

Rheological properties in discotic liquid crystalline phases of 2,3,7,8,12,13-hexa(*n*-tetradecanoyloxy)truxene

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Measurements of rheological properties, including the electrorheological effect, are made in liquid crystalline phases, discotic nematic (N_D) and discotic disordered rectangular columnar (D_{rd}) phases, of 2,3,7,8,12,13-hexa(*n*-tetradecanoyloxy)truxene. It is found that the fluidity of the N_D phase is Newtonian and an application of high electric field perpendicular to the flow direction (velocity gradient direction) causes a decrease of the viscosity. These results indicate that a flow alignment of the director occurs with its orientation near the velocity gradient direction and the signs of the Leslie coefficients α_2 and α_3 are positive. From the temperature dependence of α_2 , instability of the flow alignment structure in N_D and D_{rd} phases is also discussed.

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I. INTRODUCTION

Liquid crystals are characterized by orientational and/or positional orders, and these orders give rise to a variety of physical properties. Of various physical properties inherent to the liquid crystals, the fluidity is one of the characteristic properties of the liquid crystals, and has been studied in some liquid crystalline phases. Especially the simplest rodlike nematic (N) phase has been thoroughly explored and it has been clarified that the steady shear flow makes the director align near the flow direction, if the Leslie coefficients α_2 and α_3 fulfill the conditions of $\alpha_2 < 0$ and $\alpha_3 < 0$, with a flow alignment angle θ given by $\tan^{-1}(\alpha_3/\alpha_2)$ [1–5]. This behavior, however, is modified if the smectic A (SmA) phase exists below the N phase. In this case, the sign of α_3/α_2 becomes negative when approaching to the SmA - N phase-transition point, making the flow alignment not possible, but a tumbling motion of the director around the neutral axis [3,6–12]. Thus, the rheological properties of the N phase have been well explored, but those of the discotic nematic (N_D) phase are not fully understood.

For the rheological properties of the N_D phase, it has been theoretically suggested that a flow alignment of the director would be induced, in a similar manner to the case of the rodlike liquid crystals, if $\alpha_3/\alpha_2 > 0$ [13]. However, in contrast to the case of the rodlike nematic phase, it is indicated that the flow alignment occurs if the signs of α_2 and α_3 are positive. The flow alignment of the director in the N_D phase is also discussed in recent computer simulations for capillary Poiseuille flow [14,15] and simple shear flow [16]. Experimental studies on the rheological properties of the N_D phase have been made recently [17–19]. In an aqueous solution of cesium perfluoro octanate, it was suggested, from a small-angle neutron scattering study, that a shear deformation induces a flow alignment of the director in the N_D phase [17]. In sodium dodecyl sulfate solution, rheological and NMR measurements were made on the N_D phase, showing that a shear thinning occurs at higher shear rates and the director aligns near the velocity gradient direction [18]. In addition,

the flow properties of mesophase pitch have been measured to clarify the fluidity and its relation to the domain structure [19]. These experimental results give us important information for the rheological properties of the discotic liquid crystals, but for further understanding of the rheological properties, those for the thermotropic discotic liquid crystals are essential.

In the present study, rheological measurements were made on thermotropic discotic liquid crystalline phases of 2,3,7,8,12,13-hexa(*n*-tetradecanoyloxy)truxene, $(C_{13}H_{27}COO)_6$ -TX. This material has a disklike molecular structure composed of truxene, 10,15-Dihydrotribenzo[*a-f-k*]Triindeno[2,3:2',3':2'',3''] benzene, and six $C_{13}H_{27}COO$ chains (Fig. 1). It is reported that $(C_{13}H_{27}COO)_6$ -TX undergoes a series of phase transitions with increase in temperature; crystal (Cr)-nematic (N_D)-columnar (D_{rd})-columnar (D_{ho})-isotropic (I) [20,21]. The N_D phase is characterized by the orientational order of the director, which in the case of the discotic liquid crystals specifies the average direction of the normal to the discotic plane. On the other hand, D_{rd} and D_{ho} phases are character-

