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Liquid Crystals
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To cite this Article Faetti, S. , Gammaitoni, L. , Martinelli, M. and Rolla, P. A.(1991) 'Director reorientation in nematics studied by microwave dielectrometry', Liquid Crystals, 9: 1, 133 — 140
To link to this Article: DOI: 10.1080/02678299108036772
URL: http://dx.doi.org/10.1080/02678299108036772

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LIQUID CRYSTALS, 1991, VOL. 9, NO. 1, 133–140

Director reorientation in nematics studied by microwave dielectrometry

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(Received 20 November 1989; accepted 21 August 1990)

A new experimental method is presented which is a useful approach in studying the reorientational dynamics in liquid crystals by means of dielectric measurements at microwave frequencies. The theoretical model is developed to describe the motion of the director when it is driven by two orthogonal electric or magnetic fields. A specific set up for the experimental apparatus is described. Experiments in fairly good agreement with the theoretical model are given for a nematic mesophase at different temperatures and field values. The capabilities of the method in monitoring the slow reorientational properties of collective molecular motions in liquid crystals are demonstrated. Possible refinements of the experimental apparatus to allow more quantitative measurements of the different physical parameters of anisotropic media are indicated.

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1. Introduction

The dynamic response of liquid crystals to external driving torques allows the study of reorientational phenomena which can be directly related to the physical properties of the systems. The main parameter which is related to these phenomena is the orientational viscosity coefficient γ [1]. Several methods and experimental schemes have been proposed and used to study these reorientational properties and so to determine γ. Among the most important applied methodologies we recall dynamic measurements of the Freederickz transition by optical and thermal methods [2, 3], light scattering measurements [4, 5], measurements of the surface torques due to rotating magnetic fields (Tsvetkov's experiment) [6, 7] and bulk reorientation by sample rotation detected by magnetic resonance techniques [8–10].

Here we propose a new experimental method where director-reorientation, driven by two crossed electric or magnetic fields, switched on and off, are measured directly by the evolution of the dielectric anisotropy of the system at microwave frequencies. The experimental set up chosen for this measurement implies the use of one magnetic and one electric orthogonal fields. This choice allows measurements of both dielectric and magnetic anisotropic properties of the sample. Furthermore, if the magnetic field is supplied by an electron spin or nuclear magnetic resonance spectrometer, these measurements can be usefully supported by simulteneous magnetic resonance experiments [8, 10]. In this experimental frame the mean orientation or director rotates between the directions of the magnetic and electric field. Accordingly the resonance frequency of the microwave cavity changes and in particular the difference between its

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extreme values is proportional to the microwave dielectric anisotropy of the sample. When the field switching rate is increased, this difference is affected by the director reorientational dynamics of the liquid crystal sample.

A similar experimental method exploiting the crossed field configuration was advantageously employed for studying the director orientational dynamics of nematics by spin probe electron paramagnetic resonance techniques [9].

2. The method

The reorientational processes of an anisotropic medium can be driven by a proper field either electric **E** or magnetic **B**, whose direction is changed in time. In the method we describe, the change of the direction of the driving field is obtained by using two nearly perpendicular fields, electric and magnetic, alternately acting on the sample. The detection of the director reorientational phenomena is achieved by measuring the value of the dielectric constant of the anisotropic medium at microwave frequencies along the direction of the magnetic field. This procedure, according to the cavity perturbation method [11], requires that the sample is placed inside a resonant cavity having an appropriate distribution of the microwave electric field. The geometry of the different fields and the position of the sample are sketched in figure 1.

The theoretical treatment is specialized for a medium with positive anisotropies of both dielectric permittivity $\varepsilon_a = \varepsilon_{\parallel} - \varepsilon_{\perp}$ and magnetic susceptibility $\chi_a = \chi_{\parallel} - \chi_{\perp}$, according to the characteristics of the nematic sample used. In addition, both electric and magnetic fields are supposed to be sufficiently large as to obtain electric and magnetic coherence lengths [1],

$$\xi_H = (K/\chi_a)^{1/2}, \quad \xi_E = (K/\chi_a)^{1/2},$$

much smaller than the characteristic dimension of the sample, where K is an elastic constant. This implies that the director **n** can be assumed to be uniformly oriented over the whole sample volume and its spatial derivatives can be neglected. With these assumptions, the Leslie–Ericksen equation [1] for the director motion becomes

