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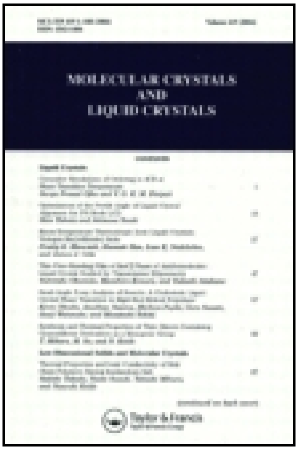

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Optical Properties of Lyotropic Poly(γ-benzyl L-glutamate) Liquid Crystals

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Optical Properties of Lyotropic Poly(γ-benzyl L-glutamate) Liquid Crystals

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To establish methodologies for determining the cholesteric sense of lyotropic poly(γ-benzyl L-glutamate) (PBLG) liquid crystals, we have examined various optical activities associated with the cholesteric twist. A colored PBLG film retaining the cholesteric structure is prepared and its pitch-band circular dichroism (CD) and optical rotatory dispersion (ORD) spectra are measured. The established relationship between cholesteric sense and ORD sign is then applied to large-pitch cholesterics in solutions where the sign of liquid crystal induced circular dichroism (LCICD) is determined. The observed LCICD signs are negative for a sidechain phenyl chromophor and positive for a guest achiral dye, acridine orange, in a left-handed PBLG liquid crystal exhibiting a negative optical rotation. We demonstrate that the ORD and LCICD signs in PBLG liquid crystals depend only on the cholesteric sense hence they can be used as simple diagnostics for the sense determination.

INTRODUCTION

Cholesteric liquid crystals are regarded as spontaneously twisted nematic mesophases in which the local director of each nematic layer has a successive slight angular displacement with respect to those of adjacent layers stacked along the axis of torsion. This structure is characterized by two principal parameters, *i.e.*, sense and pitch of the twist. In low molecular-weight, thermotropic liquid crystals, the cholesteric sense is simply dictated by the chirality of constituent molecule and remains unchanged even when temperature is changed. This empirical rule, however, does not hold true in lyotropic liquid crystals formed by a synthetic polypeptide, poly(γ-benzyl L-glutamate) (PBLG). In the pioneering work of Robinson,[1] the sign of form

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optical rotation, an indicator of the cholesteric sense, was found to reverse as the solvent dioxane was replaced by dichloromethane. Solvent-induced sense inversions have recently been reported in a variety of mixed solvent systems; in most cases the inversion is single-fold,[2,3] but even a double-fold inversion is in fact observed.[4] The cholesteric sense can also be reversed by changing temperature. Typical examples of this type of inversion are seen in PBLG liquid crystals in *m*-cresol and 1,2,3-trichloropropane (TCP).[3-5] Toward an understanding of the molecular mechanism of the sense inversion, it is important to establish the configuration of the twist, *i.e.*, its right- or left-handedness, in relation to a right-handed chirality of the PBLG α-helix.

According to a common definition,[6] the cholesteric sense is determined as follows: When an observer proceeds along the axis of cholesteric torsion to find a clockwise rotation of the local director, the structure is recognized to be right-handed. The most straightforward and decisive method of the sense determination should be a circular dichroism (CD) measurement in a cholesteric pitch band. A right-handed cholesteric liquid crystal selectively reflects a right-handed circularly polarized light within a narrow range of wavelength (pitch band) centered at

$$\lambda_0 = nP \tag{1}$$

where P is the cholesteric pitch and n is the average refractive index of the medium. A left-handed circularly polarized light, on the other hand, propagates through the medium without any major attenuation. The transmitted light in the pitch band is therefore dominated by the left-handed circular component and exhibits a negative circular dichroism.[7,8]

To date pitch-band optical properties have not been examined for PBLG liquid crystalline solutions, since their λ_0 values are large, typically 10–100 μm, beyond the wavelength range measurable by common spectrometers. In this paper we report the results of our efforts to establish the relationship between the cholesteric sense and signs of various optical activities exhibited by PBLG liquid crystals. First, the pitch-band CD and optical rotatory dispersion (ORD) signs are determined for a colored PBLG film retaining the cholesteric twisted structure. The sign of an extrinsic optical activity, “liquid crystal induced circular dichroism (LCICD)”,[9] is then determined for a sidechain phenyl chromophor and a guest achiral dye as a function of the cholesteric sense determined by using the established ORD sign. The ORD and LCICD techniques, which do not require λ_0

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within the wavelength range of visible light, are shown to be of great advantage to study the sense of large-pitch PBLG liquid crystals.

