



## Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and  
subscription information:

<http://www.tandfonline.com/loi/gmcl19>

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Version of record first published: 23 Sep 2006.

To cite this article: Masahiro Imai , Hiroyoshi Naito , Masahiro Okuda & Akihiko Sugimura (1995):  
Measurement of Rotational Viscosity and Pretilt Angle in Nematics from Transient Current, Molecular  
Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals,  
262:1, 267-274

To link to this article: <http://dx.doi.org/10.1080/10587259508033531>

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## MEASUREMENT OF ROTATIONAL VISCOSITY AND PRETILT ANGLE IN NEMATICS FROM TRANSIENT CURRENT

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**Abstract** Transient current technique is presented for measurement of rotational viscosity  $\gamma_1$  and pretilt angle  $\theta_0$  in nematic liquid crystal display devices. Theoretical expressions for the determination of  $\gamma_1$  and  $\theta_0$  from transient current are derived by taking account of the conduction of impurity ions and director rotation. Experimental results for  $\gamma_1$  and  $\theta_0$  are compared with those with other conventional methods.

## INTRODUCTION

We have developed an accurate and simple method for the measurement of rotational viscosity  $\gamma_1$  and pretilt angle  $\theta_0$  of impurity-ion free nematic liquid crystals (NLCs) in device configurations using transient current technique.<sup>1–3</sup> Since NLC materials contain impurity ions, which are generated from more or less dissociated impurities as well as from the spontaneous dissociation of the NLC molecules themselves, the influence of ionic conduction on the analysis should be considered. By means of the method, not only  $\gamma_1$ -values but also  $\theta_0$ -values can be measured in homogeneous<sup>1,2</sup> or twisted<sup>3</sup> configuration of NLC cells with common instruments. Further, the procedure for the determination of  $\gamma_1$  is much simpler than optical<sup>4,5</sup> or capacitive<sup>6</sup> methods which require differentiation of transmittance or capacitance with respect to time, respectively, to measure with the same accuracy as the present method.

In this article, we demonstrate the applicability of the present method to NLCs containing impurity ions as well.

## **THEORY**

We derive expressions for transient current in NLCs with homogeneous configuration excited by a dc step voltage  $V$ . The NLC layer is confined between the electrodes located at  $z = 0$  and  $L$ . The tilt angle  $\theta$  between the director and the  $z$ -axis is fixed to be  $\theta_0$  at the electrodes, where  $\theta_0$  is the pretilt angle.

We assume that the positive and the negative ions with the same density  $n$  in the NLC are uniformly distributed in thermal equilibrium state, and that the drift mobility  $\mu$  of the positive ions is much larger than that of the negative ions. Here we focus ourselves on the time range  $t \ll t_0$ , where  $t_0 [= L/(\mu E)]$  is the ion transit time,  $E$  is the applied electric field. In this time range, the net space charge density is zero and hence the Poisson equation is

$$\frac{\partial}{\partial z} [\varepsilon(\theta(z, t)) E(z, t)] = 0, \quad (1)$$

where  $\varepsilon$  is the dielectric permittivity of NLCs,

$$\varepsilon(\theta(z, t)) = \epsilon_0 [\epsilon_{\perp} + \Delta\epsilon \cos^2 \theta(z, t)], \quad (2)$$

and  $\epsilon_0$  is the dielectric permittivity of vacuum,  $\epsilon_{\perp}$  is the dielectric constant normal to the director, and  $\Delta\epsilon$  is the dielectric anisotropy. The conduction current due to the drift of the monovalent positive ions is written as  $Sqn\mu E$  for  $t \ll t_0$ , where  $S$  is the area of the electrodes, and  $q$  is the electronic charge.

In the absence of flow, the director motion is expressed as<sup>7</sup>

$$\begin{aligned} \gamma_1 \frac{\partial \theta}{\partial t} = & (K_{11} \sin^2 \theta + K_{33} \cos^2 \theta) \frac{\partial^2 \theta}{\partial z^2} \\ & + \left[ (K_{11} - K_{33}) \left( \frac{\partial \theta}{\partial z} \right)^2 - \epsilon_0 \Delta\epsilon E^2 \right] \sin \theta \cos \theta, \end{aligned} \quad (3)$$

where  $K_{11}$  and  $K_{33}$  are the splay and bend elastic constants, respectively. From the numerical analysis of Eqs. (1) - (3) under the strong anchoring conditions, the distributions of the director and the electric field along the  $z$ -axis are uniform during the director rotation for  $V \geq 4\pi\sqrt{K_{11}/(\epsilon_0\Delta\epsilon)}$ , except for those in the vicinity of the electrodes.<sup>2</sup> It is therefore reasonable to assume that the electric field in the bulk region  $E_b$  is approximately spatially uniform and time independent, and is given by<sup>2</sup>

$$E_b = \frac{V}{\left(1 + \frac{\beta}{V}\right) L}, \quad (4)$$

where  $\beta$  is a positive constant. If the influence of the elastic deformation on the transient current can be neglected,  $\beta$  is equal to zero.

