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Liquid Crystals

Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t713926090

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Online publication date: 11 November 2010

To cite this Article Esteves, J. and Neto, A. M. Figueiredo(2002) 'Temperature dependence of the director reorientation of dye-doped nematic liquid crystals subjected to an optical field and its relation with the non-linear absorption coefficient', Liquid Crystals, 29: 5, 733 – 742

To link to this Article: DOI: 10.1080/02678290210127760 URL: http://dx.doi.org/10.1080/02678290210127760

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Temperature dependence of the director reorientation of dye-doped nematic liquid crystals subjected to an optical field and its relation with the non-linear absorption coefficient

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(Received 21 September 2001; in final form 20 December 2001; accepted 8 January 2002)

The temperature behaviour of the torque amplification factor η in the E48-D4 dye-host/liquid crystal-guest dyed system illuminated by a laser beam has been investigated using a single beam Z-Scan experimental technique. A plot of η versus temperature showed a monotonic aspect, different from other anthraquinone dyed liquid crystals. This behaviour, in our case, is consistent with the monotonic dependence on temperature of the linear α_o and non-linear β optical absorption coefficients. In particular, the non-linear optical absorption coefficient related to the reorienting process is negative and of the order of -0.25 mm W⁻¹. The temperature dependence of η seems to be directly correlated to β and not to α_o . Since drastic conformational modifications on dye molecules are not expected on passing from the ground to the excited state, the role of the electronic structure seems to be dominant in the reorienting process in dyed nematics.

1. Introduction

An interesting phenomenon in the field of nematic liquid crystals and their electro-optical properties is the coupling between optical fields and the local director [1, 2]. The basic mechanism which takes place is the interaction between the light electric field $\mathbf{E}(t)$ and the induced (and/or intrinsic) molecular electric polarization. The electromagnetic field couples to the liquid crystal director **n** and the volume torque Γ^{opt} [3, 4] is written as:

$$\Gamma^{\text{opt}} = \varepsilon_{\text{o}} \varepsilon_{\text{a}} (\mathbf{n} \ \mathbf{E}) (\mathbf{n} \times \mathbf{E})$$
(1)

where $\varepsilon_a = n_e^2 - n_o^2$, and n_e and n_o are the extraordinary and ordinary refractive indices, respectively.

The addition of absorbing dyes to nematic liquid crystals, even in small concentrations, introduces new orienting mechanisms [5] which can modify substantially the optical torque. Dye-doped liquid crystalline systems have indeed been the subject of intense studies in recent decades [6–14]. Jánossy and co-workers [3] suggested, from a phenomenological approach, that when a dyed nematic liquid crystal interacts with $\mathbf{E}(t)$, besides the 'direct' torque Γ^{opt} , an additional torque due to the dye, Γ^{dye} , should be present, and proposed that it is

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$$\Gamma^{dye} = \eta \langle \Gamma^{opt} \rangle = \varepsilon_o \zeta \langle (\mathbf{n} \ \mathbf{E}) (\mathbf{n} \times \mathbf{E}) \rangle$$
(2)

where $\langle \rangle$ denotes the time average and $\zeta = \varepsilon_a \eta$ is a dimensionless parameter which depends on the molecular characteristics of the dye and its concentration in the nematic matrix; η is the *dye amplification factor* of the torque.

The experimental techniques generally used to investigate this phenomenon are the optical transmittance [3, 6], measuring the Fréedericksz threshold [15], and Z-Scan [8, 9, 16, 17], measuring η and β (the nonlinear absorption). It was observed that in dyed nematic samples the Fréedericksz threshold is reduced by a factor of 10^2 with respect to the undyed samples. This effect could not be explained by means of thermal mechanisms, since this threshold presented an anomalous behaviour with the temperature [6], and not even by modifications of the liquid crystal parameters like the elastic constants or ε_a . The mechanism (on the molecular scale) responsible for this behaviour is not yet completely understood. The phenomenological parameter η is commonly used to characterize the dye-orienting effect on the nematic matrix. In the case of some azo and anthraquinone dyes dissolved in thermotropic nematic liquid crystals, η has been shown to be temperature and light wavelength

dependent [6, 9], of the order of 10^2 , positive or negative. Concerning the temperature dependence of η , a puzzling behaviour not yet completely understood was observed [6] for the liquid crystal E63 doped with anthraquinone dyes (AQ1 and AQ2): despite the linear absorption presenting an increase as a function of the temperature, η starts to decrease and afterwards increases with *T*.