$$\gamma_1 \frac{d\theta}{dt} = -\varepsilon_a E^2(t) \sin(\beta - \theta) \cos(\beta - \theta) - \chi_a B^2(t) \sin\theta \cos\theta, \tag{1}$$

where γ_1 is the twist viscosity coefficient, θ is the instantaneous angle between the director **n** and the magnetic field B and β is the angle between the fields **E** and **B**. This equation can be separated in two different equations in order to account for the alternate presence of the fields. Each equation describes the orientational motion of the director driven by one of the fields, while the other field, before switching off, defines the initial orientation. The director reorientation when the electric field is switched off and the magnetic field is turned on is described by

$$\gamma_1 \frac{d\theta}{dt} = -\chi_a B^2(t - t_0) \sin\theta \cos\theta. \tag{2}$$

The solution of this equation has the form

$$\tan\theta(t - t_0) = \tan\beta \exp\left[-\frac{\chi_a B^2}{\gamma_1}(t - t_0)\right], \tag{3}$$

where the director has been supposed aligned along the direction of the electric field for $t \leqslant t_0$, where t_0 is the switch-off time.

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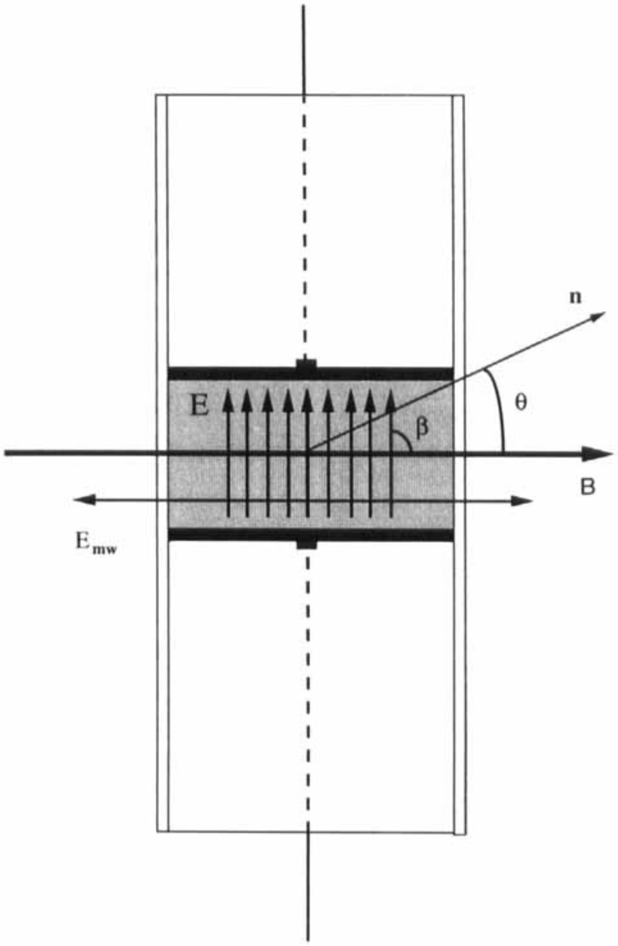

Figure 1. The fields and their distribution over the sample.

As a result of the director reorientation, the time evolution of the dielectric constant ε_m, measured along the direction of the static magnetic field **B**, by the microwave electric field $\mathbf{E}_{mw}$ is described by

$$\varepsilon_m(t) = \varepsilon_{\parallel} - \varepsilon_a \sin^2 \theta(t - t_0), \tag{4}$$

which holds separately for both real and imaginary parts of the dielectric constant. For the purposes of the present experiment, it is sufficient to take into account the variation $\Delta\varepsilon'$ of the real part of the dielectric constant in the time interval $(t - t_0)$. This quantity is calculated by combining equations (3) and (4) to give

$$\frac{\Delta\varepsilon'}{\varepsilon'_a} = \sin^2 \beta - \frac{1}{1 + (1/\tan^2 \beta) \exp[2b(t - t_0)]}, \tag{5}$$

where the coefficient b is defined by

$$b = \frac{\chi_a B^2}{\gamma_1}. \tag{6}$$

Equation (5) gives the connection between $\Delta\varepsilon'/\varepsilon'_a$ and the twist viscosity coefficient.

