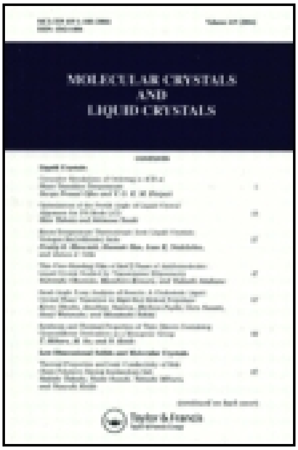

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THE CIRCULAR INTENSITY DIFFERENTIAL SCATTERING (CIDS) OF CHOLESTERIC AND BLUE MESOPHASES

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The circular intensity differential scattering of several cholesteric mixtures of CB-15 and ZLI 1612 has been been measured as a function of the scattering angle and for different ratios of pitch/wavelength. These mesophases show large CIDS ratios varying with both composition and scattering angle. Difficulties encountered in performing these experiments are discussed. The comparison of the results with a theoretical model that uses the second Born approximation shows good agreement. Preliminary studies of the cholesteric-blue phase transition as monitored by changes in the CIDS signal are also presented. The possibility to obtain information about the structural organization of these chiral mesophases and their degree of crystallinity, order parameters, etc., from the CIDS measurements is discussed.

1. INTRODUCTION

Circular Intensity Differential Scattering (CIDS) is a measure of the ability of chiral substances to scatter preferentially right or left circularly polarized light.[1-6]

CIDS is defined as the ratio:

$$\mathrm{CIDS}\ (\Theta,\Phi) = \frac{I_L(\Theta,\Phi) - I_R(\Theta,\Phi)}{I_L(\Theta,\Phi) + I_R(\Theta,\Phi)} \qquad (1)$$

where $I_L(\Theta,\Phi)$ and $I_R(\Theta,\Phi)$ are the scattered intensities at a given direction in space (Θ,Φ) for incident left and right circularly polarized light respectively. Clearly, this ratio can only take values between +1 and -1.

This effect was orginally observed indirectly as anomalies in the circular dichroism spectra of highly condensed samples.[7] A theory of this effect within the frame of Classical Electrodynamics and the first-Born approximation has been obtained.[3-4] This theory has been recently extended to the second Born approximation.[8]

The sign, magnitude, and angular dependence of the CIDS ratio contain useful information about the structure of the chiral scatterer. Analytical solutions for the regular and differential scattering intensities of randomly-oriented scatterers shows that CIDS is much more sensitive than regular scattering to the structure of the scattering aggregates.[6] The first experimental measurements of the CIDS ratio as a function of the scattering angle in aggregates of biological origin have been recently communicated.[9-10]

We present in this paper the results of a study of CIDS of cholesteric liquid crystals as a function of both angle of scattering and pitch of the cholesteric helix. We will discuss as thoroughly as allowed by the space our experimental set up, the main difficulties we have encountered in performing these experiments, as well as a preliminary interpretation of the data in light of the current CIDS theory.

1.1. The Cholesteric Mesophases

Chiral guest molecules dissolved in a nematic mesophase impose a local twist to the longitudinal axes of the host molecules which is amplified into the bulk by the long-range molecular correlation of the solvent. Equal amounts of enantiomeric guest substances of equal optical purity induce helical structures with opposite chirality and identical pitch. This long-range chirality is responsible for the selective interaction of the cholesteric mesophases and the circularly polarized radiation. The well-known selective reflection of the two circular polarizations when the wavelength of light inside the mesophases equals the pitch of the helix is a manifestation of CIDS at 180°. This selective reflection has been investigated by numerous authors.[11-14] Nityananda and Kini[15] have obtained exact expressions for the reflection and transmission cross sections by a plane parallel cholesteric film.

