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M. Kozielski<sup>a</sup>, D. Bauman<sup>a</sup>, M. Drozdowski<sup>a</sup> & Z. Salamon<sup>a</sup>

<sup>a</sup> Institute of Physics, Poznań Technical University, Piotrowo 3, 60-965, Poznań, Poland

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# The Study of the Guest Host Effect in Thin Aligned Liquid Crystal Film by Raman Spectroscopy

M. KOZIELSKI, D. BAUMAN, M. DROZDOWSKI and Z. SALAMON

*Institute of Physics, Poznań Technical University, Piotrowo 3, 60–965 Poznań, Poland*

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In the paper the nematic liquid crystal 5CB (4-pentyl-4'-cyanobiphenyl) with an embedded azo dye in the aligned thin films has been investigated by Raman scattering spectroscopy. The purpose of this paper is the determination of the dye influence on the orientational order of the host molecules in the mesophase as well as on the nematic-isotropic transition temperature of 5CB. The study was performed for the different dye concentrations and the different thicknesses of the samples. The second  $\langle P_2 \rangle$  and fourth  $\langle P_4 \rangle$  order parameters have been determined experimentally and the graphs of the molecular distribution functions of the liquid crystal molecules in the oriented films are presented.

**Keywords:** *nematic liquid crystal, azo dye, Raman scattering, absorption measurements*

## INTRODUCTION

In the recent few years liquid crystal displays utilizing “guest-host” effect<sup>1–5</sup> are widely applicable as compared to the conventional twisted-nematic displays they have two advantages: one can eliminate an outside polarizer and obtain the wide viewing angle. To get the best orientation of a guest in liquid crystalline host it is to choose the appropriate concentration of the dye and the film thickness. Then the guest-host display is characterized by the good perceived contrast that has been demonstrated in our previous paper.<sup>6</sup>

The most optical methods which are used to determine the mo-

molecular liquid crystal orientation e.g. birefringence measurements, infrared spectroscopy as well as the dichroism in visible and UV regions have a merit of providing the mean value of the second Legendre polynomial  $\langle P_2(\cos \theta) \rangle$  - the angle between the long molecular liquid crystal axis and the preferred direction/i.e. equivalent the order parameter usually reported as  $S$ .<sup>7</sup> On the other hand the fourth Legendre polynomial  $\langle P_4(\cos \theta) \rangle$  can be obtained by means of the magnetic resonance method,<sup>8</sup> the fluorescence polarization of dyes dissolved in liquid crystal<sup>9-11</sup> and the Raman scattering investigation.<sup>12-21</sup> The evaluation of the  $\langle P_4 \rangle$  parameter is very essential considering the fact that it gives more precise information on the orientation. It permits to determine the liquid crystal molecule distribution in the used samples.<sup>11,12</sup> The molecular distribution can be different for various aligned thin films of liquid crystals although the order parameter  $S$  remains the same.

The distribution function of  $f(\theta)$  may be reproduced with accuracy sufficient for most purposes by a truncated series expansion involving the Legendre polynomials  $P_l(\cos \theta)$ :<sup>9,12</sup>

$$f(\theta) = \sum_{l=0}^{\infty} \frac{2l+1}{2} \langle P_l(\cos \theta) \rangle P_l(\cos \theta). \quad (1)$$

The first few terms of the expansion include  $\langle \cos^2 \theta \rangle$  and  $\langle \cos^4 \theta \rangle$  are:

$$\langle P_0 \rangle = 1, \quad (2a)$$

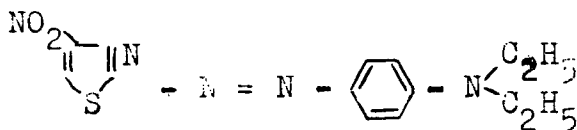
$$\langle P_2 \rangle = \frac{1}{2} (3\langle \cos^2 \theta \rangle - 1), \quad (2b)$$

$$\langle P_4 \rangle = \frac{1}{8} (35\langle \cos^4 \theta \rangle - 30\langle \cos^2 \theta \rangle + 3). \quad (2c)$$

In this paper we would like to answer the questions: how does the dye affect the host orientational order and how does the molecular arrangement depend on the thickness of the liquid crystal film and also what is the influence of the dye on the phase transition temperature? To resolve this problem we have chosen the Raman scattering technique in our present work.

