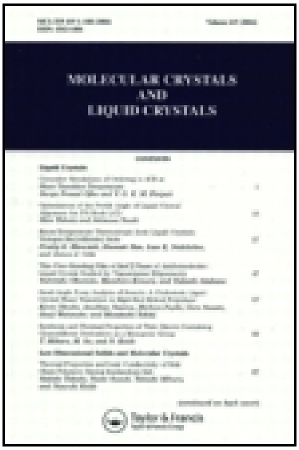

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Dielectric Properties in the Nematic and Isotropic Phases of Di-n-Propyloxyazoxybenzene (3.OAOB) and Di-n-Pentyloxyazoxybenzene (5.OAOB)

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Dielectric Properties in the Nematic and Isotropic Phases of Di-*n*-Propyloxyazoxybenzene (3.OAOB) and Di-*n*-Pentyloxyazoxybenzene (5.OAOB)†

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†The paper is dedicated to Professor Jerzy A. Janik on the occasion of his 60th birthday anniversary.

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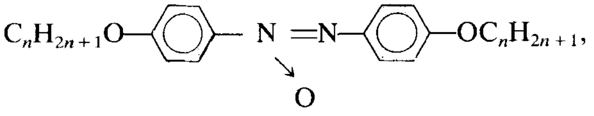
Results of dielectric relaxation studies of two members of 4,4′-*n*-alkoxyazoxybenzene homologous series, 3.OAOB and 5.OAOB, are presented. On the basis of dielectric spectra obtained for parallel ($\epsilon_\parallel^*$) and perpendicular ($\epsilon_\perp^*$) orientations of the samples with respect to measuring electric field the following problems are discussed: (i) the sign change of dielectric anisotropy when frequency increases from radio to microwave range; (ii) influence of the local field factor on the dipole correlation time in the nematic phase, and (iii) mechanism of molecular reorientations taking into consideration the dielectric spectra of three substances having different electric structures. It was concluded that the main molecular process contributing to the high frequency dielectric spectra of the nematic phases is the reorientation around the long axis of molecule as a whole, characterized by the relaxation time $\tau_\perp^{DR} \cong 85$ ps, whereas the QNS method reflects the internal reorientations of two molecular moieties around N—phenyl bonds with the correlation times $\tau^{QNS} = (4 \div 9)$ ps.

*Keywords: di-*n*-propyloxyazoxybenzene, di-*n*-pentyloxyazoxybenzene, dielectric anisotropy, dielectric relaxation, nematic, molecular motion*

INTRODUCTION

Molecular motions in 4,4′-*n*-alkoxyazoxybenzene,



(in short *n*.OAOB) homologous series are a subject of current research in our group. This class of substances seems to be a good example for studying both the reorientational motions of molecules about their principal axes and the intramolecular motions. A few experimental methods, calorimetry, dielectric relaxation (DR), quasi-elastic neutron scattering (QNS) and far-infrared spectroscopy (FIR), are systematically applied to the investigation of a given substance. The aim of these studies is to obtain a complete picture of molecular motions in liquid crystalline substances.

Part of the results of investigations of 3.OAOB and 5.OAOB have been published recently (Papers 1, 2, 3). In paper 1 the megahertz dielectric relaxation process in nematic phases of both substances is discussed, whereas in papers 2 and 3 the QNS results are presented. The results of FIR studies of some members ($n = 1, 2, 3, 5, 7$) of this series are published in paper 4. It is worth noting that 5.OAOB also possesses a solid rotator phase (the so-called ODIC phase) in which fast molecular reorientations take place.[3]

In this paper the results of microwave dielectric measurements for 3.OAOB and 5.OAOB will be presented. Some crucial points of the dielectric properties at radio frequencies will also be discussed. In the case of these substances, similarly to other members of PAA homologous series,[5] a large discrepancy between correlation times obtained by DR and QNS methods is ascertained. To shed more light on this problem we will try to estimate the influence of local field factor on the values of the dielectric relaxation times in nematic phase. Comparing the dielectric spectra of the following three substances: 5.OAOB, di-*n*-heptylazoxybenzene[22] and di-*n*-hexyloxyazobenzene,[23] having different electric structures and about the same steric shapes, will show the influence of inter- and intra-molecular motions.