Strictly speaking, a perfectly crystalline cholesteric array should only scatter light in the forward and backward direction when the light is incident along the helix axis. This is, of course, in contradiction with the large scattering cross sections exhibited by cholesteric arrays as a function of the scattering angle. This large scattering is the result of the finite range of the molecular correlations in liquid crystals. The strong scattering cross sections exhibited by cholesterics and nematics are therefore due to small amplitude orientational fluctuations[16,17] which dominate the scattering properties to the point where density fluctuations in the fluid can be easily neglected.[18] Theoretical treatments of light scattering of cholesteric liquid crystals have made use of dielectric inhomogeneities in the cholesteric to describe the observed scattering properies.[19] In the modeling of the CIDS results (see Results and Discussion sections) we have taken advantage of the fact that CIDS is a ratio technique and that in a crystalline structure the angular dependence of the CIDS patterns depends only on the structure of the unit cell.[20] The scattered intensity for left or right circularly polarized incident light ($I_{L,R}$) at a particular angle (Θ,Φ) can be written as:

$$I_{L,R}(\Theta,\Phi) = F(\Theta,\Phi)\, S_{oL,R}(\Theta,\Phi) \qquad (2)$$

where $F(\Theta,\Phi)$ is an angular-dependent scattering factor which is the same for both incident polarizations and is responsible for the extinction of the light at angles other than transmission or reflection in a perfect cholesteric crystalline array when the light is incident along the helix axis. $S_{oL,R}$ (Θ,Φ) is the scattering intensity of right or left incident circular polarization due to the unit cell.[20] Notice that when eq. (2) is replaced in eq. (1) $F(\Theta,\Phi)$ cancels in the numerator and denominator, and the CIDS is independent of $F(\Theta,\Phi)$. The theory therefore predicts differential scattering at all angles in space.

Our approach is justified inasmuch as the fluctuations are a) uncorrelated in space, and b) larger than the wavelength of light. The first condition implies that interference effects between the fluctuations do not contribute substantially to the observed angular-dependent scattering cross section. The second condition assumes that there are no important end effects in the scattering cross sections of these fluctuations, and can therefore be treated as regular, crystalline regions much longer than the wavelength of light. Our theoretical treatment is therefore equivalent to the description of one of these crystalline regions, since the CIDS ratio is unitless and independent of the actual scattering cross section of the system (see eq. 1). The main approximation involved in this model is in the description of the internal electromagnetic field. This field has been corrected up to the second Born approximation to allow for short- and long-range interactions within each crystalline region.

The study of the differential scattering properties of cholesteric liquid crystals is of interest for both a formal and practical reason. A priori, it is expected that cholesteric mesophases should exhibit rather large CIDS ratios as a function of the scattering angle. Thus, it is a unique system to confirm the predictions of the CIDS theory. Such understanding is of crucial importance in the development of CIDS into a mature optical technique. On the other hand, the use of the CIDS technique to study the cholesterics should give a new and different insight into the structure, degree of crystallinity, and order parameters of these mesophases.

1.2 The Blue Phases

The blue phases of cholesteric liquid crystals appear in a very narrow temperature range (of the order 1°C) in the transition region between the isotropic and cholesteric phases. Within this small temperature range a succession of two thermodynamically stable phases can be observed[21,22] (blue phases I and II). It was suggested by Saupe[23] that these blue phases (BPI and BPII) have a cubic structure. Further confirmation of this has come from the work of Meiboom et al,[24] Crooker et al,[25] and Hornreich and Shtrikman.[26]

Recently Nicastro and Keyes[27] have presented evidence that the types of blue phases are yet more numerous than was previously believed. Heppke et al[28] have recently communicated the existence of an electro-optic effect in blue phase systems.

The two questions appearing as the most relevant today in the study of the blue phases are: What is the structure of these phases and what are the energy constraints under which these phases become stable? Although general structural models for these phases have been proposed,[29,30] it is not as yet clear whether energy minimization computations can discriminate between them.[31] In this paper we will present a few preliminary results of the CIDS of cholesteric liquid crystals as they undergo the transition into the blue phases.