To improve the understanding of the molecular mechanisms responsible for the coupling between the dye molecules and the director of nematics it is necessary to investigate different systems (liquid crystals *and* dyes) to verify this temperature dependence and the eventual contribution of the non-linear absorption on this mechanisms. To the best of our knowledge, particularly the relation between the non-linear absorption coefficient β and η has not been reported in the literature.

In this paper, we use the Z-Scan technique to determine the temperature dependence of η and β (due essentially to the director reorienting effect) in the guest-host system D4-E48, (trade names from BDH/Merck Ltd.).

2. Experimental

2.1. Materials

The liquid crystal used was the mixture E48 from BDH Ltd., which has a mean molecular mass about 240 g and the dye was D4, N,N'-(4-methylphenyl)-1,4-diaminoanthraquinone, with molecular mass 418 g. E48 presents a nematic calamitic phase in the range from $T \leq 19^{\circ}$ C to $T_{\rm NI} = 84^{\circ}$ C ($T_{\rm NI}$ is the nematic to isotropic transition temperature—the clearing point). Doped samples had 0.5% by weight of the dye in the liquid crystal. The transition temperatures $T_{\rm NI}$ were measured by polarizing optical microscopy (POM), using a temperature controlled hot stage (Instec HS1-i) for both undoped and doped samples. The difference between these values was less than 0.5°C. At this concentration, along a given direction there is one dye molecule for every seven liquid crystal molecules. D4 is a non-dichroic dye (BDH information data sheet) and has a (slightly) isosceles triangular shape (in a planar projection), with sides of about 1.4 nm. This linear dimension is comparable to the average length of the E48 molecules. To the best of our knowledge, there are no direct measurements of the topological orientation of D4 molecules in a nematic matrix. Considering the shape anisotropy of D4 molecules and assuming that we can apply to them the concept of 'order parameter', it is expected that this parameter is rather low.

Samples were mounted between glass plates, with spacers that kept the sample thickness $L = 24 \,\mu\text{m}$. The sample holder inner glass surfaces were coated with indium tin oxide (ITO) and lecithin, allowing the application of an external electric field \mathbf{E}_{e} to the sample, besides

the optical laser field. Samples were aligned (due to the lecithin coating) in a homeotropic configuration, with **n** perpendicular to the glass surfaces. This particular liquid crystal has an anisotropy of the dielectric susceptibility that, in the presence of an external electric field, orients with $n//E_e$. The glass used in the sample holder was chosen to have a negligible index of refraction mismatch with respect to the ITO coating. This choice was made in order to avoid spurious light reflections on the different internal interfaces. This aspect is particularly important in the measurement of the sample transmittance. We checked independently that the reflection at the first air/glass interface of the sample holder was the most significant in comparison with others at the inner interfaces (front and rear).

2.2. The Z-Scan set-up

The Z-Scan apparatus is described in more detail elsewhere [17-19] and sketched in figure 1. A polarized Gaussian laser beam, propagating in the z-direction, is focused to a narrow waist with a lens. The sample is moved along the z-direction through the focal point and the transmitted intensity is measured in the far field using a photodiode behind a small iris, as a function of the z-position. As the sample moves along the beam focus, self-focusing and -defocusing modifies the wavefront phase, thereby modifying the detected beam intensity. By measuring the transmittance as a function of the time t and the position z, the values of the non-linear refractive index, non-linear absorption (with a small modification of the original set-up $\lceil 17 \rceil$) and η are obtained. To measure the non-linear absorption, the transmittance is measured by the photodetector PD3 of figure 1 and all the transmitted light is collected by it as a function of the sample position z (open-aperture condition). A CW He-Ne ($\lambda = 632 \text{ nm}$) focused laser beam is used. The beam waist and the Rayleigh length are $\omega_{\rm o} = (2.94 \pm 0.05) \times 10^{-5} \text{ m and } z_{\rm o} = (4.3 \pm 0.1) \times 10^{-3} \text{ m},$ respectively. The power illuminating the sample is 10 and 0.65 mW, respectively, in the case of undoped and doped samples. The amplitudes of the optical field are 1.8×10^5 and 1.2×10^4 V m⁻¹, respectively. A signal acquisition, with temporal resolution, is made to discard the linear effects [20]. The angle between **n** and **E** (the optical field) is 45°. The sample is placed in a two-stage temperature controlled device, with Peltier elements, which allows temperature control within an accuracy better than 0.5°C. The temperature stability during an experimental run (see definition in the next section) is better than 0.5°C. The external electric field that could be applied to the sample is $E_e = 5 \times 10^7 \text{ V m}^{-1}$, at f = 100 Hz, about 10^2 bigger than the amplitude of the optical electric field. It was verified that at this frequency no hydrodynamic instabilities are present in the sample.



