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Direct smectic A–smectic C_A^{*} phase transition

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The anticlinic smectic C_A phase belongs to the class of tilted smectic phases with an azimuthal angle alternating from one direction ($\varphi=0$) to the other ($\varphi=\pi$) in successive layers. It occurs in general at lower temperature than the uniformly tilted smectic C phase, but may be obtained directly from the untilted smectic A phase. We use the chiral nCTBB9* series synthesized in this laboratory, in order to obtain a phase transition as close as possible to second order, as revealed by DSC. We measure the temperature behaviour of the birefringence and of the optical rotatory power across the transition in order to characterize the tilt angle. We finally study the optical response to a periodic electric field which excites separately the smectic C* and C_A^{*} soft modes. The main conclusion is that the only order parameter governing the critical behaviour of the phase transition is the tilt angle θ , as we get a common divergence of both soft modes at the same temperature. This confirms previous high resolution calorimetric studies by Ema *et al.* that saw in MHPOBC an initial mean-field second order phase transition when the tilt appears, followed by sharp first order restructuring transitions between the tilted subphases.

1. Introduction

The structures of new tilted chiral smectic phases initially discovered in MHPOBC [1] are still controversial, mainly for the so-called ferroelectric phases. Two of them are well understood. The helielectric smectic C* phase has a uniform tilt angle θ and an azimuthal angle φ precessing around the layer normal together with the permanent volume polarization \mathbf{P} . In the anticlinic smectic C_A^{*} phase, θ is also uniform but the layers have to be considered as pairs with φ and $\varphi+\pi$ values; there is also a precession of φ together with a complex set of surfacic polarization densities allowed by symmetry [2–4]. The nature of the phase transition from smectic A to smectic C* is well understood; it is often mean-field second order and can be characterized by electroclinic experiments [5, 6]. When the more complicated phases like smectic C_α^{*} or C_γ^{*} appear, it seems that the mean-field character is conserved at the smectic A to smectic C_α^{*} phase transition where the tilt occurs [7], while the other phase transitions are restructuring ones with small latent heats. It has been reported that the direct transition from untilted smectic A to tilted smectic C_A^{*} can be achieved in a compound like TFMHPOBC [8], so this direct phase transition can be studied and compared to the classical A–C* transition [9]. In this paper we

briefly recall some available theories for the A–C* and A–C_A^{*} phase transitions. We then present a series of components synthesized in our group, the nCTBB9* series, which shows a weakly first order direct A–C_A^{*} phase transition. Various physical quantities are measured, such as enthalpy, birefringence, rotatory power and electroclinic response in order to study this transition. The main conclusion is that there is only one order parameter, which is the tilt angle θ , governing both the A–C* and A–C_A^{*} phase transitions.

2. Theory

It is usual practice in the physics of phase transitions in liquid crystals to define order parameters which vanish in the high symmetry phase and allow the description of the transition in terms of Landau formalism [10, 11]. In the case of the smectic A to smectic C phase transition, there is a general agreement that the order parameter has two components (3D-XY analogy). It may be expressed as a complex number $\theta \exp(i\varphi)$ or as a two component vector (θ_x, θ_y) or may be built from the unit vector \mathbf{n} , $\xi = (n_x n_z, n_y n_z)$, as long as its modulus is proportional to the tilt angle θ ($\xi^2 = n_x^2 + n_y^2 = \theta_x^2 + \theta_y^2 = \theta^2$). When the anticlinic smectic C_A phase is taken into account, theoreticians [12–14] usually apply the last expression to individual neighbouring layers 1 and 2:

$$\xi_1 = \begin{pmatrix} n_{1x} n_{1z} \\ n_{1y} n_{1z} \end{pmatrix} \quad \xi_2 = \begin{pmatrix} n_{2x} n_{2z} \\ n_{2y} n_{2z} \end{pmatrix} \quad (1)$$

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Then, two order parameters may be built up from ξ_1 and ξ_2 :

$$\xi_c = \frac{1}{2}(\xi_1 + \xi_2) \quad \xi_a = \frac{1}{2}(\xi_1 - \xi_2). \quad (2)$$

