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FTIR spectroscopic determination of quadrupolar order parameters in smectic C phases†

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By means of FTIR spectroscopy the quadrupolar order parameter $\langle\cos 2\psi\rangle$ has been determined for the smectic C and C* phases of three mesogenic compounds where the angle ψ specifies the rotation around the molecular long axis. The CO vibration and γ-CH vibration of phenyl groups have been used as spectroscopic probes. The quadrupolar ordering is rather large and different for different parts of the molecules, indicating an internal flexibility. From the spectroscopic data it is deduced that the phenyl rings of the aromatic cores are oriented mainly within the tilt plane. Using the microscopic model of Zeks, the quadrupolar parts of the rotational potential have been found to be in the order of magnitude of the thermal energy, but differ for different parts of the molecules.

1. Introduction

If liquid crystalline tilted smectic C or C* phases are formed by biaxial molecules the rotational distribution function $f(\psi)$ of the transverse molecular axes around the molecular long axis will differ from cylindrical symmetry. The angle ψ specifies the rotation of the transverse molecular axes around the molecular long axis. The molecular tilt causes steric hindrance of the rotation of the molecules around their long axes. In consequence there exists a non-vanishing averaged value of the quadrupolar order parameter $\langle\cos 2\psi\rangle$ [1–4]. The rotational potential is given by Zeks *et al.* [5]:

$$U(\psi) = -a_1\theta\cos\psi - a_2\theta^2\cos 2\psi. \tag{1}$$

Equation (1) contains two terms with the coefficients a_1 and a_2, and θ represents the tilt angle. The first term describes a polar rotational distribution of the transverse molecular axes and exists only in chiral SmC* phases. This term is responsible for the spontaneous polarization if the molecules possess a transverse electric dipole moment. The second term exists in SmC and SmC* phases, is non-chiral in character and represents the quadrupolar rotational distribution.

With the potential (1) the quadrupolar order parameter $\langle\cos 2\psi\rangle$ can be written in terms of statistical mechanics as follows:

$$\langle\cos 2\psi\rangle = \frac{\int_0^{2\pi}\cos 2\psi\exp(-U/kT)\,\mathrm{d}\psi}{\int_0^{2\pi}\exp(-U/kT)\,\mathrm{d}\psi} \tag{2}$$

where U is the Zeks potential of equation (1). The solution of the integrals in equation (2) requires a series expansion. According to references [6, 7] there results:

$$\langle\cos 2\psi\rangle = B\left(\frac{a_2\theta^2}{kT}\right). \tag{3}$$

The function $B(x)$ represents a ratio of two modified Bessel functions of the independent variable $x = a_2\theta^2/kT$ [6, 7] (see figure 1). $B(x)$ is comparable to the Langevin function but describes a two-dimensional rotation in a plane perpendicular to the molecular long axis. This two-dimensional character is reflected in an initial slope of 1/2 of $B(x)$ in contrast to the Langevin function with 1/3 [6].

The statistical calculation of the polar order parameter $\langle\cos 2\psi\rangle$ leads to the following equation [6]:

$$\langle\cos\psi\rangle = \frac{a_1\theta}{2kT}(1+\langle\cos 2\psi\rangle). \tag{4}$$

Equation (4) shows the influence of the quadrupolar order parameter on the polar order parameter which is proportional to the spontaneous polarization $\mathbf{P}_S$. Therefore it is possible to estimate the quadrupolar order parameter $\langle\cos 2\psi\rangle$ by means of polarization

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† Presented at the 24th Liquid Crystal Workshop, Freiburg i. Br. 1995.

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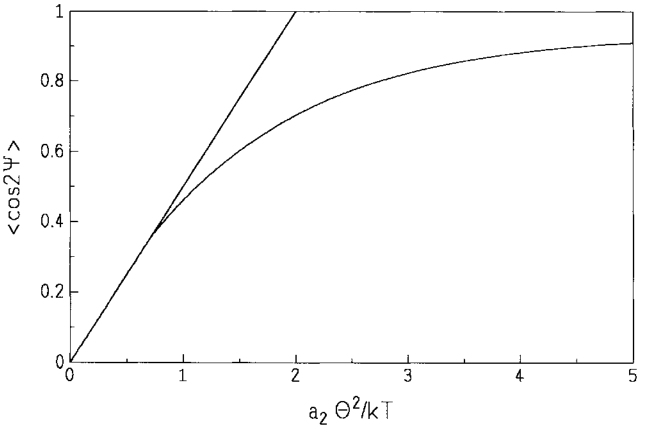


