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Structural anisotropy study by resonance Raman scattering in nematics with different dyes

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The method for the structural anisotropy determination, which is based on a comparison of the degree of polarization of absorption and resonance Raman bands, is tested with three dyes having different molecular dimensions in one nematic liquid crystal. The experimentally observed correlation between the variation of the structural anisotropy factor and the order parameters deviates from the predictions of the Segre model. This method could also be applied to determine the local field anisotropy for liquid crystals, but we must take into account the observed dependence of the structural anisotropy on the dimensions of the probe molecules.

1. Introduction

Raman scattering studies of the orientational order in nematic liquid crystals were started fourteen years ago [1-3] with the aim to measure not only one, but two coefficients in the expansion of the orientational distribution function. Already the first studies showed, that in some substances the fourth order Legendre polynomial $\langle P_4(\cos \beta) \rangle$ of the angle β between the long molecular axis and the director, thermally averaged, becomes negative near the phase transition temperature. This feature, which means that the preferred orientation of molecules is not parallel to the director, is usually observed for substances with a large birefringence [1, 2]. Therefore it was supposed, that in addition to other reasons originating from the molecular nature of nematics this effect could be due to some artifacts [4]. There were sufficiently well-founded arguments, that this effect is the result of neglecting the local field anisotropy in the mesophase [3, 5]. For some time this problem was discussed in the literature [6, 7], but no perfect way was found to determine more correctly the $\langle P_4 \rangle$ coefficient from Raman experiments. Probably this was the reason for the decrease in the interest in Raman measurements.

Later, a number of resonance Raman scattering experiments [8-10] were performed in liquid crystals. Although all of them dealt with a rather long molecule, all-trans β -carotene, which has high $\langle P_2 \rangle$ values, very low and even negative values of the $\langle P_4 \rangle$ order parameter were found in the experiments [9, 10]. It should be noted, that the local field anisotropy in nematics was absolutely neglected during the treatment of resonance Raman depolarization ratios.

Recently [11], it was supposed that the low $\langle P_4 \rangle$ values, as well as the difference between the $\langle P_2 \rangle$ order parameters determined from resonance Raman experiments and those obtained from absorption dichroism measurements for the same dye

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molecules, are due to the neglect of the local field anisotropy effects [8–10]. It was proposed [12] to use this difference of the $\langle P_2 \rangle$ values resulting from absorption and Raman scattering experiments in the isotropic local field approximation to determine the local field anisotropy.

Besides the importance of the determination of the local field anisotropy for all optical experiments in liquid crystals (cf. [13]) this method gives us the new possibility for the determination of not only single-particle properties (i.e. the order parameters), but also a two particle property, the so-called structural anisotropy. This work is devoted to the testing of the method proposed in [12] with different dye molecules. First we give a brief outline of the background of this method. Some details of the experimental set-up for resonance Raman depolarization measurements, are described in the third section. Finally, the results and their analysis are presented together with some conclusions concerning the structural anisotropy dependence on the anisometry of the probing molecule.

2. Background of the method

The absorption coefficient k and the Raman intensity I for condensed media are related to molecular properties such as the transition dipole moment \mathbf{m} and the Raman polarizability tensor α by [14, 15]:

$$k_i \sim \frac{1}{n_i} \langle (m_i f_{ii})^2 \rangle, \quad (1)$$

$$I_{ij} \sim (n_g^s + n_i^s)^{-2} (n_g + n_j)^{-2} \langle (f_{ip}^s \alpha_{pq} f_{qj})^2 \rangle, \quad (2)$$

where n_i and n_g are the refractive indices of the nematic and of the glass substrate, respectively. All subscripts except g denote the cartesian components x, y, z (I_{ij} is the scattered light intensity having i polarization while the incident light is polarized along the j axis). The superscript s indicates that the value has to be taken at the wavelength of the scattered radiation. f_{ij} is the local field correction factor, connecting the local electromagnetic field of the light wave acting on the molecule with the average field in the condensed medium. It should be noted that all corrections connected with the propagation of light through different boundaries (normal to their surfaces) are taken into account in these expressions. The equality sign is replaced by that of proportionality because all of the factors, which are identical for different polarizations of light are omitted. To study the anisotropy of physical properties in the mesophase the dichroic ratio of absorption and the depolarization ratios for Raman scattering are measured (there are two linearly independent ratios for resonance Raman). For the totally symmetric intramolecular transitions in the uniaxial medium, consisting of axially symmetric molecules these ratios are given by (compare [8] and [12])

$$N = \frac{k_z}{k_x} = \frac{n_x f_{zz}^2}{n_z f_{xx}^2} \frac{1 + 2\langle P_2 \rangle}{1 - \langle P_2 \rangle}, \quad (3)$$

$$R_1 = \frac{I_{xz}}{I_{zz}} = \frac{(n_g + n_z)^2 f_{xx}^2}{(n_g + n_x)^2 f_{zz}^2} \frac{7 + 5\langle P_2 \rangle - 12\langle P_4 \rangle}{21 + 60\langle P_2 \rangle + 24\langle P_4 \rangle}, \quad (4)$$