EXPERIMENTAL

Two PBLG samples with viscosity averaged molecular weights of $M_w = 9.6 \times 10^4$ (I) and 24.6×10^4 (II) were synthesized by the *N*-carboxyanhydride method from γ-benzyl L-glutamate using triethylamine as the initiator. All solvents were purified before use by simple distillation or vacuum distillation over proper desiccants. Acridine orange purchased from Chroma Gesellschaft Schmid & Co. was used as an achiral dye. A weighed amount of PBLG was completely dissolved in solvent, containing *ca.* 1×10^{-3} M of dye if necessary, then the solution was sealed in a cell and matured at room temperature to ensure the homogeneous development of the liquid crystaline structure. The concentration *C*, expressed in volume fraction of polymer in solution, was calculated by assuming the specific volume of PBLG to be 0.787 ml/g.

CD and ORD spectra were measured using a JASCO J-20 recording spectropolarimeter and a Shimazu QV-50 spectrometer with ORD attachments, respectively. Water-jacketed quartz cells, with pathlengths of 0.1 mm for CD and 0.2 mm for ORD, were used. The temperature of the sample was controlled by circulating water and monitored by a copper-constantan thermocouple. In order to eliminate influences of birefringence upon the optical rotation, ORD measurements were repeated for different values of the angle between the vertical position of the sample cell and the plane of polarization of the incident beam. For this purpose the cell was mounted on a holder and both the polarizer and the analyzer were designed to be rotated in a plane perpendicular to the incident beam.

RESULTS AND DISCUSSION

A. Pitch band CD and ORD spectra

It is generally recognized that the cholesteric pitch in PBLG liquid crystals decreases with increasing the polymer concentration.[4,10-12] If such a decrease in *P* could continue in the entire range of *C*, one would obtain colored liquid crystalline solutions in which *P* is as small as the wavelength of visible light. However, as evidenced by the fact that PBLG helices in most solid films are packed more or less parallel each other, the cholesteric twist in solutions will be disrupted when a twist angle between adjacent helices becomes very large at increased concentrations. Retention of the continuous cholesteric twist into the solid state therefore requires certain special treatments. Samulski and Tobolsky[13] suggested to use a plasticizer together with common casting solvents. Sasaki *et al.*[14] obtained a colored PBLG film by using *m*-cresol–dimethylformamide (1 : 100) mixture as a casting solvent. In the latter case, a three-dimensional gellike network would form during the solvent evaporation process to freeze the cholesteric supermolecular structure. With further evaporating residual solvents, the spacing between helices tends to be smaller and so does the pitch. The λ_0 value can be shortened by this method to several hundreds of millimicrons after the complete removal of solvents.

Figure 1 shows CD and ORD spectra of PBLG film prepared according to the prescription of Sasaki *et al.* Since PBLG has no absorptions at wavelengths longer than 270 mμ, the origin of ob-

