials [10], which have a phenylbenzoate backbone, demonstrated that molecular orientation, either parallel or perpendicular to the applied electric field, could be manipulated by changing the applied field frequency. X-ray scattering results showed that the application of a relatively strong a.c. electric field of approximately  $1\text{ V }\mu\text{m}^{-1}$  to the nematic compounds stabilized orientation parallel to the field at a frequency of  $10\text{ Hz}$  and perpendicular to the field at a frequency of  $10\text{ Hz}$ . We have confirmed the homeotropic orientation not only on a polymeric system but also on a monomeric system [11]. A phenylbenzoate mesogen which itself has a larger transverse dipole component (2.4 D) than its longitudinal dipole component (1.2 D) [12] is expected to show negative dielectric anisotropy. However, dielectric anisotropy is governed not only by the permanent dipole but also by molecular polarization. Because of this molecular polarization, the dielectric anisotropy of phenylbenzoate derivatives can range from negative to slightly positive [13]. Early studies on phenylbenzoate derivatives reported that the molecules displayed stationary and oscillating domains as well as electrohydrodynamic instabilities [14, 15]. On the other hand, recent X-ray studies showed that the nematic phenylbenzoates can be aligned parallel to the applied electric field in a low frequency region where the electrohydrodynamic

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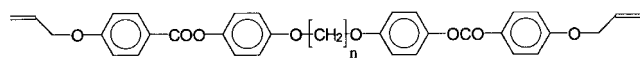
instabilities are expected to take place [7, 8, 10, 16]. A question to be raised is how to explain the discrepancy between optical observations in the early studies and recent XRD results.

To clarify this issue, we performed experiments in both optical microscopy and XRD on the same samples. Up to now, a detailed study using real-time XRD to study orientational behaviour under various electric field frequencies and amplitudes has not been carried out. The present paper not only provides additional data to the previous studies but also helps to bring a better understanding to the orientational behaviour of nematics under a.c. electric fields. The molecule studied for this purpose was 4-(2-propenyloxy)benzoic acid 1,9-nonanediybis(oxy-4,1-phenylene) ester, **Twin9a** (see figure 1). The molecule possesses a twin mesogen architecture in which two phenylbenzoate mesogens are connected with an alkyl spacer. Twin LC molecules have been studied previously as model compounds for main-chain LC polymers because twin LC molecules combine characteristics of both monomeric and polymeric systems [17–19]. In our case, we believe therefore that information obtained through this study will also be worthwhile for future discussions of both polymeric and monomeric mesogens. In this study, another twin LC molecule 4-(2-propenyloxy)benzoic acid 1,8-octanediybis(oxy-4,1-phenylene) ester (**Twin8a**) and 4-(5-hexenyloxy)benzoic acid 1,4-phenylene ester (**6a**) were also used to provide additional information.

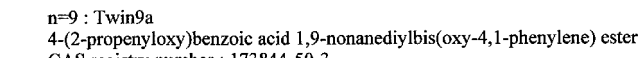
## 2. Experimental

The synthesis and purification of the nematic compounds have been described in a previous report [20].

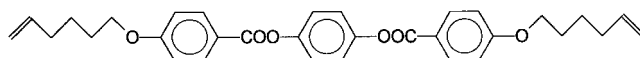
Rheological measurements were performed with the aid of a Rheometrics SR-2000 stress control rheometer using 25 mm diameter parallel-plate geometry. A



n=8 : **Twin8a**  
4-(2-propenyloxy)benzoic acid 1,8-octanediybis(oxy-4,1-phenylene) ester  
CAS registry number : 173844-49-0  
K 152 °C N 182 °C I



n=9 : **Twin9a**  
4-(2-propenyloxy)benzoic acid 1,9-nonanediybis(oxy-4,1-phenylene) ester  
CAS registry number : 173844-50-3  
K 96 °C N 159 °C I



**6a**  
4-(5-Hexenyloxy)benzoic acid 1,4-phenylene ester  
CAS registry number : 153881-42-6  
K 133 °C N 206 °C I

Figure 1. Chemical structure of the material used in this study.

pressed sample pellet (20 mm diameter) was inserted in the rheometer and measurements were conducted in a linear viscoelastic region, at a frequency of 1 Hz and a strain of 900%. The rheological measurements were carried out with a heating and cooling rate of 5°C min<sup>-1</sup>. A TA Instruments 910 differential scanning calorimeter (DSC) was used to detect the transition temperatures. The DSC study was carried out with a heating rate of 5°C min<sup>-1</sup>.

