Parameters of LC molecules' movement measured by dielectric spectroscopy in wide temperature range

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A B S T R A C T

Dielectric properties of a nematic liquid crystal (NLC) mixture ZhK-1282 were investigated in the frequency range of 10^2–10^6 Hz and a temperature range of -20 to $80\,^\circ$C. On the basis of the Debye's relaxation polarization model dielectric spectra of temperature dependence of the orientational relaxation time τ and the dielectric strength δe were numerically approximated at the parallel orientation of a molecular director relative to alternating electric field. Influence of ester components in the mixture plays crucial role in relaxation processes at low temperature and external field frequency. The activation energy of the relaxation process of a rotation of molecules around their short axis was measured in a temperature interval of -20 to $+15\,^\circ$C in which the dispersion of a longitudinal component of the dielectric constant takes place. The energy of potential barrier for polar molecules rotation in the mesophase was calculated. The value of the transition entropy from the nematic to isotropic phase was obtained from this calculation. The values of the coefficient of molecular friction and rotational diffusion were obtained by different methods. The experimental data obtained are in a satisfactory agreement with the existing theoretical models.

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1. Introduction

Dielectric spectroscopy of nematic liquid crystals (NLC) allows obtaining important dielectric characteristics, i.e. conductivity, typical relaxation times, dielectric constant (constant) and its anisotropy, as well as studying fine features of the NLC molecular dynamics processes [1–4]. In particular, the rotation dynamics of the NLC molecules causes the dispersion of dielectric constant and the change of its anisotropy sign in many nematic mixtures [5–8]. Dielectric spectroscopy is also used as a method of studying of liquid crystals unique phases such as ferroelectric [9–11] and antiferroelectric liquid crystals [12,13], blue phases [14,15] and colloid mixtures based on them.

Studying molecular dynamics processes is an important issue in the LC physics as macroscopic LC performances depend on both molecules' chemical structure and their collective behaviour and agglomeration due to intermolecular interactions.

Therefore, multicomponent NLC mixtures are of both practical and scientific interest as they comprise compounds with both positive and negative anisotropy of the dielectric constant Δe. It results in an ability to change its sign at a specific frequency of an external electric field f_c (crossover frequency). This allows controlling the time of relaxation and response in LC devices using different frequency signals (higher or lower than crossover frequency accordingly) [16–19].

2. Material and methods

This study is devoted to a measurement and analysis of both dielectric spectra and relaxation processes in a nematic LC mixture over a wide temperature range. As a study object we chose the LC mixture ZhK-1282 (NIOPIK, Russia) with the wide nematic mesophase temperature range (253.1 K–335.1 K) which allows conducting researches apart from pre-transitive processes. The ZhK-1282 [20] is composed of alkoxycyanobiphenyl (47% mass), Demus's ester (47%), and Gray's ester (4%). Each component of the mixture (Fig. 1) is a well-researched compound. It gives an opportunity to draw conclusions about each mixture component
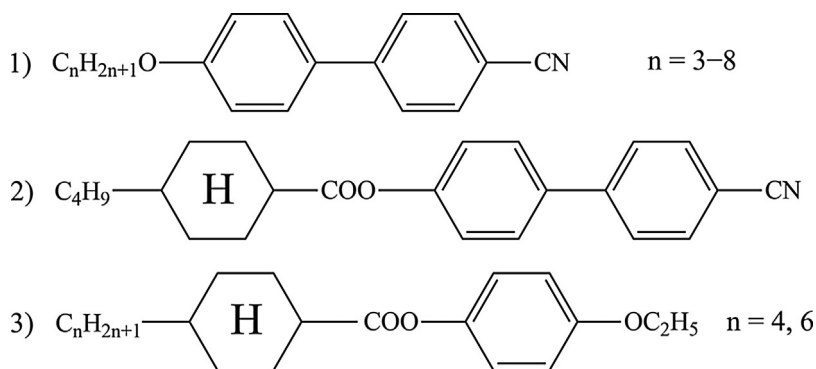