$$R_2 = \frac{I_{zx}}{I_{xx}} = \frac{(n_g + n_x)^2 f_{zz}^2}{(n_g + n_z)^2 f_{xx}^2} \frac{7 + 5\langle P_2 \rangle - 12\langle P_4 \rangle}{21 - 30\langle P_2 \rangle + 9\langle P_4 \rangle}, \quad (5)$$

where $\langle P_i \rangle$ denotes the i th rank order parameters [16]. During the derivation of these equations it was supposed that the local field is independent of the molecular

orientation. In this case the local field factor has the symmetry of the mesophase and is given by [17]

$$f_{ii} = 1 + L_{ii}(n_i^2 - 1), \quad (6)$$

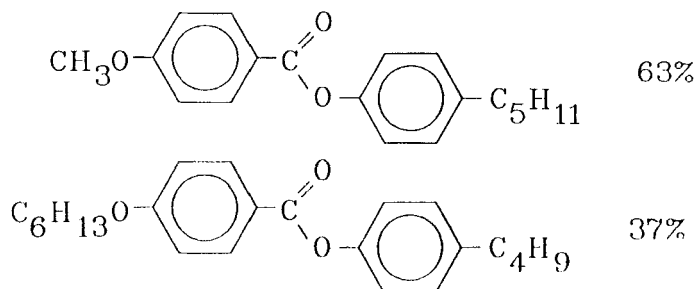
where L_{ii} are the Lorentz tensor components which obey the relation $L_{zz} + 2L_{xx} = 1$. Hence for an optically uniaxial mesophase the Lorentz tensor is determined by one component, its anisotropy, $\tau = 1/3(L_{xx} - L_{zz})$ which is related to the anisotropy of the pair distribution function [18] and because of this is called the structural anisotropy factor.

In order to be able to determine all of the molecular statistical parameters contained in relations (3)–(6) from the combined measurements of absorption dichroism and resonance Raman depolarization the following requirements (for the liquid-crystalline molecules or as it is more usual for the dye molecules dissolved in the nematic) should be satisfied:

- (i) the transitions for absorption and resonance Raman must occur between the same electronic levels and only resonance Raman depolarization ratios for totally symmetric modes should be taken into consideration. This limitation originates from the fact, that by optical methods we cannot study the orientational statistics of the molecule itself, but of some transition moment active in the optical spectra;
- (ii) only those molecules are appropriate which have such transitions active in resonance Raman scattering and which do not overlap with luminescence spectra;
- (iii) the life-time in the excited state must be much shorter than the reorientation time of the molecule so as to avoid rotational depolarization (this condition is always fulfilled for non-resonant scattering, but must be checked in resonance);
- (iv) the symmetry of the molecule should not be strictly cylindrical, because the application of the method is not based on vibrational selection rules, but the difference of the molecular properties in the directions perpendicular to the long axis should be negligible;
- (v) and the last, but not the least important, condition is that no photophysical reactions inside the probing molecules or photochemical reactions between them and the solvent molecules must occur even under laser light irradiation.

3. Experimental

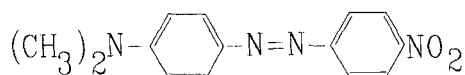
As the liquid-crystalline solvent a binary mixture of commercially available compounds have been used which have a nematic phase in the range 0–42.5°C. It should be noted that after adding the dyes the transition temperature was changed by



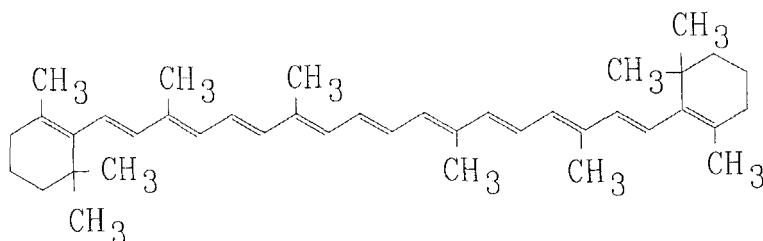
several tenths of a degree, depending on the molecular structure and the concentration of the dye. In addition the overheating of the sample in the laser beam during the resonance Raman experiments gives rise to a temperature shift of the same order of magnitude. As these effects are small they should not influence the critical behaviour of the physical parameters on a shifted temperature scale. That is why all experimental data in this paper are represented as a function of the temperature difference with respect to the actual transition temperature.

The dyes were chosen taking into account the requirements mentioned previously. To compare structural anisotropies molecules with different length-to-width ratios we have studied resonance Raman depolarization and absorption dichroism of three dyes, having approximately the same width but very different lengths

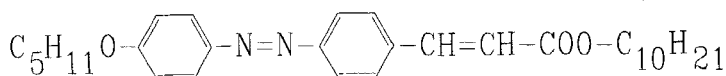
4-dimethylamino-4'-nitro-azobenzene (DMANAB),



all-*trans* β -carotene,



n-decyl ester 4-(4'-pentyloxyphenylazo)cinnamic acid (C10) [19].



Commercially available DMANAB was recrystallized while other compounds, supplied privately, were used without further purification. The concentration of dyes in the liquid-crystalline solvent was varied between 0.08–2 wt% depending on the extinction coefficient of the absorption bands. In addition the concentration and thickness dependence of the measured parameters had to be accessible in the concentration range studied.