The total current  $I(t)$  in NLCs is written as

$$I(t) = SE_b \left[ \frac{d\varepsilon(\theta_b(t))}{dt} + qn\mu \right], \quad (5)$$

where  $\theta_b$  is the tilt angle in the bulk region. We find from Eqs. (2) - (5) that the current has a peak at  $\theta_b = 45^\circ$ . The peak current  $I_p$  which is the current at the peak is

$$I_p = \frac{S(\epsilon_o \Delta \epsilon)^2 V^3}{2\gamma_1 L^3 \left(1 + \frac{\beta}{V}\right)^3} + \frac{Sq n \mu V}{L \left(1 + \frac{\beta}{V}\right)}. \quad (6)$$

The peak time  $t_p$  at which the current has the peak is obtained as

$$t_p = \frac{\gamma_1 L^2 \left(1 + \frac{\beta}{V}\right)^2 \log(\tan \theta_o)}{\epsilon_o \Delta \epsilon V^2}, \quad (7)$$

where  $\theta_o$  is the initial tilt angle, and  $\theta_o > 45^\circ$  is necessary to observe the current peak. It is noted that  $\theta_o$  does not contribute to  $I_p$  but to  $t_p$ .

## EXPERIMENT

The NLC materials used in the transient current experiment were 4-cyano-4'-pentyl biphenyl (5CB), ZLI2293 from Merck, and fluorinate mixture A, B and C obtained from Matsushita Electric Industrial Co., Ltd. The NLC was introduced between two pieces of glass with a transparent  $\text{In}_2\text{O}_3$  electrode, whose area was  $2 \text{ cm}^2$ . We prepared three 5CB cells (5.3, 12 and  $44 \mu\text{m}$ ), a ZLI2293 ( $5.8 \mu\text{m}$ ) cell, and fluorinate mixture A ( $4.8 \mu\text{m}$ ), B ( $5.7 \mu\text{m}$ ) and C ( $5.1 \mu\text{m}$ ) cells, where the numerals in parentheses are the thickness of the cells. Polyimide alignment layers with 100 nm in thickness were coated on the electrodes except for the  $12 \mu\text{m}$  5CB cell. Substrate surfaces were unidirectionally rubbed in an antiparallel manner to produce homogeneous configuration.

A dc voltage pulse was applied to the cells at constant temperatures, where the NLCs were in the nematic phase. Transient currents were observed through a series load resistor by means of a wideband preamplifier and a digital storage oscilloscope. The measured data were transferred to a microcomputer for the analysis and the storage.

Representative current transients are shown in Figures 1(a) and 1(b) for the  $12 \mu\text{m}$  5CB cell at 303 K and the fluorinate mixture A cell at 293 K, respectively. A prominent current peak induced by director rotation is observed in each transient. Current baselines

due to ionic conduction are not negligible in 5CB, but negligible in fluorinate mixtures and ZLI2293.

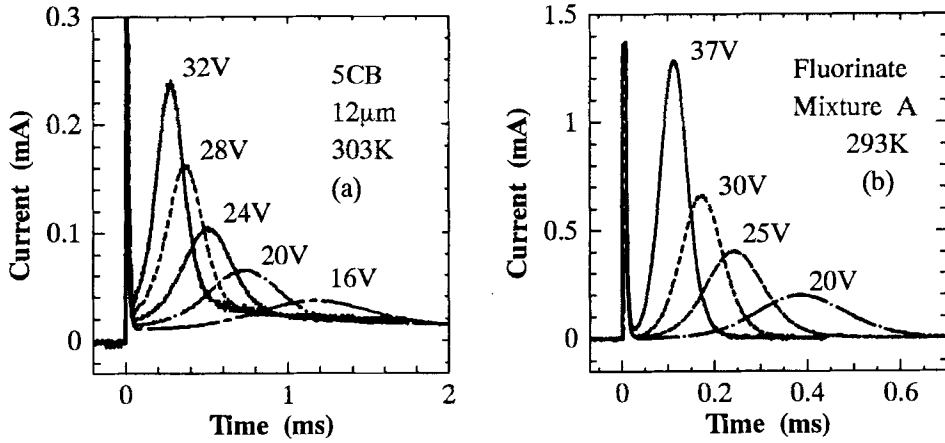


FIGURE 1 Transient current shapes of the 12  $\mu\text{m}$ -thickness 5CB cell at 303 K (a) and of the fluorinate mixture A cell at 293 K (b).