Figure 1. Function $\langle \cos 2\psi \rangle = B(a_2\theta^2/kT)$, initial slope 1/2 (after Meister [6], Ch. B1).

measurements [6]. It must be stressed that the values of $\langle \cos 2\psi \rangle$ obtained by $\mathbf{P}_S$ measurements are due to the averaged contributions of all dipolar groups within the molecule.

By means of FTIR spectroscopy used in this paper the quadrupolar order parameters of particular molecular groups (spectroscopic probes) can be determined, and this generates the possibility of obtaining information about the flexibility of different parts of the mesogenic molecules.

2. Theoretical considerations

The quadrupolar order parameters $\langle \cos 2\psi \rangle$ have been determined by means of FTIR spectroscopy and tilt angle measurements. As spectroscopic probes we used the C=O vibrations and the γ-CH out of plane vibrations of the phenyl groups. By the following procedure it is possible to determine $\langle \cos 2\psi \rangle$ by measuring the IR absorption in the smectic C/C* phase and in the isotropic phase using unpolarized IR radiation.

Let us consider the orientation of an arbitrary transition dipole moment **M** within a molecular frame:

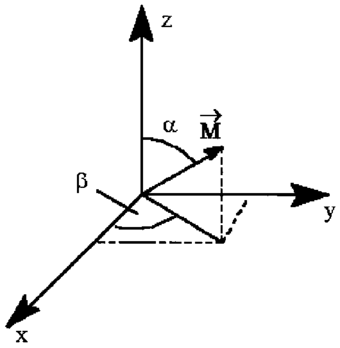


Figure 2. Orientation of the transition moment **M** within the molecular frame *xyz* and definition of the angles α and β [see equations (5), (6) and (8)].

The components of the oscillator strength f within the molecular frame which are proportional to $\mathbf{M}^2$ then can be written as follows:

$$f_x = f_0 \sin^2 \alpha \cos^2 \beta \tag{5a}$$

$$f_y = f_0 \sin^2 \alpha \sin^2 \beta \tag{5b}$$

$$f_z = f_0 \cos^2 \alpha \tag{5c}$$

where f_0 represents the maximum oscillator strength. The application of the Euler transformation matrix [8] to the components in equation (5) leads to the corresponding expressions within the laboratory frame:

$$\begin{aligned} f_X = f_0[&(\cos\psi\cos\phi - \cos\theta\sin\psi\sin\phi)^2 \sin^2\alpha\cos^2\beta \\ &+ (-\sin\psi\cos\phi - \cos\theta\cos\psi\sin\phi)^2 \sin^2\alpha\sin^2\beta \\ &+ \sin^2\theta\sin^2\phi\cos^2\alpha] \end{aligned} \tag{6a}$$

$$\begin{aligned} f_Y = f_0[&(\cos\psi\sin\phi + \cos\theta\sin\psi\cos\phi)^2 \sin^2\alpha\cos^2\beta \\ &+ (-\sin\psi\sin\phi + \cos\theta\cos\psi\cos\phi)^2 \sin^2\alpha\sin^2\beta \\ &+ \sin^2\theta\cos^2\phi\cos^2\alpha] \end{aligned} \tag{6b}$$

where ϕ denotes the azimuthal angle. ψ is equal to the rotation angle around the molecular long axis and θ represents the known tilt angle (see § 1).

The oscillator strength component f_z within the laboratory frame is not necessary in our mathematical treatment, because we consider IR light which is irradiated parallel to the smectic layer normal (laboratory Z axis). In this case the oscillator strength for unpolarized IR light is given as $f_{\mathrm{LC}} = 1/2\ (f_X + f_Y)$. For the isotropic phase one gets $f_{\mathrm{I}} = 1/3 f_0$. Taking into account that the absorption peak area is proportional to f, the ratio of the absorption peak areas in the liquid crystalline and the isotropic state can be written as:

$$d = \frac{f_{\mathrm{LC}}}{f_{\mathrm{I}}} = \frac{3}{2}\,\frac{f_X + f_Y}{f_0} \tag{7}$$

where d is denoted as the relative absorption.