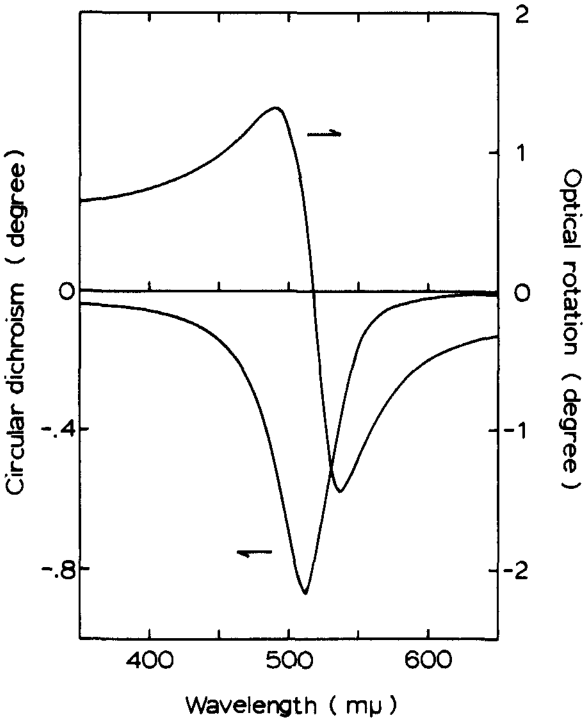

FIGURE 1 Pitch-band CD and ORD spectra of a colored PBLG-I film (50 μm thick) cast from a *m*-cresol–dimethylformamide (1 : 100) solution at 60°C.

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served CD effect is assigned to a selective light reflection. The center wavelength of the CD band $\lambda_0 = 515$ mμ, corresponding to green cholesteric color visible under natural light, calculates $P = 340$ mμ for $n = 1.5$. The sense of the retained twist is of right-handed: A negative CD sign indicates that the right-handed circularly polarized light is selectively reflected by this right-handed cholesteric slab. The corresponding ORD spectrum shows an anomalous dispersion with the sign changing from negative to positive as the wavelength is decreased passing through the pitch band, *i.e.*, a negative anomalous dispersion. Note that types (or signs) of CD and ORD effects are concordant, similar to the case of intrinsic optical activities. The crossover point of the anomalous dispersion is located exactly at λ_0. It is also seen that, while the circular dichroism falls rapidly to zero outside the pitch band, the optical rotation retains appreciably large values even at wavelengths far from λ_0. This characteristic of the ORD spectrum enables us to determine the sense of large-pitch PBLG liquid crystals, based on the ORD sign versus sense relationship established above.

B. ORD spectra of PBLG liquid crystalline solutions

De Vries[7] has formulated the wavelength λ dependence of the optical rotation exhibited by a cholesteric liquid crystal as follows,

$$\Theta = \frac{\pi(\Delta n)^2 P}{4} \frac{1}{\lambda^2\{1 - (\lambda/\lambda_0)^2\}} \tag{2}$$

where $\Theta = d\Psi/dZ$ is the optical rotatory power per unit thickness and Δn is the birefringence of each nematic layer. The sign of P should be taken as positive for the right-handed cholesterics. This equation predicts that, in the $\lambda < \lambda_0$ region, Θ initially decreases toward a minimum value (see Figure 1) then begins to increase with a further decrease in λ. For PBLG liquid crystalline solutions which have λ_0 values larger than several microns, only the latter part of dispersion can be observed in the λ range of visible light. Under a condition $\lambda \ll \lambda_0$, Eq. 2 reduces to a simple form,

$$\Theta = \pi(\Delta n)^2 P/4\lambda^2 \tag{3}$$

The results of ORD measurements in TCP and 1,2-dichloroethane (EDC) are shown in Figure 2 where the rotation angle is plotted against the square inverse of λ according to Eq. 3. A positive rotation in TCP varying its magnitude in proportion to λ^{-2} indicates the presence of a right-handed cholesteric twist. A solution in *m*-cresol–