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3. Experimental apparatus and procedure

Figure 2 shows the block diagram for the experimental apparatus. The microwave source (klystron reflex Varian mod. V297W) is frequency locked to the reflection cavity by means of an automatic frequency control loop with a response time of 10 ms. The cavity oscillates in the TE_{102} mode at a frequency of about 9·5 GHz and the resonance frequency is measured by means of the frequency meter (Systron Donner mod. 6054B). The cavity is inserted in the gap of an electromagnet (Bruker mod. BMN40) able to supply a maximum magnetic field of about 2 T in the direction parallel to the microwave electric field (see figure 1). The sample is contained in a cylindrical tube (internal diameter 4 mm) between two 3 mm spaced electrodes, which generate an orienting electric field **E** nearly perpendicular to the magnetic field **B** and are in direct contact with the sample. A 1 kHz electric field is used in order to avoid electrohydrodynamic effects [1] and electrode polarization phenomena. The frequency of the field has been chosen to be much larger than the inverse of the relaxation time τ_E of the director, so a mean orientation like that produced by a DC field of proper intensity is obtained. The voltage applied to the electrodes is drawn from a high voltage transformer driven by a function generator (ENERTEC mod. 4046).

Drawbacks related to fast switching of magnetic fields were overcome by sizing the electric field so that the orienting effects of **B** are negligible as long as **E** is active; this corresponds to using $E \gg (\chi_a/\varepsilon_a)^{1/2} B$. The electric field intensities employed (about $15 \times 10^3\ V_{pp}/cm$) were sufficient to guarantee the validity of this condition for all magnetic fields used (up to 0·8 T). The electric field was gated on for time periods large enough for complete molecular orientation and gated off for time periods comparable with the time relaxation of the director in the magnetic field. The gating function was performed by a square wave with a period T allowing complete alignment of the

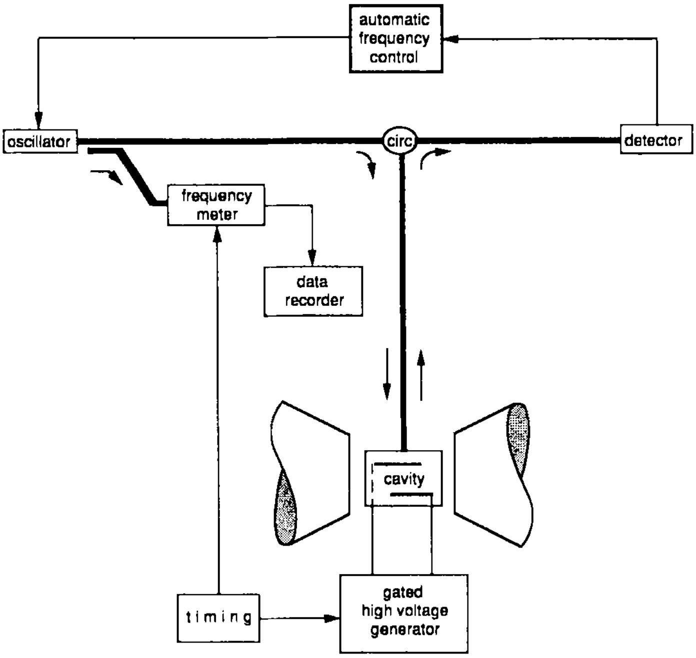

Figure 2. A block scheme for the experimental apparatus.

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director in the presence of the electric field and appreciable changes in orientation when the magnetic field alone was active.