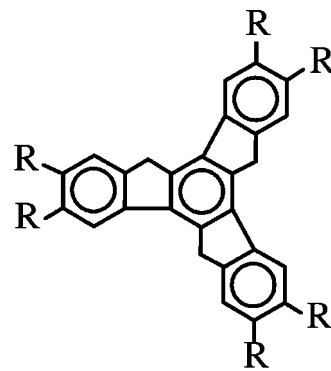


FIG. 1. Molecular structure of 2,3,7,8,12,13-hexa(*n*-tetradecanoyloxy)truxene, $(C_{13}H_{27}COO)_6$ -TX.

ized by columnar structures with a rectangular arrangement of the columns having a disordered molecular stacking and with a hexagonal arrangement of the columns having an ordered molecular stacking, respectively. Of these liquid crystalline phases, rheological measurements were made on the N_D and the D_{rd} phases to clarify the temperature dependence of the viscosity, the shear stress versus shear rate relationship, and the effect of the electric field on the rheology [electrorheological (ER) effect]. Based on these results, rheological properties of discotic liquid crystalline phases, mainly those of N_D phase, are discussed.

II. EXPERIMENTS

$(C_{13}H_{27}COO)_6$ -TX was synthesized according to the method by Lee *et al.* [22]. The purification was made by successive recrystallization from ethanol-chloroform solution and its purity was determined to be better than 99.8% by the elemental analysis. The phase-transition temperature from the N_D to the D_{rd} phase was determined from viscosity measurement to be 359.6 K. The viscosity, the shear stress versus shear rate relationship, and the ER effect were measured with a concentric cylinder viscometer. To observe the ER effect, ac high voltages were applied to the gap (1 mm) between the inner and the outer cylinders of the viscometer to generate an electric field of a few $kV\ mm^{-1}$ along the velocity gradient direction, i.e., perpendicular to the velocity (flow) direction. For supplying ac high voltages, an ac voltage of 0.1–2 V from an oscillator (3325B, HP, USA) was amplified 1000 times with a high-voltage amplifier (HVA4321, NF, Japan). In some cases, a phase-sensitive detection of the current passing through the specimen was made with a lock-in amplifier (LI5640, NF, Japan) to obtain the dielectric permittivity under a shear deformation. In order to measure the temperature dependence of the rheological properties, the temperature of the sample was controlled with a precision of 0.01 K using a temperature controller (340, Lakeshore, USA) and a heater and a thermocouple (chromel-constantan) attached on the outer cylinder. Throughout this article the amplitude of the ac electric field is expressed in root mean square, which gives an effective strength of the ac field.

III. RESULTS AND DISCUSSION

A. Rheology

Temperature dependence of the viscosity is measured at a shear rate of $32.95\ s^{-1}$ and the result is given in Fig. 2. As can be expected from the molecular shape of $(C_{13}H_{27}COO)_6$ -TX, a large viscosity (a few Pa s), which is about two orders magnitude larger than that of the N phase of 5CB(4-*n*-pentyl-4'-cyanobiphenyl) [5] was observed. As apparent from this figure, the temperature dependence of the viscosity is characteristic. With an increase in temperature, the viscosity decreases almost linearly with temperature in N_D phase, but above the N_D - D_{rd} phase-transition point, $T_C = 359.6\ K$, it increases largely with a change of the temperature coefficient near 362 K (T_A), which is specified by an arrow in Fig. 2. Although the sign of the temperature coefficient

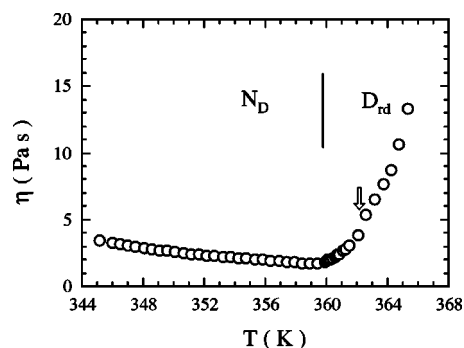


FIG. 2. Temperature dependence of the viscosity (η) measured at a shear rate of $32.95\ s^{-1}$. The nematic (N_D) to columnar (D_{rd}) phase-transition point (T_C) and the temperature (T_A), at which a change of the temperature dependence is observed, are specified by a solid line and an arrow, respectively.