Domain formation and light scattering intensity were observed using a Nikon polarizing optical microscope (POM) OPTIPHOT2-POL at 100× or 200× magnification; it was equipped with a Mettler FP-82HF hot stage. In addition, light scattering intensity was recorded as a signal from a Mettler 17517 photomonitor mounted on the POM. Liquid crystal cells were constructed from two transparent indium tin oxide (ITO) glass electrodes and a polyimide insulator of 72.5 μm thickness. Voltage and frequency generated by a HP 8116A function generator were amplified ×1000 by a Trek model 10/10 high voltage operational amplifier and applied to the cell. The light scattering intensity, the voltage and frequency of the applied electric field and the temperature were monitored and recorded simultaneously by a personal computer system. Both voltage and frequency sweep experiments were carried out with a 35 s interval between each set of measurements in order to associate with the real-time X-ray diffraction experiments.

The real-time XRD data were obtained at the F1 beamline of the Cornell High Energy Synchrotron Source (CHESS). X-ray optical cells consisted of two 500 μm wide stainless steel electrodes separated by a controlled gap spacing of 72.5 or 145 μm with a polyimide film. The cell was mounted in a Mettler FP-82HT hot stage to regulate the sample temperature. Voltage and frequency generated by a HP 8116A function generator were amplified ×1000 by a Trek model 10/10 high voltage operational amplifier and applied to the cell. The flux of a monochromated beam of 0.910 Å wavelength attained 27 000 cps after collimation by a 0.3 mm diameter collimator, allowing exposure times of less than 1 s. XRD images were recorded on a flat CCD detector every 30 to 40 s, including data transfer and processing time, and were finally saved in 16 bit resolution tiff format. Orientation parameters (*S*) were calculated from azimuthal scans passing through the wide angle reflections on the flat-images detailed by McNamee *et al.* [7, 8].

## 3. Results and discussion

### 3.1. Phase behaviour of the nematic compound **Twin9a**

First let us briefly describe the phase behaviour of **Twin9a**. Among the three LC compounds examined in this study (figure 1), **Twin9a** possesses the lowest melting

point at 96°C. Its nematic range of 63°C on heating and 81°C on cooling, as shown in figure 2(A) was broad enough adequately to observe the temperature dependence of the molecular response as a function of applied electric field. However, rheological measurements revealed that there existed a relatively broad transitional phase from melting up to 130°C, which was

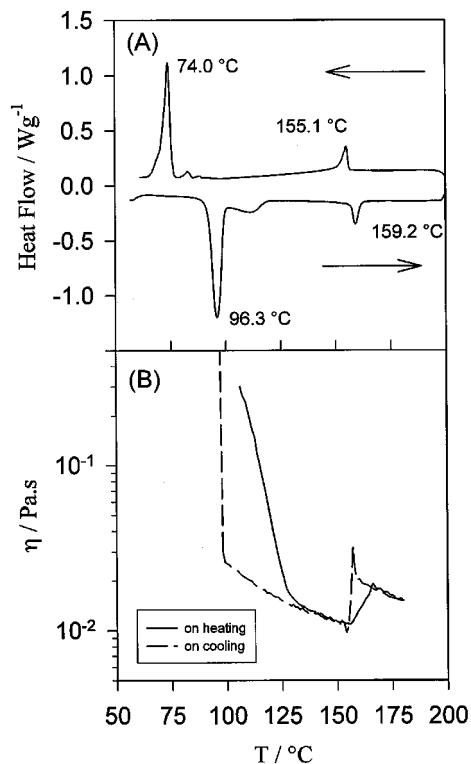


Figure 2. DSC and viscometric measurements for **Twin9a** compound.

not observed in the cooling process. DSC traces likewise revealed this transitional state as a small peak in the endotherm at the same temperature. In this transitional state, **Twin9a** had a high viscosity as well as a relatively small loss tangent ( $< 10$ ), similar to a low molar mass fluid. This fact suggests that the molecules are entangled in the transitional state and that such entanglement may exist in the crystalline state. A similar observation was reported by Lin *et al.*, that a twin LC molecule exhibited an unusually high elastic modulus, and was ascribed to entanglement of the molecules [21]. Because of this transitional state during the heating process, temperature regulation for experiments with optical cells and X-ray optics was carried out so that the samples were first heated to 150°C, and then cooled to the target temperature. During rheological measurements, crystallization of **Twin9a** occurred approximately 20°C above the crystallization temperature detected by DSC. We believe that the large 900% strain amplitude may trigger crystallization without supercooling.