## RESULTS AND DISCUSSION

### Influence of Ionic Conduction on Current Transients

We describe the results for 5CB, in which the influence of ionic conduction on the current cannot be neglected. It is assumed that the elastic deformation does not contribute to the current ( $\beta = 0$ ) at high applied voltages. The applied voltages used here satisfy  $V \gg \mu\gamma_1 \log(\tan \theta_o) / (\epsilon_o \Delta\epsilon)$ , which is derived from  $t_p \ll t_o$ . At extremely high applied voltages, the initial charging current affects the current peak because the peak time becomes very short above certain applied voltage. Substituting  $\beta = 0$  into Eqs. (6) and (7), we have

$$\frac{I_p}{E^3} = \frac{S(\epsilon_o \Delta\epsilon)^2}{2\gamma_1} + Sqn\mu \frac{1}{E^2}, \quad (8)$$

$$t_p = \frac{\gamma_1 L^2 \log(\tan \theta_o)}{\epsilon_o \Delta\epsilon V^2}. \quad (9)$$

We show plots of  $I_p/E^3$  vs  $1/E^2$  and  $t_p$  vs  $1/E^2$  for the three 5CB cells at 303 K in Figures 2(a) and 2(b), respectively. Using  $\Delta\epsilon$ -value in the literature,<sup>8</sup> we obtain  $\gamma_1$ -values from the extrapolation of  $I_p/E^3$  to  $1/E^2 = 0$ . We can also determine  $qn\mu$ - and  $\theta_o$ -values from the slope of  $I_p/E^3$  vs  $1/E^2$  and of  $t_p$  vs  $1/E^2$ , respectively. These results at 303 K are summarized in Table I. The  $qn\mu$ -values are in the range  $10^{-9}$ - $10^{-10}$  S·cm<sup>-1</sup>, consistent

with those in the literature.<sup>10</sup> As listed in Table I, the  $\gamma_1$ -values for the different 5CB cells are the same, and the  $\theta_o$ -values are the same for the cells with the same surface treatment.

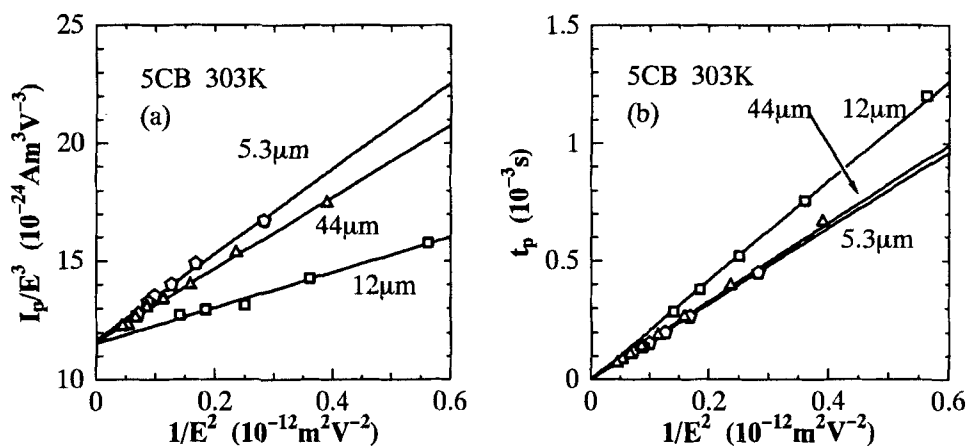


FIGURE 2 Plots of  $I_p/E^3$  vs  $1/E^2$  (a) and  $t_p$  vs  $1/E^2$  (b) for the 5CB cells at 303 K.

TABLE I The rotational viscosity  $\gamma_1$ , the product of the density and drift mobility of the ions  $qn\mu$ , and the pretilt angle  $\theta_o$  for the 5CB cells measured at 303 K.