of the viscosity changes at T_C , a continuous and reversible change is observed in the viscosity when passing through T_C , indicating that the N_D - D_{rd} phase transition is second-order or second-order like. To investigate the fluidity in these phases, the shear stress versus shear rate relationship is measured at 345.2 K (N_D phase) and at 360.5 and 362.6 K (D_{rd} phase), and the results (log-log plot) are given in Fig. 3. In this figure, straight lines, which represent Newtonian flows ($\tau \propto \dot{\gamma}$), are also given to understand whether the flow is Newtonian or not. As this figure shows, the data at 345.2 K (N_D phase) are just on the straight line, indicating that the flow in this phase is Newtonian. This flow property is contrasted to the experimental results for the lyotropic N_D phase [18] and mesophase pitch [19]; it is reported that with increase in shear rate a shear thinning occurs with the viscosity decreasing at higher shear rates. The non-Newtonian flow in the N_D phase is also suggested from computer simulations [14–16]. Referring to these results, a possibility of

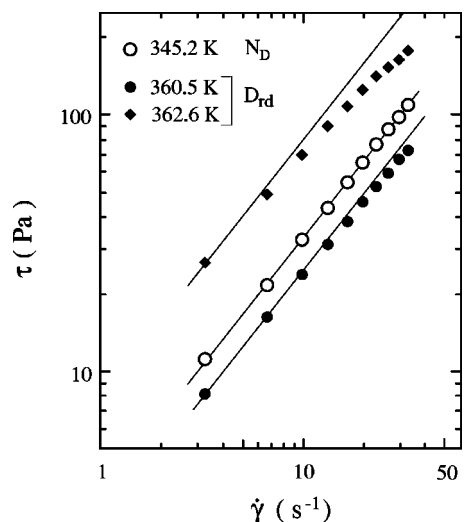


FIG. 3. The shear stress (τ) vs shear rate ($\dot{\gamma}$) relationship in N_D and D_{rd} phases (log-log plot). Straight lines representing Newtonian flows ($\tau \propto \dot{\gamma}$) are given to understand whether the flow is Newtonian or not. The flow in the N_D phase is Newtonian and that in the D_{rd} phase is non-Newtonian.

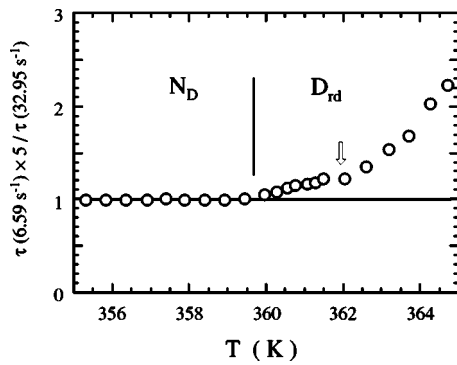


FIG. 4. Degree of deviation from the Newtonian flow: $\tau(6.59 \text{ s}^{-1}) \times 5 / \tau(32.95 \text{ s}^{-1})$. A deviation from a Newtonian flow occurs gradually above T_C and shows a change of the temperature dependence at T_A . T_C and T_A are specified by a solid line and an arrow, respectively.

non-Newtonian flow in $(C_{13}H_{27}COO)_6$ -TX is not ruled out if the measurement is made in much wider shear rate region. While in the D_{rd} phase, non-Newtonian flow is observed with a slight deviation from Newtonian flow at 360.5 K and a large deviation at 362.6 K, indicating that more ordered columnar structure grows up with increase in temperature. To make clear the temperature dependence of the fluidity change, the shear stress at a shear rate of 6.59 s^{-1} is also measured as a function of temperature to get temperature dependence of a shear stress ratio $[\tau(6.59 \text{ s}^{-1}) \times 5] / \tau(32.95 \text{ s}^{-1})$ (Fig. 4). This quantity specifies a degree of the deviation from the Newtonian flow; in the case of the Newtonian flow it is unity but deviates from unity if the flow becomes non-Newtonian. As this figure shows, the flow is Newtonian at all temperatures in the N_D phase, but it continuously changes to non-Newtonian above T_C and shows a change of the temperature dependence around T_A . Even from this result, it is confirmed that the N_D - D_{rd} phase transition is second-order or second-order like and there is some shear-induced structural change around T_A .