### 3.2. Investigations with RTXRD

Figure 3 gives typical XRD patterns for the **Twin9a** molecule aligned both parallel (A) and perpendicular (B) to an applied electric field. Amplitude and frequency of the applied electric field was  $1 \text{ V } \mu\text{m}^{-1}$  at 100 Hz for the parallel orientation and  $0.75 \text{ V } \mu\text{m}^{-1}$  at 30 kHz for the perpendicular orientation. A high flux, 300  $\mu\text{m}$  diameter collimated X-ray beam with 1 s exposure time was sufficient to obtain orientation data for the LC placed between electrodes with a 145  $\mu\text{m}$  cell gap under an a.c. electric field. The cross-over (cut-off) frequency of **Twin9a** at 100°C was observed at  $\sim 2 \text{ kHz}$  using a frequency sweep from 10 Hz upward to 40 kHz in an

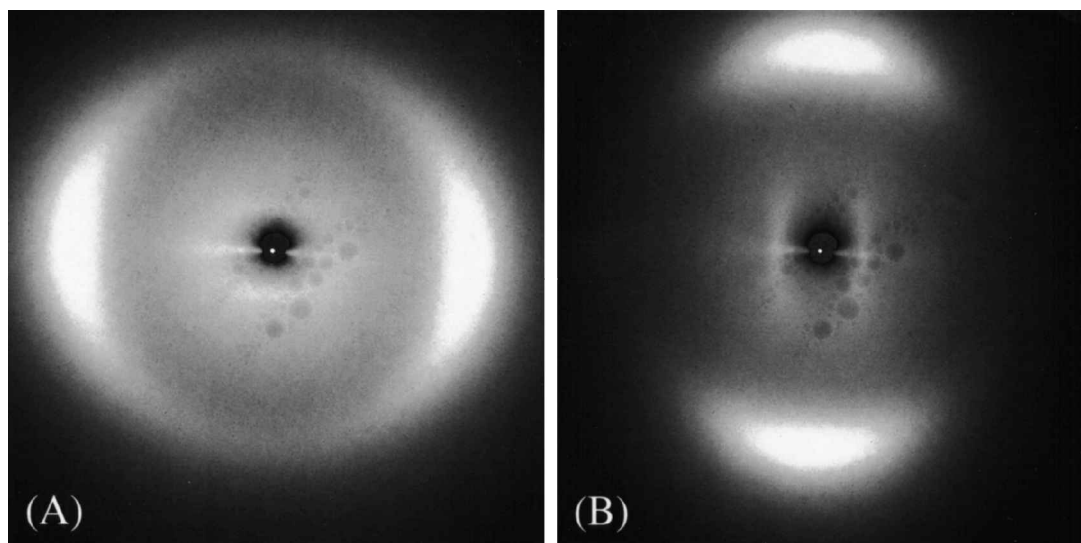


Figure 3. XRD image for **Twin9a** under an applied electric field at 100°C. (A) Electric field of  $1 \text{ V } \mu\text{m}$  and 100 Hz (conduction regime, instability region); (B) electric field of  $0.75 \text{ V } \mu\text{m}^{-1}$  and 30 kHz (dielectric regime, stability region).

electric field of  $1 \text{ V } \mu\text{m}^{-1}$  (see figure 4). The cross-over frequency of 2 kHz suggests that the compound possesses a positive dielectric anisotropy with a relatively low relaxation frequency for  $\varepsilon_{//}$  which might be ascribed to the long molecular shape of **Twin9a**. De Jeu *et al.* [13] reported a phenylbenzoate derivative having a positive dielectric anisotropy which exhibits a Fréedericksz type reorientation at its cross-over frequency from planar to homeotropic alignment under an a.c. electric field.

Of interest is the dependence of the orientation parameter on field amplitude, in particular in the low frequency region, because the field amplitude of  $1 \text{ V } \mu\text{m}^{-1}$  seemed strong enough to generate electrohydrodynamic instabilities. As displayed in figure 5, increasing the electric field at 10 Hz stabilized orientation and yielded a higher orientation parameter. The highest orientation parameter for the homeotropic orientation was approximately 0.48, and the orientation was saturated above a field strength of  $0.8 \text{ V } \mu\text{m}^{-1}$ .

Figure 6 shows azimuthal scans along the wide angle diffraction ring for the XRD patterns displayed in figure 3. As shown in figure 6, the minimum value of the distribution function (at  $\beta = 90^\circ$ ) is not zero for the homeotropic orientation, whereas no diffraction was observed at  $\beta = 90^\circ$  for the planar orientation. This suggests that homeotropic alignment under a low frequency electric field is less perfect than the planar orientation obtained under a high frequency electric field due to molecular agitation. If any flow induced by an applied a.c. electric field was taking place and produced