2. INSTRUMENTATION

The CIDS instrumentation used for the liquid crystal studies is shown in the block diagram (Fig. 1). This system has been described in more detail recently.[32] We used an argon ion laser at 488nm as the light source (1). The laser beam was expanded to increase the area of illumination at the sample and to avoid turbulance in the sample by local heating effects (2). The beam is then passed through a polarizer (3) in order to optimize the linear polarization incident on the modulator. An electro-optic modulator (4) (Pockels' cell) was used to produce alternately right and left circularly polarized light with an alternation frequency of 327 Hz. A modulator circuit and two power supplies produce the necessary signal for the operation of the Pockels' cell (7). The modulated light incident upon a chiral sample is differentially scattered depending on the incident polarization, and the scattered intensity for each incident polarization is detected at a head-on photomultiplier tube (6). The photomultiplier and tandem double slit system are mounted on a goniometer (13) so that detection may occur as a function of angle about the scattering volume.

The signal from the PMT is processed to obtain the ratio $(I_L-I_R)/(I_L+I_R)$ by the method of synchronous demodulation.[32-34] The PMT signal is electronically processed (8) and connected to a lock-in analyzer (9). The lock-in analyzer acts as a cross-correlator, i.e., it associates the PMT signal with the original modulation signal. In this way differences in scattered intensities from incident left and right circularly polarized light are detected.

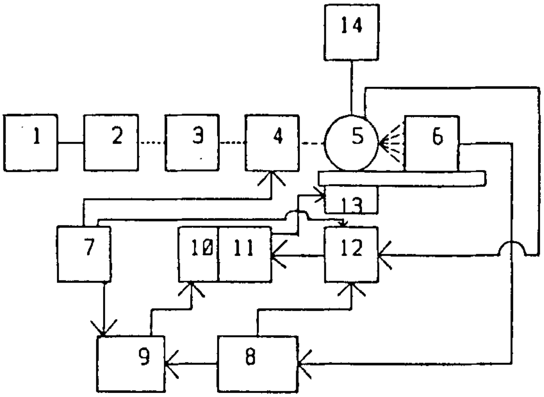


FIGURE 1 Schematic diagram of CIDS instrument

The CIDS ratio, scan angles, and various instrumental monitor values (12) are sent to a MINC 11/23 computer (11) via an A/D converter (10) for data storage and further processing. The computer also controls the motion of the goniometer during the experiment.

During the study of blue-phase liquid crystals a temperature control bath (14) was connected to a sample holder (5) with a water jacket. The temperature of the sample was maintained to within 0.02°C and monitored via a digital thermometer interfaced to the computer.

The temperature was measured by placing a platinum film sensor in direct contact with one of the quartz plates used as a mounting surface for the liquid crystals.

Some instrumental difficulties were encountered in measuring the CIDS patterns. The argon ion laser heated the sample sufficiently to disturb its structure and cause significant variations in the CIDS signals with time. This problem was satisfactorily solved by increasing the cross-sectional area of the light beam with the beam expander. Also, when the polarization of the incident light is not exactly circular (i.e., if there are linear components in the polarization), there can be contributions to the apparent CIDS signal from linear anisotropies in the sample. The electro-optic modulator used to produce circularly polarized light in our instrument creates circularly polarized light with a stray ellipticity of about one percent. This is sufficient to introduce some linear anisotropy contributions into our data when the CIDS signals are smaller than about 5 percent.

3. MATERIALS AND METHODS

All seven of the liquid crystal samples investigated were composed of mixtures of the chiral cholesteric p-2-methylbutyl-p-cyanobiphenyl CB15 (BDH) and the nematic ZLI 1612 (Merck).

The mixtures were prepared according to the weight percent of CB15 added to the nematic ZLI 1612. Five cholesteric (9.8, 19.3, 29.2, 41.2, 48.5 wt%) and two blue phase mixtures (53.9, 59.9 wt%) were prepared based on the phase diagram presented by Stegemeyer and Pollman.[35] This phase diagram was based on bulk viscosity measurement of CB15/ZLI 1612.