## EXPERIMENTAL

The commercial nematic liquid crystal 4-pentyl-4'-cyanobiphenyl (5CB, BDH) without further purification was used as a host while the blue azo dye†



as a guest. This dye is however unstable of UV light, but we have decided to take it for our investigation because of good orientation in 5CB as well as the convenient absorption region with respect to the light exciting Raman scattering. The azo dye used absorbs the light in region of 480–720 nm with maximum at  $\lambda_{\max} = 593$  nm in 5CB.

The dye has been dissolved in liquid crystal and sandwiched by two glass plates using the spacer of 10–120  $\mu\text{m}$  in thickness. The concentration was changed from  $2 \cdot 10^{-4}$  M/l to  $2 \cdot 10^{-2}$  M/l (e.g. from 0.005% wt to 0.5% wt). The planar orientation of liquid crystal and dye molecules was obtained by oblique evaporation of  $\text{SiO}_x$  at an angle of about  $60^\circ$  to the normal to the glass surfaces and by rubbing procedure additionally. This procedure gives a good homogenous orientation of liquid crystal molecules in thin layer that has been supported by observation by means of the polarizing microscope.

The absorption spectra of the dye in liquid crystal were with a SPECORD M40 (Carl Zeiss Jena) equipped with polarizers. Polarized Raman measurements were carried out by means of the conventional apparatus. Experiments were performed in the back scattering geometry ( $180^\circ$  scattering angle). An ILA-120 Carl Zeiss Jena laser operating at 488 nm was the exciting source. Occasionally, the 458 nm line was also used. In order to reduce the local heating of the sample at the laser beam, the light power was kept down as far as possible. The Raman scattering was excited by 20 mW because such a laser power has not perturbed the appropriate arrangement of liquid crystal molecules in aligned layer.

Figure 1 shows the liquid crystal sample in the laboratory coordinates system. The light beam incidents along the  $x$  axis, the  $yz$  plane

†Synthesized in Institute of Dyes, Łódź Technical University.

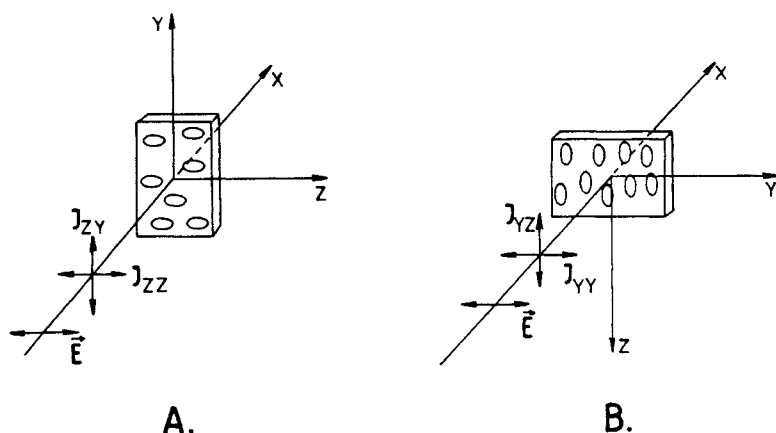


FIGURE 1 Experimental geometries for measuring the scattering anisotropies  $R_1$  (A) and  $R_2$  (B). Arrows indicate the vibration direction of the electric vectors of incident and scattered light.

is the plane of the sample and the order direction coincides with the  $z$  axis. The polarized Raman spectra were recorded in the sample setting where the electric vector  $\vec{E}$  of the incident light was taken as the  $z$  direction (Figure 1A) or the  $y$  direction after  $90^\circ$  rotation of the sample in  $yz$  plane (Figure 1B).  $J_{zz}$ ,  $J_{zy}$ ,  $J_{yz}$ ,  $J_{yy}$  are the appropriate scattering intensities.

All measurements were made in thermostat filled with glycol ( $\pm 0.2^\circ$ ). The temperature was changed from  $25^\circ\text{C}$  to  $40^\circ\text{C}$  in  $0.2^\circ$  stepwise. The scattered radiation was detected by GDM 1000 Carl Zeiss Jena double grating monochromator and a cooled by liquid nitrogen EMI 9658 photomultiplier tube. The Raman band positions were estimated with resolution of  $\pm 4\text{ cm}^{-1}$ .