If one reasonably assumes that the tilt angle is the same in successive layers, so that $n_{1z} = n_{2z} = \cos \theta$, one has to obey the constraints $\xi_a^2 + \xi_c^2 = \theta^2$ and $\xi_a \xi_c = 0$. It is easy to see now that the smectic C phase is described by $\xi_c \neq 0$ and $\xi_a = 0$, while the smectic C_A obeys $\xi_c = 0$ and $\xi_a \neq 0$ and that intermediate ferroelectric phases could be described in a two layer model by requesting simultaneously $\xi_c \neq 0$ and $\xi_a \neq 0$. The usual procedure when writing a Landau free energy describing the phase transitions is to start with quadratic terms:

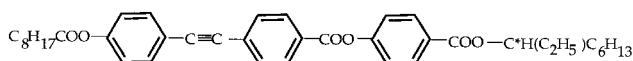
$$F = \frac{1}{2} a_c \xi_c^2 + \frac{1}{2} a_a \xi_a^2 \quad (3)$$

followed by higher order and coupling terms[†] that would allow one to describe the full phase diagram [12–14]. One would then introduce two temperatures T_c and T_a where the Landau coefficients vanish: $a_c = \alpha_c(T - T_c)$ and $a_a = \alpha_a(T - T_a)$, and try to explain the experimental phase sequences by a classification based on the values of T_a and T_c . On the other hand, the constraint $\xi_a^2 + \xi_c^2 = \theta^2$, together with high resolution calorimetry results [7] suggest that the two order parameters are not independent and that there is only one critical temperature $T_a = T_c$ where the tilt angle θ appears. The aim of this study is to try to derive experimentally a proof of this assertion.

3. Material

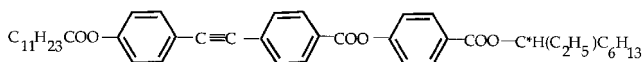
We have used successively two members of the n CTBB9* series ($n = 8$ and $n = 11$) [15] that show a direct phase transition from the smectic A to the smectic C_A phase.

8CTBB9*:



Crystal $\rightarrow < 50^\circ C \rightarrow SmI_A^* \rightarrow 68.5^\circ C \rightarrow SmC_A^* \rightarrow 82.6^\circ C \rightarrow SmA \rightarrow 106.9^\circ C \rightarrow$ Isotropic.

11CTBB9*:



Crystal $\rightarrow 74.3^\circ C \rightarrow (SmI_A^* \rightarrow 58.4^\circ C) \rightarrow SmC_A^* \rightarrow 85^\circ C \rightarrow SmA \rightarrow 93.4^\circ C \rightarrow L_1 \rightarrow 96^\circ C \rightarrow$ Isotropic.

[†] Note that in reference [13] the first coupling term reads $(\xi_a \cdot \xi_c)^2$ and is identically null under the constraint of constant tilt angle.

The second compound presents a new phase (named L_1), which has been discovered for the first time [15] and characterized in this chemical series.

4. Experimental

4.1. Calorimetry

The transition enthalpies were measured with a Perkin-Elmer DSC7 apparatus and show (figure 1) that the $SmA-SmC_A^*$ phase transition is weakly first order and should reveal pretransitional phenomena in the experiments. These enthalpies are respectively 0.24 kJ mol^{-1} for 8CTBB9* and 0.54 kJ mol^{-1} for 11CTBB9* [15].

The DSC results compare well with the high resolution calorimetry results of Ema *et al.* [7] for the reference compounds MHPOBC and MHPOCBC. These authors found that the excess heat capacity ΔC_p linked to the emergence of the tilt angle θ at the $SmA-SmC_A^*$ phase transition is well described by a mean-field second order Landau equation with a sixth order term. The total integrated enthalpy in MHPOBC is reported [7] to be 0.37 kJ mol^{-1} , while the latent heats of the first order restructuring transitions between tilted subphases go from 9 to 16 J mol^{-1} . In the case of the compounds under study here, the order of magnitude of the integrated transition enthalpy is the same as the main contribution in MHPOBC, suggesting that it contains an excess heat capacity ΔC_p and a latent heat contribution due to the weakly first order character of the $SmA-SmC_A^*$ phase transition; this contribution is much smaller for the $n = 8$ compound which may present a second order phase transition.