To find the pitches of these mixtures we used a formula of the form

$$P = \frac{k}{(wt\%)^c}$$

We evaluated the constants k and c by first putting the formula in a linear form,

$$\ln P = \ln k - c\ln(wt\%),$$

then plotting lnP vs ln(wt%) for four compositions whose pitches had been measured by spectroscopic methods and finding the least-squares slope and intercept. We obtained k and c values of 18.27 and 0.9714 respectively, with a correlation coefficient of 0.99990.

CIDS studies were performed at room temperature for the cholesteric samples. The blue phase samples were examined at different temperatures near room temperature and changes noted as a function of temperature.

All of the mixtures studied were sandwiched between a pair of square, fused quartz plates separated by a circular 10 micron spacer (Helma Cells Inc., 0.001 mm path length cell, Suprasil Quartz, 210-3-QS). The plates were cleaned before each mounting by sonication in petroleum ether (about 15 min.) to remove liquid crystals, then soaked overnight in $Na_2Cr_2O_7/H_2SO_4$. The plates were then soaked (about 15 min.) in dilute NH_4OH to remove residual ions, then rinsed with deionized, filtered water and dryed with N_2 gas.

The quartz plates were coated with polyvinyl alcohol (1g/liter H_2O) prior to application of the liquid crystals, and the plates were then rubbed on polystyrene foam to orient the PVA molecules in a uniform direction. This coating helps the samples achieve a monodomain, planar orientation with respect to the plate surfaces.

The CB15/ZLI 1612 sample was then clamped into a cell holder that maintains uniform pressure on the quartz plates. To reduce linear birefringence in the sample, the plates were oriented in the holder so that the PVA coatings of the two quartz plates were oriented at 90° with respect to each other.

The sample holder was placed on a stage at the center of the goniometer device of the CIDS instrument, and the CIDS was then measured.

Baseline measurements were performed regularly by mounting a sample of polystyrene liquid (.109µm) in the same manner as the liquid crystal sample. The baseline signal typically ranged between 0.01% to 0.1% of the full-scale signal. Most of the baseline magnitude may be attributed to stray reflections from the light entry port on the sample holder. Careful alignment of the sample cell and mounting eliminated most of these reflections, but it seems likely that the CIDS signals are still affected by stray light at angles close to 0° or 180°. A new design is currently being considered to reduce these reflection problems.

Also, linear birefringence from nonuniform PVA coatings and other defects may contribute to individual sample differences. We are currently studying techniques to control the variables that cause detectable CIDS differences from sample to sample.

It should be mentioned that the kinetics of the samples studied may be very important to the signal obtained by CIDS. In many cases we observed that the signal changed with time for several hours after the sample mounting. After about 10 to 20 hours these signals became stable. Also, in the study of the blue phases we had to allow ample time between each temperature change for the systems to give a stable signal.

The geometry of the sample cell also presented difficulties to the data collection. At scattering angles greater than about 26° or less than about 154° (26° from the backward direction) the scattered light does not pass cleanly through the plates but is mostly reflected at the air-quartz interface. This diminishing of the overall scattered intensity does not affect the CIDS ratio, but at high angles the observable light intensity drops too low for our detector. We have therefore not been able to collect data between 27° and 153°. Also, even at scattering angles less than 26° or greater than 154° the air-quartz interface tends to selectively reflect the scattered light depending on its polarization, and this can introduce a systematic artifact into the CIDS signals. We estimate that this error is at most one percent in the range of scattering angles reported here. Finally, the scattered light is refracted at the air-quartz interface so that the apparent scattering angle is considerably larger than the actual scattering angle. This effect has been corrected for by the use of Snell's law.

None of the difficulties listed in the last two sections is fundamental, and methods are currently being developed which should greatly increase the accuracy and sensitivity of our measurements.