B. ER effect

The effect of the electric field on the rheology, ER effect, has also been studied in N_D and D_{rd} phases. In Fig. 5,

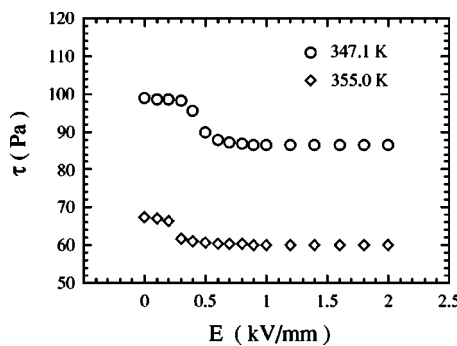


FIG. 5. ER effect in the N_D phase: a change of the shear stress upon application of the electric field (1 kHz) under a shear rate of 32.95 s^{-1} . The shear stress decreases above $\sim 0.3 \text{ kV/mm}$ and becomes constant at high fields.

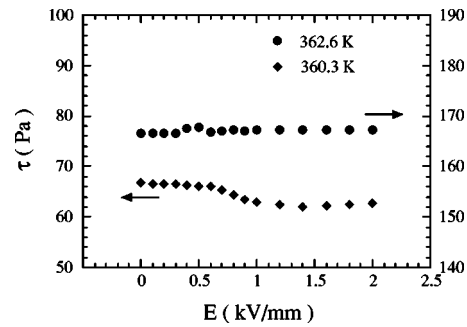


FIG. 6. ER effect in the D_{rd} phase: a change of the shear stress upon application of the electric field (1 kHz) under a shear rate of 32.95 s^{-1} . The ER effect in the D_{rd} phase is smaller than that in the N_D phase.

electric-field dependence of the ER effect in the N_D phase observed at a shear rate of 32.95 s^{-1} , is given. At 347.1 K (N_D phase, far from T_C), the shear stress gradually decreases above $\sim 0.3 \text{ kV/mm}$ and becomes constant at higher electric fields larger than 1 kV/mm. Such a decrease of the shear stress is also confirmed at 355.0 K (N_D phase, near T_C), although the field-induced shear stress change occurs at lower electric field with its amount smaller than that for 347.1 K. In D_{rd} phase, a smaller ER effect than those in N_D phase is observed with its magnitude being negligible at temperatures far from T_C (Fig. 6). To make clear the temperature dependence of the ER effect, the difference in the shear stress between at 0 kV/mm and at 2 kV/mm, $\Delta\tau = \tau(2 \text{ kV/mm}) - \tau(0 \text{ kV/mm})$, is measured as a function of temperature (Fig. 7). In this figure, the corresponding viscosity change $\Delta\eta = \Delta\tau / \dot{\gamma}$, where $\dot{\gamma}$ is the shear rate, can also be read from the scale in the right-hand side. As this figure shows, with an increase in temperature the ER effect becomes smaller in the N_D phase, and after a change of the temperature coefficient at T_C it decreases to almost zero around T_A .

C. Flow alignment and its instability

With respect to the rheological properties of discotic liquid crystalline phases, some theoretical studies have been

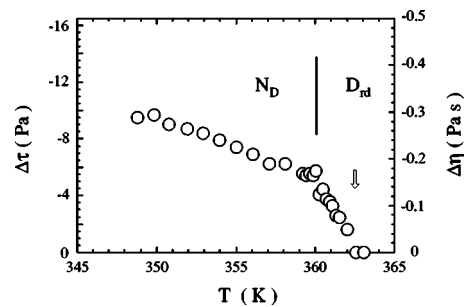


FIG. 7. Temperature dependence of the shear stress change induced by the electric field (1 kHz), $\Delta\tau = \tau(2 \text{ kV/mm}) - \tau(0 \text{ kV/mm})$, under a shear rate of 32.95 s^{-1} . The corresponding viscosity change $\Delta\eta = \Delta\tau / \dot{\gamma}$ can be read from the scale in the right-hand side. $\Delta\eta$ corresponds to $-\alpha_2$. T_C and T_A are specified by a solid line and an arrow, respectively.