Liquid-crystalline samples were placed between quartz glass plates. The spacing was measured by recording interference fringes in the visible range. Homogeneous alignment of the liquid-crystalline mixtures was achieved by oblique vacuum evaporation of SiO_2 on the glass plates and was verified under a polarizing microscope. For the temperature dependent measurements the samples were placed in an electrically heated oven and the temperature was maintained with an accuracy of $\pm 0.03^\circ\text{C}$.

Resonance Raman scattering was excited with 488 nm line of an argon-ion laser (for C10 with 457 nm) to ensure sufficient resonance enhancement. The power of the incident light on the sample was varied from 5 to 20 mW depending on the absorption

coefficient of the sample. The light intensity was chosen to keep the overheating of the sample as low as a few tenths of a degree. For the same reason a cylindrical lens was used as shown in figure 1. Raman spectra were recorded in the back scattering geometry with the scattered light collected in a solid angle of less than 0.1 sr in order to prevent additional depolarization (see figure 1). The Raman intensities were determined in two ways: by integration of the spectral line intensity with the background supposed to be straight line or by fitting the experimental spectrum with lorentzian lineshape with the help of the Levenberg–Marquardt procedure. The depolarization ratios obtained with both methods are the same within the experimental accuracy, which was of the order of 5 per cent (the systematic error was not larger than 2 per cent when checked by measuring the depolarization ratios of CCl_4). Correction on the reabsorption, when it was necessary, was performed as described in [11]. It was as large as 15 per cent for the R_1 ratio of DMANAB and less than the experimental error for C10.

Absorption spectra were recorded with a Specord M40 spectrophotometer. The edge of the absorption bands of the liquid-crystalline solvent was compensated by using the sample without the dye. Dichroic ratios were computed from peak intensity measurements with an accuracy of up to 2 per cent.

Refractive indices in the range of transparency of the liquid-crystalline mixtures were determined with an Abbe refractometer IRF 454B (the prisms were treated by lecithin to provide the possibility for the measurement of both indices). For the measurements at different wavelengths monochromatic light was obtained with a Spekol monochromator and corrections for the refractometer readings were calculated from the prism refractive indices data. The resulting error after all of these precautions was less than ± 0.001 . For DMANAB the wavelength of the scattered light was within the absorption band. In this case refractive indices were determined with an interferometric method as described in [11]. In all cases, because of the low dye concentration, the refractive indices of the mixtures, determined by the same interferometric method, differed from that of the pure liquid crystals by not more than 0.3 per cent. It was possible, therefore, to take for the refractive indices at the wavelength of the dye absorption band in equation (3) their values at the same wavelength in the liquid-crystalline mixture without the dye.

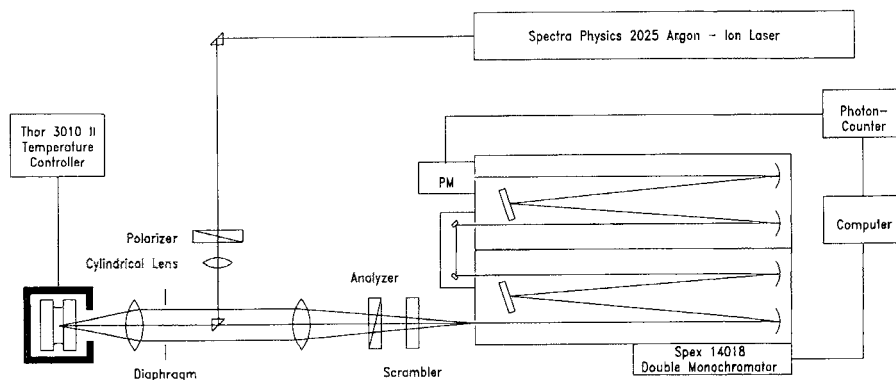


Figure 1. Experimental set-up.

4. Results and discussion

Resonance Raman and absorption spectra of β -carotene and C10 are shown in figures 2 and 3 (experimental data concerning DMANAB and some analysis of the results obtained can be found in [11, 12]). For both compounds the absorption dichroism is positive, this means that the transition moments must be parallel to the long molecular axes. Absorption band widths are larger than 100 nm and in this case additional depolarization of the Raman bands due to molecular rotation can be neglected. Because of the absence of vibronic structure in the absorption spectrum of C10 it was possible to use only near-resonant excitation. Nevertheless the resonance enhancement in both cases was larger than 10^3 . For β -carotene the position of the Raman bands is dependent on the excitation wavelength [20]. We found that depolarization ratios in the mesophase also show such a dependence on the excitation