4. RESULTS

4.1. Cholesteric Mesophases

The experimental data for the cholesteric phase are shown in figures 2a-2d and 3. All data were collected with an incident wavelength of 488nm, at room temperature. As can be seen, data are lacking in the region from about 27° to 153°. Also, at angles near 0° or 180° the CIDS signals tend to decrease significantly in all the samples. This is probably due to stray light reflected from the sample holder as mentioned above.

To help understand our results we have developed a simple theory for the CIDS of cholesteric liquid crystals. This theory treats the liquid crystal as a twisted field of anistropic polarizable point scatterers described by the polarizability density

$$\underline{\underline{\alpha}}(\underline{r})=\alpha_1\hat{t}(z)\hat{t}(z)+\alpha_2[\underline{\underline{1}}-\hat{t}(z)\hat{t}(z)] \tag{1}$$

where $\hat{t}(z)=\cos\left(\frac{2\pi z}{P}\right)\hat{x}+\sin\left(\frac{2\pi z}{P}\right)\hat{y}$,

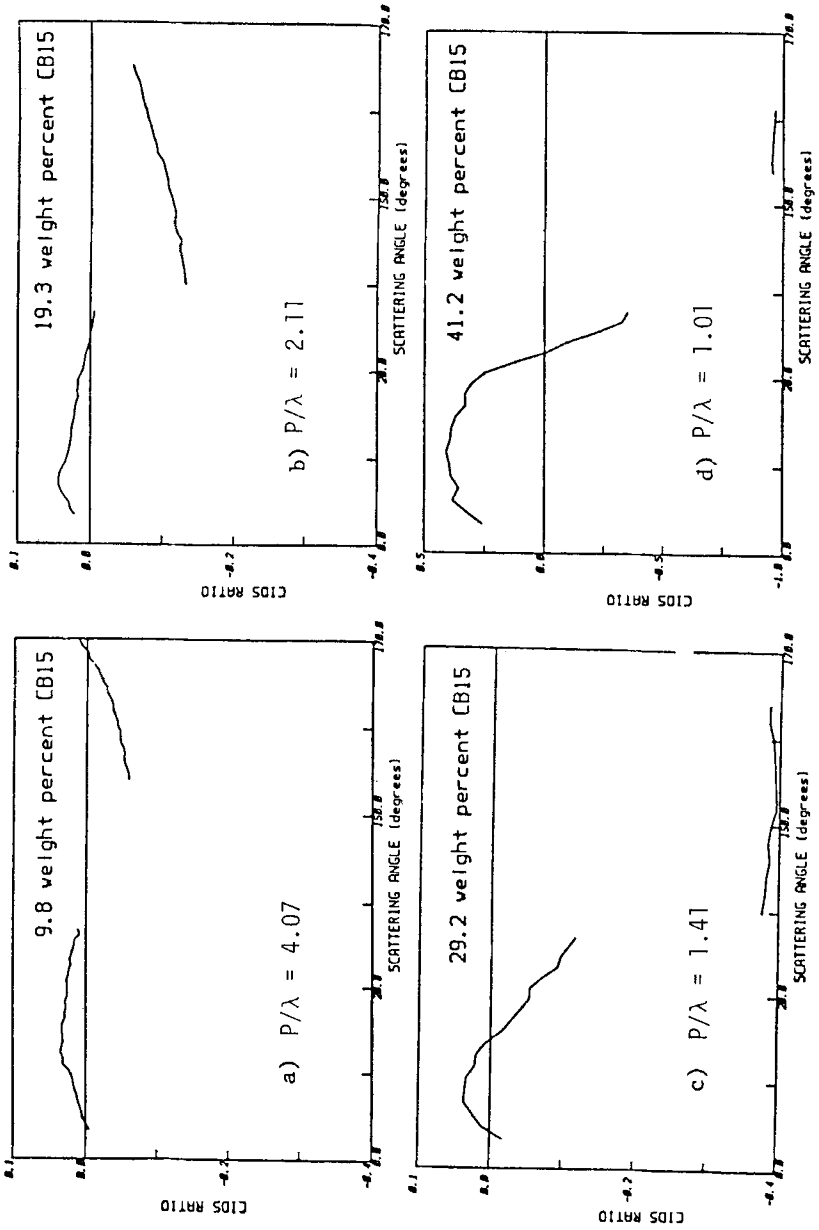


FIGURE 2 CIDS vs scattering angle of cholesteric samples

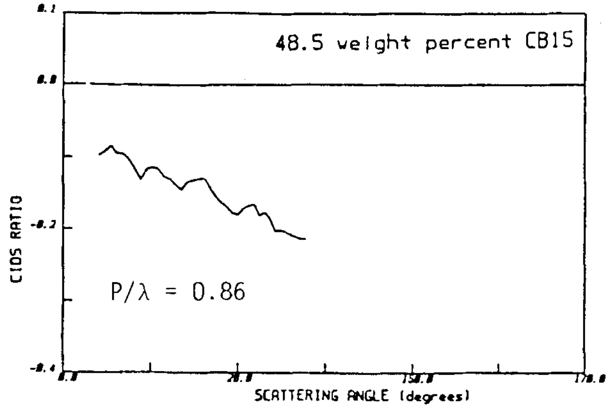


FIGURE 3 CIDS vs scattering angle for the 48.5% cholesteric sample

where (ˆ) indicates a unit vector, P is the pitch, and α_1 and α_2 are polarizability density magnitudes. An important general property of CIDS is that for periodic systems the CIDS pattern of a single unit cell is equal to the CIDS of the entire periodic array.