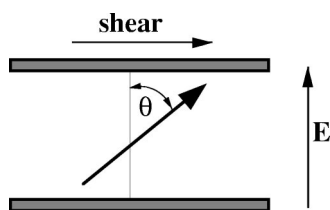


FIG. 8. Geometry specifying the orientation of the director and the directions of the steady shear flow and the electric field. The angle between the director and the velocity gradient (electric field) direction is θ .

made on the N_D phase but little is known on the columnar phases [13,23–25]. In the present study, therefore, discussion is focused on the rheological property in the N_D phase, referring to the theoretical studies on N_D phase and well-studied ER effect of the N phase. In the N phase it has been suggested that the ER behavior is clearly understood on the basis of the flow alignment of the director, which is determined by a balance of electric and viscous torques exerted on the director [3,4]. In the case of the liquid crystal having a positive dielectric anisotropy, e.g., 5CB, the electric field makes the director align along the velocity gradient direction, leading to an increase of the viscosity [5]. While if the dielectric anisotropy is negative as in the case of MBBA (4-methoxybenzylidene-4'-*n*-butylaniline), a high electric field makes the director align along the velocity direction and causes a decrease in the viscosity [26]. If a steady shear flow and an electric field are applied as shown in Fig. 8, the ER behaviors can be understood in terms of following equations [4]:

$$(\alpha_3 \sin^2 \theta - \alpha_2 \cos^2 \theta) \dot{\gamma} - 1/2 \epsilon_a E_0^2 \sin 2\theta = 0, \quad (1)$$

$$\eta(\theta) = 1/2 [(\alpha_3 + \alpha_6) \sin^2 \theta + (\alpha_5 - \alpha_2) \cos^2 \theta + \alpha_4]. \quad (2)$$

Here, $\alpha_2 - \alpha_6$ are Leslie coefficients, θ is the angle between the velocity gradient direction and the optical axis of the director, $\dot{\gamma}$ is the shear rate, ϵ_a is the dielectric anisotropy, E_0 is the amplitude of the electric field, and $\eta(\theta)$ is the viscosity. Equation (1) is derived from the balance of viscous and electric torques exerted on the director and determines the flow alignment angle θ_0 . The viscosity $\eta(\theta)$ can be calculated from Eq. (2) if the flow alignment angle θ_0 is derived from Eq. (1). Here in Eq. (2), $\alpha_1 \cos^2 \theta \sin^2 \theta$ is not included, since α_1 is generally much smaller than other Leslie coefficients [3].

For discussing the ER effect in the N_D phase, it would be necessary to consider the flow alignment behavior in the absence of the electric field, $E=0$. In such a case, Eq. (1) reduces to $\alpha_3 \sin^2 \theta - \alpha_2 \cos^2 \theta = 0$ and the flow alignment angle θ_0 is given by Eq. (3),

$$\tan \theta_0 = \pm \sqrt{\alpha_2 / \alpha_3}. \quad (3)$$

As for the sign of this Eq. (3), it is theoretically suggested that it depends on the signs of α_2 and α_3 [13]. In the case of the N phase, where $\alpha_2 < 0$ and $\alpha_3 < 0$, the plus sign in Eq. (3) gives a stable solution of θ_0 with its angle being in the range of $45^\circ < \theta_0 < 90^\circ$. While, if $\alpha_2 > 0$ and $\alpha_3 > 0$, a stable solu-

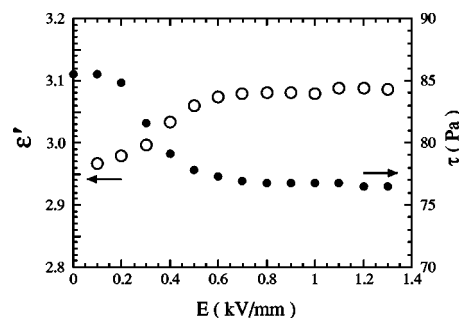


FIG. 9. Electric-field dependence of the dielectric permittivity and the shear stress obtained simultaneously at a shear rate of 32.95 s^{-1} in the N_D phase (351.3 K). The frequency of the electric field is 1 kHz.

tion is given by minus sign with $-45^\circ < \theta_0 < 0^\circ$, and this type of flow alignment occurs in the discotic N_D phase. The sign of Eq. (3), thus, is closely related to the molecular shape, but the viscosity when the flow alignment occurs can be obtained without considering it. It can be expressed, using Eqs. (2) and (3), as Eq. (4),

$$\eta(\theta_0) = \frac{1}{2} (-\alpha_3 + \alpha_4 + \alpha_6). \quad (4)$$

If we use Miesowicz viscosities (η_1, η_2, η_3), $\eta(\theta_0)$ can be expressed in terms of η_1 and η_2 as $\eta(\theta_0) = \eta_2 - (\eta_2 - \eta_1) \cos^2 \theta_0$. Here, the Miesowicz viscosities η_1 , η_2 , and η_3 specify the viscosities when the director aligns along the velocity gradient, the flow, and the neutral directions, respectively, and are related to the Leslie coefficients as $\eta_1 = (\alpha_4 + \alpha_5 - \alpha_2)/2$, $\eta_2 = (\alpha_3 + \alpha_4 + \alpha_6)/2$, and $\eta_3 = \alpha_4/2$ [1].