4.2. Birefringence

The smectic A phase is truly uniaxial with the birefringence $(n_e - n_o)$ while the smectic C_A^* phase is biaxial at the local level with three main indices $n_1 > n_2 > n_3$. When the characteristic helix is wound around the layer normal, it becomes uniaxial by compensation with an apparent birefringence [16] $\Delta n = n_1 - (n_2 + n_3)/2 \approx (n_e - n_o)(1 - 3 \sin^2 \theta/2)$. Therefore the emergence of the tilt angle θ induces a decrease of the birefringence that may be used to measure θ . We have done this experiment with $5 \mu\text{m}$ thick planar samples which were mostly helicoidal in the bulk and probably unwound at the surfaces. We used a technique of compensation of the birefringence in monochromatic light (632.8 nm) with a quarter-wave plate. The result obtained with 8CTBB9* is shown in figure 2:

The birefringence [figure 2(a)] shows a plateau in the SmA phase followed by a step decrease in the SmC_A^* phase, like the layer spacing in an X-ray experiment. The low temperature part of the corresponding θ curve [figure 2(b)] can be fitted to a power law $\theta_0 [(T_a - T)/T_a]^{0.28}$. The exponent is close to the tricritical

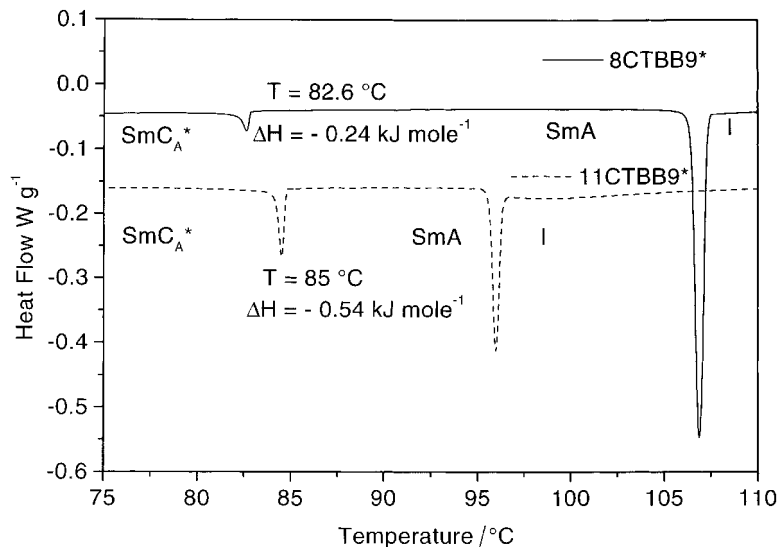


Figure 1. Differential scanning calorimetry diagrams for cooling scans ($3^{\circ}\text{Cmin}^{-1}$) obtained with 8CTBB9* and 11CTBB9*.