Substitution of equations (6) into equation (7) and forming the expectation value finally leads to the following expression for the quadrupolar order parameter $\langle \cos 2\psi \rangle$.

$$\langle \cos 2\psi \rangle = \frac{\frac{4}{3}d - 2\cos^2\alpha\sin^2\theta - \sin^2\alpha(1+\cos^2\theta)}{\cos 2\beta\sin^2\alpha(1-\cos^2\theta)}. \tag{8}$$

Details are described in reference [9].

It is reasonable that equation (8) is not sensitive to the azimuthal angle ϕ because we have considered unpolarized IR radiation in our calculations. In equation (8), α and β represent the orientation of a transition dipole moment within the molecular frame acccording to figure 2. In the tilted smectic C/C* phase, the angle β can be arbitrarily set to 0° or 90° for symmetry reasons. The angle α can be determined from the relative absorption in the uniaxial SmA (d_A) or nematic phase (d_N) [9]:

$$\sin^2 \alpha = \frac{2}{3} d_A \tag{9}$$

$$\sin^2 \alpha = \frac{2}{3} - \frac{\frac{2}{3}(1 - d_N)}{S_2}. \tag{10}$$

Equation (9) is only valid for the SmA phase because the value of the orientation order parameter S_2 is rather large and tends to unity in this case (about 0·9 for our substances) and thus may be neglected. In the nematic phase, for comparison, one has to consider the orientational order parameter S_2 [equation (10)]. Of course for $S_2 \to 1$, equation (10) transforms back into equation (9).

3. Experimental

The quadrupolar order parameter $\langle \cos 2\psi \rangle$ in the smectic C/C* phases has been measured for the liquid crystalline compounds ZLI 4421, DOBAMBC and 8OO7, the structures of which are shown below. The phase transition temperatures are given in °C. The investigated spectroscopic probes are marked by arrows. Unfortunately, the signal for the CN vibration in ZLI 4421 was so weak that we could not use it as a probe.

ZLI 4421 was purchased from E. Merck. Syntheses of the substances DOBAMBC and 8OO7 are described in the literature [9, 10]. The test samples were prepared between HTAB (hexyltrimethylammonium bromide) coated NaCl plates (Optispec). The HTAB coating supports the homeotropic orientation of the smectic layers between the NaCl plates. The suitable sample thickness of about 12 μm was realized by mylar spacers. The temperature of the sample was controlled by a hot stage (Mettler FP 52). The spectra were scanned by an FTIR spectrometer (Nicolet 5 MX) and the appertaining software (Spectrafile IR, Heyden & Sohn). The unpolarized IR beam was irradiated parallel to the smectic layer normal.

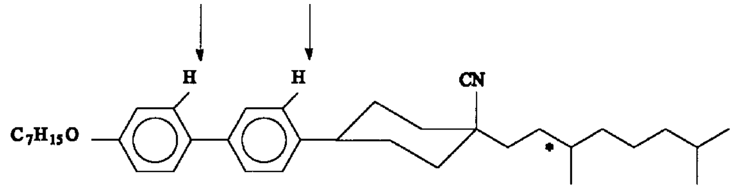
For the tilt angle measurements, commerical cells (E.H.C. Co., Ltd) were used. In the ferroelectric SmC* phases of ZLI 4421 and DOBAMBC, the tilt angle was derived from the optical switching angle which can be observed using the polarizing microscope [12, 13]. For 8OO7 the tilt angle data have been taken from references [6, 13].

4. Results and discussion

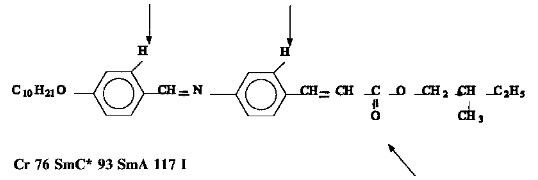
4.1. *Spectroscopic measurements and quadrupolar order parameters*

The relative absorption d and the quadrupolar order parameters of the chiral molecules ZLI 4421 and DOBAMBC and of the achiral molecule 8OO7 were investigated for the SmC* or SmC phase and are presented in figures 3–5.