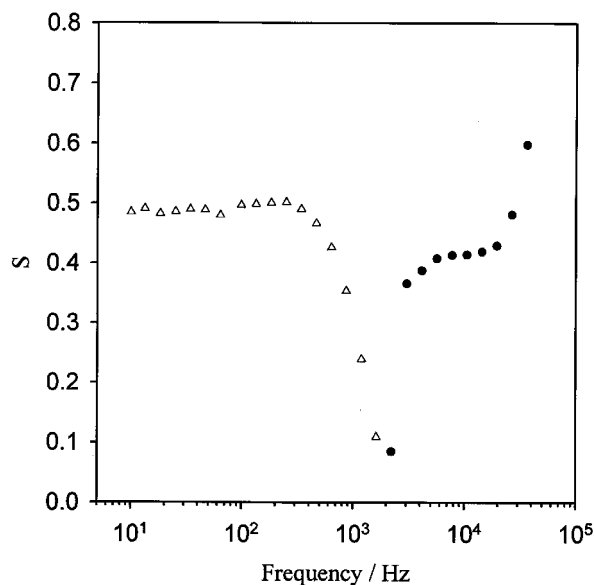


Figure 4. Dependence of the orientation parameter on the frequency of an applied electric field. An electric field of  $1 \text{ V } \mu\text{m}^{-1}$  was applied. (●) The director is perpendicular to the applied electric field. (Δ) The director is parallel to the applied electric field.

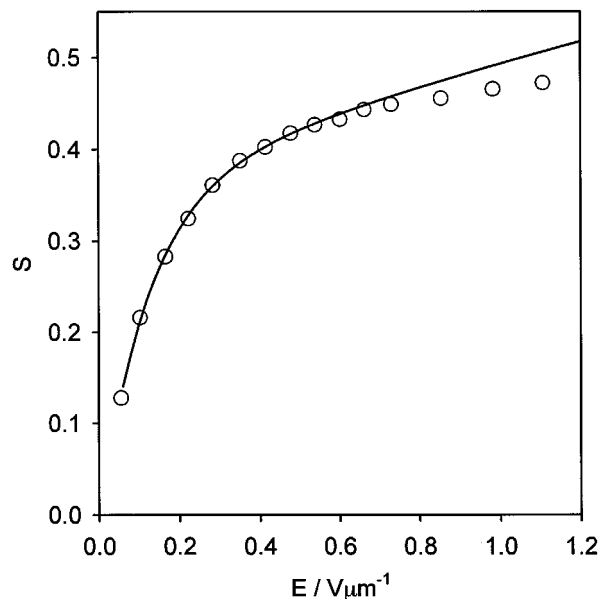


Figure 5. Dependence of orientation parameter on the amplitude of an applied electric field of 10 Hz at  $100^\circ\text{C}$  for **Twin9a**.

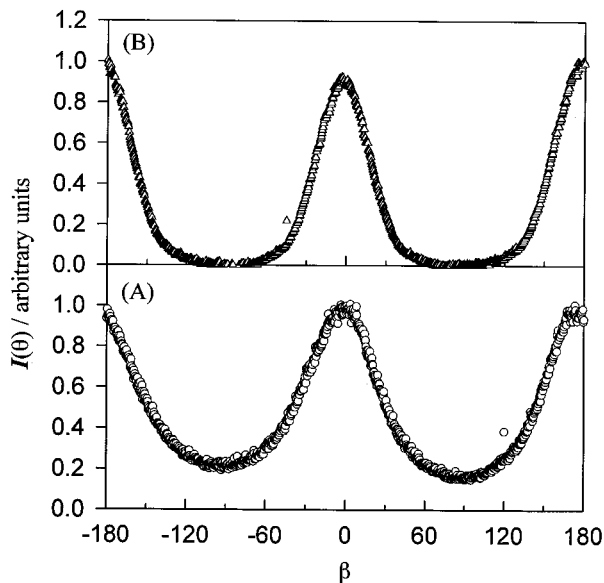


Figure 6. Distribution function of  $I(\theta, \beta)$  at  $2\theta$  of  $11.7^\circ$  ( $d$ -spacing  $4.46 \text{ \AA}$ ) for **Twin9a** molecule. (A) Electric field of  $1 \text{ V } \mu\text{m}^{-1}$  and 100 Hz (conduction regime, instability region). (B) Electric field of  $0.75 \text{ V } \mu\text{m}^{-1}$  and 30 kHz (dielectric regime, stability region).

lower orientation in the low frequency region, it would be of interest to determine whether orientation could be retained on cooling from a nematic state to a crystalline state.