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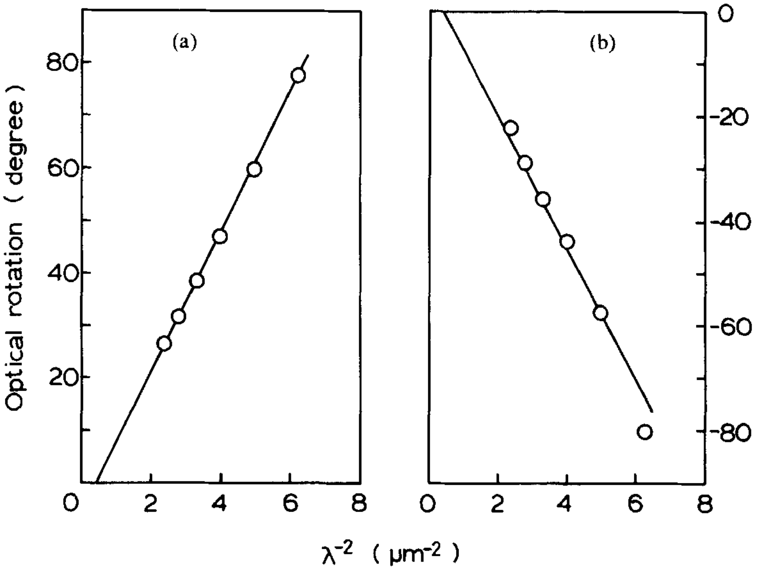

FIGURE 2 De Vries plots of ORD data for PBLG-II liquid crystals ($C = 0.15$) in (a) TCP and (b) EDC at room temperature.

dimethylformamide (1 : 100) also exhibits a positive rotation confirming that both cholesteric phases in this solution and in films cast from the same solvent system (cf. Figure 1) have the right-handed structure. On substituting EDC for TCP, the cholesteric sense reverses to left as demonstrated by a negative optical rotation shown in Figure 2b.

The slope of the Θ vs. λ^{-2} plot, $\pi(\Delta n)^2P/4$, depends on both pitch and birefringence. P can be directly determined from microscopic measurements, while the determination of Δn requires to use Eq. 3 itself since this quantity can vary from specimen to specimen depending upon a choice of solvent, polymer concentration and orderedness of the cholesteric twist. We therefore constructed a wedge-shaped cell, 10 × 50 mm rectangular and 1 mm thick at one end, to estimate Δn through the thickness dependence of the $\pi(\Delta n)^2P/4$ value. Solutions matured in this cell developed regions of parallel striation lines (focal conic texture) and of optically uniform appearance (planar or Grandjean texture); known as two typical textures of cholesteric liquid crystals. The former texture appears when the cholesteric twisted structure is viewed from the direction perpendicular to the torsion axis, while the latter appears when the same structure is seen along this axis. A large single domain of the uniform region was

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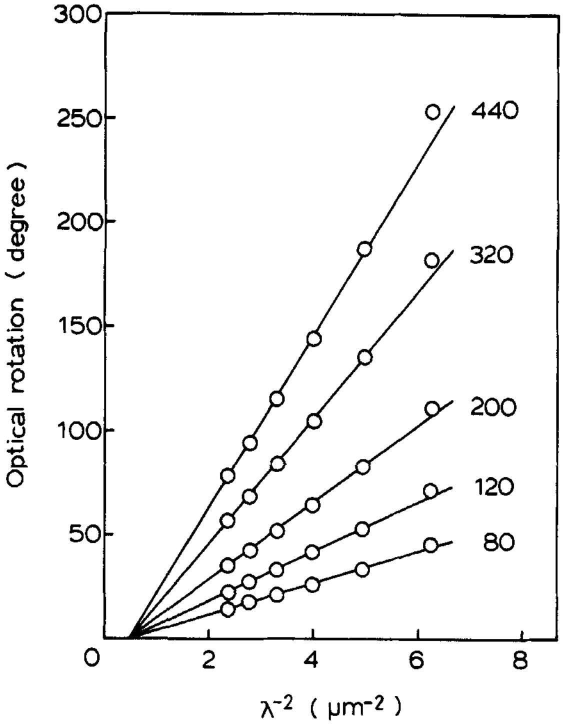

FIGURE 3 De Vries plots of ORD data for a liquid crystalline PBLG-I solution in *m*-cresol ($C = 0.20$) measured in a wedge-shaped cell at room temperature. Numerals indicate the thickness in μm.