EXPERIMENTAL

A complex dielectric permittivity, $\epsilon^* = \epsilon' - i\epsilon''$, of 3.OAOB and 5.OAOB was measured at the following frequencies: 1.1 and 1.8 GHz (coaxial line) and 7.38 and 9.38 GHz (rectangular waveguides). A standing-wave method has been applied.[6] In the nematic phase the samples were oriented by means of a magnetic field. In the case of the coaxial line a solenoid giving B = 0.25 T was used; therefore in this case only the perpendicular orientation ($\mathbf{E} \perp \mathbf{B}$) was attained. For rectangular waveguides both principal orientations ($\mathbf{E} \parallel \mathbf{B}$ and $\mathbf{E} \perp \mathbf{B}$) were obtained by changing the orientation of a magnet by 90° (B = 0.5 T). The sample holders were filled with a substance at the temperature of about 10 K above the clearing point. The measurements were carried out on gradual cooling down to the freezing point and also on heating. The reproducibility of measurements was good. The temperature stabilization was better than 0.5 K. The substances used were the same as those described in the previous paper.[1] The temperatures of phase transitions are the following: for 3.OAOB $T_{KN} = 388.8$ K and $T_{NI} = 396.8$ K, for 5.OAOB $T_{KN} = 348.8$ K and $T_{NI} = 396.3$ K.

RESULTS AND DISCUSSION

The results of the dielectric measurements for both substances are presented in Figures 1–5. In the case of 3.OAOB only the temperature range of about 5 K of the nematic phase was suitable for further data processing, due to the pretransitional effects observed near the melting point (Figure 2). Therefore, the conclusions drawn from the experimental results, especially concerning the values of activation energies, are less dependable for 3.OAOB than for the 5.OAOB having broad range of the nematic phase.

Dielectric anisotropy

Figure 1 presents a temperature dependence of the dielectric permittivity ϵ' measured at two frequencies: 0.1 MHz (it corresponds to static ϵ_0 value) and 9.4 GHz for both substances in isotropic and nematic phases. In both cases strong dispersions of $\epsilon'_\perp$, $\epsilon'_\parallel$ and ϵ'_{is} are observed. It is a consequence of the existence of dipole relaxation

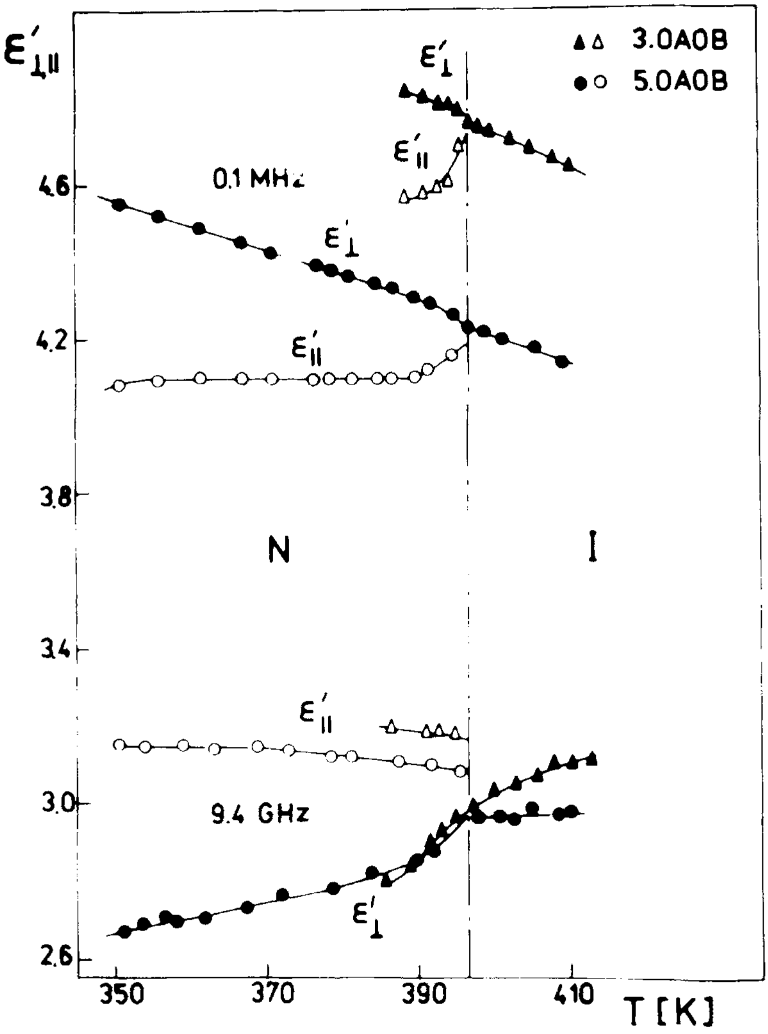


FIGURE 1a Temperature dependences of components of the dielectric permittivity tensor measured for 3.OAOB and 5.OAOB in the megahertz[1] and gigahertz frequency ranges.

processes discussed below. In Figure 1b the dielectric anisotropies, $\Delta\epsilon' = \epsilon'_{\parallel} - \epsilon'_{\perp}$, measured at radio and microwave frequencies are shown. It can be seen that the dielectric anisotropy is negative in the radio frequency range. It is consistent with the findings of Jeżewski,[9] Kast[10] and Maier and Meier.[11] On the other hand, in the microwave frequency range the dielectric anisotropy is positive in agreement with the results obtained by Carr and Spence[12] and Axmann[13] for other substances of the same homologous series.