Fig. 1. Structural formulas of components of mixture ZhK-1282 under study: 1) Derivatives of alkoxycyanobiphenyl; 2) Gray's ether; 3) Demus's ether.
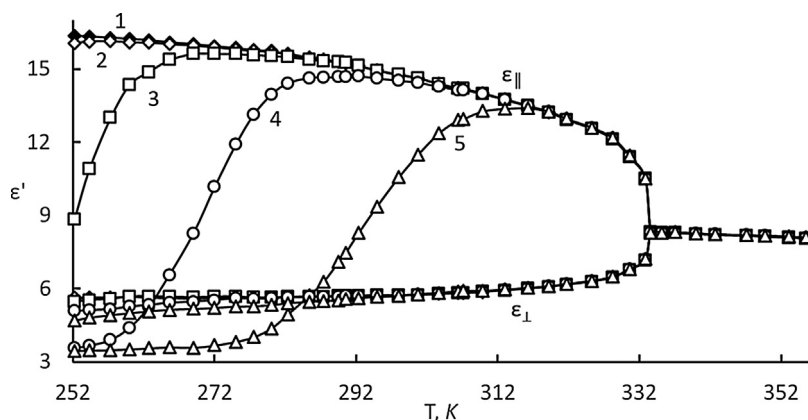

Fig. 2. Temperature dependence of the real part of dielectric constant calculated at different frequencies of the electric field of $1–10^2$; $2–10^3$; $3–10^4$; $4–10^5$; $5–10^6$ (Hz).

contribution, as well as their collective interaction and behaviour [1,21–24].

Dielectric spectra measurements were taken in a wide frequency f range and temperatures ($10^2–10^6$ Hz and -20 to $80\,^\circ$C, accordingly). Calculation of a dielectric constant was done by means of the comparison of an empty capacitor and a capacitor filled with the sample studied. The LC director orientation in the mixture was measured by using a static magnetic field with the induction of 0.52 T in the range of angles of the magnetic field rotation from 0° to 90° with a 10° step. The magnetic field that sets LC mixture orientation is considerably larger than the saturation field and the distance between the capacitor plates is greater that the magnetic coherence length. The dielectric constant values measured by using the static magnetic field and in the NLC cells with both planar and vertical LC alignment are in a good agreement within the ε accuracy of 0.1. Voltage of the applied electric signal was of 1 V. To increase the accuracy of measurements in the calculations, the capacity of connecting wires was taken into account and the stabilization of fluid cryothermostat was set at $\pm0.5\,^\circ$C.

3. Results and discussion

Fig. 2 demonstrates temperature dependence of the real part of a dielectric constant measured at different frequencies of the electric field. Longitudinal $\varepsilon'_{\parallel}$ and transverse $\varepsilon'_{\perp}$ components correspond respectively to the parallel and perpendicular orientation of long axes of molecules relative to the direction of electric field.

When the temperature reduces, we get in the area of transitive frequencies in nematic mesophase at which the anisotropy of dielectric constant changes its sign (Fig. 2). For example, the crossover frequency for the temperature of 284 K is 10^6 Hz. When

the temperature is lowered the crossover frequency decreases according to the Arrhenius law:

$$f_c = f_0 \, \exp\left[-\frac{E}{RT}\right], \tag{1}$$

where E is the activation energy, R is the universal gas constant, and f_0 is the high-temperature ($T \to \infty$) crossover frequency.

The reason for this phenomenon is a dipole-dipole molecular correlation resulting in to their pairing of oppositely directed dipole moments of alkoxycyanobiphenyl and ester molecules within the mixture. Such correlation decreases the positive anisotropy at the increasing of the order parameter. This phenomenon is observed well at the temperature lowering when the dipoles of alkoxycyanobiphenyl stop their rotation along the field; therefore, the main contribution to polarizability is made by the esters with the transverse dipole moment. The dependence observed indicates the influence of molecular interaction mechanisms on the LC mixture anisotropy at the temperature lowering.

There is a discontinuous change of dielectric constant near the transition from the nematic phase to the isotropic phase. The frequency dispersion is not observed in the isotropic phase at the measured range.