formed in a *m*-cresol solution at $C = 0.20$ allowing us to carry out ORD measurements at several different thicknesses. Figure 3 summarizes the results. Slopes read from this figure are then plotted against thickness to give $\Delta n = 0.011$ or $\Delta n/C = 0.055$ for a value of $P = 16.2$ μm determined from the spacing between striation lines in the same specimen. Our Δn value is slightly larger than the value obtained by Robinson,[1] $\Delta n/C = 0.040$ in *m*-cresol at $C = 0.24$, suggesting the correspondingly higher orderedness of the twisted structure. In this estimation the wavelength dispersion of birefringence[15] is left outside of consideration, hence the observed value should be regarded as an average in the wavelength range of measurement.

The cholesteric slab in the wedge cell, when placed between two polars, displays a series of colors changing continuously with thickness. For example, between parallel polars, colors ranging from violet to red through blue, green and yellow toward a thicker end of the cell are seen. The color does not change when the sample is rotated on the observation stage, but does change if the angle between polars is varied. These phenomena can be readily accounted for based on the de Vries theory. By integrating Eq. 3 with respect to Z and, for example, by putting $\Psi = n\pi$ (n being zero or any positive integer) for the above case, we find that the wavelength of the transmitted light λ becomes longer in proportion to $Z^{1/2}$.

C. LCICD spectra of sidechain phenyl chromophors

Figure 4 shows ultraviolet CD spectra of the two PBLG liquid crystals whose cholesteric senses have been established in Figure 2. Intense CD triplets in the 240–270 mμ region are assigned to the LCICDs induced upon sidechain phenyl chromophors of PBLG, since they have the identical shape and peak wavelengths (254, 259 and 265 mμ) with those of an absorption spectrum and, since they disappear when the PBLG concentration is decreased to convert the liquid crystalline phase to the isotropic phase.[4] This assignment is further supported by a sign inversion from positive in TCP to negative in EDC, corresponding to the cholesteric sense inversion from right to left. A similar observation is also reported in different solvent systems.[16] The sidechain phenyl groups in an isotropic solution may exhibit very

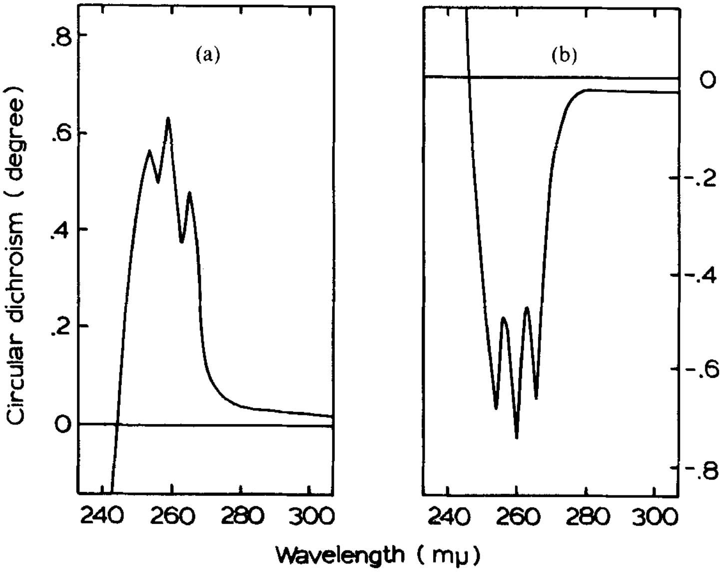

FIGURE 4 Ultraviolet LCICD spectra of PBLG-II liquid crystals ($C = 0.15$) in (a) TCP and (b) EDC at room temperature.