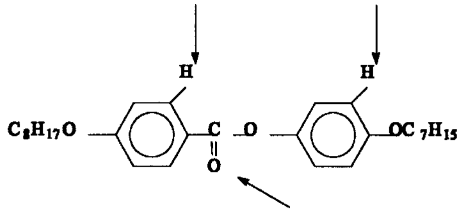
The quadrupolar order parameters obtained for the different molecular groups exhibit the rather large values



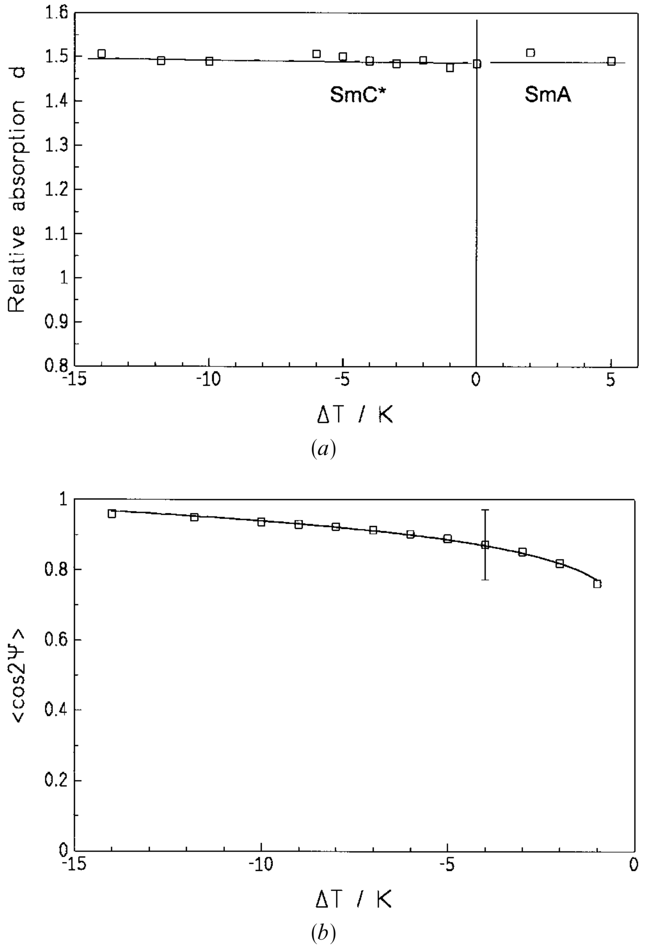
ZLI 4421 Cr 89 SmC* 109 SmA 118 I



DOBAMBC Cr 76 SmC* 93 SmA 117 I



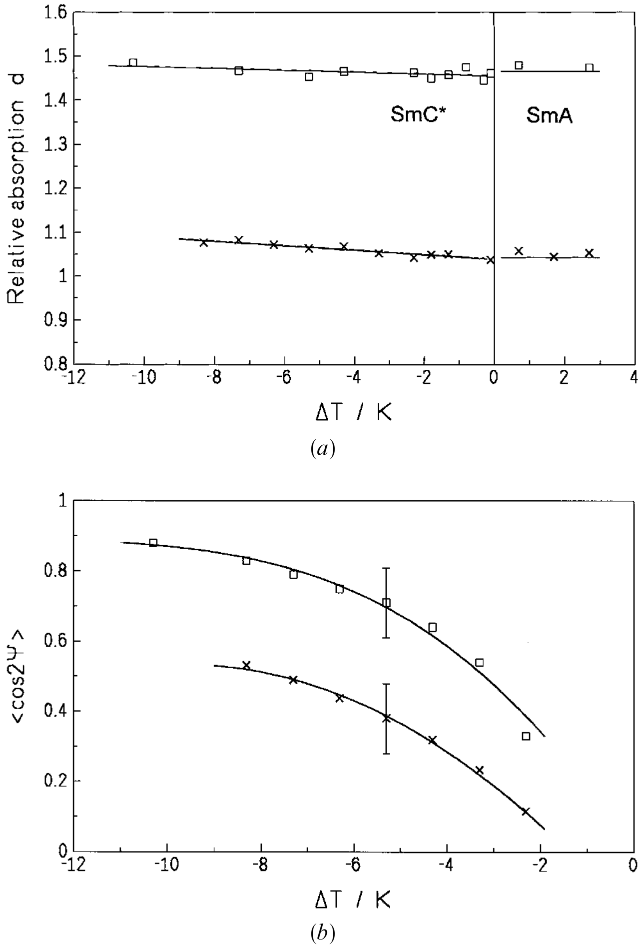
8OO7 Cr 62.9 SmC 69.1 N 86.6 I



(*a*)