Calculation of the imaginary component ε'' of dielectric constant was done by using the values of a loss angle tangent and the real part of the dielectric constant on the basis of the following equation [25]:

$$tg\varphi = \frac{\epsilon''}{\epsilon'} + \frac{\sigma}{2\pi f \epsilon_0 \epsilon'}, \tag{2}$$

where σ is the ionic conductivity, ε' is the real component of the dielectric permitivity, and ε_0 is the electric constant.
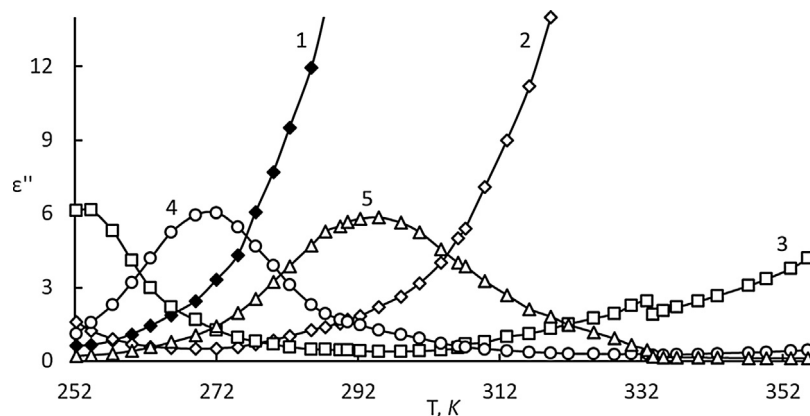
Fig. 3. Temperature dependence of the imaginary component of the transverse dielectric constant at different frequencies of the electric field of $1–10^2$; $2–10^3$; $3–10^4$; $4–10^5$; $5–10^6$ (Hz).
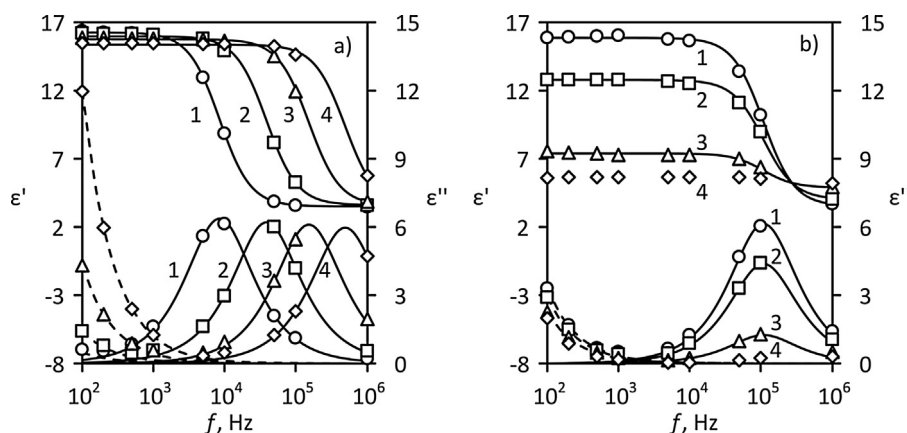

Fig. 4. Frequency dependence of the real and imaginary components of dielectric constant a) temperature with director orientation angle $\theta = 0°$ 1–252 K; 2–263 K; 3–275 K; 4–285 K and at director orientation angles with temperature of 273 K b) $4-\theta = 0°$, $3-\theta = 30°$, $2-\theta = 60°$, $1-\theta = 90°$.

In the low temperature area the maxima of the imaginary component of the dielectric constant are observed (Fig. 3), which characterize the relaxation time corresponding the LC molecules rotation around their short axis. It is important to notice that this process does not make considerable contribution to the dielectric constant at the director perpendicular orientation in relation to the electric field (the dispersion is not observed at the same temperature interval). When the temperature is increased at low frequencies, the ionic conductivity starts to make larger contribution to the ε value, causing difficulties in its separation from the imaginary component of the dielectric constant of the material studied.

The frequency dispersion of the ε, as well as the relaxation processes causing it, may be described in detail with a known Debye's equation [26] for $\varepsilon^* = \varepsilon' - i\varepsilon''$– complex component of the tensor of the dielectric constant:

$$\varepsilon^* = \varepsilon_\infty + \frac{\delta\varepsilon}{1 + i\omega\tau}, \qquad (3)$$

where $\delta\varepsilon = \varepsilon_S - \varepsilon_\infty$ is the dielectric strength, ε_∞ is the high frequency dielectric constant, ε_S is the low frequency dielectric constant (static dielectric permittivity), τ is the relaxation time, $\omega = 2\pi f$ is the electric field angular frequency.

By using optimization methods, such values of unknown parameters as $\varepsilon_\infty, \varepsilon_S, \tau, \sigma$ may be found, at which the minimum is observed for a standard deviation from the experimental data obtained with the help of Debye's Eq. (3). According to these parameters with using Eq. (3) one can build a theoretical curve, which is given in Fig. 4 in the form of solid lines.