The effect of changing the sign of the dielectric anisotropy at the microwave frequencies can be qualitatively explained by taking into account the Maier–Meier formula[7]

$$\Delta\epsilon' = \epsilon'_{\parallel} - \epsilon'_{\perp} = N_0Fh\left[\Delta\alpha - F\frac{\mu^2}{2kT}(1 - 3\cos^2\beta)\right] S, \quad (1)$$

where N_0 denotes the number of the molecules per unit volume, S is the nematic order parameter, $\Delta\alpha$ is the anisotropy of the molecular

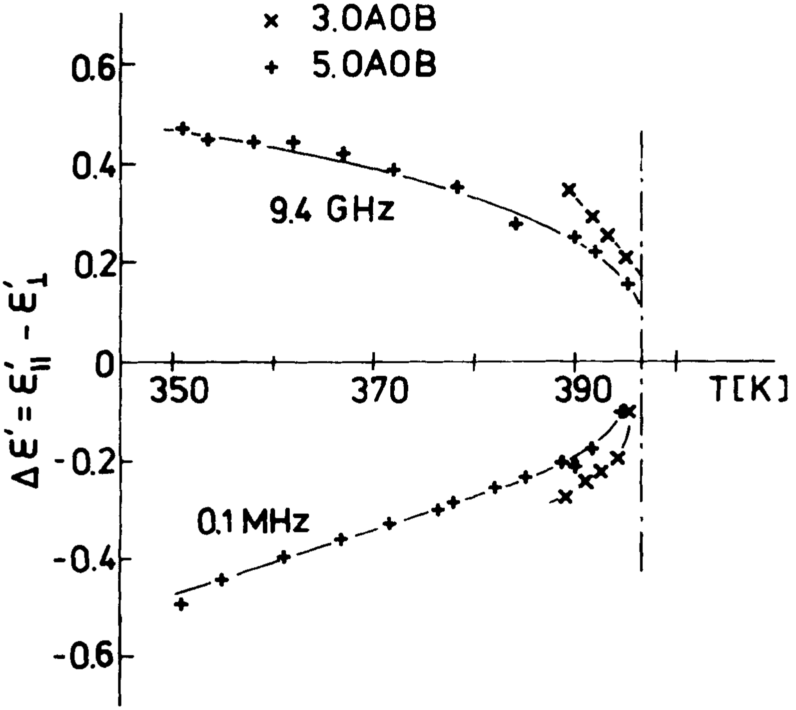


FIGURE 1b Temperature dependences of the dielectric anisotropies acquired for 3.OAOB and 5.OAOB at radio[1] and microwave frequencies.

polarizability, β stands for the angle between the total dipole moment μ and the long axis of molecule, and F and h are the local field factors depending on the mean values of $\bar{\epsilon}_0$ and $\bar{\epsilon}_\infty$.

From the dielectric increments, $\delta\epsilon = \epsilon_0 - \epsilon_\infty$, of the low frequency (l.f.) and high frequency (h.f.) relaxation processes, using the expressions derived in Maier–Meier theory,[7] it is possible to estimate the angle β. It was found that $\beta \cong 70°$. At low frequencies the dipolar polarization contribution, $F\,\mu^2/2kT\,(1 - 3\cos^2\beta)$, is bigger than $\Delta\alpha$ and the sign of $\Delta\epsilon'$ is determined by the angle β. When frequency increases the dipolar polarization diminishes, due to passing through the subsequent relaxation processes and at certain frequency it becomes equal to anisotropy of the polarizability which is positive for rod-like molecules. Such is the case for 3.OAOB and 5.OAOB at the frequency of about 1 GHz where $\Delta\epsilon'$ vanishes.

High frequency relaxation process

Dispersion effects observed in the isotropic phase and at the two orientations of the nematic phases are accompanied by the absorption effects. Figure 2 representatively shows such a behaviour for 3.OAOB. A frequency dependence of the complex dielectric permittivities will be discussed using the Cole–Cole modification of the Debye equation

$$\epsilon_\lambda^* = \epsilon_\lambda' - i\epsilon_\lambda'' = \epsilon_{\infty,\lambda} + \frac{\epsilon_{0,\lambda} - \epsilon_{\infty,\lambda}}{1 + (i\omega\tau_\lambda)^{1-\alpha_\lambda}}, \quad \lambda = \parallel, \perp. \tag{2}$$

The α_λ parameter is commonly interpreted as a measure of a symmetric distribution of the dielectric relaxation times, τ_λ.