Figure 1. Sketch of the Z-Scan set-up. I, P, BS, L and PD are the iris, polarizers, beam splitters, lens and photodetectors, respectively.

3. Results and discussion

Figure 2 shows typical Z-Scan results with the undoped sample at different temperatures (T = 20, 55 and 70°C), without (a) and with (b) the external electric field. The waiting time between two successive z-positions of the sample to measure the transmittance is 2 s. An experimental run consists of a sequence of three independent experiments, with the sample moving successively along the z-axis. A data set is then obtained considering the mean values of the three experiments. The evaluated error in the normalized transmittance is of the order of 2% of the quoted value. This accuracy (and the others presented hereafter) takes into account not only the usual error propagation, but also the reproducibility of the experiments. The non-linear refractive index n_2 is obtained [17] from the peak-to-valley distance (ΔT_{pv}) of the Z-Scan curve according to equation (3):

$$n_{2} = \frac{\Delta T_{\rm pv} \lambda}{0.812 \pi L_{\rm eff} I_{\rm o}}$$
$$L_{\rm eff} = \frac{1 - \exp(-\alpha_{\rm o} L)}{\alpha_{\rm o}}$$
(3)

where α_o and I_o are the linear absorption coefficient and the optical field intensity at the focus (z = 0), respectively. α_o was calculated (see the table) from the measurements of the transmittance (doped samples), with (T_s) and without (T_b) the sample, as a function of the temperature, with a low laser beam intensity— $T_s = T_b [\exp(-\alpha_o L)](1-R)$, where $R \sim 0.04$ is the evaluated reflection loss factor. It is important to notice that the α_o of the undoped and doped samples are about 10 and 350 cm⁻¹, respectively,

Table. Experimental values of α_0 , η and n_2 for the undoped liquid crystal E48 and for doped (n_2^d) samples, as a function of temperature.