Fig. 4a) shows that with the temperature increasing the dielectric spectra shift to high frequencies. In the area of low temperatures the dispersion of the real part of ε is observed. The dotted lines demonstrate the contribution of the ionic conductivity to the experimental data of the imaginary part of the ε. Temperature dependence of the specific ionic conductivity is well-described by the Arrhenius' exponential law.

Angular dispersion of the real part of the dielectric constant is shown in Fig. 4b). Relaxation frequency is determined by the maximum of the imaginary part of the dielectric constant. Fig. 4b) demonstrates that the relaxation frequency does not depend on the director orientation. It is important to notice that numerical approximations according to the Debye's equation coincide well with the experimental data at all orientation angles.

By using the data obtained we plotted the temperature dependencies of the relaxation time corresponding to the LC molecules rotation around their short axis (Fig. 5) and the dielectric strength of this relaxation process (Fig. 6).

The activation energy value calculated by the tangential angle of the logarithmic straight line $\ln[\tau(1/T)]$ (Fig. 5b), is $E_| = (72 \pm 5) \cdot 10^3$ J/mol; for the activation energy of the relaxation process in the isotropic phase it is equal to $E_{IS} = (30 \pm 5) \cdot 10^3$ J/mol. The activation energy of this relaxation process in the mesophase is constant at the whole mesophase temperature range studied. It corresponds to the data for other NLC mixtures [27–29]. The differences in the activation energy value $\triangle U = E_| - E_{IS} = (42 \pm 5) \cdot 10^3$ J/mol characterizes the intermolecular interaction energy, which the polar molecules surmount while
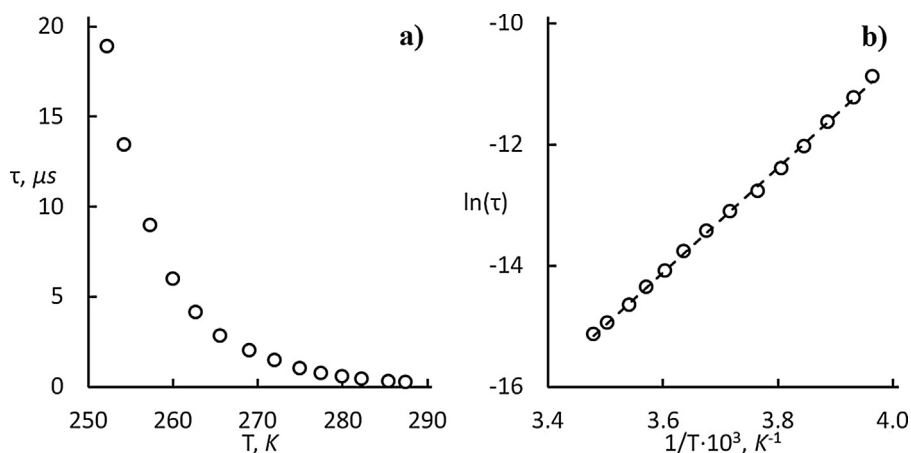
Fig. 5. Temperature dependency of relaxation time $\tau(T)$ a) and $\ln[\tau(1/T)]$ b).
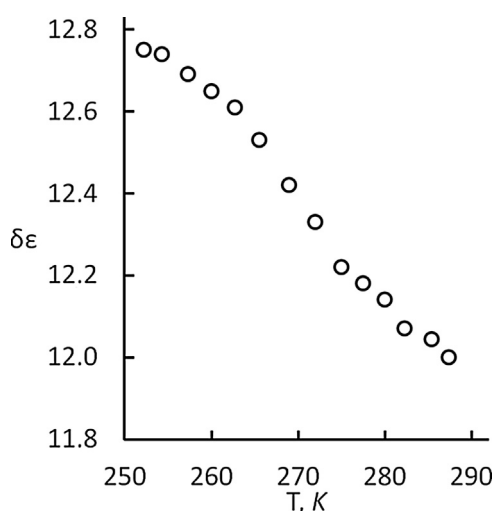

Fig. 6. Temperature dependence of the dielectric strength $\delta\varepsilon$ of the studied relaxation process.

their rotating in the mesophase. Knowing the energy change at the transition from the nematic phase to the isotropic phase ΔU, it is not difficult to calculate an increment of the system entropy $\Delta S \sim 125\,\mathrm{J}/(\mathrm{K\,mol})$ of this process, which corresponds to the studies [30] in the similar mesophases.