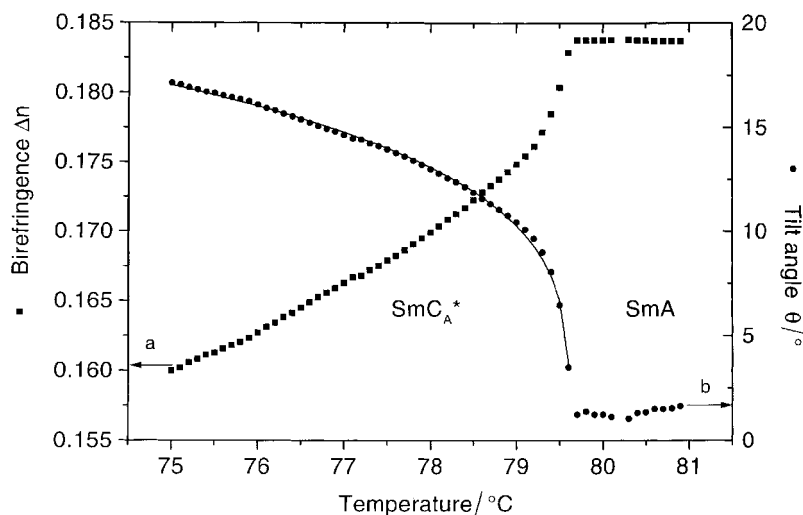


Figure 2. Birefringence and tilt angle θ versus temperature for a $5\ \mu\text{m}$ thick planarly aligned 8CTBB9* sample. The phase transition is weakly first (or second) order, as confirmed by the vanishingly small discontinuity of Δn .

value 0.25 in accordance with the high resolution calorimetry results of Ema *et al.* [7] who fitted the MHPOBC data with a mean-field Landau equation with a large sixth order term. If the sixth order term cannot be neglected, it is reasonable to assume that the vanishing of the fourth order term at the tricritical point is not too far away.

4.3. Optical rotatory power

The biaxiality of the anticlinic phase may be evidenced by the measurement of the optical activity induced by the helicoidal rotation about the layer normal [16]. The rotation per unit length of the polarization for a beam propagating normal to the layers reads, far away from any selective reflection:

$$\rho = -\pi \frac{\epsilon_a^2 \sin^4 \theta \lambda_0^3}{16n_{\perp}^3 \lambda^2 (\lambda_0^2 - \lambda^2)} \quad (4)$$

where $\epsilon_a \sin^2 \theta = n_2^2 - n_3^2$ is a measure of the in-plane anisotropy, $n_{\perp} = (n_2 + n_3)/2$, λ is the laser wavelength at 632.8 nm and $\lambda_0 = n_{\perp} p$ where p is the helical pitch.

The ORP has been measured for 11CTBB9* (figure 3) showing that there is a discontinuity at the A–C_A^{*} phase transition (i.e. a jump of the tilt angle θ) with a small coexistence region about 85°C. This confirms the first order character of the transition. In figure 3 is also plotted the minimum light intensity after the crossed analyser. One sees that there is only a small increase at 85°C due to the coexistence and a regular divergence at lower temperatures due to the approach of the selective reflection at 632.8 nm that renders the polarization elliptic. This also shows that there is no divergence of the depolarized Rayleigh scattering at the transition which could have been interpreted [16] as the signature of a critical phenomenon linked to the uniaxial to biaxial character of the phase transition.

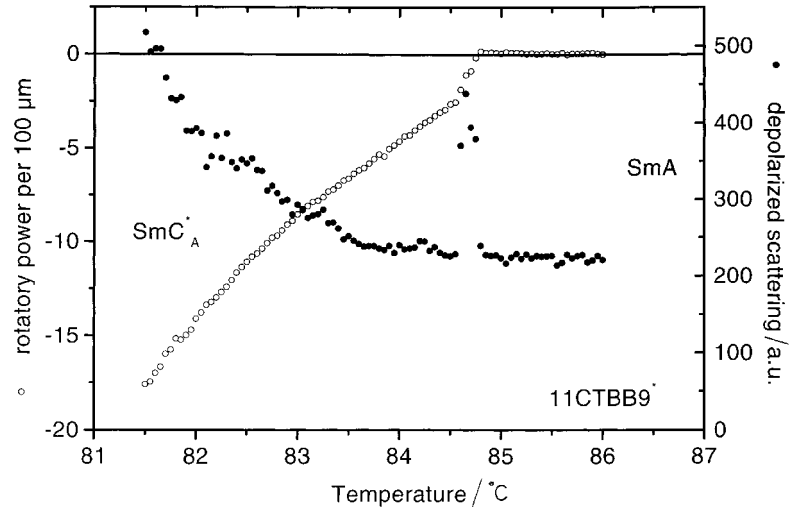


Figure 3. Optical rotatory power and depolarized Rayleigh scattering in a homeotropic 100 μm thick 11CTBB9* sample. The phase transition is first order as revealed by the discontinuous ORP. There is no divergence of depolarized scattering ruling out the biaxiality as being the primary order parameter of the transition.