Figure 7 displays a series of XRD images taken at various temperatures. (A) shows an XRD picture taken in the nematic phase with homeotropic orientation at

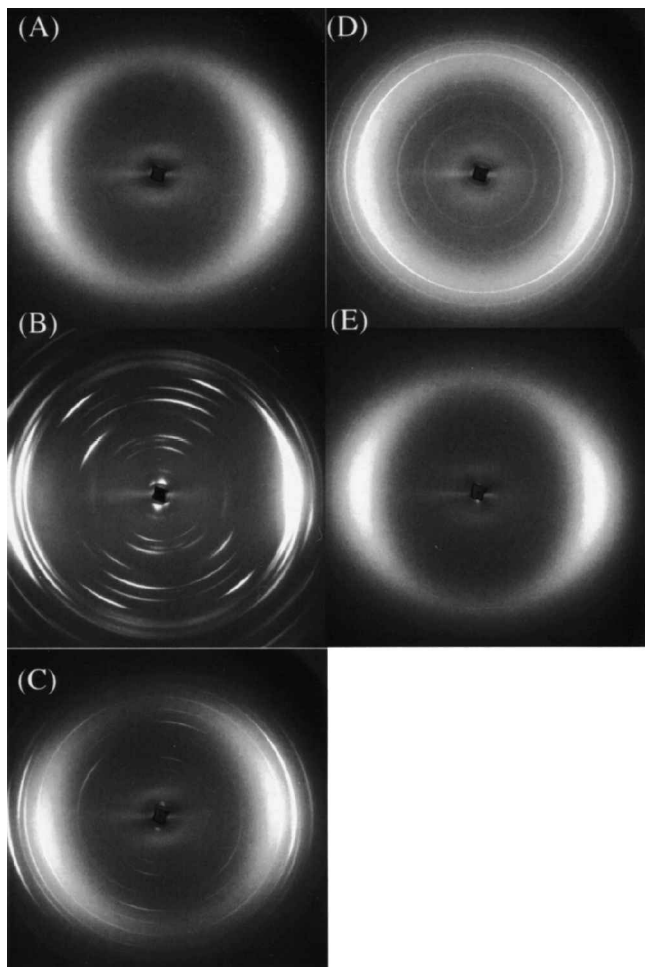


Figure 7. A series of XRD pictures for **Twin9a** over a temperature sequence under an electric field of  $1 \text{ V } \mu\text{m}^{-1}$  and 10 Hz. (A) at  $100^\circ\text{C}$  → (B) at  $75^\circ\text{C}$  → (C) at  $100^\circ\text{C}$  → (D) at  $120^\circ\text{C}$  → (E) at  $130^\circ\text{C}$ .

$100^\circ\text{C}$  aligned under an electric field of  $1.0 \text{ V } \mu\text{m}^{-1}$ . The sample under the electric field was subsequently cooled to  $75^\circ\text{C}$ . (B) shows that the orientation was retained and even improved in the crystalline state. Likewise, as displayed in figure 8, orientation was enhanced with decreasing temperature in the nematic phase. This means that an increase of viscosity upon cooling did not affect orientation and suggests that little or no flow occurred. Figure 7(C) shows that melting started on reheating the sample to  $100^\circ\text{C}$ , as shown by the diffuse diffraction at wide angles. Heating further to  $120^\circ\text{C}$  destroyed any sample orientation, figure 7(D). High viscosity in the transitional state does not allow the sample to reorient after losing orientation during melting. Eventually, orientation was recovered by heating to  $130^\circ\text{C}$ , figure 7(E). This homeotropic orientation could be retained up to  $140^\circ\text{C}$  where it is, however, only  $20^\circ\text{C}$  below the clearing temperature detected by DSC measurements

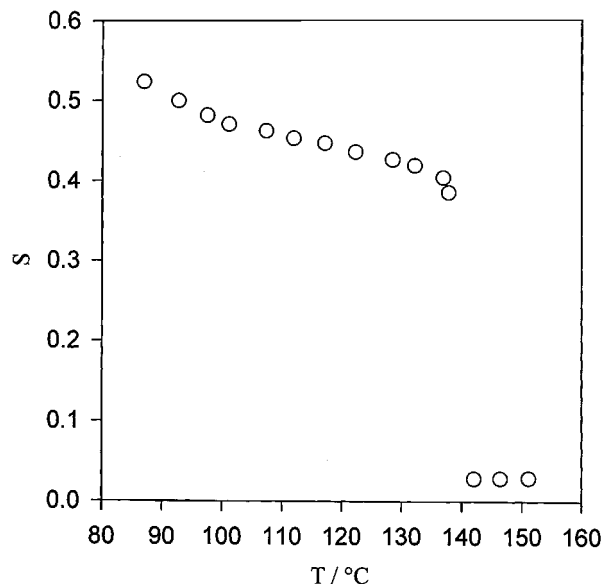


Figure 8. Dependence of the orientation parameter on temperature for **Twin9a**. An electric field of 10 Hz and  $1 \text{ V } \mu\text{m}^{-1}$  was applied.