The dielectric strength value of the relaxation corresponding to the LC molecules rotation around their short axis slowly decreases with the temperature lowering.

An important task of the dielectric spectroscopy is determination of different parameters of the NLC molecular dynamics, such as the coefficient of molecular friction, rotational diffusion et al. on the basis of to-date theories. Several approaches exist to calculate the coefficient of the molecular friction ζ. Here are some expressions for calculating it which were obtained in different ways [31–34].

$$\zeta = 2kT\tau_D, \qquad (4)$$

$$\zeta = \frac{\gamma_1}{3nf_1(S)} = \frac{\gamma_1 \left[q + (2+S)S^2\left(1+\frac{bq}{6}\right) + 6S^2 - 1.5bS^4\right]}{3nS^2\left(1+\frac{bq}{6}\right)}, \qquad (5)$$

$$\zeta = \frac{2}{9}\frac{(1+2\gamma)}{\gamma}(1+p)\left[1-\frac{5}{p^2+6}\right]d^4\sqrt{\pi kTmn}\,\exp\frac{E}{kT}, \qquad (6)$$

$$\zeta = 8\pi\gamma_1 L^3 f(p) \approx \frac{16\pi\gamma_1 L^3}{6\ln\left[1+\sqrt{p^2-1}\right]-3}, \qquad (7)$$

where τ_D is the typical relaxation time of molecules, k is the Boltzmann constant, T is the temperature, γ_1 is the coefficient of rotational viscosity, S is the order parameter, $p = L/d$ is the ratio of both long and short molecular axes length, n is the molecular concentration, m is the molecular weight, E is the potential barrier which is to be surmounted at the reorientation of a molecular cluster according to Maier-Saupe model [35–39].

The $f_1(S)$ function, as well as the parameters γ and p are estimated in Ref. [40]. The p average value for the mixture studied is taken as 3, $d \sim 0.4$ nm, and $n \sim 2.04 \cdot 10^{27}\,\mathrm{m}^{-3}$.

However, the molecular friction coefficient also is determined by the rotational diffusion D by the proportion [41].

$$\zeta = \frac{kT}{D}. \qquad (8)$$

By using Eqs. (4)–(8) the values of the molecular friction coefficient, as well as the corresponding values of rotational diffusion were calculated at the temperature T = 297 K. The data obtained are presented in Table 1.

The molecular friction coefficient values determined by both dielectric relaxation and rotational viscosity with the usage of the Perrin's function have the same order of magnitude $10^{-27}\,\mathrm{kg\,m^2/s}$. The ζ values, determined by rotational viscosity according to Eq. (5) given in Refs. [32,33,42] are about one order of magnitude lower. This value corresponds to the ζ value, given for azoxy compounds, Schiff bases, pentyl-cyanobiphenyl (5CB) and a bicyclooctane derivative in Ref. [42].

The values calculated by different formulas for the rotational diffusion D are in the range of $2\cdot10^7\,\mathrm{s}^{-1} - 7\cdot10^9\,\mathrm{s}^{-1}$. For the substances studied in Ref. [42] the D value at room temperature is in the range of $8\cdot10^7\,\mathrm{s}^{-1} - 7\cdot10^8\,\mathrm{s}^{-1}$. In Refs. [43,44] one can see the values of both parallel and perpendicular component of the rotational diffusion D for hexyloxy-cyanobiphenyl (6OCB), where $D_| \sim 10^8\,\mathrm{s}^{-1}$, $D_\perp \sim 3\cdot10^7\,\mathrm{s}^{-1}$ at 314 K. For pentyl-cyanobiphenyl (5CB) $D_| \sim 8\cdot10^8\,\mathrm{s}^{-1}$, $D_\perp \sim 4\cdot10^7\,\mathrm{s}^{-1}$ at 297 K. These data are in the best correspondence with the results in column 3 of Table 1.

Somewhat larger values of $D_\perp \sim 4\cdot10^9\,\mathrm{s}^{-1}$ for 5CB at 297 K were obtained in Ref. [45] by the method of the neutron scattering in the NLC. Such difference can be explained by the fact that during this process the scattering takes place on single LC molecules, while during the process of molecular relaxation at both dielectric and viscosimetric measurements the LC molecular clusters' reorienta-

Table 1
Comparison of both molecular friction and rotational diffusion coefficients obtained by using data of Refs. [31–34,41] at T = 297 K.