(*b*)

Figure 3. (*a*) Relative absorption d of the γ-CH vibration band in ZLI 4421 as a function $\Delta T = T - T_C$. (*b*) Quadrupolar order parameter $\langle \cos 2\psi \rangle$ as a function of $\Delta T = T - T_C$ in the SmC* phase of ZLI 4421 ($\alpha = 90°$).



(*a*)

(*b*)

Figure 4. (*a*) Relative absorption d as a function of $\Delta T = T - T_C$ for DOBAMBC; □: γ-CH, phenyl groups, ×: CO group. (*b*) Quadrupolar order parameter $\langle \cos 2\psi \rangle$ as a function of $\Delta T = T - T_C$ in the S_C^* phase of DOBAMBC; □: aromatic core ($\alpha = 85°$), ×: CO groups ($\alpha = 56{\cdot}8°$).

which have been theoretically predicted by Osipov and Pikin [4] because of the existence of strong quadrupolar ordering in the smectic C phases. However, by NMR and NOR methods, which are due to the ordering of single atoms, smaller values of $\langle \cos 2\psi \rangle$ have been obtained [2, 3, 14]. Values of $\langle \cos 2\psi \rangle$ obtained from measurements of the spontaneous polarization [6, 15] are also smaller than those derived from the FTIR data. On the other hand, from the polarization values of DOBAMBC taken from [16] we estimated a value of about 0·8 for $\langle \cos 2\psi \rangle$. However, it is of note that these values are due to the ordering of the whole molecule.

The relative absorption d of the γ-CH and the CO vibration is rather independent of temperature in the smectic C/C* phase for all the molecules under investigation [cf. figures 3 (*a*), 4 (*a*), and 5 (*a*)]. The values of $\langle \cos 2\psi \rangle$ decrease with increasing temperature. This temperature dependence obviously is due to the influence of the tilt angle θ which decreases with increasing temperature. As a consequence of equation (1), the steric hindrance of the rotation around the molecular long axis decreases and as a result $\langle \cos 2\psi \rangle$ decreases [see figures 3 (*b*), 4 (*b*) and 5 (*b*)].

Furthermore the results show that $\langle \cos 2\psi \rangle$ depends on the internal position of the spectroscopic probe within the molecule. In the case of DOBAMBC, the CO group is separated from the core and consequently the corresponding quadrupolar order parameter is considerably smaller than $\langle \cos 2\psi \rangle$ of the more rigid aromatic core [see figure 4 (*b*)]. The large values of $\langle \cos 2\psi \rangle$ of the aromatic core derived from the γ-CH vitractions†

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† Note that the γ-CH band we used is due to both phenyl groups in the compounds under investigation because, according to Bellamy [17], the position of this band is determined by the position of substituents in the phenyl ring and independent of the nature of the substituents.