The previous sections have shown that when one isolates the direct transition SmA (untilted) to SmC_A^* (tilted), the natural order parameter ξ_a can be identified with the tilt angle θ and that the memory of the usual SmC order parameter ξ_c seems to be lost. Let us show now that the SmC character is still underlying and can be revealed by the coupling to an external electric field.

4.4. Electroclinic experiments

It is well known since Garoff and Meyer [5] that an electric field applied in the smectic A phase of a chiral compound induces a polarization parallel to itself and a tilt angle in a plane normal to it. Let us define an axis frame where z is the layer normal and y the direction of the applied field. Close to the tilted–untilted phase transition, if the applied field is E_y , one may introduce two soft modes, the ferroelectric SmC^* mode [5] that describes a uniform tilt $\theta_x = |\xi_c|$ and the anticlinic SmC_A^* mode, with an alternating tilt $\theta_y = |\xi_a|$ that may appear if the dielectric anisotropy is positive. The free energy density that describes these soft modes may be written as:

$$F = \frac{1}{2}(a_c \theta_x^2 + a_a \theta_y^2) - CP_y \theta_x - P_y E_y + \frac{P_y^2}{2(\chi_\perp + \chi_a \sin^2 \theta_y)} + \frac{1}{8\pi} \varepsilon_{yy}^\infty E_y^2 \quad (5)$$

where a_a and a_c vanish at T_a and T_c , P_y is the number of permanent dipoles aligned by the field E_y , $\chi_{yy} = \chi_\perp + \chi_a \sin^2 \theta_y$ is the susceptibility in the y direction and ε^∞ is the dielectric constant without the contribution of permanent dipoles. Within the mean-field

approximation, one gets:

$$\tilde{p}_y = \frac{\chi_{yy} E_y}{1 - \frac{\chi_{yy} C^2}{a_c}} \quad (6)$$

$$\tilde{\theta}_x = \frac{C}{a_c} \frac{\chi_{yy} E_y}{1 - \frac{\chi_{yy} C^2}{a_c}}$$

In these expressions, if the anisotropy of the susceptibility χ_a is negative (i.e. $\chi_\parallel < \chi_\perp$), one should simply get $\theta_y = 0$ and $\chi_{yy} = \chi_\perp$. Otherwise, θ_y has to adjust in order to minimize:

$$\tilde{F} = \frac{1}{2} a_a \theta_y^2 - \frac{1}{2} \frac{\chi_{yy} E_y^2}{1 - \frac{\chi_{yy} C^2}{a_c}} \quad (7)$$

and as $\chi_{yy} = \chi_\perp + \chi_a \sin^2 \theta_y$ is a non-linear function of θ_y , the solution has to be found numerically or graphically (figure 4) as:

$$a_a \tilde{\theta}_y = \chi_a \left(\frac{a_c}{a_c - \chi_{yy} C^2} \right)^2 E_y^2 \sin 2\tilde{\theta}_y. \quad (8)$$

The optimal value of θ_y is found by the crossing of the straight line and the sine function in figure 4. If the phase transition is dominated by the anticlinic order parameter, the Landau coefficient a_a vanishes at a higher temperature T_a than a_c ($T_a > T_c$). In that case, θ_y should diverge as soon as the slope of the straight line in figure 4 is small enough to cross the sine function; on the contrary, equation (6) shows that there is no dramatic effect to be expected on θ_x as the effective Landau coefficient [17] ($a_c - \chi_{yy} C^2$) does not vanish.