## RESULTS AND DISCUSSION

Figure 2 presents the polarized components of the Raman scattering spectra ( $\lambda_{\text{exc}} = 488\text{ nm}$ ) for nematic liquid crystal 5CB in the  $60\text{ }\mu\text{m}$  sample at temperature of  $25^\circ\text{C}$  (sample setting as in Figure 1A) without correction for the anisotropy of the refractive index and of the apparatus polarization. As shown in Figure 2 the band intensity at  $1602\text{ cm}^{-1}$  is the most intensive and is assigned to the C—C stretching mode of benzene ring.<sup>22–24</sup> Moreover, the line at  $2222\text{ cm}^{-1}$  attributed to the C $\equiv$ N vibration mode of cyanin group<sup>12,13,22,24</sup> as well as the bands at  $1282\text{ cm}^{-1}$  and  $1175\text{ cm}^{-1}$  assigned to the C—C stretching vibration of biphenyl link and the aromatic C—H in plane defor-

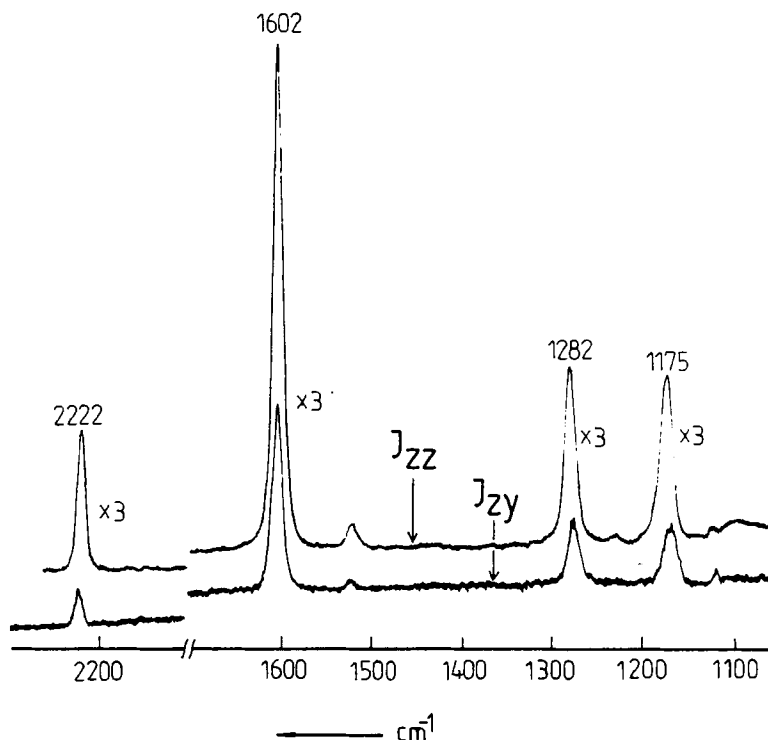


FIGURE 2 The polarized Raman spectra ( $J_{zz}$ ,  $J_{zy}$ ) of 5CB in the 60  $\mu\text{m}$  layer.

mation, respectively, are observed.<sup>24</sup> The band intensities of  $J_{zz}$  component are about 5.5 times larger than those of  $J_{zy}$  (regarding the corrections). Therefore one can assume that the vibration directions of each group are nearly parallel to the  $z$  axis. Since the Raman band intensities vary with the molecular orientation they can reflect the molecular order of liquid crystal in thin aligned film.

The scattering anisotropy was calculated on the basis of the Raman line intensities measurements and expressed as following:

$$R_1 = \frac{J_{zz} - J_{zy}}{J_{zz} + 2J_{zy}}, \quad (3)$$

for the excitation with light polarized parallel to the  $z$  axis (Figure 1A), and

$$R_2 = \frac{J_{yy} - J_{yz}}{J_{yy} + 2J_{yz}} \quad (4)$$

for the excitation with light polarized parallel to the  $y$  axis (Figure 1B).