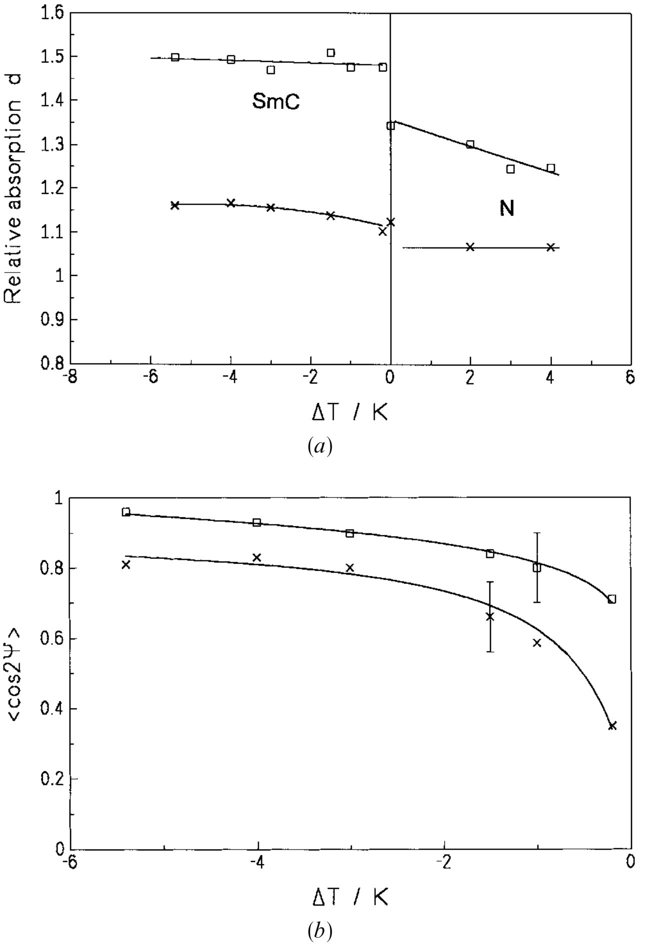

Figure 5. (*a*) Relative absorption *d* as a function of $\Delta T = T - T_C$ for 8OO7; □: γ-CH, phenyl groups, ×: CO group. (*b*) Quadrupolar order parameter $\langle \cos 2\psi \rangle$ as a function of $\Delta T = T - T_C$ in the SmC phase of 8OO7; □: aromatic core ($\alpha = 90°$), ×: CO group ($\alpha = 59{\cdot}5°$).

can be explained by dispersion interactions between the aromatic groups of neighbouring molecules. Obviously this interaction hinders the rotation of the aromatic core around the molecular long axis.

The quadrupolar order parameters of the two spectroscopic probes γ-CH and CO in DOBAMBC [cf. figure 4 (*b*)] differ by a factor of 2 as the CO dipole is decoupled from the aromatic core. This clearly shows that the molecule is far from being a rigid rod and possesses an internal flexibility [14].

In ferroelectric mixtures of chiral guest and non-chiral host substances, molecules like DOBAMBC are denoted as guest molecules of type I [18]. It has been shown experimentally [19], as well as by a theoretical treatment [20], that in so-called induced ferroelectric SmC* phases with guest molecules of type I the tilt angle reduced polarization $\mathbf{P}_0 = \mathbf{P}_S/\sin\theta$ is independent of the nature of the host phase, because of a decoupling between the core and the transverse dipole. This decoupling is supported by the different observed quadrupolar order parameters.

In 8OO7 the values of $\langle \cos 2\psi \rangle$ for the CO group are only slightly smaller than those for the γ-CH of the aromatic groups [cf. figure 5 (*b*)]. This is reasonable because the CO group is included in the core of the mesogenic molecule. Hindered rotations of molecular groups are also discussed in reference [21], but without specifying the corresponding quadrupolar order parameters.

For all compounds under investigation we found that the relative absorption *d* of the aromatic out-of-plane C–H vibration is rather large and nearly independent of temperature in the smectic C/C* phases [figures 3 (*a*), 4 (*a*), and 5 (*a*)], despite the strong temperature dependence of the tilt angle θ in these compounds (cf. [12], [16] and [13]). As the IR light beam was parallel to the smectic layer normal, one must conclude that the transition moment **M** (CH) is located within the layer plane. More specifically, **M** (CH) must be oriented perpendicular to the tilt plane *ZY* (see figure 6). Consequently, the phenyl groups of the mesogenic molecules are mainly oriented parallel to the tilt plane because **M** (CH) is perpendicular to the phenyl rings (figure 6). Further, the large value of *d* (CH) indicates that this orientation of the phenyl groups is rather fixed, leading to the observed large quadrupolar ordering.

In the nematic phase of 8OO7 *d*(CH) is smaller than in the smectic phase and does depend on temperature [see figure 5 (*a*)]. This result is reasonable because the phenyl groups librate around the director and S_2 is smaller than in the smectic C phase and depends on temperature.