The Cole–Cole plots for the isotropic phase and for two orientations of the nematic phase of 3.OAOB and 5.OAOB are presented in Figures 3 and 4, respectively. For the parallel orientation the semicircle connected with the low frequency (Debye) process is also shown. The results of the analysis of the dielectric spectra of Figures 3 and 4 are collected in Table I. Since there are only two experimental points available for the high frequency process seen for the parallel orientation, this process will only be discussed qualitatively.

The temperature dependences of the relaxation times of both l.f. and h.f. processes in 3.OAOB and 5.OAOB are shown in Figure 5. The application of the Arrhenius equation gives the following values for the activation energy for the h.f. processes:

for 3.OAOB, $E_{\mathrm{is}} = 48 \pm 9$ kJ/mol, $E_\perp = 24 \pm 9$ kJ/mol,

for 5.OAOB, $E_{\mathrm{is}} = 37 \pm 4$ kJ/mol, $E_\perp = 25 \pm 3$ kJ/mol.

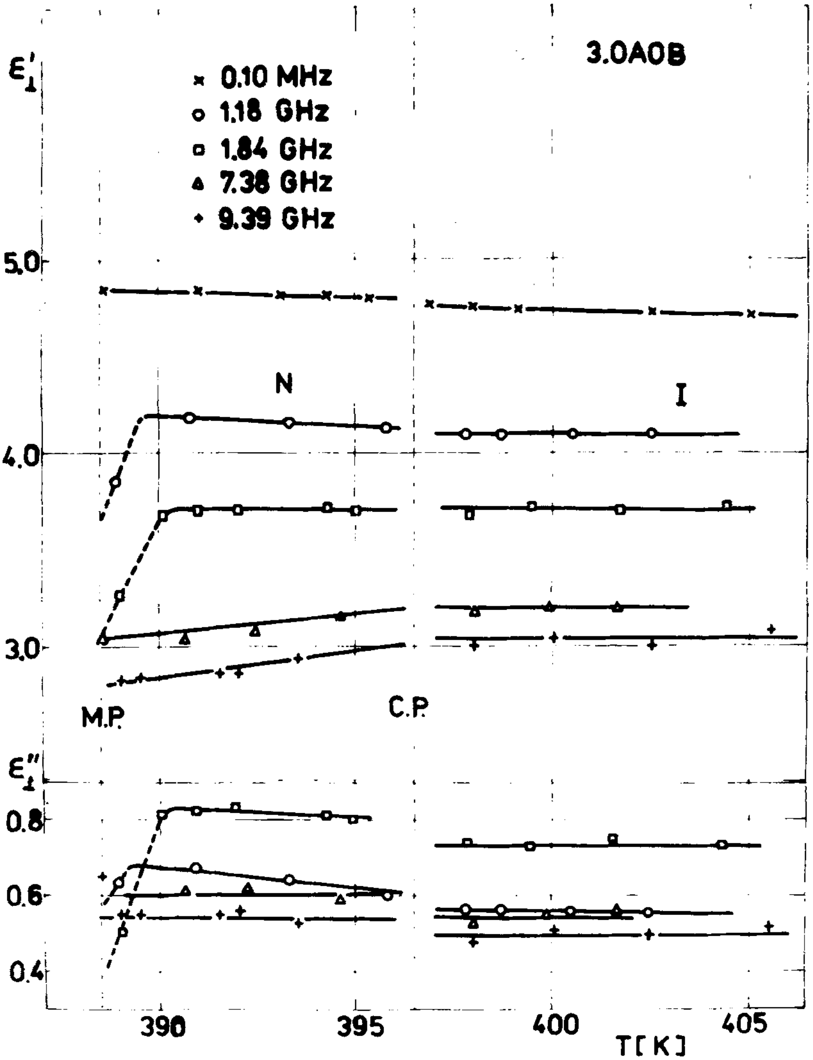


FIGURE 2 The dielectric permittivity (ϵ') and loss (ϵ'') measured for 3.OAOB as a function of temperature at different frequencies.