Since the reflection loss at the cell-sample interface is anisotropic, due correction factor is incorporated to obtain the above scattering anisotropies from the recorded intensities  $J^{\text{exp}}$  as follows:<sup>12,14,17</sup>

$$R_1 = \frac{J_{zz}^{\text{exp}} C_n - J_{zy}^{\text{exp}}}{J_{zz}^{\text{exp}} C_n + 2J_{zy}^{\text{exp}}}, \quad (5)$$

$$R_2 = \frac{J_{yy}^{\text{exp}} - C_n J_{yz}^{\text{exp}}}{J_{yy}^{\text{exp}} + 2C_n J_{yz}^{\text{exp}}}, \quad (6)$$

where:

$$C_n = [(n_g + n_e)/(n_g + n_o)]^2; \quad (7)$$

here  $n_g$  is the refractive index of the glass plate, and  $n_o$  and  $n_e$  represent the ordinary and extraordinary refractive indices of liquid crystal at given temperature and wavelength of exciting light respectively.

The Raman scattering tensor related to the molecular frame for any particular vibration have the diagonal form:<sup>12,18-21</sup>

$$T = \begin{vmatrix} a & 0 & 0 \\ 0 & b & 0 \\ 0 & 0 & 1 \end{vmatrix}. \quad (8)$$

Assuming the liquid crystal molecules to be cylindrical rods, we have  $a = b$ . This value can be easily determined on the basis of the scattering anisotropy measurements in the isotropic phase.<sup>12</sup> The value obtained in our paper for the  $1602 \text{ cm}^{-1}$  line is  $a = -0.045$ , thus it is small comparing with 1. Therefore we assumed  $a = 0$  in our consideration. We assume also that the angle between the long molecular axis and the principal axis of the normal mode of vibration  $\beta_0 = 0^\circ$ , which seems reasonable because the vibration  $C - C$  ( $1602 \text{ cm}^{-1}$ ) has its long axis along the axis of the rigid part of liquid crystal molecules.

Using such assumptions the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters can be related with the  $R_1$  and  $R_2$  scattering anisotropies through experimentally obtained  $J_{zz}$ ,  $J_{zy} = J_{yz}$  and  $J_{yy}$  as follows:

$$\langle P_2 \rangle = \frac{2 + 7R_1 - 14R_2 + 5R_1R_2}{23 - 14R_1 + R_2 - 10R_1R_2}, \quad (9)$$

$$\langle P_4 \rangle = \frac{-12 + 21R_1 + 21R_2 - 30R_1R_2}{23 - 14R_1 + R_2 - 10R_1R_2}, \quad (10)$$

However, the approximation applied here gives only the roughly estimated  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$ , but we are interested in the changing of the liquid crystal 5CB orientation *versus* the thickness and dye concentration rather than in the absolute  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  values, previously obtained by other authors.<sup>16,19,21</sup>

The  $\langle P_2 \rangle$  parameter can also be evaluated based on the dichroic ratio of the dye in liquid crystal<sup>9</sup> for the rod-like molecules of the pleochroic dye reflect the host orientational order in thin aligned film as has been shown by Rumyantsev *et al.*<sup>25</sup>

Figure 3 presents the dependence of  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters on the sample thickness for neat 5CB and 5CB with the dye ( $c = 6.7 \cdot 10^{-4}$  M/l). These parameters were calculated from Eqs. (9) and (10) using the scattering anisotropy values  $R_1$  and  $R_2$  measured for the line  $1602 \text{ cm}^{-1}$  at  $25^\circ\text{C}$  ( $\lambda_{\text{exc}} = 488 \text{ nm}$ ). The  $R_1$  and  $R_2$  values are the same for other 5CB lines within experimental error. However, the line  $1602 \text{ cm}^{-1}$  is most intensive and therefore we have taken it into account for our further consideration. In Figure 3 is also shown  $\langle P_2 \rangle$  parameter values calculated from the dichroic absorption ratio of samples with dye in 5CB of concentration  $c = 6.7 \cdot 10^{-4}$  M/l (dashed curve).

Figure 4 presents  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters estimated on the base of the Raman study as well as  $\langle P_2 \rangle$  obtained from the absorption dichroic ratio as a function of the dye concentration for the  $20 \text{ }\mu\text{m}$  samples. In this case the  $458 \text{ nm}$  line was used for the excitation of Raman scattering to avoid the overlapping of the dye absorption band and the exciting line. The results obtained using the  $488 \text{ nm}$  line were not too reliable, especially for concentration higher than  $10^{-3}$  M/l, because of the laser heating of the samples.

