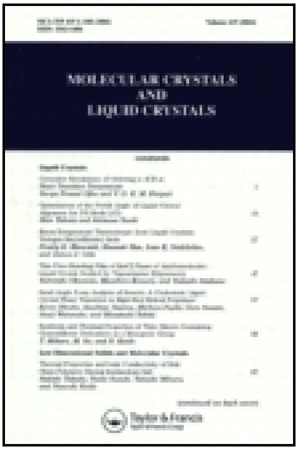

Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

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Version of record first published: 24 Sep 2006

To cite this article: G. K. Johri, M. Johri, J. A. Roberts & Katsumi Yoshino (2001): Experimental Study of Dielectric Relaxation in 4-Cyano-4-n-Hexyl Biphenyl Nematic Liquid Crystal, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 367:1, 711-717

To link to this article: http://dx.doi.org/10.1080/10587250108028692

Mol. Cryst. and Liq. Cryst., 2001, Vol. 367, pp. 711-717
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Experimental Study of Dielectric Relaxation in 4-Cyano-4-n-Hexyl Biphenyl Nematic Liquid Crystal

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Microwave cavity spectrometer has been used to measure the width of resonant profile and the shift of the resonant frequency. The observed data are analyzed to determine permittivity, dielectric loss and relaxation time. The relative variation of dielectric loss and permittivity at different temperatures has been studied for the purpose to monitor the phase changes in the range 282°K to 314°K and to identify the transition temperatures. We have used two frequencies 9.0 GHz and 29.867 GHz to see the frequency dependence for the purpose to compare relaxation mechanism. The technique used is useful as it needs a small quantity ($< 0.001\ cm^3$) of the sample and provides fruitful information about the macroscopic structure of the liquid crystal. The transition temperatures are $T_{C\rightarrow N} = 287.6°K$ and $T_{N\rightarrow I} = 301.9°K$.

Keywords: Dielectric properties; Phase transition; Liquid crystals; Microwave energy; Electrical polarization; Dielectric technique

INTRODUCTION

The dielectric technique [1] is the most important one to study the characteristics of materials for many industrial, scientific and medical applications at microwave frequencies. The dielectric data helps in understanding the molecular structure of compounds and thus it is a tool for fundamental research. Nematic liquid crystals are characterized by the presence of the orientational order of the elongated rod-like molecules and this phase is the least ordered of liquid crystalline phases. Liquid crystals [2] combine the material properties of solids with the flow properties of liquids. As such they have provided the foundation for a revolution in low power flat panel display technology i.e. Liquid Crystal Devices (LCDs). One of the most useful techniques [3] is the cavity perturbation technique. A material sample, when introduced into a cavity, alters its characteristic parameters namely the resonance frequency (f_0), and the loaded quality factor Q_L. The changes depend upon the real and imaginary parts of the complex dielectric constant and on the geometry of the sample and the cavity. For a sample with very small volume as compared to that of the cavity the first order perturbation theory [3-5] could be used.

In this paper, we have experimentally measured the dielectric response in 4-Cyano-4-n-Hexyl Biphenyl nematic liquid crystal at different temperatures using microwave cavity spectrometer. The observed data of the width of the resonant profile (Δw) and shift of the resonant frequency (Δf) are analyzed to measure permittivity (ϵ'), dielectric loss(ϵ'') and relaxation time (τ). The first order perturbation theory is used to relate the shift of resonant frequency and the width of resonant profile to ϵ' and ϵ'' respectively. The ratio of dielectric loss and permittivity corresponds to the ratio of Δw and $2\Delta f$ (i.e. $\Delta w/2\Delta f$) or tan δ.The relaxation time (τ) has been determined using Debye's single relaxation time mechanism [6]. The free energy of activation (ΔG) has been computed using the data of relaxation time at different temperatures. The values of τ and ΔG at different temperatures have been used for the purpose to monitor the phase transition temperatures.

EXPERIMENTAL DETAILS AND METHOD OF MEASUREMENT

The sample of 4-Cyano-4-n-Hexyl Biphenyl nematic liquid crystal chosen, was procured from B.D.H. Limited, Broom Road, Poola, B.H. 124 MN, England.

A block diagram of microwave cavity spectrometer is shown in Fig. 1.The detailed description is given elsewhere [5].