These values are of the same order of magnitude as the ones obtained for 2.OAOB[8] and 7.OAOB.[18]

A mechanism of molecular reorientations

From the geometry of the measurements as well as from the low values of activation energy and short relaxation times ($\tau_\perp \sim 10^{-10} \div$

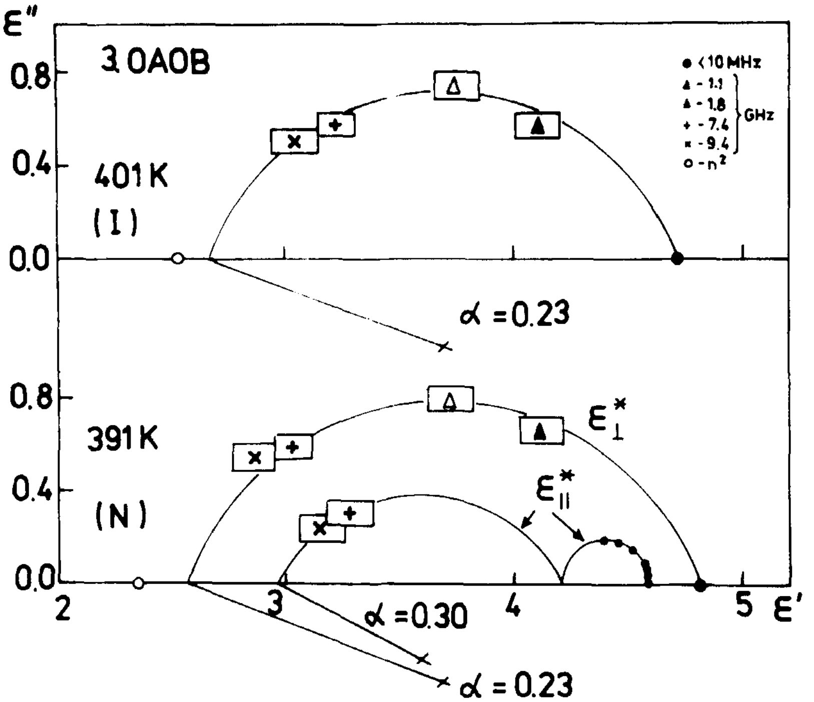


FIGURE 3 The Cole–Cole diagrams obtained for 3.OAOB for the isotropic phase and for two orientations of the nematic phase.

10^{-11} s) one can suppose that the h.f. relaxation process observed in the perpendicular orientation is mainly connected with the reorientation of molecule as a whole around its long axis. As the value of the $\alpha_\perp$ parameter found for this process is ca. 0.25 one can state that it is a rather complex process. According to theoretical considerations[19,20] the precession of the long axis around the director undoubtedly contributes to the main process.[26] In the case of *n*.OAOB this contribution is estimated to be of about 7% in comparison with the reorientation around the long axis.[21] Another process which could influence the distribution of the relaxation times is the intramolecular rotation of the end groups.

The *n*.OAOB molecules possess three group dipole moments: μ_1 connected with the bridging group and two dipole moments μ_2 connected with the alkoxy groups. The geometrical distribution of these moments is schematically shown in the inset of Figure 6a. The μ_1 and μ_2 form angles 65° and 70° with the long molecular axis and their values are 1.72 and 1.25 D, respectively. In principle, the internal motions can substantially affect the observed relaxation spectra if the

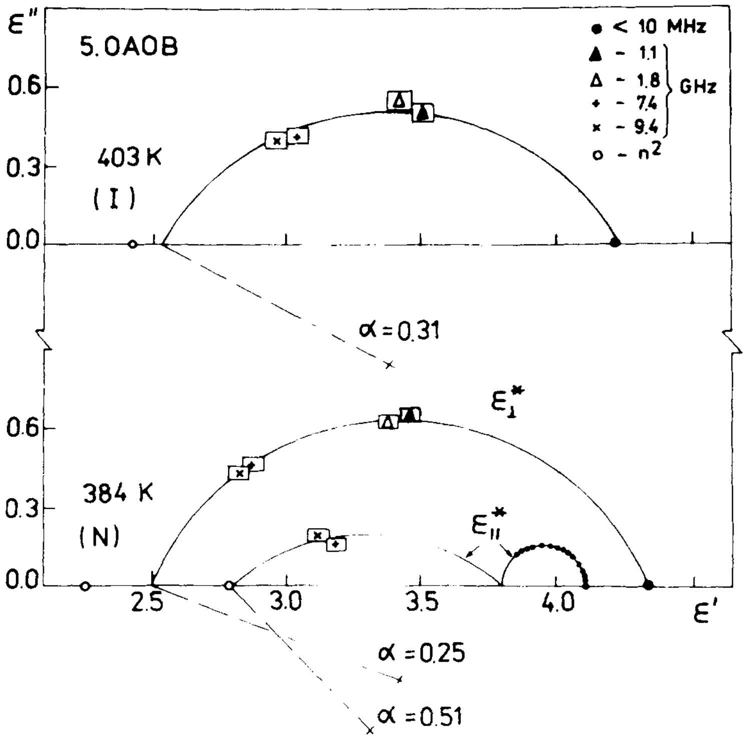


FIGURE 4 The Cole–Cole diagrams obtained for 5.OAOB for the isotropic phase and for two orientations of the nematic phase.

peripheral dipole moments reorient independently of the molecular core and with different (higher) velocity. This effect should be more visible when one considers two other substances which consist of similar molecules from the steric standpoint but with different electric structures.