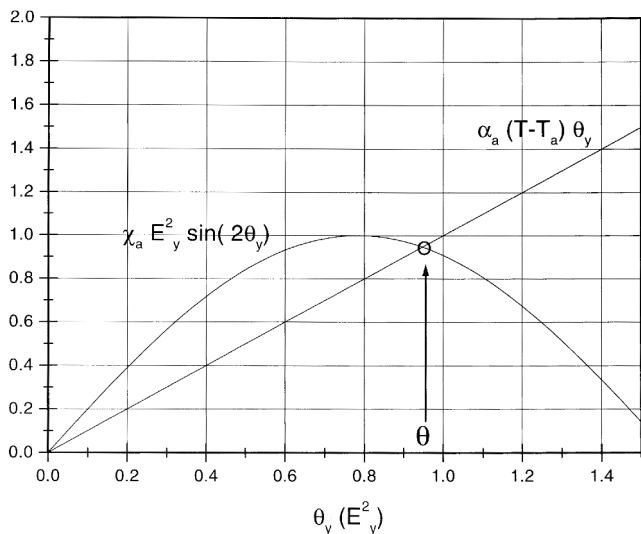


Figure 4. Graphical determination of the anticlinic tilt angle θ_y as a function of the applied field E_y , provided $\chi_a > 0$.

On the contrary, if the transition temperatures T_a and T_c are the same, the Landau coefficient $(a_c - \chi_{yy} C^2)$ will drive a simultaneous divergence of θ_x via equation (6) and θ_y via equation (8) and figure 4.

4.4.1. Characterization of the electroclinic responses under an a.c. field

Let us consider application of a sinusoidal field $E_y \cos(\omega t)$ in a planar cell. On the one hand, the ferroelectric soft mode [6] will lead via equation (6) to a uniform periodic tilt θ_x in the x direction, proportional to the electric field and modulated at the same frequency. On the other hand, the anticlinic soft mode (provided that $\chi_{\parallel} > \chi_{\perp}$) would induce an alternating tilt angle θ_y , normal to the first one, which is also periodic but even with respect to E_y . So, one may write down that θ_y is of the form $\lambda \langle E_y^2 \rangle + \mu \langle E_y^2 \rangle \cos(2\omega t) + \dots$; i.e. θ_y has a zero frequency and 2ω components quadratic in the applied electric field $E_y \cos(\omega t)$. At the same level of approximation, one may assume that $\sin(\theta_y)$, which can be revealed by birefringence measurements, has the same field dependence.

4.4.2. Experiment

In order to obtain evidence for both the θ_x and θ_y contributions, we have used the classical electroclinic set-up [6] shown in figure 5:

When the polarization of the incoming light is at an angle α_0 from the layer normal z , the intensity recovered after the crossed analyzer reads:

$$I = I_0 \sin^2 2(\alpha_0 + \theta_x) \sin^2 \left(\pi \frac{\Delta n e}{\lambda} \right) \quad (9)$$

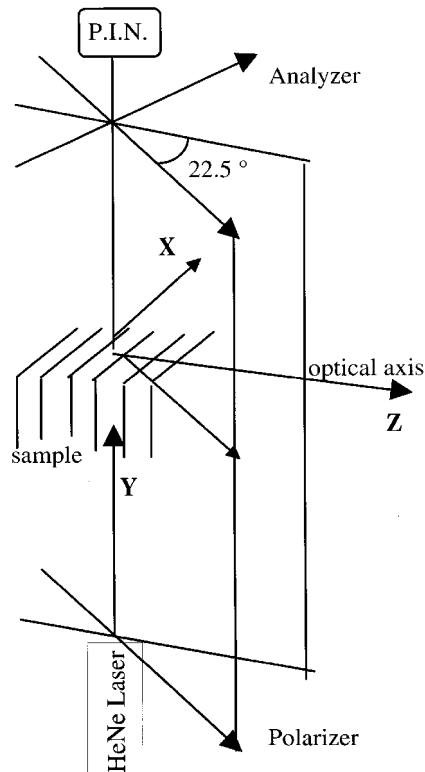


Figure 5. Electroclinic set-up. The axis frame reads $Oxyz$ where z is the layer normal, y is the in-plane direction normal to the glass holders (which is the direction of both the laser beam propagation and the applied electric field) and x is the remaining in-plane direction. The response to the field is the sum of a uniform tilt θ_x in each layer and of an alternating tilt $\pm \theta_y$ in adjacent layers.

where e is the sample thickness of about $5 \mu\text{m}$ and λ is the HeNe laser wavelength of 632.8 nm . The first sine factor depends only on θ_x and can be linearized if one chooses $\alpha_0 = \pi/8$; when the applied field has the form $E_y \cos(\omega t)$, this factor is modulated at the frequency of the field without any harmonic component.