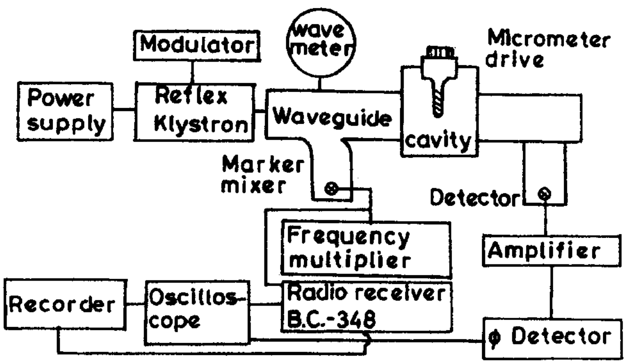

FIGURE 1 Block diagram of Microwave Cavity Spectrometer

After setting voltage and mechanical tuning, a signal frequency is generated by a Reflex Klystron near cavity resonance. Saw tooth ramp voltage is gained by time base of Tektronix Model 561A oscilloscope. To produce an a.c. signal of that frequency at detector, simultaneously a chopper signal of 31 KHz is impressed upon the Klystron repeller electrode. The arrangement allows for the synchronized sweep of frequency with the voltage scan of oscilloscope. Moreover, 31 KHz signal enables high gain tuned amplifier to be employed to detect the signal arriving at r.f. detector. The second derivative of the signal is displayed on one channel of the oscilloscope. Signals from the Klystron are sent to the marker mixture along with harmonics of the frequency standard. A tuned radio receiver [Hammurlund Model HQ 180A] is used for the comparison between these two signals. Two markers ($f_k - nf_s$), ($nf_s - f_k$), where f_k is Klystron frequency and nf_s is the proper harmonic of the standard signal source, were generated and displayed on the second channel of the oscilloscope. The separation of these markers is set to 4 MHz. A cylindrical microwave cavity is shown in Fig. 2 with internal diameter 4.9 cm and a quality factor $Q_L \approx 5000$ was operating in the TM_{010} mode served as the test cavity.

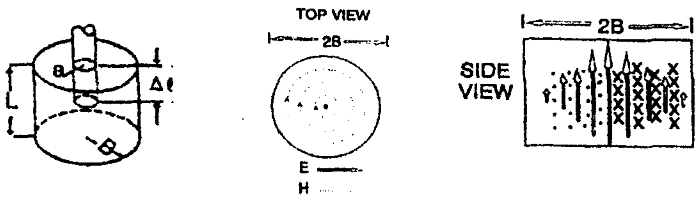

FIGURE 2 A sketch of the resonance cavity with assumed fields in TM_{010} mode pattern. Δl is controlled by a micrometer drive mechanism attached to the exterior of the cavity.

A dual pen chart recorder trace of the second order derivative of cavity resonance is given in Fig. 3.

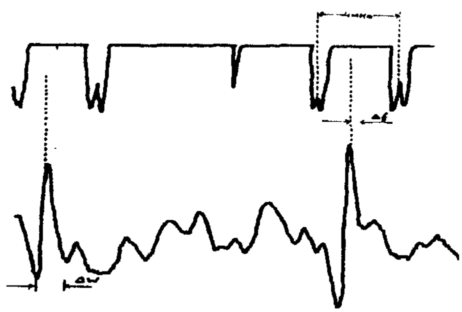

FIGURE 3 A dual chart trace of the second derivative of the absorption power. The markers are 4.0 MHz apart, where f_0 = resonant frequency, Δf = shift in the resonant frequency and Δw = width of resonance profile.

Temperature control of the sample was achieved by flushing chilled or steam heated air around the resonant cavity with the help of copper pipe heat exchanger placed in Dewar Flask or in boiling water. The rate of flow was adjusted as required to maintain the sample and the cavity at equilibrium temperature. The cavity was thermally insulated and shielded in order to maintain the stability of temperature. The temperature of the sample is measured by a thermocouple lead using an analog voltmeter.