TABLE I

Parameters obtained from the analysis of different high frequency relaxation processes observed for 3.OAOB and 5.OAOB. The refractive index values were taken from 29.

Substance	T K	Orientation	τ ps	α	ϵ_0	ϵ_∞	n^2
3.OAOB	401 (I)	—	64 ± 13	0.23	4.73	2.66	2.35
	392 (N)	⊥	74 ± 12	0.23	4.83	2.59	2.35
		∥	40 ± 15	0.33	4.22	3.10	2.84
5.OAOB	403 (I)	—	73 ± 30	0.31	4.22	2.54	2.42
	384 (N)	⊥	97 ± 40	0.23	4.34	2.49	2.26
		∥	47 ± 10	0.51	3.80	2.80	2.80

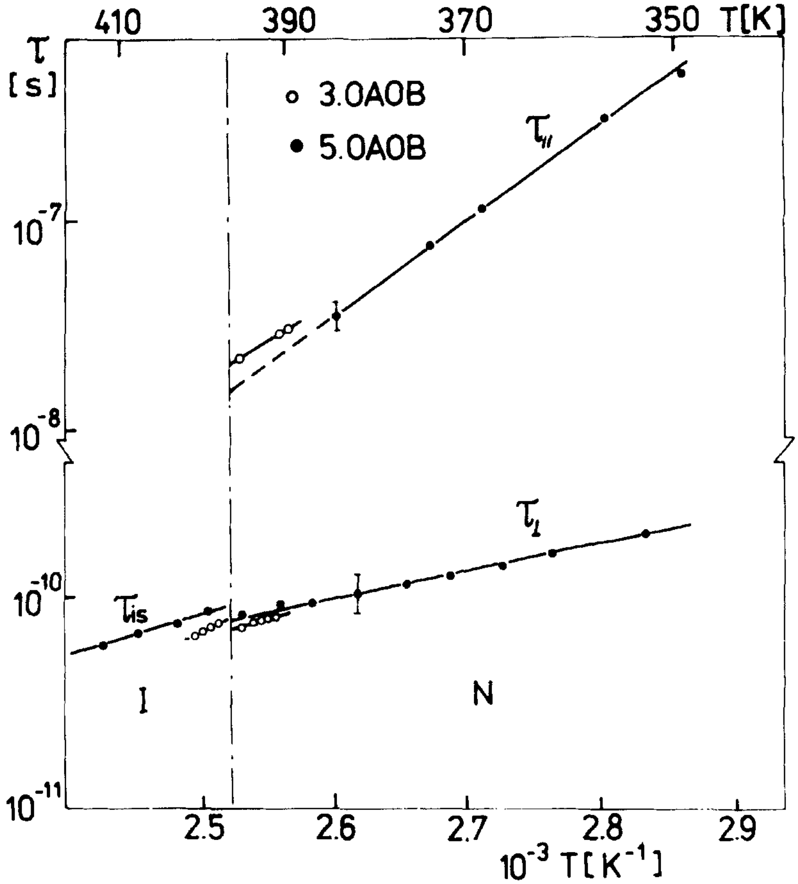


FIGURE 5 Arrhenius plots for the low and high frequency relaxations of 3.OAOB and 5.OAOB.

Figure 6b shows the experimental results obtained by Parneix and Chapoton[22] in the nematic phase of the substance with central dipole moment only, viz. di-*n*-heptylazoxybenzene (in short 7.AOB). On the contrary, Figure 6c, shows the results obtained by Axmann[23] in the substance with only outer dipole moments, i.e. di-*n*-hexyloxyazobenzene (in short 6.OAB). As one can notice the *n*.OAB molecules possess no longitudinal dipole moment component and therefore the l.f. relaxation process is not to be observed here.