The director reaches its maximum alignment along the electric and magnetic fields, respectively, just at the end of each semi-period $T/2$. The difference between two successive orientation values, measured at the end of two successive semi-periods when the period T is varied, accounts for the dynamic behaviour of the director. According to equation (5), the director orientation is measured by the real part ε' of the dielectric constant of the sample along the microwave electric field. In our application ε' is determined by measuring the resonance frequency of the microwave cavity, according to the cavity perturbation method. Under proper geometrical conditions the extension of this for time dependent processes shows [12] that the total variations of ε' and cavity frequency ν in a time interval $t_1 \to t_2$ are connected by

$$\varepsilon'(t_2)-\varepsilon'(t_1)=-\frac{2}{\eta}\frac{\nu(t_2)-\nu(t_1)}{\nu(t_1)}, \qquad (7)$$

where η is the filling factor [11] which is related to the ratio between sample and cavity volumes. In our experimental frame, measurements were performed every time the electric field is switched off $[t_1(n)=nT]$ and switched on $[t_2(n)=(2n+1)T/2]$. In this way the variation of ε' determined in each period of the switching frequency and defined by

$$\Delta\varepsilon'=\varepsilon'\left[(2n+1)\frac{T}{2}\right]-\varepsilon'[nT]$$

can be related to the frequency variation by equation (7)

$$\Delta\varepsilon'=-\frac{2}{\eta}\frac{\nu[(2n+1)(T/2)]-\nu[nT]}{\nu[nT]}=-\frac{2}{\eta}\left(\frac{\Delta\nu}{\nu}\right)_n. \qquad (8)$$

Measurements of the cavity resonant frequency are obtained by triggering the frequency meter at times $t_1(n)$ and $t_2(n)$ by pulses synchronous with the rise and fall edges of the square wave gating, on and off, of the electric field.

Since the sample has a positive anisotropy ε_a, $\Delta\varepsilon'$ is positive and $\Delta\nu$ is negative. Equation (8) can be conveniently rearranged by resorting to the dielectric anisotropy ε'_a, we obtain

$$\frac{\Delta\varepsilon'}{\varepsilon'_a}=-a\frac{\Delta\nu}{\nu}, \qquad (9)$$

where

$$a=\frac{2}{\eta\varepsilon'_a} \quad \text{and} \quad \frac{\Delta\nu}{\nu}=\frac{\sum_{n=1}^{N}(\Delta\nu/\nu)_n}{N}$$

is the relative frequency shift averaged over N measurements performed in successive constant time intervals $T/2$. From equation (9) the ratio $(\Delta\varepsilon'/\varepsilon'_a)$ can be determined from the experiments. By inserting into equation (9) the quantity $(\Delta\varepsilon'/\varepsilon'_a)$ obtained from equation (5) with the substitution $(t-t_0)=T/2$, we obtain

$$-a\frac{\Delta\nu}{\nu}=\sin^2\beta-\frac{1}{1+(1/\tan^2\beta)\exp(bT)}. \qquad (10)$$

This equation can be usefully employed in order to compare the theoretical predictions of equation (5) with the experimental results.

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4. Results and discussion

Measurements have been performed with the nematic liquid crystal 4-*n*-hexyl-4′-cyanobiphenyl (6CB) (commercial grade). The temperature range of the nematic phase for this sample is between 13·8 and 29·3°C, this latter being the nematic–isotropic transition temperature T_{NI}. The director reorientational dynamic behaviour of this material was studied at different temperatures below T_{NI}.

Figure 3 shows the experimental results obtained for the temperature $T_{NI}-T=7°C$ in the nematic range. The figure reports also the result of fitting the experimental data to equation (10); the value of the filling factor was determined experimentally and the dielectric anisotropy ε_a is given in [13]; these are used in this fitting procedure. The agreement between the experimental results and the theoretical predictions confirms the validity of the model for director reorientation in nematics. The fit procedure gives the values of the parameter $b=(\chi_a B^2/\gamma_1)$ at different temperatures. The table summarizes the results obtained in the nematic range with a magnetic field intensity of 0·18 T.

These achievements are reinforced by results of a further series of measurements performed with different intensities of the magnetic field B, at the fixed temperature $T_{NI}-T=7°C$. The values of the parameter b calculated from the fit of this series of experiments, are plotted in figure 4 as a function of B^2. The continuous line represents the best fit of the b values; its strongly linear trend shows the good agreement of the experimental data with the expected theoretical trend with B^2.