[8,20] This often simplifies model computations considerably. In the model considered here the unit cell is a "twisted ladder", i.e., a single line of rod-like liquid crystal molecules with their long axes perpendicular to the line joining their centers (the "rungs" of the ladder) and rotated in a helical fashion about the line joining their centers (hence "twisted ladder"). The unit cell is only one turn of helix in length. The derivation of the scattered intensities and the CIDS for this unit cell is given by Keller,[20] for the special case where $\alpha_2=0$. Only slight modifications are required for the case where

$\alpha_2 \neq 0$. According to the modified theory the CIDS pattern depends on three parameters: r, γ, and a, where r is the ratio of the pitch to the wavelength (P/λ), $\gamma=(\alpha_1+\alpha_2)/(\alpha_1-\alpha_2)$ and controls the anisotropy of the scattering groups, and $a = (\pi/2)(\alpha_1-\alpha_2)$ and controls the degree to which groups are coupled to each other by multiple scattering and interactions between induced dipole moments. Our calculations were done using the second Born approximation, so the effects of multiple scattering and induced dipole interactions are not fully accounted for.

The CIDS is given by

$$\Delta(\theta)=\frac{I_L-I_R}{I_L+I_R}=\frac{\Delta_o(\theta)+\Delta(0)}{1+\Delta_o(\theta)\Delta(0)} \tag{2}$$

where

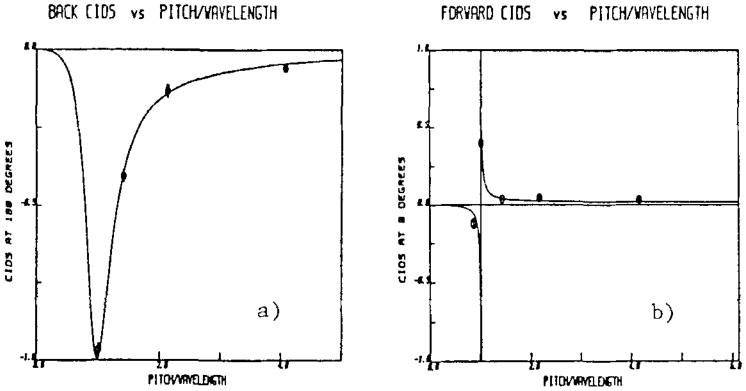
$$\Delta_o(\theta) = \frac{x^3}{\frac{1+|\gamma|^2}{2}\,x^4+\frac{1-2|\gamma|^2}{2}\,x^2+\frac{1}{2}\,|\gamma|^2}, \tag{3}$$