In Figures 3 and 4 the parameter  $\langle P_4 \rangle$  obtained from the experimental values of  $\langle P_2 \rangle$  using Maier-Saupe mean field theory<sup>26</sup> as a function of the layer thickness and the dye concentration, respectively, is drawn.

From results presented in Figures 3 and 4 one can see the following indications: 1) the Raman results are in accordance with absorption results in experimental uncertainties both for thickness and dye concentration dependence of  $\langle P_2 \rangle$ , 2) the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  values are decreased with the increasing of thickness both for neat liquid crystal and for liquid crystal with dye, 3) presence of dye in liquid crystal matrix caused measurable changes of the initial molecular orientation,

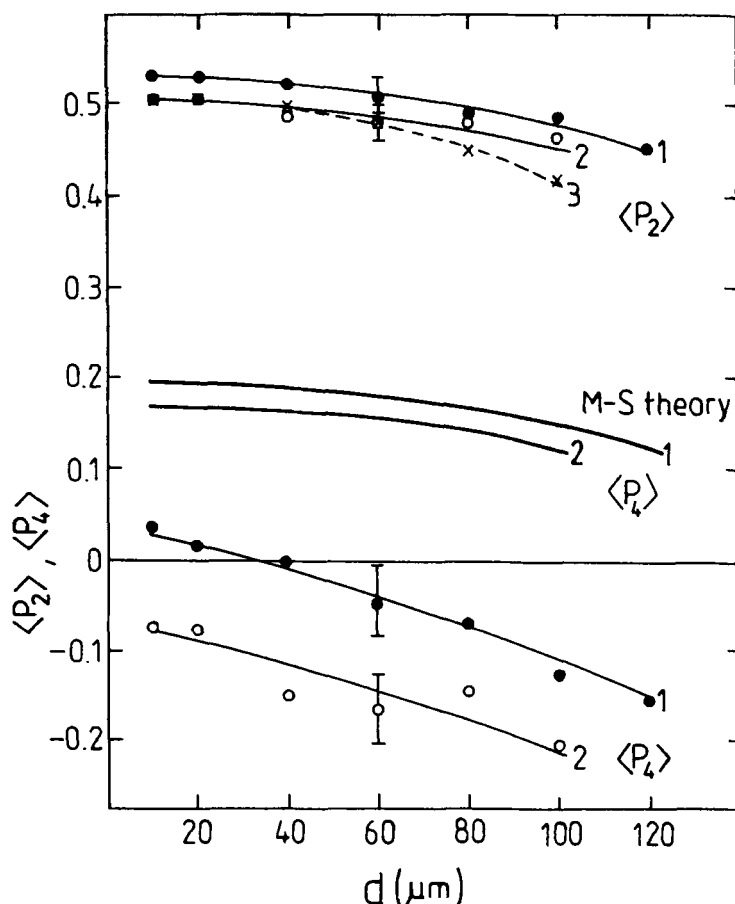


FIGURE 3 The values of order parameters  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  obtained from Raman scattering (—●—●—, —○—○—) and visible dichroic ratio (—x—x—) as a function of the sample thickness for: 1-5CB, 2,3-5CB with dye ( $c = 6.7 \cdot 10^{-4} \text{M/l}$ ). Solid lines:  $\langle P_4 \rangle$  from Maier-Saupe theory.

however stronger perturbation is observed for  $\langle P_4 \rangle$  parameter (Figures 3 and 4). The increase in dye concentration decreases the molecular orientation in thin layers (Figures 4 and 5). The experimental values of  $\langle P_4 \rangle$  are much more lower than that obtained from the mean field theory and they are predominantly negative.

Other authors studying the liquid crystal orientation by Raman spectroscopy<sup>12,13,16</sup> have suggested that the decrease of the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters with the increasing thickness is connected with the multiple scattering by the direction fluctuations which generate spu-