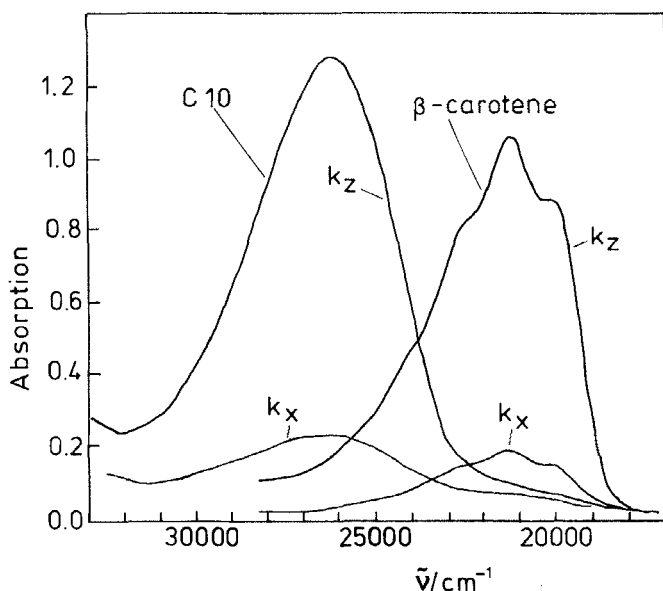


Figure 2. Polarized absorption spectra of 0.073 per cent β -carotene (sample thickness, $40\ \mu\text{m}$) and 2 per cent C10 (sample thickness, $30\ \mu\text{m}$ in M1 at 24°C).

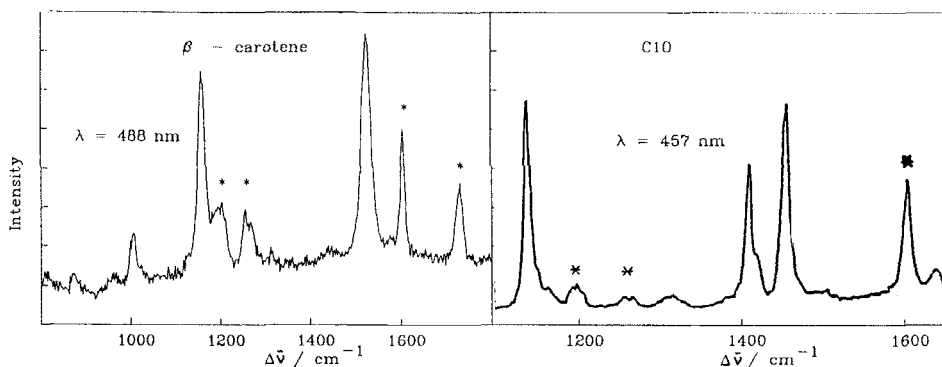


Figure 3. Resonance Raman spectra of β -carotene (0.073 per cent and $40\ \mu\text{m}$ thick) and C10 (2 per cent and $30\ \mu\text{m}$ thick) in M1. Lines marked with * correspond to M1.

wavelength. We tried, therefore, to excite Raman scattering as near to the absorption band maximum as possible (taking into account reabsorption effects). Despite the pronounced vibronic structure, the experimentally determined dichroic ratios for the maxima near $21\,000$ and $22\,000\text{ cm}^{-1}$ are the same allowing us to excite resonance Raman scattering away from the main maximum of the β -carotene absorption band. The stability of β -carotene to photoisomerization and the presence only of the all-*trans* configuration was checked during the experiment by comparison of its absorption and resonance Raman spectra with literature data [20,21]. Raman depolarization measurements in the isotropic phase have shown that for all bands studied the depolarization ratio, ρ is 0.333 ± 0.01 . All of these observations prove the assumptions made concerning the Raman polarizability tensor and ensure the applicability of equations (3)–(5).

Resonance Raman depolarization measurements were performed only for the most intense bands. These are the 1522 cm^{-1} band of all-*trans* β -carotene, belonging to the totally symmetric C=C stretching mode of the polyene chain [20], and the bands of C10 at 1140 , 1406 and 1453 cm^{-1} , the first two bands could be attributed to the C–O–C and N=N stretching vibrations, respectively. The ratios for the band near 1600 cm^{-1} were not considered because of possible presence of a contribution from the solvent molecules (as is evident from a comparison with spectra in figure 3). For all of the bands studied for C10 the depolarization ratios coincide within the experimental accuracy. In figure 4(b) only the values averaged over three bands are given. Precautions have been taken in order to avoid additional depolarization due to multiple scattering [1]. As we can see from figure 4(a) (and also figure 3 of [11]) no thickness dependence of the depolarization ratios is observed for the liquid-crystalline solvent under consideration. Rather small cross-sections for light scattering by director fluctuations can be due to the relatively small optical anisotropy and, probably, large elastic constants. In this