and

$$\Delta(0) = \frac{-2ar^2[1+(a-1)r^2]}{(r^2-1)^2-ar^2[ar^2(1+r^2)-2(r^2-1)]} \tag{4}$$

with $x=r/2(\cos\theta-1)$. The quantity $\Delta_o(\theta)$ vanishes at $\theta=0$, and so the CIDS in the forward direction is given by $\Delta(0)$. At $\theta=180^\circ$, $x=-r$, so both $\Delta_o(\theta=180^\circ)$ and $\Delta(0)$ are simple rational functions of r.

Figures 4a and 4b show plots of the CIDS in the



FIGURES 4a,b Backward and forward CIDS vs P/λ

backward and forward directions as a function of r, for both experiment and theory. The experimental values of CIDS in the "forward" and "backward" directions are actually the largest values of the CIDS in the forward and backward regions respectively. We have not used the CIDS values at precisely 5° and 170° because of the stray light problem at these angles mentioned earlier. Theoretical calculations using eq.(2) indicate that the CIDS should be quite constant at angles near 0° and 180°, so we expect that in the absence of stray light the CIDS patterns at 0° and 180° would be similar to the largest values in the forward and backward regions. For the theoretical curves in figures 4a and 4b the quantitites γ and a were treated as adjustable parameters to obtain the closest agreement with the experiments. The curves are very distinctive, and the theory and experiment are clearly in agreement despite the uncertainty in the data and the simplicity of the theory.

4.2. Blue Phases

Some cholesteric mixtures of CB15/ZLI 1612 (CB15 > 46 wt%) can undergo a thermotropic transition into a blue phase region before becoming isotropic. The temperature of the transition depends on the relative composition of the mixture. This system is ideally suited for our studies since the phase diagram has been well characterized,[35] and it exhibits blue phases within a large temperature range. Below 55% CB15 the transition temperature is higher than room temperature so that a careful control of the sample temperature can be achieved. We have performed a preliminary CIDS study of two compositions of the mixtures: 53.9% and 59.9% CB15. The latter showed the transition near room temperature, and the results were somewhat less reproducible - presumably due to the inherent difficulty to control the temperature of the sample below room temperature. In both cases after the characterization of these CIDS patterns, the samples were cooled off and the same temperature program applied to the measurement of the circular dichroism. The temperature of the samples was controlled to within 0.02 °C. The samples showed a marked hysteresis, reflected in the CIDS and CD measurements. To reduce these effects, at each temperature the samples were allowed to equilibrate for at least 30 minutes prior to the measurement.

Although the phase diagram presented by Stegemeyer and Pollman [35] showed two blue phases indicated by two dramatic viscosity changes, CIDS did not always indicate both changes. One explanation for this may be that the blue phase I and II coexist. During the transition to blue phase II the CIDS signal may become large due to an increasing number of blue phase II structure(s) which may dominate and mask the disappearance of the blue phase I signal pattern. In this case, the CIDS would appear to show only one blue phase when, in fact, two phases exist but overlap in their respective transitions. Also, this would make the temperature range of the transition appear broader; indeed this appears to be true in the CIDS.

During the CD experiments on the 59.9% CB15 sample we observed the appearance of a large peak around 515nm indicating the transition to blue phase I at about 22.3°C. As the temperature approached 22.6°C the blue phase I peak began to diminish and a large peak around 440nm appeared indicating a blue phase II transition. In the process of trying to reproduce the data we twice observed a transition to blue phase II, while the peak indicating blue phase I did not appear. We were able to regain the blue phase I peak only after allowing the sample to equilibrate for about four hours or more. The equilibration time appeared to depend on whether we approached the blue phase I by raising or lowering the temperature.