It is worth pointing out that the h.f. relaxation processes have been observed in all three substances shown in Figure 6 for both orientations. Let us consider in detail the processes detected for the sam-

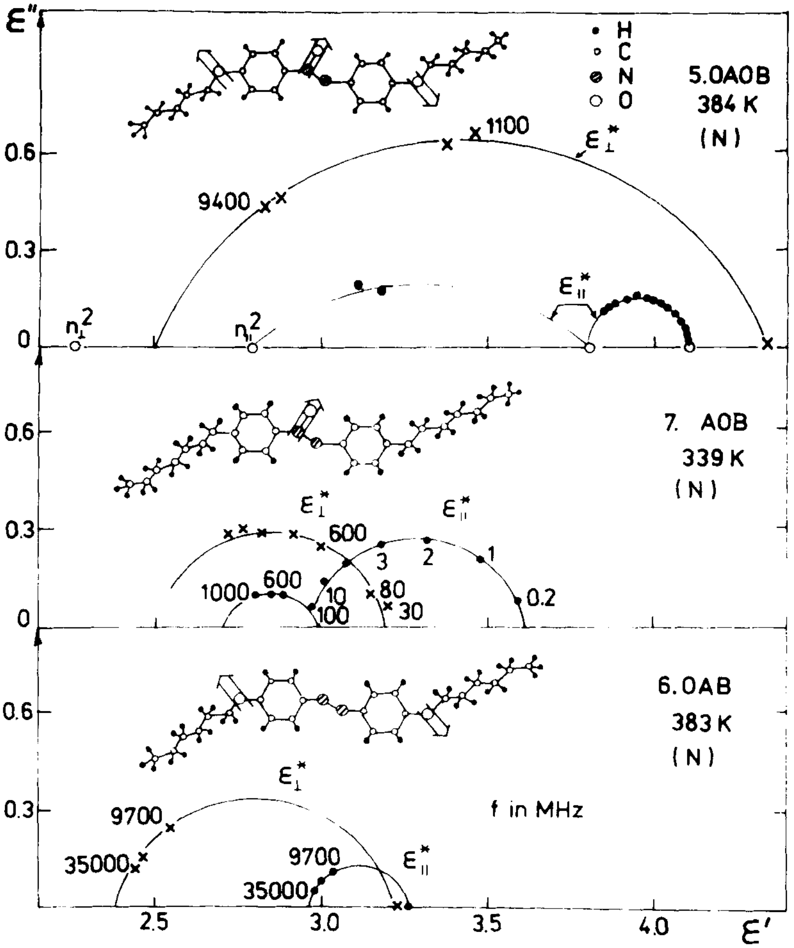


FIGURE 6 Comparison of the dielectric spectra obtained for three compounds: a) 5.OAOB (our results), b) 7.AOB[22] and c) 6.OAB[23] having different electrical structures. Numbers by the experimental points are frequencies in MHz.

ples oriented perpendicularly to the measuring electric field. Table II contains some results calculated on the basis of the experimental data. From this comparison the following conclusions can be deduced:

1. All dipole moments participate in h.f. relaxation process as the values of the dielectric increments, $\delta\epsilon_\perp = \epsilon_{0\perp} - \epsilon_{\infty\perp}$, reflect the molecules' electric structure. For instance, in the case of 5.OAOB the estimation based on Maier–Meier expressions[7] gives $\mu_t = 2.25$ D. Taking the longitudinal component of the middle dipole moment

TABLE II

Comparison of the dielectric increments ($\delta\epsilon$) and relaxation times (τ) of dielectric relaxation processes observed for three substances having similar shape of molecules but different electric structures

	l.f. domain			h.f. domain			
Substance	$\delta\epsilon_{\parallel}$	$\tau_{\parallel}$ ns	$\alpha_{\parallel}$	$\delta\epsilon_{\perp}$	$\tau_{\perp}$ ps	$\alpha_{\perp}$	Reference
5.OAOB	0.30	28	0	1.80	90	0.25	this work
7.AOB	0.63	80	0	0.7	200	0	22
6.OAB	0	—	—	0.7	45	0.17	23

$\mu_1 = 0.72$ D (the components of azoxy groups cancel), the total value of μ equals 2.36 D, which is in good agreement with the value 2.38 D obtained from the measurements on solutions.[14]

2. The dielectric relaxation times are roughly of the same order of magnitude $(5 \div 20) \cdot 10^{-11}$ s. The differences in $\tau_{\perp}$ must certainly be connected with the differences in viscosity coefficients. Nevertheless, one can note that the substance with only peripheral dipole moments (6.OAB) exhibits a small lowering of the relaxation time value. This effect can be explained if one assumes the existence of internal reorientations in the molecule. Because the reorientation of the molecule as a whole is seen via dipole moments which take part in the simultaneous faster internal motions, the net relaxation time is then shorter.

3. The above conclusion is substantiated when the $\alpha_{\perp}$ parameters are taken into consideration. If the molecular reorientations are seen via the central dipole moment only (7.AOB) no distribution of the relaxation times is observed.

4. The main molecular process contributing to the h.f. relaxation domain is the same for all three substances and it is the reorientation of molecule as a whole around the long axis.