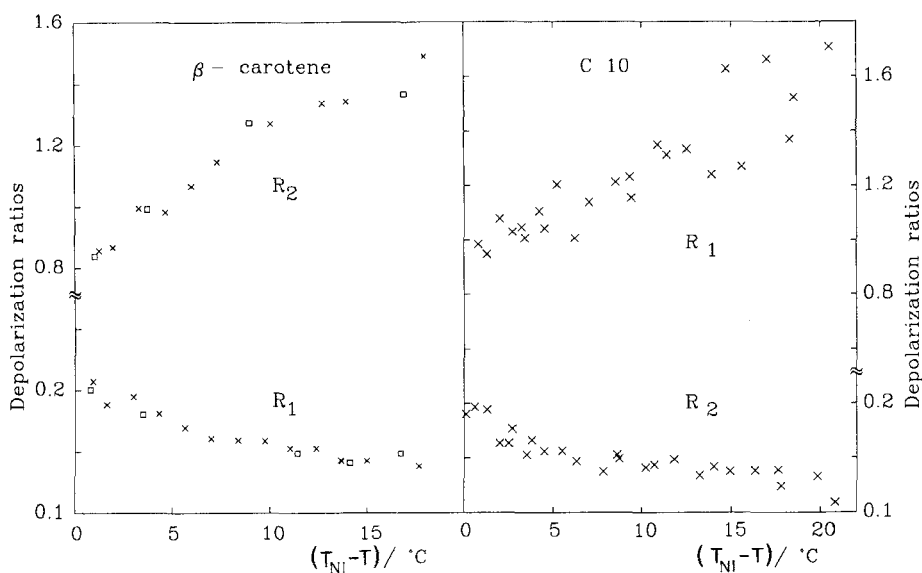


Figure 4. Depolarization ratios for the 1522 cm^{-1} band of 0.125 per cent β -carotene for the sample thickness $10\ \mu\text{m}$ (\square) and $20\ \mu\text{m}$ (\times) and depolarization ratios for the 1140 , 1406 and 1453 cm^{-1} bands of 2 per cent C10.

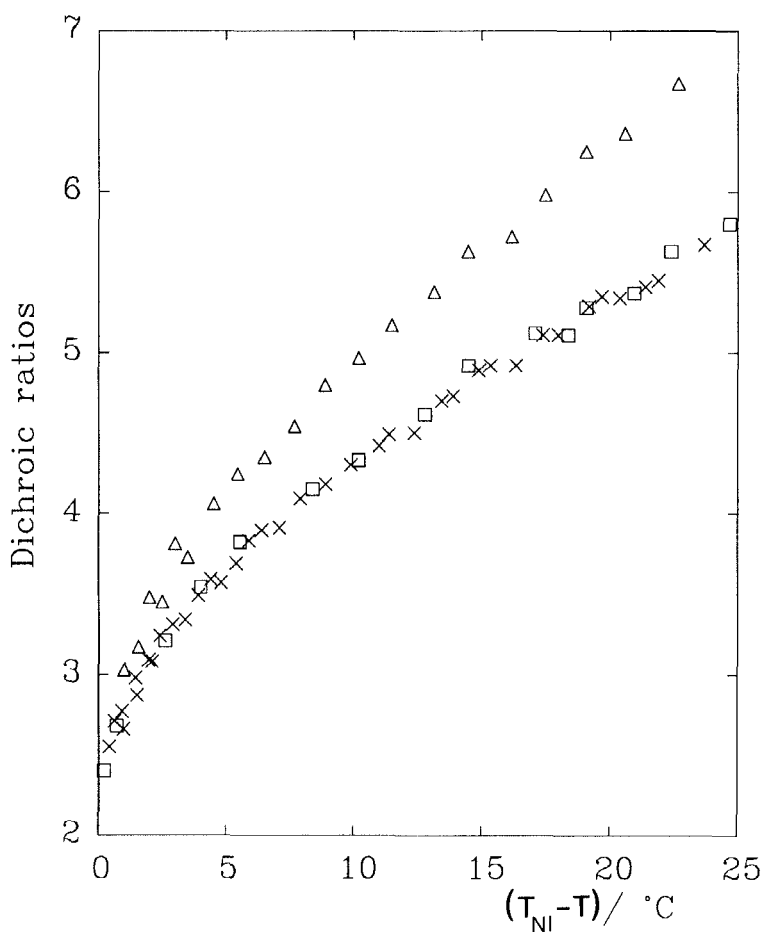


Figure 5. Dichroic ratios for 30 μm (\times) and 40 μm (\square) sample thicknesses of β -carotene in M1 with 0.126 and 0.073 per cent concentration, respectively; and for 2 per cent C10 (\triangle) in M1.

solvent an additional depolarization due to elastic scattering was not observed even for very precise absorption dichroism measurements, as shown for β -carotene in figure 5.

The order parameters can be obtained from equations (3)–(5) and the absorption and Raman data independently in the isotropic local field approximation, i.e. when $f_{zz}/f_{xx} = 1$ (the refractive indices necessary for this are depicted in figure 6). As we can see from figure 7 (see also [12] for DMANAB) for all of the dyes in the nematic solvent studied the $\langle P_2 \rangle$ order parameter calculated from absorption dichroism is somewhat larger than that determined from Raman data. This means that the local field anisotropy in this nematic is negative, i.e. $f_{zz} < f_{xx}$. The difference is obvious but not much larger than the scatter in the experimental points. For that reason the precision of the structural anisotropy determined from the combined resonance Raman and absorption measurements is not very high (see figure 8). In addition the experimentally