5. Conclusions

The method which we have presented indicates a new approach to the study of director reorientational dynamics in nematic liquid crystals by means of dielectric

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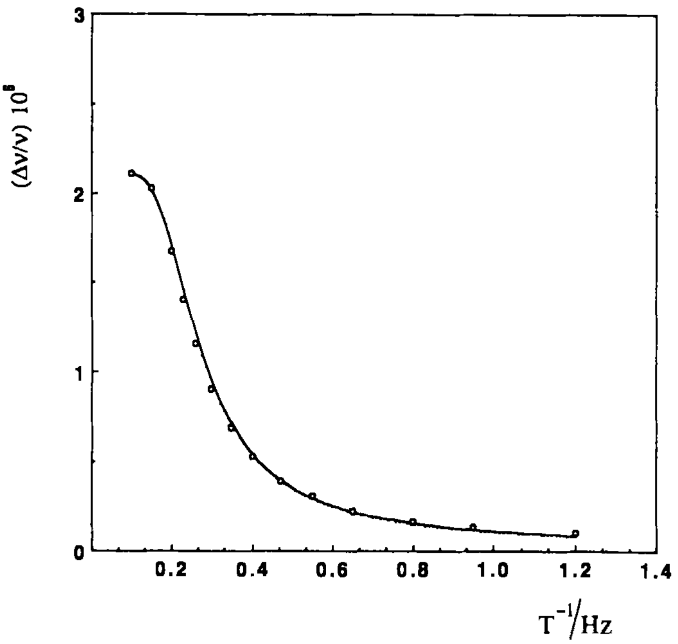

Figure 3. Relative values of $\Delta\nu$ (○) measured as a function of the reciprocal of the switching period T at the shifted temperature $T_{NI}-T=7°C$. The solid line represents the fit of the experimental data to equation (10).

Values of the parameter *b* obtained from the fit of the experimental data at different temperatures in the nematic phase of 6CB. The intensity of the magnetic field was 0·18 T in all measurements.

$(T_{NI}-T)/°C$	7	4·3	1·6	0·1
b/s^{-1}	0·225	0·233	0·29	0·50

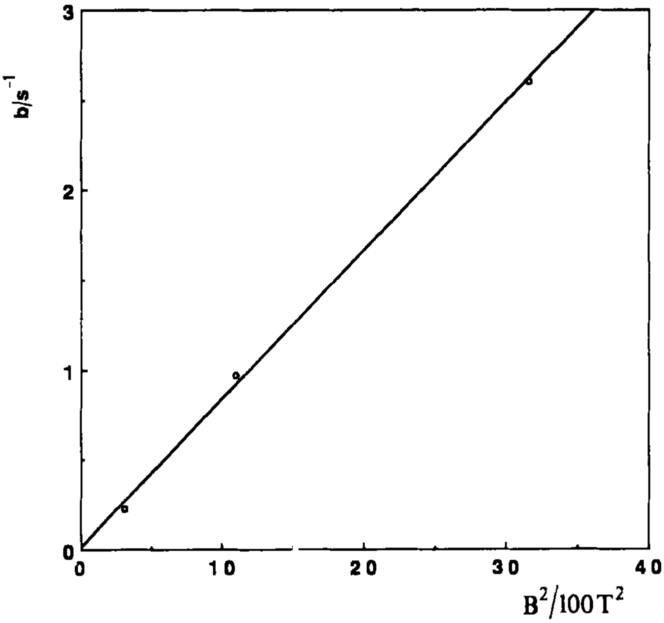

Figure 4. Values of the parameter *b* (O) determined from the fit of the experimental data at different values of the magnetic field *B*. The solid line represents the linear fit to the *b* values.

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measurements at microwave frequencies. Our discussion demonstrates very attractive capabilities of the method for characterizing in detail the director reorientation phenomena. In fact, the experimental results favourably match the theoretical model derived from the Leslie–Eriksen theory that has proved effective in describing the dynamic behaviour of liquid crystals. The present method appears very promising for measuring the characteristics of nematic liquid crystals, such as the magnetic and dielectric anisotropy, the viscosity coefficient and the order parameters. On the other hand, in order to obtain accurate determinations, some experimental refinements are required, particularly in the data acquisition procedure and in the geometrical arrangement of the fields over the sample volume. Finally, we remark that this method, in that it uses the cavity mostly employed in electron paramagnetic resonance experiments, is particularly suitable for measuring simultaneously liquid crystal anisotropic properties by means of both dielectric and paramagnetic resonance techniques.

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