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weak CD effect whose sign remains always negative irrespective of solvent.[17] The origin of this extrinsic CD effect may be assigned to an asymmetric perturbation from the backbone α-heliz and/or a regular arrangement of sidechain chromophors around the backbone helix. On the other hand, the apparent dependence of sign on solvent demonstrated in Figure 4 confirms that LCICD has its origin in the cholesteric supermolecular structure formed by PBLG rods, to which the sidechain phenyl groups are attached. This LCICD technique has been applied in our previous study to prove the thermally-induced cholesteric sense inversion in PBLG-TCP liquid crystals.[4]

Sackmann and Voss[18] have shown by expanding the de Vries theory to an absorbing system that the LCICD intensity increases when either the cholesteric pitch (hance λ_0) in the region of $\lambda_0 > \lambda$ or a linear dichroism in each nematic layer is increased. The orientational order of the sidechain phenyl ring, responsible for determining the magnitude of linear dichroism, has been shown to decrease with temperature.[19] Therefore, if we employ a system in which the cholesteric pitch increases with temperature to enhance the LCICD intensity, in opposition to the effect of a reduced linear dichroism, we can see which of these two effects is predominant to determine the

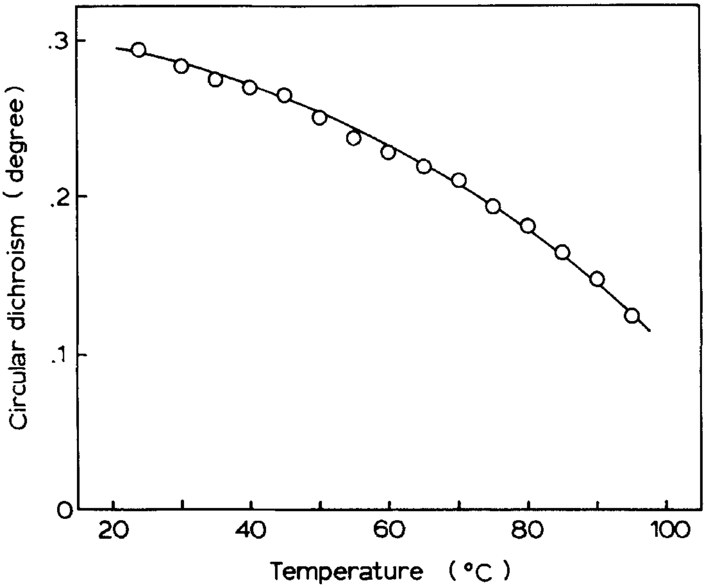

FIGURE 5 Temperature dependence of the LCICD intensity of the sidechain phenyl band at 254 mμ in a PBLG-II liquid crystal in dioxane at $C = 0.20$.

LCICD intensity at elevated temperatures. A right-handed PBLG liquid crystal in dioxane is chosen as such a system based on the observation that its pitch has a positive temperature coefficient: $dP/dT > 0$.[4] The observed LCICD intensity in this system shows a monotonic decrease (Figure 5), indicating that the reduction of linear dichroism dominates the effect of the increase in pitch. In the present case, chromophors are located at the termini of sidechains, hence we have to consider both inter- and intramolecular mechanisms for explaining this reduction of linear dichroism; the former is the reorientational motion of the helix as a whole and the latter is the sidechain segmental motion which would randomize the orientation of phenyl chromophors relative to the helix axis.[20]

D. LCICD spectra of acridine orange dissolved in a PBLG liquid crystal

Figure 6 shows the temperature dependence of the LCICD spectrum in a PBLG-EDC-acridine orange system. There appear an intense positive band at 495 mμ with a shoulder at 470 mμ and, a relatively