(see figure 8). The homeotropic orientation seemed to be controlled by a delicate balance between thermal fluctuation and torque induced by an alternating electric field. These forces are deeply related to the elasticity of the domains and even of the molecule itself. For the **Twin9a** molecule, a threshold voltage where a regular domain gives way to turbulence was expected to be located below  $0.1 \text{ V } \mu\text{m}^{-1}$ . Electrohydrodynamic instabilities may be a transient phenomenon related to regular flow in the homeotropic orientation. We believe that a further increase in voltage brings about a stabilized homeotropic orientation. A detailed discussion on this point is given below, with the corresponding experimental results from POM observations.

A sweep of electric field amplitude was performed in a high frequency region, producing a planar orientation. An electric field amplitude dependence of the orientation parameter was also observed in the high frequency region (see figure 9). However, the trends were different from those observed in the low frequency region. The highest orientation parameter for planar orientation of approximately 0.65 was obtained at electric fields of  $< 1 \text{ V } \mu\text{m}^{-1}$ . The molecules could be well aligned even at fields of  $0.1 \text{ V } \mu\text{m}^{-1}$ . On the other hand, electric fields greater than  $1 \text{ V } \mu\text{m}^{-1}$  gave poorer orientation with an orientation parameter of 0.45, that was almost constant above the threshold. This threshold voltage changed with frequency: a higher frequency gave a higher threshold voltage. We believe that a strong alternating field perturbed the molecules and caused the reduced orientation above the threshold voltage whereas, below the threshold,

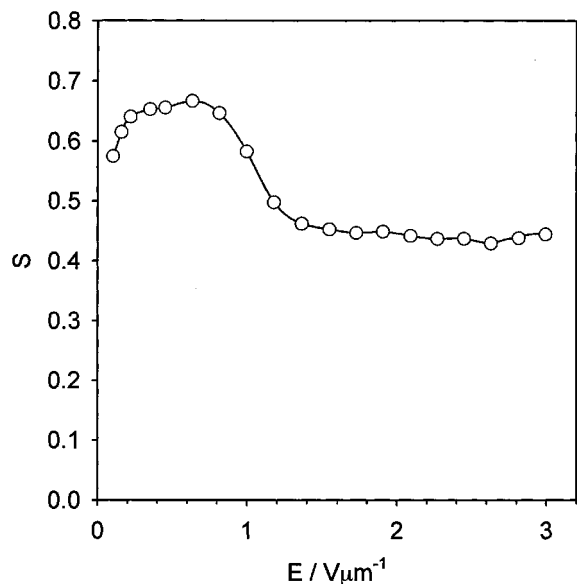


Figure 9. Dependence of the orientation parameter of the amplitude of the applied electric field of 10 kHz at 100°C for **Twin9a**. The data were obtained with an increasing field.

an electric field was able to couple with the dipole moment of the molecule, and maintain the orientation of the molecules.

### 3.3. Investigations with an optical LC cell

In order to determine how the optical behaviour of **Twin9a** would correspond with XRD experiments, investigations with an optical LC cell were carried out. In these investigations, we assumed that light intensity correlated with orientation direction of the molecules, as a high light intensity is associated with a planar orientation and a low light intensity indicates homeotropic alignment. Though low light intensity may also indicate a disordered state, we have already confirmed

with XRD measurements that the molecules were aligned homeotropically under a low frequency electric field.

Figure 10 illustrates the frequency dependence of light intensity through a crossed polarizer under an electric field of  $1 \text{ V } \mu\text{m}^{-1}$  at various temperatures. A cross-over frequency from homeotropic to planar orientation depended on temperature and showed Arrhenius-type temperature behaviour (see figure 11). Early studies of electrohydrodynamic behaviour revealed that the cross-over frequency was a function of both conductivity [22] and viscosity [14] of molecules. Raising the temperature causes increased conductivity as well as decreased viscosity, and resulted in elevation of the cross-over frequency. Interestingly, the cross-over frequencies of

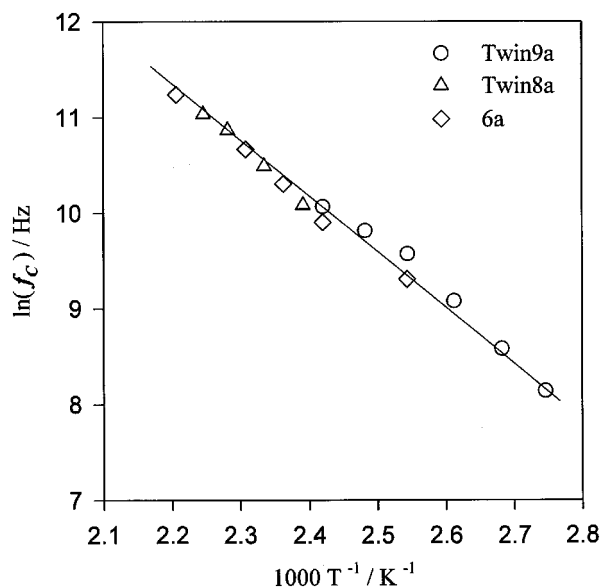


Figure 11. Temperature dependence of the crossover frequency  $f_c$  for three different nematic phenylbenzoate derivatives.