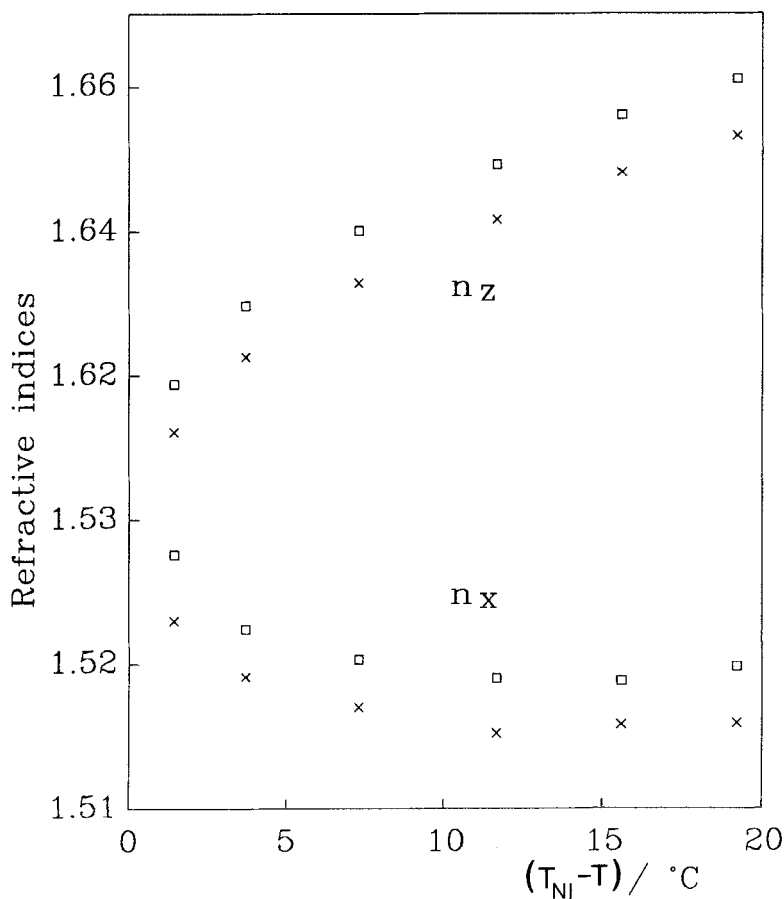


Figure 6. The refractive indices of M1 at the wavelengths of the absorption bands of (×) β -carotene (500 nm) and (□) C10 (400 nm).

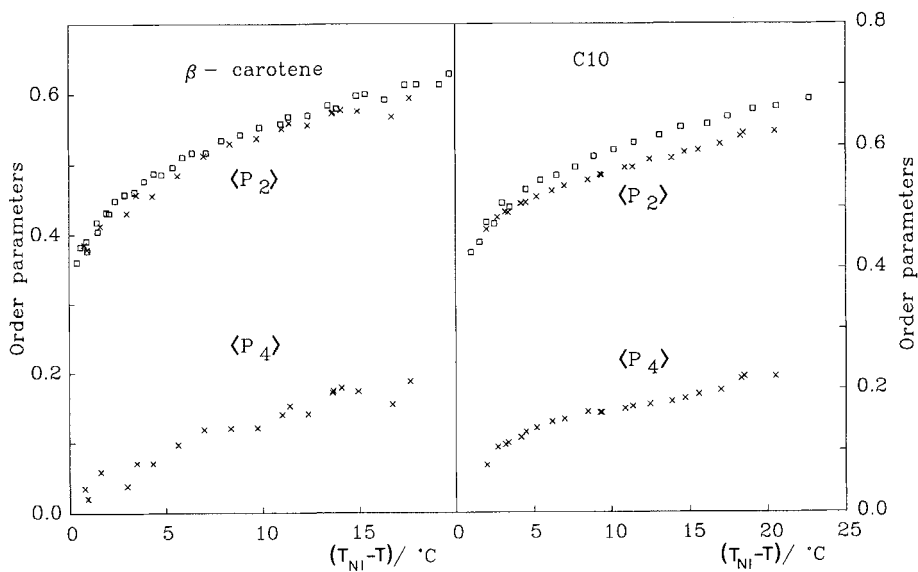


Figure 7. The order parameters, calculated from absorption dichroism (□) and Raman depolarization (×) measurements with the isotropic local field approximation.

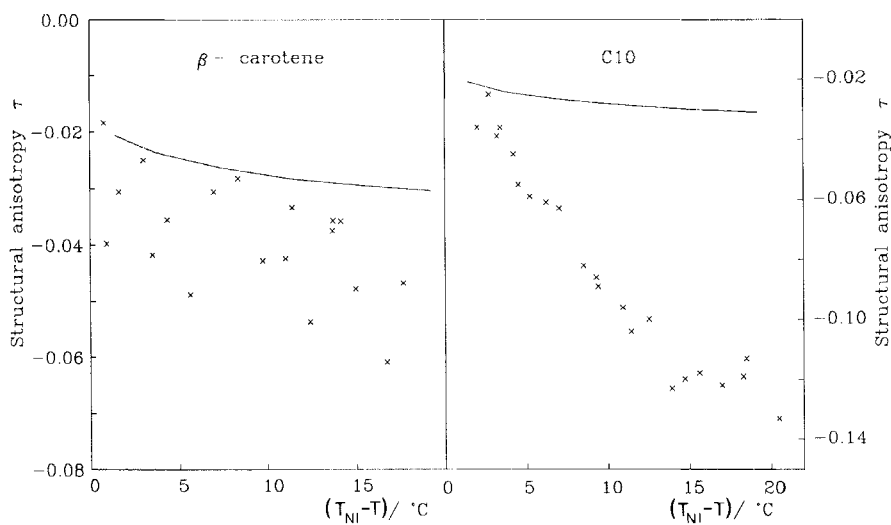


Figure 8. The temperature dependence of the structural anisotropy, obtained from combined absorption and Raman measurements. The curve corresponds to τ_i , calculated from equation (7).