The measurements of the resonance frequency, shift and width are taken before and after putting the sample in the cavity. The temperature is recorded in suitable steps over a desired temperature range from 282°K to 314°K. For this temperature range, the second derivative of the profile is not deformed and location of exact points for width are convenient. The observed data have been analyzed using Slater's [4] perturbation equations given below:

$$\epsilon' = (2\Delta f / f_0)[F(E)]^{-1} + 1 \qquad (1)$$

since the first term is much greater than 1, therefore, we can ignore 1 for the liquid crystal chosen and the modified eq.(1) for the value of ϵ' is

$$\epsilon' \approx (2\Delta f / f_0)[F(E)]^{-1} \qquad (2)$$

$$\epsilon'' = (\Delta w / f_0)[F(E)]^{-1} \qquad (3)$$

where, $$F(E) = \left[\int_v \mathbf{E}_s.\mathbf{E}_0\, dv\right] / \left[\int_V \mathbf{E}.\mathbf{E}_0\, dV\right], \qquad (4)$$

Δf is frequency shift, Δw is the change in the resonance width and f_0 is the cavity resonance frequency, ϵ' and ϵ'' are the real and imaginary parts of the dielectric response. F(E) is the functional form of the field interacting with the sample loading the cavity and energy stored per cycle in the cavity. **E** represents the electric field applied to the cavity, $\mathbf{E}_0$ is the unperturbed component of the electric field in the cavity volume without the sample, v is the volume of the sample and V is the cavity volume.

The relaxation time is obtained from this relation using Debye's mechanism [6] i.e. $$\tan \delta = \omega\tau \tag{5}$$

$$= \Delta w / 2\Delta f \tag{6}$$

Hence, the relaxation time

$$\tau = (1/\omega)(\Delta w / 2\Delta f) \tag{7}$$

here, $\omega = 2\pi f$.

RESULTS AND DISCUSSION

We have measured width of the resonance profile, shift of the resonant frequency at 9 GHz and 29.867 GHz using microwave cavity spectrometer. Since this technique does not give absolute value of permittivity and dielectric loss due to involvement of the form factor F(E) defined in eq.(4), we have determined the rélaxation time (τ) and free energy of activation (ΔG). The relaxation time and free energy of activation have been computed for both the frequencies i.e. 9GHz and 29.867 GHz and their variation with temperature are plotted in Fig. 4 and 5.

From these preliminary measurements the phase transition temperatures for 4-cyano-4-n-Hexyl Biphenyl nematic liquid crystal are not much different at 9.0 GHz and 29.867 GHz and on an average transition temperatures are around 278 ± 3.2°K to 301.9 ± 2.1°K for

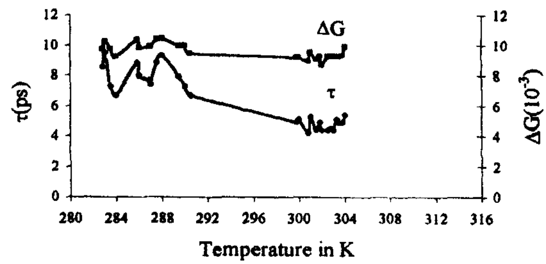

FIGURE 4 Variation of τ and ΔG with temperature at 9.0 GHz.

crystal to nematic and nematic to isotropic phases respectively. The microwave cavity spectrometer technique requires Slater [4] perturbation equations for the analysis of relative measurements of permittivity and dielectric loss for lowest possible perturbations and it gives absolute values of relaxation time.

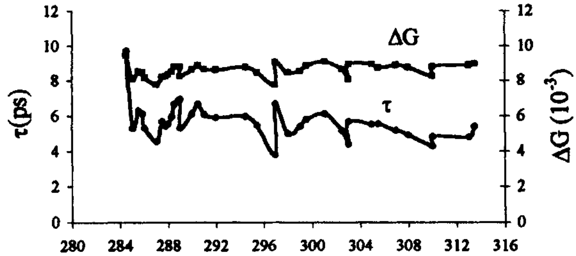

FIGURE 5 Variation of τ and ΔG with temperature at 29.867 GHz.

CONCLUSION

It is concluded that the microwave cavity spectrometer is one of the good techniques which is accurate to monitor phase transition in liquid crystals relative change in dielectric response and it needs very small quantity of sample (< 0.001cm^3). This technique is also useful to study biological molecules, agricultural products, plasma and ionic solutions.

Acknowledgements
The author (MJ) is grateful to the University Grants Commission, New Delhi, India for the financial assistance.

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