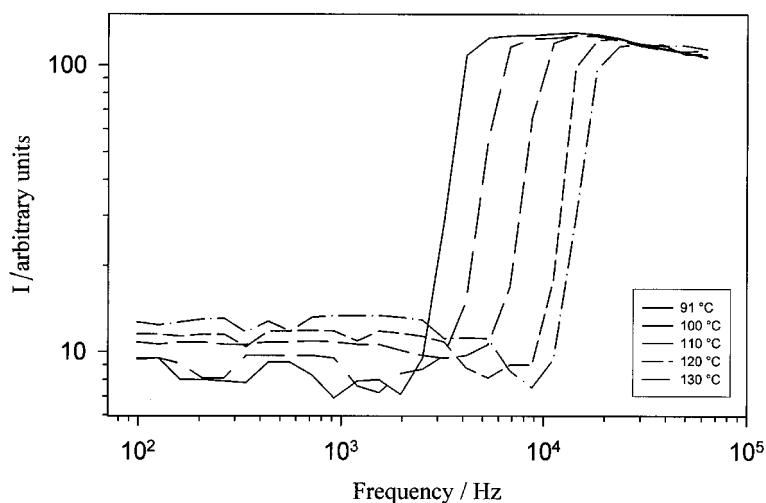


Figure 10. Dependence of the light scattering intensity  $I$  on the frequency of an applied electric field under various temperature conditions for **Twin9a**. An electric field of  $1 \text{ V } \mu\text{m}^{-1}$  was applied to a liquid crystal cell with  $72.5 \mu\text{m}$  gap. The light intensity  $I$  was obtained as transmittance through crossed polarizers.

another twin LC **Twin8a** and a single mesogen **6a** could be fitted to the same Arrhenius plot as **Twin9a**. This fact suggests that the **Twin9a**, **Twin8a** and **6a** molecules possess a similar conductivity and viscosity dependence with temperature. In figure 10 we can also observe that light intensity strengthens as the temperature is raised. This result corresponded well with XRD data shown in figure 8.

POM observations on **Twin9a** revealed more detailed phenomena under a frequency sweep at an electric field of  $1 \text{ V } \mu\text{m}^{-1}$ . Upon sweeping from low frequency to high frequency, a chevron domain was observed directly above the cross-over frequency. Further sweeping of the frequency upward resulted in the disappearance of the chevron domain and produced a bright uniform texture. On the other hand, below the crossover frequency, scattered depolarized light was observed as numerous fine dots which were dynamically flashing in the dark. This observation suggests that molecular motion was quite high and the mesogens were not perfectly aligned parallel to the applied electric field. The low orientation parameter of approximately 0.45 (obtained by XRD) measured under these conditions can be attributed to a non-equilibrium situation which may be induced by both an alternating field of the appropriate frequency and thermal fluctuation. This behaviour was consistent over the temperature range examined ( $90\text{--}130^\circ\text{C}$ ) and was noted in the other two compounds.

Figure 12 displays the light intensity dependence for **Twin9a** on frequency and amplitude of an applied electric field at  $91^\circ\text{C}$ . Below the cross-over frequency, the light scattering intensity was independent of frequency over the region but it was strongly dependent on the amplitude of the applied electric field. This trend corresponded well with results obtained by XRD. Amplifying the

applied electric field to  $0.8 \text{ V } \mu\text{m}^{-1}$  caused an exponential reduction of light scattering intensity, whereupon the intensity became saturated by further increasing the electric field (see figure 13). Using POM, upon increasing the electric field, the Williams domains [4] were observed around  $0.1 \text{ V } \mu\text{m}^{-1}$ . At this electric field, no orientation could be observed by X-ray diffraction. An X-ray beam collimated to  $300 \mu\text{m}$  diameter was clearly too large to focus on a streamline of this electrically induced convective flow, and yielded a diffraction pattern without orientation as a result of averaged molecular orientation. Subsequent increase of the amplitude caused