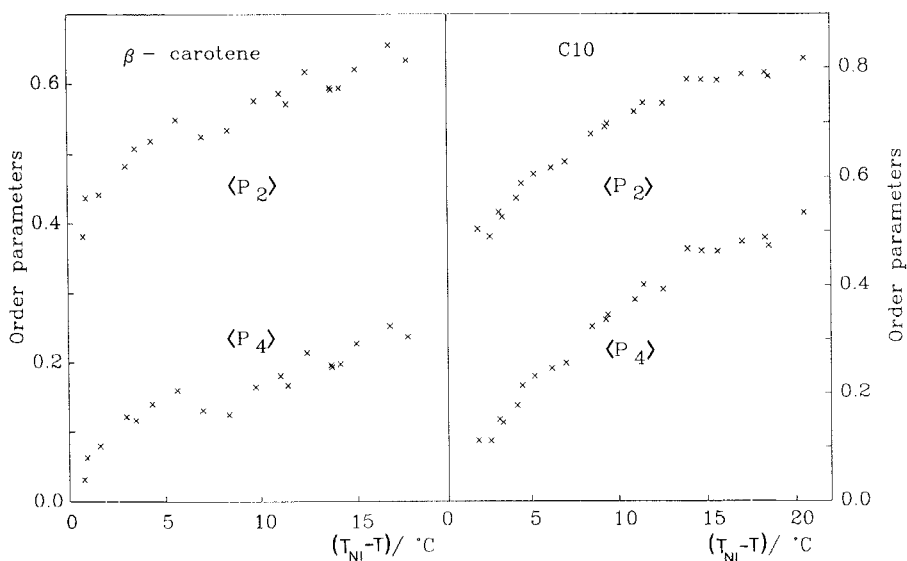


Figure 9. The order parameters, obtained from combined absorption and Raman measurements.

determined structural anisotropy is much larger than that, calculated from the local field isotropy condition

$$\tau_i = \frac{1}{3} \frac{n_x^2 - n_z^2}{2n_z^2 + n_x^2 - 3} \quad (7)$$

Taking into account the local field anisotropy effect when treating the optical data the order parameters are increased (compare the data in figures 7 and 9). This effect is particularly pronounced for the $\langle P_4 \rangle$ order parameter.

Different models have been developed to determine the structural anisotropy to take into account the local field anisotropy when treating the different optical data (these models are reviewed in [13]). One of the most well-founded models is that of Segre [22]. Following Onsager, he has solved the electrostatic problem for an anisotropic spheroid which is not perfectly oriented in a uniaxial dielectric continuum. After averaging over the orientational distribution function of the spheroid he obtained equation (6) for the local field correction factor, but with an apparent structural anisotropy different from the anisotropy τ_{\max} of the depolarizing tensor of the spheroid

$$\tau = \langle P_2 \rangle \tau_{\max}, \quad (8)$$

where

$$\tau_{\max} = \frac{a_1 a_2}{2} \int_0^\infty dv (v + a_2^2)^{-3/2} (v + a_1^2)^{-1} - 1/3, \quad (9)$$

and a_1, a_2 are the long and short semi-axes of the spheroid, respectively. The proportionality of the structural anisotropy values to the order parameters, predicted by equation (8), can be compared with the experimental data in figure 10. For DMANAB and β -carotene it is difficult to draw any reliable conclusions due to insufficient accuracy. But, as it is evident from figure 10, the structural anisotropy for C10 molecules is not proportional to the order parameter. The experimental data for the dyes studied are more likely to be approximated by some linear function. Different effects could be responsible for such a deviation from the proportionality. First, the molecular statistical definition of the structural anisotropy [18] in our notation can be written as

$$\tau = \frac{1}{4\pi} \int \frac{d\mathbf{r}}{|\mathbf{r}|} g_{12}(\mathbf{r}) P_2[\cos(\mathbf{r}, \mathbf{n})], \quad (10)$$

where $g_{12}(\mathbf{r})$ is the two particle, centre of mass distribution function. Because of the local ordering effects the relative positions of the neighbouring molecules closely follow more the anisotropy of the molecular repulsive core. When the temperature is lowered not only the long range orientational order, but also the local order increases and, as seen from equation (10), this will lead to an additional increase of the absolute value of structural anisotropy. Another probable reason for the observed effect is the flexibility of the long-chain molecules. Lowering the temperature the aliphatic chains tend to adopt an all-*trans* configuration and this will lead to an increase of the effective anisotropy of the molecular repulsive core and the absolute value of τ_{\max} . At present we have no possibility to separate these two mechanisms experimentally.