If a high electric field is applied along the velocity gradient direction, the viscosity varies due to a change of the flow alignment direction. In the N_D phase of $(C_{13}H_{27}COO)_6$ -TX, the viscosity decreases to a constant value upon application of the electric field as shown in Fig. 5, suggesting that at high electric fields the director aligns along the velocity gradient direction, i.e., $\theta_0 = 0^\circ$. This alignment under high electric fields is also confirmed from our preliminary result on the simultaneous measurements of the shear stress and the dielectric permittivity along the velocity gradient direction in the N_D phase (Fig. 9). With an increase in the amplitude of the electric field, the dielectric permittivity increases and becomes constant at high electric fields, which is contrasted to the behavior of the shear stress. This behavior of the dielectric permittivity also suggests that at high electric fields the director aligns along the direction of the electric field (velocity gradient), since the dielectric anisotropy of the discotic liquid crystal is generally positive unless some special group is attached to the molecule [27]. The viscosity at high electric fields, thus, can be characterized by $\theta_0 = 0^\circ$ and is expressed using Eq. (2) and the Parodi's relation $\alpha_2 + \alpha_3 = \alpha_6 - \alpha_5$ as $\eta(0^\circ) = 1/2 (-\alpha_3 + \alpha_4 + \alpha_6 - 2\alpha_2)$. From $\eta(0^\circ)$ and $\eta(\theta_0)$, the change of the viscosity under the application of the electric field can be expressed as $\Delta\eta = \eta(0^\circ) - \eta(\theta_0) = -\alpha_2$. This relation and the experimental result of $\Delta\eta < 0$ indicates that α_2 of $(C_{13}H_{27}COO)_6$ -TX is positive. Furthermore, if we consider the fact that a flow alignment is

achieved in $(C_{13}H_{27}COO)_6$ -TX, i.e., $\alpha_2\alpha_3 > 0$, we can suggest that α_3 is also positive. The positive signs of α_2 and α_3 thus obtained coincide with the theoretically expected ones for the discotic N_D phase. We can estimate the value of α_2 from $\Delta\eta$ (Fig. 7); $\alpha_2 = 0.38$ Pa s at 347.1 K, which is contrasted to $\alpha_2 = -70$ mPa s reported for the N phase (300 K) of 5CB [28].

In the rodlike nematic phase, α_2 and α_3 are negative in the flow alignment region, but if the sign of α_3 becomes positive, the flow alignment becomes unstable, leading to a tumbling motion of the director. While in the case of discotic nematic phase, it is theoretically suggested that the role of the Leslie coefficients α_2 and α_3 may be interchanged; the flow alignment occurs if α_2 and α_3 are positive but it becomes unstable if the sign of α_2 changes to negative [13]. With respect to this instability of the flow alignment, the result of the temperature dependence of α_2 (Fig. 7) is suggestive. With an increase in temperature, α_2 decreases and becomes zero around T_A . Above T_A , precise measurement of the shear stress was not possible, owing to a large fluctuation of the observed value. Considering these facts and the anomalous temperature dependence of the viscosity around T_A (Fig. 2), it is expected that some shear-induced structural change as-

sociated with some instability of the flow alignment is brought about.

IV. SUMMARY

In the N_D and D_{rd} phases of $(C_{13}H_{27}COO)_6$ -TX, measurements of viscosity, the shear stress versus shear rate relationship, and ER effect are made, showing that there appear characteristic behaviors associated with the structures of these liquid crystalline phases. From the result of the ER effect, it is suggested that Leslie coefficients α_2 and α_3 in the discotic liquid crystal are positive, as expected from the theoretical analysis, and that the flow alignment of the director occurs with its behavior characterized in a similar manner to that of the rodlike nematic phase. A possibility of a flow alignment instability is also discussed in terms of the sign of α_2 .

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