The second factor depends only on the anticlinic tilt angle θ_y by means of the birefringence $\Delta n = n_1 - n_3$, where $n_1^2 = n_c^2 \cos^2 \theta_y + n_0^2 \sin^2 \theta_y$ and $n_3 = n_0$ are the refractive indices seen by the laser beam. It cannot be linearized at will like the first one, but one realizes that the birefringence Δn behaves like $(n_c - n_0) - \mu \langle E_y^2 \rangle + \nu \langle E_y^2 \rangle \cos(2\omega t) \dots$ under a periodic field.

So the light intensity I must reflect, by its component at the frequency $\omega/2\pi$, the uniform tilt angle θ_x and by the harmonic at $2\omega/2\pi$, the anticlinic angle θ_y . We have analysed the light intensity recovered by a photomultiplier with a lock-in amplifier (EGG PAR5301) for the two members of the $n\text{CTBB9}^*$ series.

4.4.3. Results

With 8CTBB9^* we made frequency scans up to 100 kHz at fixed temperatures (figure 6) with an EGG-PAR 5301

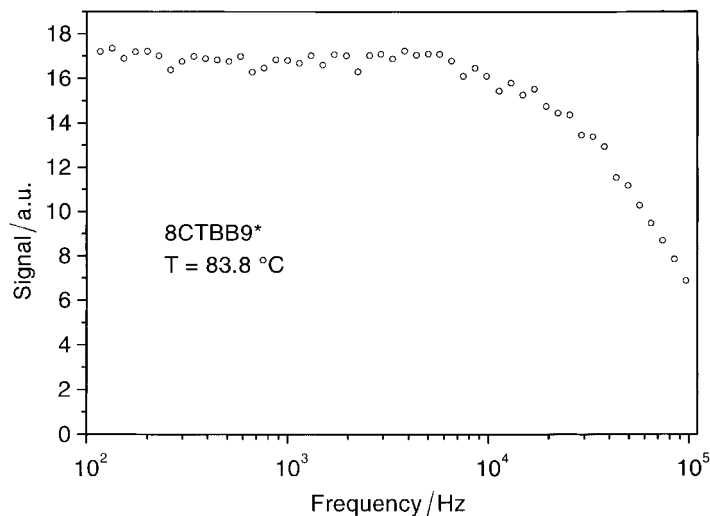


Figure 6. Typical Lorentzian frequency scan in 8CTBB9* for the fundamental harmonic.

lock-in amplifier. We obtained for the fundamental harmonic $f = \omega/2\pi$ the classical Lorentzian shape (figure 6) with a plateau amplitude diverging at the approach of the A-C_A phase transition and the relaxation frequency f_c decreasing linearly in parallel. The only exception was a scan at 86.9°C (slightly above the phase transition) for which the signal amplitude decreased dramatically and the Lorentzian shape was lost. For the second harmonic at $2f = \omega/\pi$, a signal was detected at the magic angle $\alpha_0 = \pi/8$ where no signal should exist in the pure A-C* phase transition [6]. This signal also showed a tendency to increase when approaching the phase transition. Due to an uncertainty in the determination of the relaxation frequency caused by too high a value of the RC time constant of the cell ($RC > 2 \mu\text{s}$), we did the next series of experiments on 11CTBB9* at a constant frequency of 1 kHz, but by varying the temperature on a very slowly decreasing ramp at -0.2 K h^{-1} and recording the amplitude of the signal versus the temperature. For the fundamental harmonic [figures 7(a) and 7(b)] this leads to a very peculiar shape of the light signal which vanishes at a given temperature above the phase transition and then diverges close to it. The applied voltages were respectively 0.5 V peak-to-peak and 1 Vpp, with a vanishing temperature further away from T_c in the latter case. The second harmonic signal was also recorded under the same conditions (figure 8) and shows a simple divergence when approaching the phase transition.