Because of the non-proportionality effect it is difficult to evaluate τ_{\max} in equation (8) from the experimental data. Such estimates could be made in the way shown in figure 10. The values obtained from extrapolation of the experimental data together with those, calculated from equation (9) from the molecular structure and the

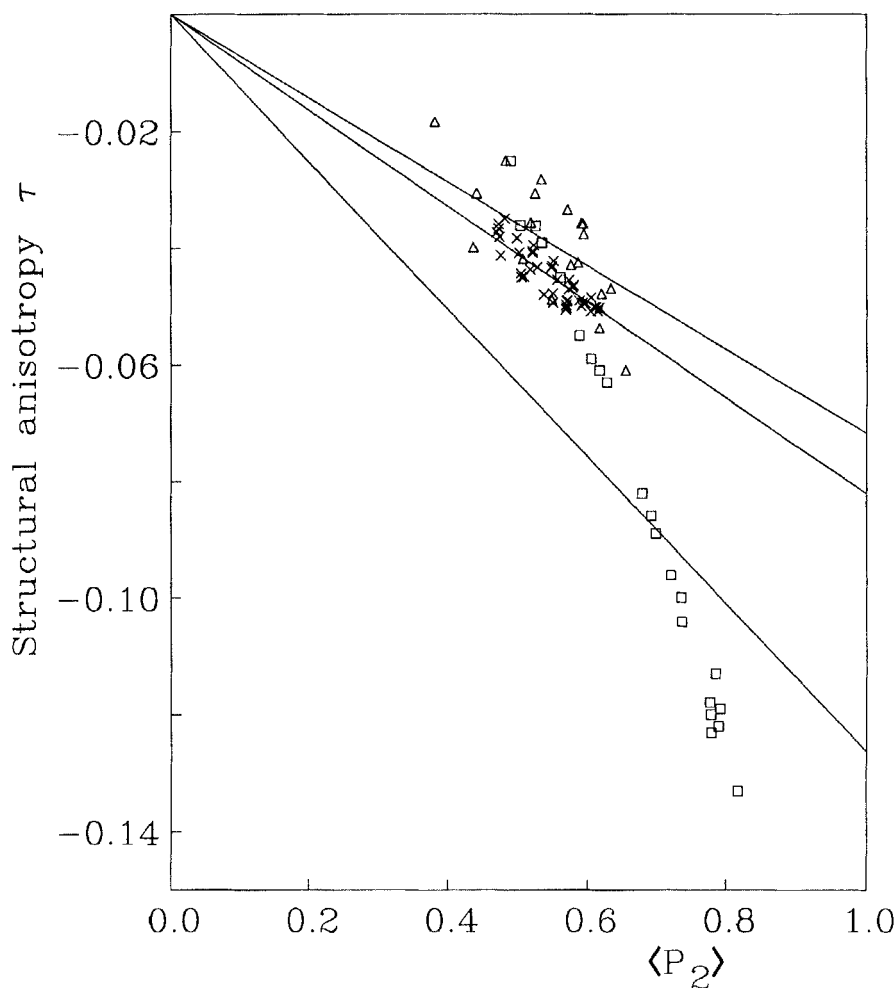


Figure 10. The dependence of the structural anisotropy on the order parameter $\langle P_2 \rangle$. The lines are the extrapolation to the perfectly ordered state. The values, calculated from equations (8) and (9) for the dyes and the liquid-crystalline solvent molecules anisotropy respectively, are (x) DMANAB -0.112 , (Δ) β -carotene -0.318 , \square C10 -0.144 ; solvent -0.121 .

interatomic data [23] are also depicted in the figures. It is evident, that the model estimates are in all cases too high. Probably, somewhat lower values will be obtained when account is taken of the molecular flexibility. In principle this could be done using literature data on *cis-trans* rotational barriers. But to perform the thermal equilibrium averages the molecular interactions in the mesophase and their influence on the molecule conformation should be taken into consideration. In addition the deviation of the molecular shape from a spheroid should be taken into account while calculating the depolarizing factors. This makes the problem of estimating the structural anisotropy factor from the model much more complicated.

While discussing the structural anisotropy it has been compared with the anisotropy of the dye molecules. However as we can see from equation (10) it must contain a contribution from the solvent molecule's structural anisotropy also. Probably the fact that the experimental data for all of the dyes in the solvent studied lie

approximately on a single straight line (see figure 10) can be regarded as a manifestation of this contribution. But for such analysis, as well as for the experimental determination of the structural anisotropy for nematogenic molecules themselves, some generalization of the local field theories is necessary.

5. Conclusions

In this paper the method for determining the structural anisotropy, which is based on a comparison of the degree of polarization of absorption and resonance Raman bands, has been applied to three dyes having different molecular dimensions in a single nematic solvent. Correlation between the variation of the structural anisotropy factor and the order parameters are observed but the experimental behaviour deviates from the predictions of the Segre model. Because of the lack of methods for investigating the structural anisotropy of solute molecules of different dimensions these experimental results could be compared directly only with some computer simulation data. In principle, this method could be applied for the local field anisotropy determination also for liquid-crystalline molecules, but we must take into account the observed dependence of the structural anisotropy on the probing molecule's dimensions.

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