4.2. *Quadrupolar order parameter and tilt angle*

Additionally, we investigated the tilt angle dependence of $\langle \cos 2\psi \rangle$ for the spectroscopic probes of the compounds presented. The results are shown in figures 7, 8 and 9.

According to figure 1 the dependence $\langle \cos 2\psi \rangle$ on θ^2

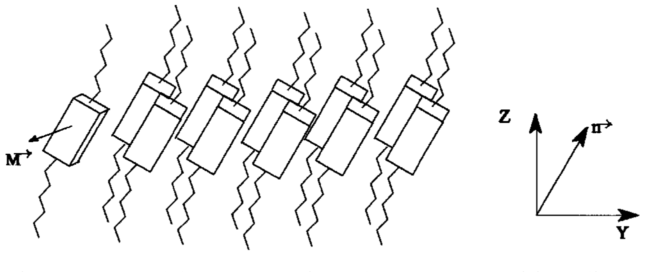

Figure 6. Model of a smectic C layer. **M**: transition dipole moment of the γ-CH out of plane vibration perpendicular to the aromatic planes; the aromatic planes lie within the tilt plane *ZY*, **n** director.

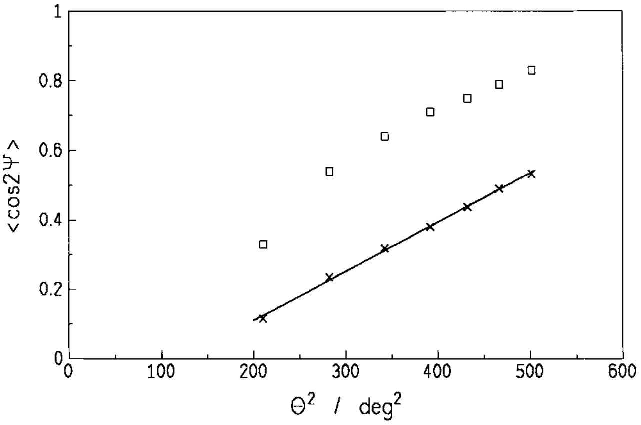

Figure 7. Quadrupolar order parameter $\langle \cos 2\psi \rangle$ as a function of θ^2 in the SmC* phase of DOBAMBC; □: aromatic core, ×: CO group.

should be linear up to values of $\langle \cos 2\psi \rangle$ of approximately 0·4–0·5 (the Boltzmann factor $1/kT$ is nearly constant under the experimental conditions). On the basis of the Zeks model [15], the slope of the linear function in figure 7 should be $a_2/2kT$. This leads to a value of the Zeks parameter of $a_2 = 4{\cdot}6 \times 10^{-20}$ J rad^{-2} ($T = 360$ K) for the CO group of DOBAMBC in the SmC* phase. Thus for the quadrupolar part of the Zeks potential, equation (1), we find $a_2\theta^2 = 5{\cdot}6 \times 10^{-21}$ J ($\theta = 20°$, 0·35 rad). This value is comparable to the thermal energy kT at $T = 360$ K ($4{\cdot}97 \times 10^{-21}$ J). For the aromatic core of DOBAMBC the $\langle \cos 2\psi \rangle$ values are significantly higher than for the CO group [see figure 4 (*b*)]. Therefore the potential $a_2\theta^2$ should be higher for the aromatic core as well. For $\theta = 20°$ we found a value of $a_2\theta^2$ of about 10^{-20} J ($\approx 2kT$) for the aromatic core, twice as large as that for the CO group.

The results for the potentials of the compounds ZLI 4421 and 8OO7 (see figures 8 and 9) are similar to that of the aromatic core of DOBAMBC. For $\theta = 20°$ we found $a_2\theta^2$ 1–$1{\cdot}25 \times 10^{-20}$ J ($\approx$2–$2{\cdot}5kT$). The result that the quadrupolar rotational potential of the phenyl groups in the molecules under investigation is larger than the thermal energy is consistent with the suggestion of aromatic cores which are rather tightly located within the tilt plane.