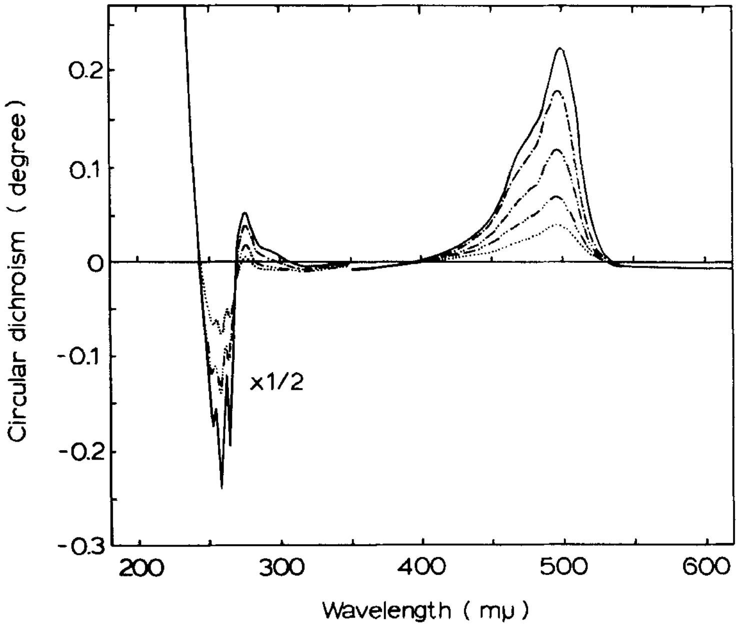

FIGURE 6 Temperature dependence of the LCICD spectrum in a PBLG-I (C = 0.15)-EDC-acridine orange system: (———) 31°C, (-·-) 42°c, (-··-) 50°C, (-····-) 56°C and (· · ·) 60°C.

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weak positive band in the 270–300 mμ region partially superimposed upon a negative triplet. The host PBLG-EDC liquid crystal is responsible only for the negative triplet, thus all positive LCICD bands are attributed to acridine orange. The decrease in the magnitude with increasing temperature is observed for every band, and should again be explained in terms of the reduction of the orientational order. Linear dichroism studies[21] have shown that absorptions at 495 and 470 mμ are associated with electronic transitions along the long, in-plane axis of monomeric and dimeric acridine oranges, respectively. The 270–300 mμ band includes two transitions polarized parallel and perpendicular to the long molecular axis.[21-23] However, the observed LCICD spectra do not reflect these characteristics. They have a single sign, positive in this left-handed cholesteric phase, and follow the shape of an absorption spectrum. The LCICD sign versus cholesteric sense relationship reported for the 495 mμ band by Tsuchihashi *et al.*[24] is in agreement with our observation. The single-sign LCICD spectrum has also been reported for anthracene.[25] All of its absorption bands, irrespective of the direction of polarization, exhibit positive circular dichroisms in a left-handed cholesteric phase and negative ones in a right-handed phase.[26] The LCICD signs of these two guest achiral dyes are thus found to be identical and vary systematically with the cholesteric sense of the host PBLG liquid crystal. Details of the chemical structure and the direction of polarization within each molecule do not influence the LCICD sign. Further experiments are in progress to interpret the observation for anthracene[25,27] that it dissolved in thermotropic cholesterics exhibits the LCICD spectrum consisting of positive and negative bands, thereby

TABLE I

Signs of optical activities exhibited by PBLG liquid crystals ($\lambda < \lambda_0$) at room temperature

Cholesteric sense	ORD sign	LCICD signs		Solvents
		Phenyl groups	Guest dyes	
Right-handed	+	+	−	Dioxane Chloroform *m*-Cresol[a] TCP[a]
Left-handed	−	−	+	EDC Dichloromethane

[a] In these solvents the cholesteric sense reverses to left at elevated temperatures.

differing from the single-sign spectrum in lyotropic PBLG liquid crystals.

Signs of various optical activities determined in this study are summarized in Table I as a function of the cholesteric sense. Also included are some typical solvents supporting right- and left-handed cholesteric phases taken from a more complete list in Ref. 4. It should be noted that the ORD and LCICD signs depend only on the cholesteric sense provided $\lambda < \lambda_0$. The sense of large-pitch PBLG cholesterics can therefore be uniquely determined by these methods, even if we can not observe the pitch-band CD spectrum directly. The LCICD technique may have some advantages over ORD measurements: It can be measured by using convensional CD spectrometers without any particular attachments and, is less influenced by the local birefringence in the specimen. The LCICD technique may also be applied to determine orientational order parameters of aromatic chromophors located at the specific position of the liquid crystalline molecule.[18,27]

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