$L$ ( $\mu\text{m}$ )	Substrate surfaces	$\gamma_1$ (Pa·s)	$qn\mu$ (S·cm <sup>-1</sup> )	$\theta_o$ (°)
5.3	Polyimide	0.063	$9.1 \times 10^{-10}$	83.5
12	In <sub>2</sub> O <sub>3</sub>	0.064	$3.7 \times 10^{-10}$	86.5
44	Polyimide	0.063	$7.6 \times 10^{-10}$	83.9

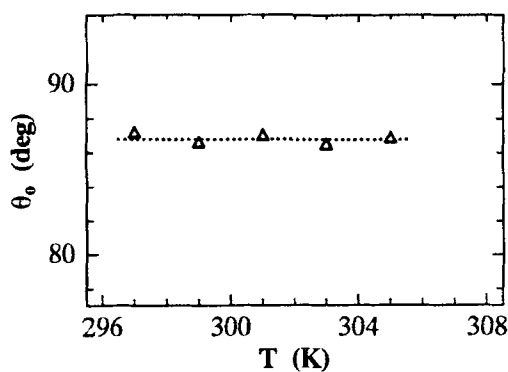


FIGURE 3 Temperature dependence of  $\theta_o$  for the 5CB cell without polyimide alignment layers.

Temperature dependence of  $\theta_o$ ,  $\gamma_1$  and  $qn\mu$ , which are measured in the 12  $\mu\text{m}$  5CB cell, is shown in Figures 3 and 4. We see that  $\theta_o$ -values are independent of temperature because the temperature range is narrow and far from the transition temperature,<sup>11</sup> while the  $qn\mu$ -values exponentially vary with reciprocal temperature, whose activation energy is 0.77 eV. The  $\gamma_1$ -values are in agreement with those in the literature,<sup>5,8,9</sup> and especially, in excellent agreement with those by the electro-optic method.<sup>5</sup>

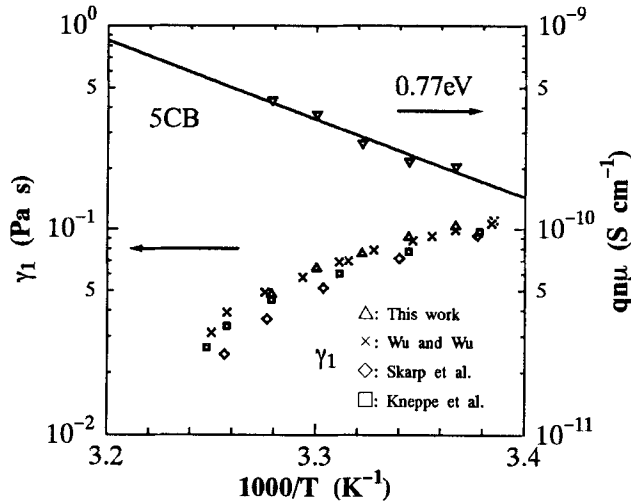


FIGURE 4 Temperature dependence of  $\gamma_1$  and  $qn\mu$  for the 5CB cell with 12  $\mu\text{m}$  in thickness.  $\triangle$ ,  $\times$ ,  $\square$  and  $\diamond$  represent the data points of  $\gamma_1$  measured in this work, by Wu and Wu (Ref. 5), by Skarp *et al.* (Ref. 8), and by Knepe *et al.* (Ref. 9), respectively.  $\nabla$  represents the data points of  $qn\mu$  measured in this work.

### Ionic Conduction Free Current Transients

We describe the results for the fluorinate mixtures and ZLI2293, in which the conduction current can be neglected in comparison with the displacement current. Thus, we reduce Eqs. (6) and (7) to

$$I_p^{-\frac{1}{3}} V = L \left( \frac{2\gamma_1}{S(\epsilon_o \Delta \epsilon)^2} \right)^{\frac{1}{3}} \left( 1 + \frac{\beta}{V} \right), \quad (10)$$

$$\sqrt{t_p} V = L \sqrt{\frac{\gamma_1 \log(\tan \theta_o)}{\epsilon_o \Delta \epsilon}} \left( 1 + \frac{\beta}{V} \right), \quad (11)$$

respectively. If the contribution of the conduction current to the peak current is not negligible, the sign of  $\beta$  is negative. We can therefore experimentally check whether or not the conduction current is negligible from the sign of  $\beta$ .

Plots of  $V/I_p^{1/3}$  vs  $1/V$  and  $\sqrt{t_p}V$  vs  $1/V$  are shown in Figures 5(a) and 5(b), respectively, using the  $I_p$ - and  $t_p$ -values in the fluorinate mixture A, B and C at 293 K. We find that experimental data points lie on the straight lines, which are almost parallel to the horizontal axis, indicating that the elastic deformation does not greatly contribute to the current peak at high applied voltages. Using  $\Delta\epsilon$ -value, the  $\gamma_1$ -values can be determined from the extrapolation of  $V/I_p^{1/3}$  to  $1/V = 0$ . The  $\theta_o$ -values can also be determined from the extrapolation of  $\sqrt{t_p}V$  to  $1/V = 0$ . The  $\beta$ -values do not affect the determination of  $\gamma_1$  and  $\theta_o$ . These results are shown in Table II.  $\theta_o$ -values by this method are in good agreement with those by the crystal rotation method.<sup>12</sup>