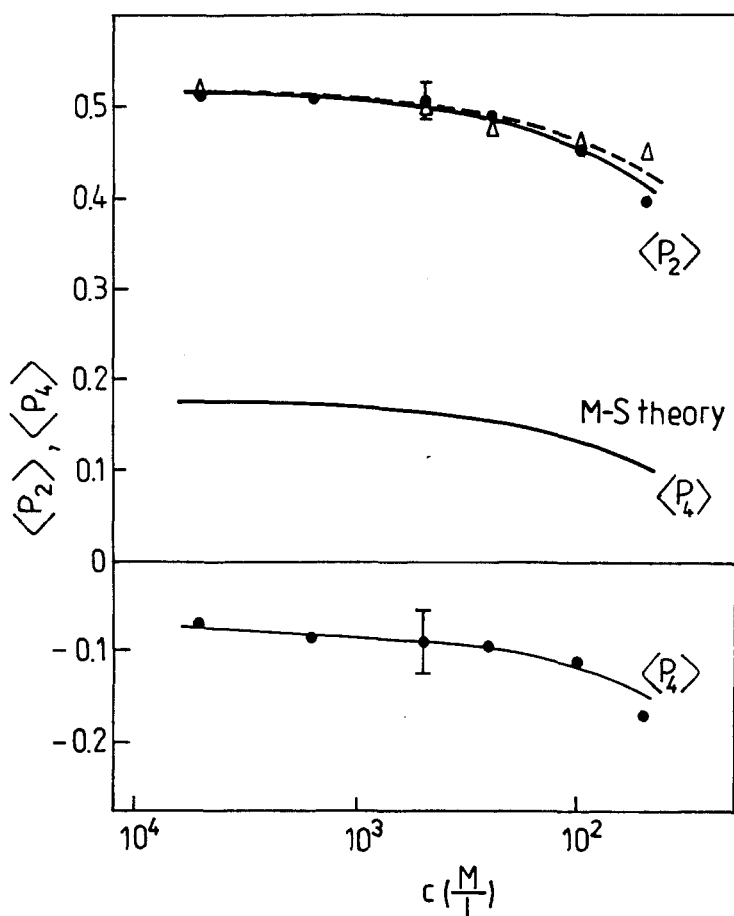


FIGURE 4 The  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  versus the dye concentration (for  $20 \mu\text{m}$  sample thickness) obtained from Raman ( $\bullet$ ) and absorption ( $\Delta$ ) results. Solid line:  $\langle P_4 \rangle$  from Maier-Saupe theory.

rious depolarization of the incident and scattered light. However, we have observed the identical of the Raman and absorption results, it is necessary to suppose that another effect plays also some role: the increase of the sample thickness causes the decreasing of the orientational order of the molecules in the investigated samples. Many authors have noted<sup>27-30</sup> that the molecular order of liquid crystal molecules as a result of the surface interaction is larger near by the glass plate surface than in the volume of the sample, where the flexible mesogenic molecules are more mobile. The order parameters obtained from the absorption dichroic ratio and by Raman scattering

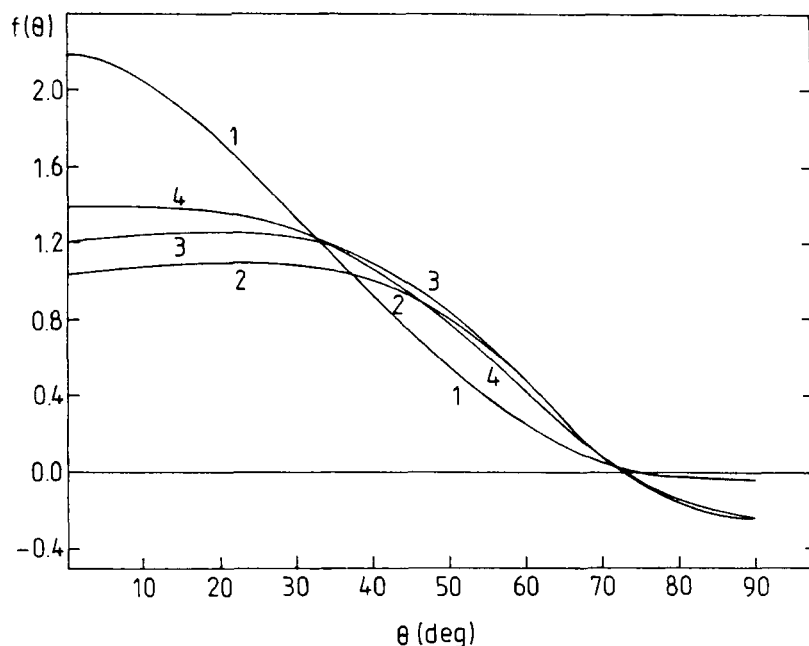


FIGURE 5 The molecular distribution function  $f(\theta)$  for: 1,2-5CB in 20  $\mu\text{m}$  sample at  $T = 25^\circ\text{C}$  and  $T = 33^\circ\text{C}$ , respectively; 3-5CB in 100  $\mu\text{m}$  sample,  $T = 25^\circ\text{C}$ ; 4-5CB with dye in 20  $\mu\text{m}$  sample,  $T = 25^\circ\text{C}$ .

anisotropies are the averaging values over all molecules. For the contribution of the surface orientational order is decreased with increasing the sample thickness, then the order parameter taken as a whole is decreased.