1.	2.	3.	4.	5.
Coefficient		Values obtained		
	[24] according to formulae (4) and (8)	[25] according to formulae (5) and (8)	[26] according to formulae (6) and (8)	[27] according to formulae (7) and (8)
$\zeta \cdot 10^{27}$, kg m^2/s	0.734	0.107	0.00057	1.97
$D \cdot 10^{-6}$, s^{-1}	5.59	38.35	7190	2.08

Table 2
Physical parameters of the LC mixture ZhK–1282 and its components (alkoxycyanobiphenyl derivatives).

Substance	Demus's ester	5OCB	6OCB	7OCB	8OCB	ZhK-1282
$\varepsilon_{\infty \parallel}$	–	[a]3.8 [21]	[a]3.1 [21]	[a]3.1 [21]	[b]3 [c]3.8 [21]	[e]3.4
$\Delta \varepsilon$	[a]1.01	–	–	–	[d]8.9 [23]	[e]9.8
$\varepsilon_{s \perp}$	–	[a]7.1 [22]	–	–	–	[e]5.7
$\varepsilon_{s \parallel}$	–	[a]19.8 [21]	[a]16.6 [21]	[a]15.5 [21]	[b]13.7 [c]13.4 [21]	[e]15.4
Δn	[a]0.0752	–	–	–	[d]0.15 [23]	[f]0.142
γ_1, Pa s	[a]0.385	–	–	–	–	[f]1.37
K_{11}, 10^{-12} N	[a]7.89	–	–	–	[d]8.8 [23]	[f]10.401
K_{33}, 10^{-12} N	–	–	–	–	[d]15 [23]	–
$E_{\perp}$, kJ/mol	–	5 [21]	6 [21]	4 [21]	–	–
$E_{\parallel}$, kJ/mol	–	70 [21]	68 [21]	65 [21]	[a]75 [b]56 [21]	72
T_M, K	–	321.2 [21]	330.2 [21]	327.15 [21]	327.7 [21]	253.1 [20]
T_{SmA-N}, K	–	–	–	–	340.7 [21]	–
T_{N-I}, K	–	341.2 [21]	348.7 [21]	347.15 [21]	353.7 [21]	335.1 [20]

[a] 324 K.
[b] 330 K.
[c] 346 K.
[d] 341.7 K.
[e] 285.4 K.
[f] 297.4 K.

tion takes place. The evaluation of the number of the molecules in the cluster according to Ref. [42] results in the value of 5–7, which corresponds to X-ray crystallography data.

$$N = \left(\frac{\zeta^{\gamma}}{\zeta'_{mol}} \right)^{\frac{6}{11}} \approx 17.4. \qquad (9)$$

The estimation of ratios of the molecular friction coefficients in columns 3 and 4 of Table 1 according to the procedure described in Ref. [42], gives 17.4 as the molecules' number in the cluster in the mixture ZhK-1282.

Below one can see the comparative data of physical parameters of alkoxycyanobiphenyl derivatives presented in other papers and the LC mixture ZhK–1282 studied. Table 2 demonstrates the typical contribution of derivatives of alkoxycyanobiphenyl to viscous and dielectric properties of the LC mixture.

4. Conclusions

The relaxation process of the LC molecular rotation around their short axis was studied in detail in the LC mixture ZhK–1282 by using the dielectric spectroscopy method. The intermolecular movements in the LC influence the dispersion of dielectric constant which is determined mainly by the molecular rotation around their short axis. At lowering the temperature, the ZhK–1282 dielectric anisotropy changes its sign from positive to negative owing to dipole-dipole molecular correlations resulting in to association of alkoxycyanobiphenyl and ester molecules.

The values of both relaxation time and dielectric strength in the temperature range studied were calculated; the value of activation energy of dipole relaxation in nematic and isotropic phases was determined. The value of activation energy is constant within the studied range, which corresponds well to data obtained for other NLC mixtures. There is quantitative correspondence between the experimental data and the results of numerical calculations.

The contribution of ionic conductivity to the imaginary component of the dielectric constant in the area of low frequencies was measured. The temperature dependence of specific ionic conductivity is described by the exponential law.

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