α_o/cm^{-1}	η	$n_2 \times 10^{-7}/$ cm ² W ⁻¹	$n_2^{ m d} imes 10^{-5} / { m cm}^2 { m W}^{-1}$
306 ± 28	-104 ± 16	1.49 ± 0.22	-2.37 ± 0.19
320 ± 15	-96 ± 13	1.48 ± 0.16	-2.28 ± 0.21
320 ± 15	-89 ± 10	1.63 ± 0.16	-2.29 ± 0.16
320 ± 15	-83 ± 14	1.65 ± 0.22	-2.17 ± 0.24
320 ± 15	-75.7 ± 9.8	1.65 ± 0.18	-1.97 ± 0.15
335 ± 15	-76 ± 14	1.60 ± 0.26	-2.00 ± 0.19
335 <u>+</u> 15	-64.6 ± 8.3	1.91 ± 0.22	-1.98 ± 0.13
350 ± 16	-66.6 ± 6.3	1.79 ± 0.14	-1.96 ± 0.14
354 ± 16	-51.6 ± 7.9	2.06 ± 0.31	-1.77 ± 0.09
354 + 16	-52+7	2.19 ± 0.32	-1.85 + 0.11
370 ± 17	$-47.\overline{6} \pm 5.7$	2.31 ± 0.21	-1.81 ± 0.17
	$\begin{array}{c} \alpha_{o}/cm^{-1} \\ \hline 306 \pm 28 \\ 320 \pm 15 \\ 320 \pm 15 \\ 320 \pm 15 \\ 320 \pm 15 \\ 335 \pm 15 \\ 335 \pm 15 \\ 355 \pm 16 \\ 354 \pm 16 \\ 354 \pm 16 \\ 370 \pm 17 \\ \end{array}$	$\begin{array}{cccc} \alpha_{o}/cm^{-1} & \eta \\ \hline & & \\ 306 \pm 28 & -104 \pm 16 \\ 320 \pm 15 & -96 \pm 13 \\ 320 \pm 15 & -89 \pm 10 \\ 320 \pm 15 & -83 \pm 14 \\ 320 \pm 15 & -75.7 \pm 9.8 \\ 335 \pm 15 & -76 \pm 14 \\ 335 \pm 15 & -64.6 \pm 8.3 \\ 350 \pm 16 & -66.6 \pm 6.3 \\ 354 \pm 16 & -51.6 \pm 7.9 \\ 354 \pm 16 & -52 \pm 7 \\ 370 \pm 17 & -47.6 \pm 5.7 \\ \end{array}$	$\begin{array}{cccc} n_2 \times 10^{-7} / \\ \alpha_o/cm^{-1} & \eta & cm^2 W^{-1} \end{array} \\ \hline 306 \pm 28 & -104 \pm 16 & 1.49 \pm 0.22 \\ 320 \pm 15 & -96 \pm 13 & 1.48 \pm 0.16 \\ 320 \pm 15 & -89 \pm 10 & 1.63 \pm 0.16 \\ 320 \pm 15 & -83 \pm 14 & 1.65 \pm 0.22 \\ 320 \pm 15 & -75.7 \pm 9.8 & 1.65 \pm 0.18 \\ 335 \pm 15 & -76 \pm 14 & 1.60 \pm 0.26 \\ 335 \pm 15 & -64.6 \pm 8.3 & 1.91 \pm 0.22 \\ 350 \pm 16 & -66.6 \pm 6.3 & 1.79 \pm 0.14 \\ 354 \pm 16 & -51.6 \pm 7.9 & 2.06 \pm 0.31 \\ 354 \pm 16 & -52 \pm 7 & 2.19 \pm 0.32 \\ 370 \pm 17 & -47.6 \pm 5.7 & 2.31 \pm 0.21 \end{array}$

i.e. doped samples present a linear absorption coefficient about 30 times larger than that of undoped samples. When \mathbf{E}_{e} is present, i.e. **n** is orientationally frozen, there is no Z-Scan classical response behaviour and $\Delta T_{pv} \rightarrow 0$, figure 2(b). In the absence of \mathbf{E}_{e} , the non-linear Z-Scan curve is characteristic of an induced convergent lens in the sample, with $n_2 > 0$; moreover, n_2 increases with T. These results indicate that the optical non-linearity observed is mainly due to reorientation of **n** from the coupling to the optical field.

Figure 3 shows a typical Z-Scan result with the doped sample at different temperatures (T = 60, 65 and 70°C), without (a) and with (b) the external electric field. Now the Z-Scan response is typical of a divergent lens (with



Figure 2. Typical Z-Scan results with the undoped sample at different temperatures (T = 20, 55 and 70°C): (*a*) without the external electric field, (*b*) with the external electric field.

 $n_2 < 0$) and the absolute value of n_2 decreases with T. When \mathbf{E}_e is present, the same behaviour is observed, i.e. the absolute value of n_2 decreases with T, but n_2 still reaches positive values, for example, at $T = 70^{\circ}$ C, figure 3(b). The local heating of the sample due to the laser beam was evaluated, taking into account its heat absorption coefficient, and was less than 0.5°C during an experimental run. We independently checked, by using POM (texture observations), that the sample does not undergo a nematic to isotropic phase transition due to the laser heating, during the time of an experimental run and for all the temperatures investigated.

It was verified that in doped and undoped samples, the ΔT_{pv} of the Z-Scan curves, figure 3(*b*), remain the same (within our experimental accuracy) for external fields until about $\mathbf{E}_{e}/3$. This result indicates that the homeotropic alignment is not expected to be significantly perturbed by the optical field in all the cases investigated, when the external field \mathbf{E}_{e} is present. The non-linear phase shift, present in the Z-Scan results with the doped samples in the presence of \mathbf{E}_{e} , indicates that besides the orientational origin, other mechanisms exist to account for this non-linear response.