Figure (5) shows the CIDS versus temperature at a scattering angle of 13° and 21° for the 59.9% CB15 sample. At 13° two maxima appear precisely at the temperatures observed for the BPI, BPII transitions observed in the CD experiments. At 21° a single maxima appears at the temperature observed for the BPI

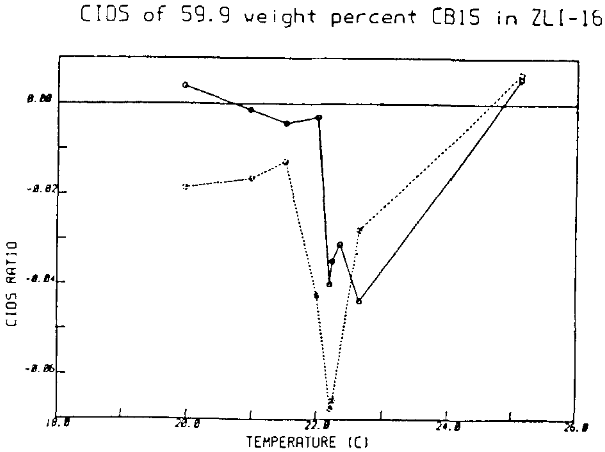


FIGURE 5 CIDS vs temperature near the blue phase transition point

transition. It is evident that the sensitivity of CIDS varies from angle to angle. The information necessary to detect blue phase transitions may be observed at some angles and not others. Therefore, BPI, BPII transitions may be detectable at their transition temperatures at certain scattering angles. In general, the CIDS grew very large during the temperature range necessary to produce the blue phase transitions, then decreased rapidly as the sample became isotropic. The ability to discriminate between the different phase transitions will require careful analysis of thermotropic activity at individual scattering angles.

5. DISCUSSION

Our purpose here has been to describe some preliminary attempts to use circular intensity differential scattering as a method for investigating the structure and properties of liquid crystals. Our data are still very rough, but considerable improvement in instrumentation and sample prepartion techniques should be possible in the near future. However, even at this stage two important conclusions can be drawn:

1) CIDS signals of liquid crystals in cholesteric and blue phases are measurable and are quite sensitive to structure. In particular, it has been possible to follow the transition between the cholesteric phase and the blue phases of mixtures of CB15 and ZLI 1612 as a function of temperature.

2) The CIDS signals can be successfully modeled with relatively simple theories. A major reason for the simplicity of our expressions for the CIDS of a cholesteric liquid crystal in the planer orientation is that in general CIDS patterns for periodic systems depend only on the properties of one unit cell of the periodic array. If models for the CIDS of the blue phases are successful, it may be possible to use CIDS to follow the changes in structure of the liquid crystal as it makes transitions between the cholesteric and blue phases.

The preliminary results carried out in our laboratory have established the sensitivity of CIDS to follow and characterize the phase transitions of cholesteric liquid crystals. We have found that the CIDS signal at the transition from the cholesteric to the blue phase increases ten-fold, and the angular distribution of the differential signal changes dramatically. We have observed that these phases are fairly stable with a temperature control of 0.02°C. We plan to carry out a detailed study of the temperature dependence of the CIDS of the blue phases as a function of the scattering angle. Our preliminary results indicate that different scattering angles show different sensitivity to the structural reorganization that occurs through the phase transition. It is expected therefore that this angular selectivity could give valuable information on the structure of these phases. Recently Flack, et al[36] carried out a detailed measurement of the Mueller matrices of the blue phases of a mixture of CB15 and E9 15 liquid crystals. This study has been done as a function of temperature at a fixed scattering angle. It is interesting to point out that the term M_{14} in Flack's study can be shown to correspond to the CIDS. However, the phase detection demodulation method used in our instrument gives much more sensitive mesurements than the rotating compensator technique used by these authors. Our instrumental setup will allow us to obtain the critical angular dependences of the differential scattering pattern. From this pattern and by modeling the CIDS of the different structures which have been so far proposed for the blue phases, we expect to be able to discriminate among these models.

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