Comparison of the DR and QNS correlation times

To answer the question what type of internal motions take place in liquid crystalline phases considered above one should compare the results of measurements carried out with the use of the DR and QNS methods. Such complementary studies are systematically performed in our group for some members of *n*.OAOB series.[2,3,5]

However, the DR method gives information on the dielectric relaxation time τ^{DR} which is a polymolecular (macroscopic) value, whereas the time constant determined from QNS measurements is a monomolecular (microscopic) value. Therefore, it is worthwhile to discuss the relation between the dielectric relaxation time and the dipole correlation time τ_1 (monomolecular value).

It is well known that finding an appropriate relation between both times is very difficult even in the case of isotropic liquids.[15,16] The known approaches to this problem mention two main factors relating τ_1 with τ^{DR} [16]

$$\tau_1/\tau^{DR} = f(\epsilon_0, \epsilon_\infty)/g, \tag{3}$$

where $f(\epsilon_0, \epsilon_\infty)$ depends on the local field model and g is the Fröhlich-Kirkwood dipole correlation factor. For the simple liquids with medium values of $\epsilon_0 \sim 5$ the $f(\epsilon_0, \epsilon_\infty)$ factor is a little smaller than unity and usually $g \cong 1$.

In the nematic phase the situation is additionally complicated due to the anisotropy of the medium surrounding a given dipolar molecule. Let us assume, however, that the relation (3) is roughly fulfilled for nematic phase. In particular it should be valid for the h.f. relaxation time $\tau_\perp = \tau_\perp^{DR}$ which characterises a diffusive reorientational motion of molecules around the long axes. Therefore the correlation factor for perpendicularly oriented sample $g_\perp$ should be taken into consideration. We calculated the g factors for both substances under study according to the formulae described in paper 17. The results are given in Table III. As it is seen the $g_\perp$ values are less than unity which means that in both nematic phases the perpendicular components of the dipole moments show a tendency to antiparallel correlations. Let us assume for nematic phase $f(\epsilon_0, \epsilon_\infty) = f(\bar{\epsilon}_0, \bar{\epsilon}_\infty) = (2\bar{\epsilon}_0 + \bar{\epsilon}_\infty)/3\bar{\epsilon}_0 \cong 0.9$[27] which is consistent with the analysis done for other mid polar liquid crystals.[25] As it is seen that τ_1 values are a little larger than $\tau_\perp^{DR}$. Therefore, we are inclined to conclude that the dielectric relaxation time $\tau_\perp^{DR}$ fairly well characterizes reorientational motion of individual molecule around long axis.

The correlation times obtained from QNS experiments, τ^{QNS}, appear to be distinctly shorter than the dielectric ones, $\tau_\perp^{DR}$. In the case

TABLE III

The Fröhlich-Kirkwood dipole correlation factors for the isotropic and nematic phases of 3.OAOB and 5.OAOB.

Substance	g_{is}	$g_\perp$	$g_\parallel$
3.OAOB	0.71	0.70	1.08
5.OAOB	0.44	0.48	0.69

of 3.OAOB the ratio $p = \tau_{\perp}^{DR}/\tau^{QNS} = 80/9 \cong 9$,[2] whereas for 5.OAOB $p = 90/4 \cong 22$.[3] The differences between both correlation times are certainly beyond the experimental errors.

On the other hand, the same values of the τ^{QNS} have been obtained for protonated molecules as well as for the chain-deuterated ones (the motions of hydrogen atoms in the C_nH_{2n+1} groups were not seen). This shows that the internal motions observed in QNS experiments are the reorientations of two moieties around the N—phenyl bonds, whereas DR method mainly detects a slower reorientation of the molecule as a whole around the long axis. It should be added that such an interpretation is consistent with theoretical calculations done for PAA (1.OAOB) free molecule[24] and for HOAB (7.OAOB).[28]

CONCLUSIONS

1. 3.OAOB and 5.OAOB exhibit in their nematic phases both the l.f. relaxation and the h.f. one which differ markedly in the values of the relaxation times as well as in the values of activation energies. The l.f. and h.f. relaxations are connected with the reorientations of molecule around the short and long molecular axes, respectively.
2. The dielectric relaxation time, $\tau_{\perp}$, measured for perpendicularly oriented sample characterizes well the reorientation of individual molecule around long axis.
3. The difference between the h.f. relaxation time ($\tau_{\perp}^{DR}$) and the QNS correlation time (τ^{QNS}) can be explained in terms of intramolecular motion of the moieties around the N—phenyl bonds.
4. From comparing the dielectric spectra of 5.OAOB, 7.AOB and 6.OAB one can conclude that both inter- and intramolecular motion influence the dielectric relaxation process. This leads to a distribution of the relaxation times observed for the nematic and also for the isotropic phases of 3.OAOB and 5.OAOB.

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