The response of the medium (dye-doped liquid crystal) to the light electromagnetic field can be due to the lightinduced director reorientation and to any thermal lensing contributions. These contributions can be separated by superimposing the external field \mathbf{E}_{e} onto the optical field [7]. In this framework it is assumed that thermal effects remain the same with *and* without the external field. To study the non-linear response of the sample due *only to the reorienting effect* from the coupling between \mathbf{n} and \mathbf{E} , the experimental data are treated in the following way [7]: the Z-Scan results with \mathbf{E}_{e} present are subtracted from the results obtained when \mathbf{E}_{e} is absent. Typical Z-Scan results obtained with this particular



Figure 3. Typical Z-Scan results with the doped sample at different temperatures (T = 60, 65 and 70°C): (a) without the external electric field, (b) with the external electric field.

treatment are presented in figures 4(a) (undoped sample) and 4(b) (doped sample). In this framework we expect to isolate the non-linear phase shift due essentially to the orienting effect. The values of the non-linear refractive index of the undoped (n_2) and doped (n_2^d) samples are presented in the table. It is observed that in undoped samples n_2 (which is positive) increases with increasing T. The n_2^d values are negative, two orders of magnitude larger than the values obtained with undoped samples and with absolute values decreasing as T increases. Another possible procedure to treat the experimental Z-Scan results is to state the n_2 values for each Z-Scan profile and subtract the numbers to get the contribution from the orientational effect only. This procedure could be more appropriate if the magnitude of the thermal lensing contribution strongly depends on the orientation. We calculated the values of n_2^d following this latter procedure and verified that they differ from those presented in the table by less than 3%.

The torque amplification factor η can be obtained from the Z-Scan experiments (only due to the reorienting effect, as described above) [7]:

$$\eta = \frac{\Delta T_{\rm pv}^{\rm dye} P^{\rm host}}{\Delta T_{\rm pv}^{\rm host} P_{\rm mean}^{\rm dye}} - 1 \tag{4}$$

$$P_{\rm mean}^{\rm dye} = P^{\rm dye} \frac{1 - \exp\left(-\alpha_{\rm o}L\right)}{\alpha_{\rm o}L}$$
(5)

where ΔP_{pv}^{dye} , ΔP_{pv}^{host} , P^{dye} and P^{host} are the peak-tovalley transmittances and the input powers of the dyedoped and undoped samples, respectively. Their values are presented in the table and plotted, as a function of the temperature, in figure 5. It is observed that the bigger the linear absorption, the smaller the absolute value of η , as far the temperature dependences of these parameters are concerned. The decrease of η with increasing temperatures could be partially due to the approach to the nematic to isotropic phase transition of the host. A



Figure 4. Typical Z-Scan results at different temperatures obtained by subtracting the transmittance values (at a given z) with \mathbf{E}_e present from the values measured without \mathbf{E}_e . T = 20 and 65°C: (a) undoped sample, (b) doped sample.



Figure 5. Amplification factor due to the reorienting effect as a function of the temperature.

decrease of the order parameter S as T increases is expected, until the jump to S = 0 at $T = T_{\rm NI}$. As $\eta < 0$, the dye torque is opposite to the optical torque. This behaviour is different from that observed [6] in the doping of E63 with AQ1 and AQ2 (He-Ne light) as a function of the temperature: despite the linear absorption presented with an increase as a function of the temperature (as for our results), η (which is positive) starts to decrease and afterwards increases with T. As the hosts E48 and E63 have essentially the same physicochemical properties (clearing points, flow viscosity, dielectric anisotropy, optical anisotropy, threshold voltage [21]), the different temperature behaviours observed in η versus T seem to be characteristics of the dye molecule. Although the cores of the AQ(1, 2) and D4 molecules are essentially the same, D4 has two additional aromatic rings. Drastic conformational changes are not expected to take place to these dye molecules due to the optical field. On



Figure 6. Typical Z-Scan results with doped samples for the measurement of the non-linear absorption coefficient at different temperatures: (a) without \mathbf{E}_{e} , T = 20, 55 and 65°C; (b) with \mathbf{E}_{e} , T = 20, 55 and 65°C; (c) data corresponding to the reorienting effect due to the coupling between **n** and \mathbf{E}_{e} , T = 20 and 65° C.

the other hand, their electronic structure in the excited state is different from that in the ground state, changing the interaction energy with the liquid crystal host.