4.4.4. Discussion

These unusual results may be interpreted as showing that there exist two simultaneous soft modes diverging at the same temperature (see for example figures 7 and 8), thus proving that there is only one relevant order

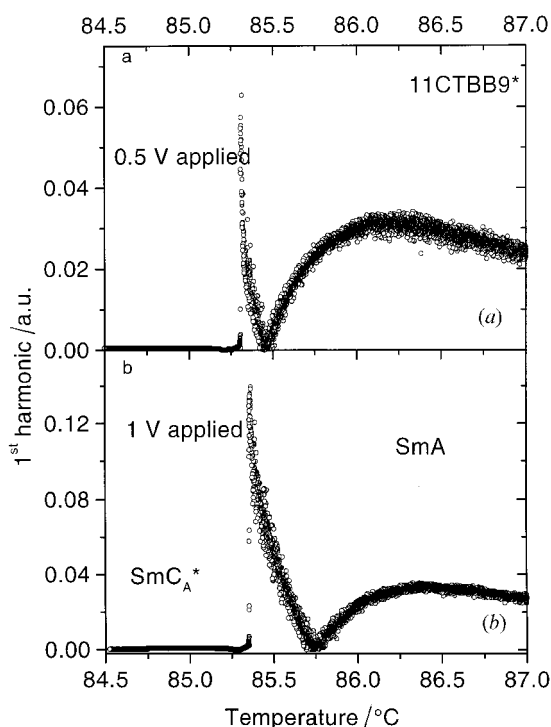


Figure 7. Temperature scans in 11CTBB9* for the fundamental harmonic (a) at 0.5 V, (b) at 1 V. The signal vanishes in the SmA phase due to the contribution of the field dependent birefringence to the intensity.

parameter which is the tilt angle θ . The vanishing of the fundamental harmonic signal close to the transition backs up this interpretation as it is probably due to the vanishing of the second sine term of equation (9), when the birefringence, which is a function of the applied field and of the temperature via the anticlinic angle θ_y , is such that (Δne) is a multiple of the wavelength λ .

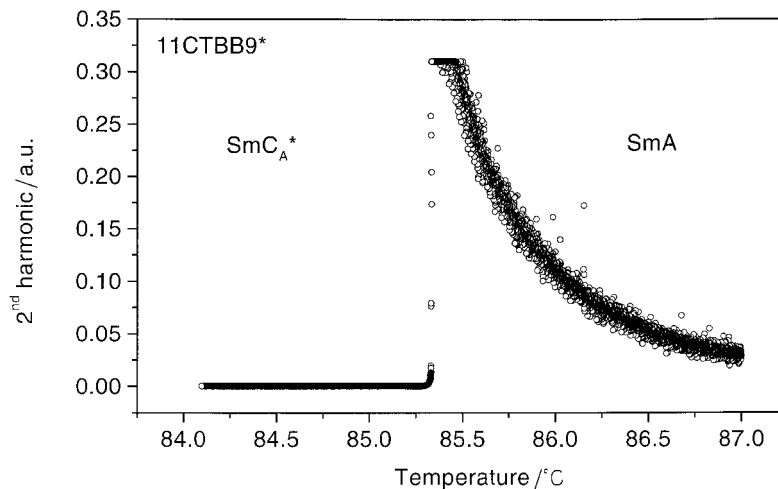


Figure 8. Temperature scans in 11CTBB9* for the second harmonic showing the (2ω) component of the birefringence, see equation (9).

5. Summary

We have shown with two members of a new series of compounds that the direct transition from the untilted smectic A phase to the tilted anticlinic smectic C_A is governed by the tilt angle θ . This same order parameter governs the A–C* phase transition that may coexist with the previous one under application of an external electric field.

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