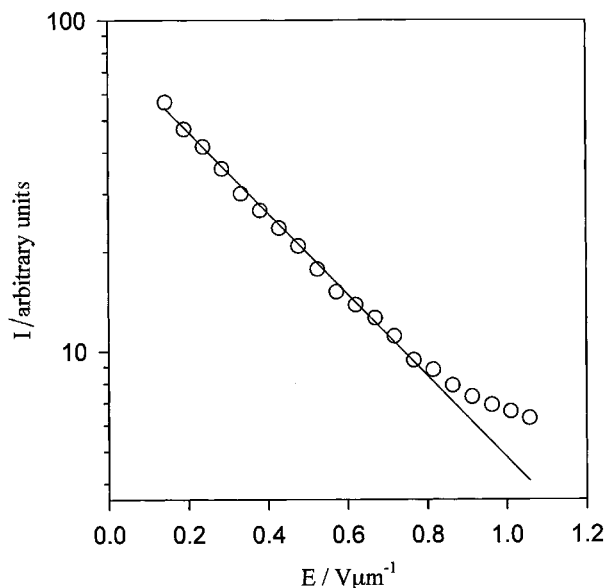


Figure 13. Dependence of light scattering intensity  $I$  on the amplitude of an applied electric field of  $10 \text{ Hz}$  at  $100^\circ\text{C}$  for **Twin9a**.

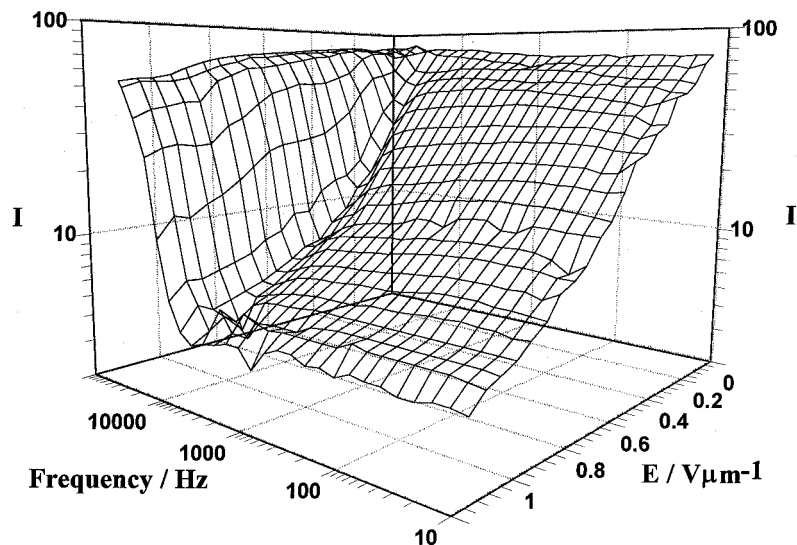


Figure 12. Dependence of light scattering intensity  $I$  on the amplitude and frequency of applied electric fields at  $90^\circ\text{C}$  for **Twin9a**.



the disappearance of the Williams domain and, instead, the emergence of turbulent flow. Electrohydrodynamic instabilities were certainly observed in this system. Further amplification of the field to  $0.2 \text{ V } \mu\text{m}^{-1}$ , however, resulted in the disappearance of turbulent flow and led to an exponential decrease in light intensity, indicating a gradual stabilization of homeotropic orientation as the electric field was amplified. The electrohydrodynamic instability is accordingly a transient phenomenon which occurs in passing from regular flow to homeotropic orientation. Decreased light intensity was observed as a reduction in the number of dynamically flashing fine dots. The flashing dots never fully disappeared even on applying an electric field of over  $1 \text{ V } \mu\text{m}^{-1}$ . This observation corresponded to the X-ray results where the orientation parameter for homeotropic orientation remained as low as 0.48. In figure 12, one can see a slight decrease of light intensity above the cross-over frequency at  $\sim 1 \text{ V } \mu\text{m}^{-1}$ , which corresponds to a decrease of orientation under high electric field, as observed by XRD.

#### 4. Conclusion

The present study combines data from both POM and RTXRD examinations to study a.c. electric field effects on the alignment of phenyl benzoate nematogens. With a nematic compound having a positive dielectric anisotropy, the relationship between optical behaviour and orientation as well as differences in the orientation process between homeotropic and planar alignment were examined. Hydrodynamic instabilities and domain pattern formation were observed as transient phenomena. A high orientation parameter ( $S = 0.65$ ) was obtained only for planar orientation under a low magnitude electric field; otherwise, the applied electric field combined with thermal motion perturbed the LC domains and yielded lower macroscopic alignment. In using electric fields to process LC materials, therefore, temperature, field strength and frequency must all be considered in maximizing orientation.

The authors would like to thank Dr T. J. Bunning for discussion of the results, Dr J. Navaie and the Cornell High Energy Synchrotron Source (CHESS) staff for their great technical assistance concerning the CHESS experiments, and the Cornell Materials Science Centre

for use of computer facilities. This research is partially supported by Japan Synthetic Rubber Co., Ltd and the National Science Foundation, Division of Materials Research.

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