Typical Z-Scan (open-aperture condition) results used to measure the non-linear absorption coefficient are presented in figure 6 as a function of temperature, for



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Figure 7. Temperature dependence of the non-linear absorption coefficient of the dyed sample: (a) with \mathbf{E}_{e} , (b) without \mathbf{E}_{e} , (c) contribution of the reorienting effect only.

doped samples without (a) and with \mathbf{E}_{e} (b). Undoped samples do not give, within our accuracy, a measurable value of β . An interesting feature is observed in the Z-Scan profiles of doped samples for $T < 50^{\circ}$ C with-

out \mathbf{E}_{e} applied, figure 6(*a*): there is a peak (increase in transmittance) at $z \sim 0$. This behaviour is not observed when \mathbf{E}_{e} is present. Similar behaviours were observed for dye solutions of chloroaluminum phthalocyanine in

methanol [22] and some organic materials [23], for smaller time-scale regimes (ps) and larger incident light energy pulses. Differently from our experiment, these results dealt with isotropic systems. The physical process which takes place for such materials is related to electronic transitions (saturable absorption—SA) with β written as a function of the imaginary part of the third order susceptibility $\chi^{(3)}$ [23]. SA in dyes occurs at power densities of about 1 MW cm⁻², much higher than that used in our experiment (24 W cm^{-2}). Since this behaviour is not observed in our samples when E_e is present, i.e. when the optical electric field reaches the dye molecules in a particular relative orientation, we will interpret our observations as a consequence of the anisotropy of the medium. As discussed in $\S2$, the D4 molecule can be sketched as a straight parallelepiped with a (slightly) isosceles base (its height being smaller than any typical dimension in the base). Anisometric dye molecules can be oriented in the nematic matrix [24] by means of a mechanical coupling between them and n. In this condition, the optical electric field reaches dye molecules that are in different relative orientations, depending on the nematic matrix orientation. Our results indicate that the non-linear absorption depends on this relative orientation.

Referring to figure 7, in doped samples, both with (a) and without (b) \mathbf{E}_{e} , β increases with T. In particular with \mathbf{E}_{e} applied (a), $\beta \sim 0$ for $T < 50^{\circ}$ C. In the case of the samples without \mathbf{E}_{e} present, $\beta < 0$ for $T < 50^{\circ}$ C. Following the same procedure described above, we evaluate the contribution of the reorienting effect due to the coupling between **n** and **E** in β , subtracting from the Z-Scan results without E_e the results with E_e . These results are presented in figure 6(c). The contribution of the reorienting effect in β , as a function of T is shown in figure 7(c). The values obtained following this procedure differ less than 3% from those calculated simply by subtracting the results in figure 7(a) from those in figure 7(b), as discussed in the calculation of n_2^d above. There is a slight tendency to decrease the absolute value of β as T increases. In almost all the temperature range investigated (except at $T = 70^{\circ}$ C), β is negative and small.

Let us now analyse the complete set of absorption data available—linear, table 1; non-linear related to the orienting effect, figure 7 (c)—and the torque amplification factor η , figure 5, as a function of T. The absolute value of η decreases for increasing temperatures, while α_o increases with T and the absolute values of β related to the orienting effect decrease with T. Our results indicate that the higher the non-linear absorption due to the orienting effect, the higher the torque amplification. This correlation is summarized in figure 8. The temperature behaviour of η is not directly correlated with α_o . The effectiveness of the dye in promoting a torque in the



Figure 8. Temperature dependences of the torque amplification factor η and the non-linear absorption related to the orienting effect.

nematic matrix appears to be mostly related to its non-linear absorption.

In summary, the temperature behaviour of η in the E48-D4 host-guest system showed a monotonic aspect, different from other anthraquinone dyed liquid crystals (AQ1,2 in E63). This behaviour, in our case, is consistent with the monotonic dependence on temperature of the linear and non-linear optical absorption coefficients. In particular, the non-linear optical absorption coefficient related to the reorienting process is negative and of the order of -0.25 mm W^{-1} . The temperature dependence of η seems to be directly correlated with β and not α_{o} . Since drastic conformational modifications of dye molecules are not expected to occur from the ground to the excited state, the role of the electronic structure seems to be dominant in the reorienting process in dyed nematics.

Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP), CNPq and PRONEX supported this work. It is a pleasure to thank Dr T. Kósa and Msc. S. Alves for very helpful discussions. One of us (J.E.) also thanks Dr R. Horowicz.

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