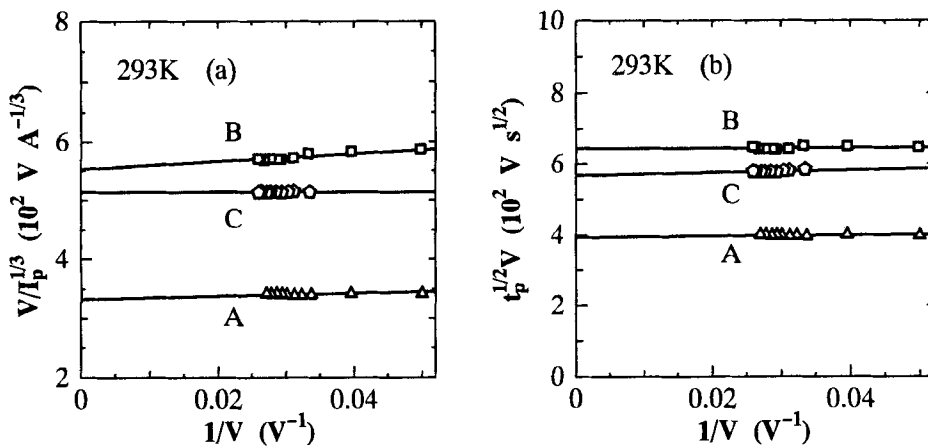


FIGURE 5 Plots of  $V/I_p^{1/3}$  vs  $1/V$  (a) and  $\sqrt{t_p}V$  vs  $1/V$  (b) at 293 K. A, B and C represent the fluorinate mixture A, B and C, respectively.

TABLE II Physical properties of fluorinate mixture A, B and C at 293 K. The dielectric anisotropy  $\Delta\epsilon$  measured at 1 kHz by the dielectric method (Ref. 13). The pretilt angle  $\theta_o$  measured by the crystal rotation (CR) method and the present transient current (TC) method. The rotational viscosity  $\gamma_1$  measured with the TC method.

Sample	$L$ ( $\mu\text{m}$ )	$\Delta\epsilon$	$\theta_o$ ( $^\circ$ ) [CR]	$\theta_o$ ( $^\circ$ ) [TC]	$\gamma_1$ (Pa·s) [TC]
A	4.8	7.6	87.1	87.1	0.15
B	5.7	5.2	85.4	87.2	0.19
C	5.1	5.5	87.1	85.4	0.24

We also measure  $\gamma_1$ - and  $\theta_o$ -values of the ZLI2293 cell by the same procedure as those for the fluorinate mixtures.<sup>1,2</sup> The  $\gamma_1$ -values measured in this work are in excellent agreement with the Merck data.<sup>14</sup> The temperature dependence of  $\theta_o$ -values is also in



excellent agreement with that in the literature.<sup>11</sup>

## CONCLUSIONS

We have derived the expressions relating the peak current and peak time in the transient current to the physical quantities ( $\gamma_1$ ,  $\Delta\epsilon$ ,  $\theta_o$  and  $qn\mu$ ). Based upon these equations, we have developed the procedure to determine the physical quantities such as  $\gamma_1$  and  $\theta_o$  of the NLC cells and have examined the procedure for NLCs with or without impurity ions.

We have measured the transient current of 5CB, ZLI2293 and fluorinate mixtures (A, B and C) at nematic temperatures. The conduction current is not negligible in 5CB, but negligible in ZLI2293 and the fluorinate mixtures. We have determined the  $\gamma_1$ -,  $\theta_o$ - and  $qn\mu$ -values of the 5CB cells and the  $\gamma_1$ - and  $\theta_o$ -values of the ZLI2293 cell and the fluorinate mixture cells with this method. The  $\gamma_1$ -values of 5CB and ZLI2293 are in agreement with those in the literature. The  $\theta_o$ -values of the fluorinate mixtures are in agreement with those using the crystal rotation method.

## ACKNOWLEDGMENTS

The authors would like to thank Mr. K. Hattori, Mr. H. Wakemoto and Mr. H. Kubota, Matsushita Electric Industrial Co., Ltd., for the preparation of the samples and for the crystal rotation measurement. The authors would like to thank E. Merck for supplying ZLI2293. One of the authors (M. I.) would like to thank Association for Promotion of Electrical, Electronic and Information Engineering for financial support.

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