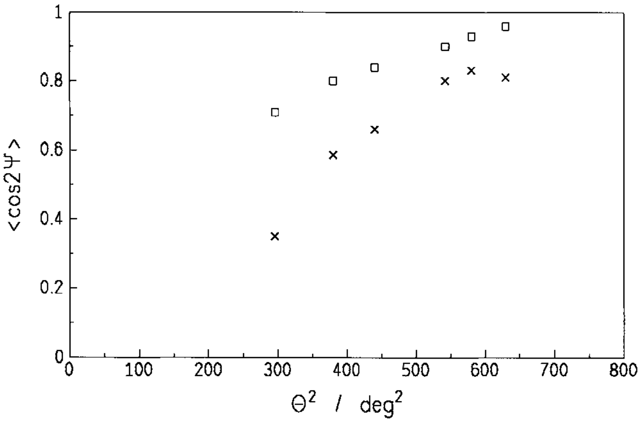

Figure 8. Quadrupolar order parameter $\langle \cos 2\psi \rangle$ as a function of θ^2 in the SmC phase of 8OO7; □: aromatic core, ×: CO group.

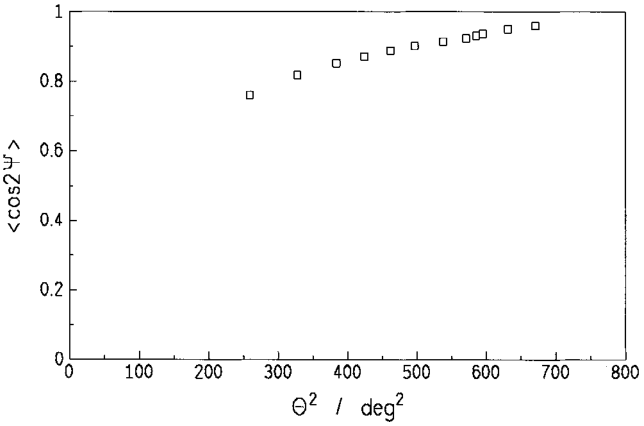

Figure 9. Quadrupolar order parameter $\langle \cos 2\psi \rangle$ as a function of θ^2 in the SmC* phase of ZLI 4421; aromatic core.

By means of spontaneous polarization measurements, Buka *et al.* [22, 23] obtained a quadrupolar potential of $a_2\theta^2 = 2 \times 10^{-21}$ J for ZLI 4421 at $\theta = 20°$ using a fitting procedure. This value is comparable with our result for the CO group in DOBAMBC, but is a factor of 5 smaller than that found here for the aromatic core of ZLI 4421. This difference can be easily understood if one considers the molecular structure of ZLI 4421. The spontaneous polarization $\mathbf{P}_S$ is caused mainly by the CN dipole which is located away from the aromatic-containing core and to a certain degree is decoupled from it. Thus the CN dipole is more flexible than the core. As the potential derived by Buka *et al.* from $\mathbf{P}_S$ is due to this dipole, it is reasonable that our potential derived from the γ-CH vibrations of the aromatic core is larger. Again, the advantage of methods using molecular groups as probes over integral methods is demonstrated.

5. Summary

The quadrupolar order parameters $\langle \cos 2\psi \rangle$ of the CO groups and phenyl groups of DOBAMBC and 8OO7 and the phenyl groups of ZLI 4421 have been determined for the smectic C/C* phase by means of FTIR spectroscopy and tilt angle measurements. The values of $\langle \cos 2\psi \rangle$ are rather large and decrease with increasing temperature and decreasing tilt angle θ. The rather large values of $\langle \cos 2\psi \rangle$ are in agreement with theoretical predictions [4]. The different $\langle \cos 2\psi \rangle$ values for the CO and aromatic groups of DOBAMBC provide evidence for an internal molecular flexibility [14].

The observed temperature independence of the relative absorption *d* indicates that the phenyl planes of the investigated molecules are oriented within the tilt planes (figure 6).

Using the microscopic model developed by Zeks *et al.* [5] we found different quadrupolar potentials for the restricted rotation around the molecular long axis for molecular groups at different molecular sites. The potential of the CO group of DOBAMBC is in the order of magnitude of the thermal energy kT. As this CO group is located within a terminal chain of the molecule its flexibility is reasonable. On the other hand, for spectroscopic probes within the more rigid aromatic molecular core, this potential is found to be about $2kT$. This result also indicates an internal flexibility of the molecules.

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