The non-typical behaviour of the  $\langle P_4 \rangle$  parameter has also been found by other authors studying the orientation of 5CB by using Raman method.<sup>16,19,21</sup> Three possible reasons of deviation of  $\langle P_4 \rangle$  parameter of the neat liquid crystal from the Maier-Saupe theory were suggested: 1) the local field anisotropy,<sup>18-20</sup> 2) molecular flexibility, especially due to the alkyl chain,<sup>17,19,20</sup> 3) molecular association ("dimerization"), due to strongly dipolar CN group of 5CB.<sup>19-21</sup> Recently Dalmolen *et al.*<sup>21</sup> studying the order parameters in homologous series of alkylcyanobiphenyls gave some evidence that the anomalously low values observed for  $\langle P_4 \rangle$  in some nematics might be related to dimerization. Then one can expect two effects: 1) the long molecular axis of the dimer is not parallel to the direction of the Raman vibration anymore, 2) steric interactions lead to non-parallelism of the long axis of a dimer and of a neighbouring monomer.

We are in agreement on the molecular association in the case of 5CB. It seems to be the main reason for the low value of  $\langle P_4 \rangle$ . However, the question why such a low concentration (for example  $c = 10^{-3} \text{ M/l}$  means one molecule of the dye per 5000 molecules of liquid crystal) changes so much the  $\langle P_4 \rangle$  value cannot be resolved assuming for example a dissociation process caused by the guest molecules. The other two suggestions concerning the anomalous behaviour of  $\langle P_4 \rangle$  cannot also explain the observed effects. This question needs to be further investigated.

Since the  $\langle P_4 \rangle$  parameter diminishes with the layer thickness as well as the dye concentration one can expect the broadening of the distribution function  $f(\theta)$  (Figure 5). The curve 1 and 3 present  $f(\theta)$  for neat 5CB in the 20  $\mu\text{m}$  and 80  $\mu\text{m}$  samples, respectively, while curve 4 for 5CB with dye ( $c = 6.7 \cdot 10^{-4} \text{ M/l}$ , 20  $\mu\text{m}$ ), at 25°C. On the other hand the curve 2 shows the distribution of the liquid crystal molecules after heating to 33°C for the sample of the 20  $\mu\text{m}$  in thickness. As one can see the increase in temperature caused the changes in the molecular arrangement of liquid crystal molecules in the thin layer. We have studied in detail the temperature dependence of the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters as a function of the sample thickness and the dye concentration.

Figure 6 shows the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters and the scattering anisotropies  $R_1$  and  $R_2$  versus temperature for the neat liquid crystal 5CB in the sample of the 100  $\mu\text{m}$  thickness in the region of the nematic-isotropic phase transition. In the figure two temperatures  $T_1$  and  $T_2$  are distinguished. At  $T_1$ , the  $\langle P_4 \rangle$  order parameter is observed to have the lowest value while the  $\langle P_2 \rangle$  parameter is found to be  $\approx 0.34$ . The similar changes of  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  have been observed by other authors.<sup>12-21</sup> They have studied the temperature dependence of the order parameters to  $T_1$  temperature only. We have applied the higher temperature  $T_2$  and observed that the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  parameters reach zero values and the scattering anisotropy values are equal to be  $R_1 = R_2 \approx 0.4$ . It is confirmed that  $T_2$  is the temperature above which only isotropic liquid exists. In the range between minimal value of  $\langle P_4 \rangle$  and  $\langle P_4 \rangle = 0$  the alignment of the sample which was observed by transmission pattern of a laser beam light was neither typical for homogenous nor for homeotropic arrangement and also nor for isotropic case. Therefore, we suggest that this region of the  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  changes are correlated with a two-phase system. In the range of temperatures  $T_1 \rightarrow T_2$  both nematic and isotropic phases coexist.<sup>31</sup> As our results indicate this region of phase separation can be

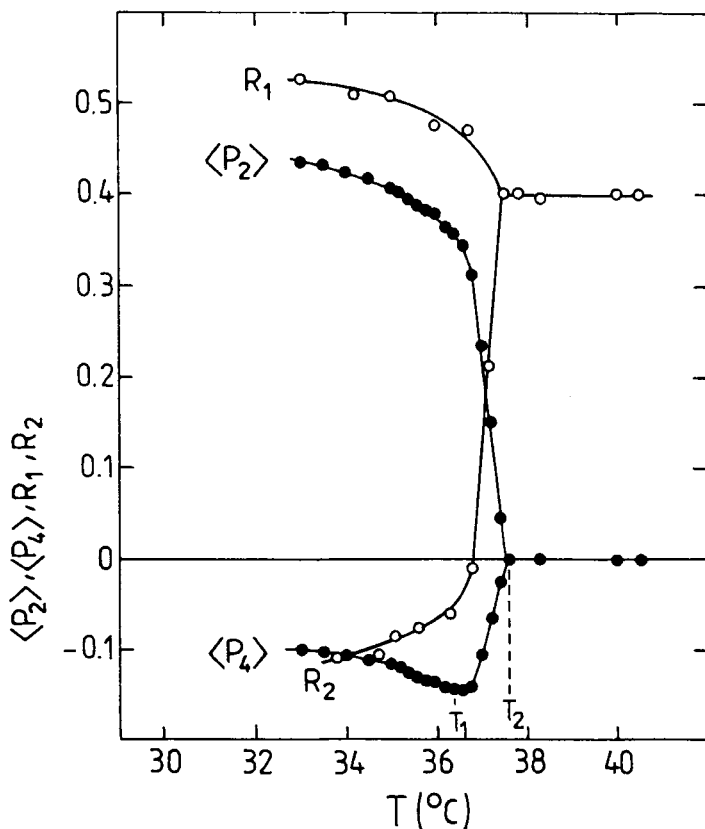


FIGURE 6 The temperature dependence of scattering anisotropies  $R_1$ ,  $R_2$  and order parameters  $\langle P_2 \rangle$  and  $\langle P_4 \rangle$  for neat 5CB in the sample of 100  $\mu\text{m}$  in thickness.

affected both by thickness of the sample and by the concentration of the guest dye (see Table I).

As it is seen from Table I for neat 5CB the temperature of the beginning of the phase separation region ( $T_1$ ) is independent of the layer thickness, whereas the temperature region from  $T_1$  to  $T_2$  is slightly extended. For the samples of the low dye concentration (of order of  $10^{-4}$  M/l)  $T_1$  is considered to be independent of the thickness (within experimental uncertainties). However, the enlargement of the phase separation region with the increasing cell thickness is observed.

The addition of dye causes the decrease in  $T_1$ , i.e. the temperature  $T_1$  is remarkably diminished with the increasing dye concentration. The rise in the dye concentration also causes the very large temper-

TABLE I  
The values of  $T_1$  and  $T_2$  for investigated samples

$d$ [ $\mu\text{m}$ ]	$c$ [M/l]	$T_1$ [ $^{\circ}\text{C}$ ]	$T_2$ [ $^{\circ}\text{C}$ ]
20	0	36.4	37.2
100	0	36.4	37.6
20	$6.7 \cdot 10^{-4}$	34.2	35.8
80	$6.7 \cdot 10^{-4}$	34.0	36.8
20	0	36.4	37.2
20	$6.7 \cdot 10^{-4}$	34.2	35.8
20	$4.0 \cdot 10^{-3}$	30.8	34.0

ature region expansion from  $T_1$  to  $T_2$  (from  $\Delta T = T_2 - T_1 = 0.8^{\circ}\text{C}$  for the neat liquid crystal to  $\Delta T = 3.2^{\circ}\text{C}$  for the liquid crystal with the dye of concentration  $c = 4 \cdot 10^{-3}$  M/l). Also other authors<sup>32</sup> observed the changes of the transition temperature due to the increasing dye concentration.

Obtained results permit us to draw a conclusion that for guest-host liquid crystal display construction, the very thin sample and the dye concentration as low as possible should be used, so as not to destroy the molecular order of liquid crystal and not to decrease too much the nematic-isotropic phase transition temperature.

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