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Elastic deformations of nematic laterally attached dimers

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Temperature dependences of the optical anisotropy and elastic constants of nematic laterally connected dimers, in which two rod-like 2,7-disubstituted fluorene moieties are linked through their 9-positions by flexible –OOC$(CH_2)_m$COO– residues, were determined by using the Fréedericksz method of threshold transitions in a magnetic field. It was shown that the possibility of rotation of the mesogenic groups about their short axes influences the dependence of the order parameter on spacer length. No marked dependence of splay and bend elastic constants on spacer length was observed. The results are compared with literature data for polymer and low molecular mass nematics.

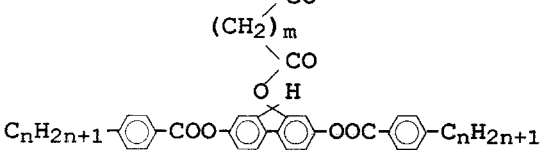
1. Introduction

Thermotropic liquid crystalline polymers are known to exhibit several specific properties distinguishing them from low molecular mass mesophases. In order to elucidate the behaviour of high molecular mass liquid crystals (LCs), it is very useful to study the properties of their low molecular mass analogues, the molecules of which simulate polymer chain fragments, and to compare the properties of these compounds and polymers. In such cases particular attention is usually devoted to the investigation of the relationship between the characteristics of polymeric and corresponding model low molecular mass LCs and the structure of their molecules, in particular the structure and size of the mesogenic core and the spacer length and flexibility.

The process of 'accumulation' of polymer properties by the mesophase has been studied in comparative investigations of the liquid crystalline monomer, dimer and polymer [1–3]. It was established that dimers already exhibit some specific properties characteristic of a high molecular mass LC. On the whole, the behaviour of the dimer is closer to that of the polymer than to that of the monomer. The effect of spacer length on the properties of mesophases formed by dimers [4, 5] and trimers [6] based on cyanobiphenyl has been investigated in detail. It was found that with increasing length of the flexible alkyl fragment, the orientational order parameter changes jumpwise and a distinct odd–even effect is observed. This experimental fact is due to changes in the angle formed by the *para*-aromatic axes of the mesogenic groups when the spacer length increases by one $-CH_2-$ group [4–6]. It is very important that in the nematic phase even relatively long alkyl chains in the molecules of these dimers are mainly in the extended *trans*-conformation [7].

Orientational elastic constants K_i are fundamental values determining the main properties of a LC. They characterize the forces of intermolecular orientational interaction in a nematic, ensuring long range order in the mesophase. Fréedericksz threshold transitions are widely used for the experimental determination of elastic constants [8–10]. The application of these methods to low molecular mass LCs has made it possible to follow the effect of the chemical structure, shape, and size of their molecules on the values of K_i [11–16]. However, papers in which the dependence of K_i values on the structure of macromolecules or their elements has been studied for nematic polymers and their low molecular mass models are very few.

In the present work the Fréedericksz method of threshold transitions in a magnetic field [8] was used to study the birefringence and orientational elastic deformations in the nematic phase for dimers with the following structure



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where $m = 3$ and $n = 6$ for a D-3-6 dimer, $m = 4$ and $n = 7$ for a D-4-7 dimer, $m = 5$ and $n = 7$ for a D-5-7 dimer. The structure of the molecules of these LCs differs from that of the molecules of linear dimers [4, 5] by the way in which the mesogenic groups are joined together. In this case rigid anisotropic cores are bound 'laterally' via the flexible fragment. The spacer is virtually bound to the geometric centre of the mesogenic groups. Moreover, both ends of these groups are relatively long and flexible.

The synthesis and phase behaviour of these dimers have been described in ref. [17].

2. Experimental

The compounds investigated each form a nematic phase by a monotropic phase transition from the isotropic melt. The temperatures of melting, T_m, and of the isotropic (I)–nematic (N) phase transition, T_{IN}, determined by polarizing microscopy are given in the table.

The procedure used for investigating orientational elastic deformations in this work has already been described in detail [8, 18–21]. Wedge-shaped plane–concave nematic layers were used; the LC being investigated was placed between the flat and spherical surfaces of quartz plates. Planar textures were obtained in the usual way by rubbing the surfaces of the plates. In the case of homeotropic textures, the situation was more complex; the surfaces of lenses and glasses were successively etched in sulphuric acid, washed with distilled water, treated with a lecitin solution in alcohol, and dried at 80°C. Even this relatively complex treatment made it possible to obtain uniform homeotropic layers of considerable thickness only for dimers D-4-7 and D-5-7. For D-3-6, the thickness of homeotropic layers obtained did not exceed 10×10^{-4} cm under any conditions of glass treatment. Hence, it was impossible to study quantitatively orientational elastic deformations of D-3-6 in homeotropic layers.

Each sample prepared was observed in a polarizing microscope in parallel light normal to the layer plane. The wavelength of the light λ was 546 nm. The temperature T of the sample was controlled to within 0.5°C, and measurements were carried out using a magnetic field with a strength up to 26 000 Oe.

Orientational elastic deformations appearing in the nematic phase under the influence of a magnetic field applied normal to the director are of the threshold type. A minimum critical thickness z_c of the layers exists in which deformation in a magnetic field with a strength H is possible. According to the Fréedericksz law [8], the product z_cH is constant at a given temperature and determines the ratio of the elastic constant K_i to the diamagnetic anisotropy $\Delta\chi$ for unit volume of nematic

$$z_cH = \pi(K_i/\Delta\chi)^{1/2} \tag{1}$$

where $i = 1, 3$. The values of the splay K_1 and bend K_3 elastic constants can be obtained by studying the deformation of planar and homeotropic layers, respectively.

Table. Molecular masses and phase transition temperatures for the dimers investigated.

Dimer	m	n	M_0	T_m/°C	T_{IN}/°C
D-3-6	3	6	1252	151.0	110.0
D-4-7	4	7	1321	147.0	128.0
D-5-7	5	7	1335	138.5	136.5

In figure 1 the values of the critical thickness z_c are plotted against inverse values of the magnetic field strength H. For both deformation types, at a given temperature, the experimental points fall on the same straight line passing through the origin. This fact is very important because it indicates that strong cohesion of the nematics being investigated to the supporting surfaces exists for both planar and homeotropic textures [11]. The slope of each straight line determines the value of z_cH and, correspondingly, the $K_i/\Delta\chi$ ratio at the given temperature $\Delta T = T - T_{IN}$.

Another important characteristic of the mesophase which can be established by investigating orientational deformations by this procedure is the difference between the two main refractive indices $n_e - n_0 = \Delta n$ or the optical anisotropy of the nematic. For this purpose it is convenient to use polarizing photomicrographs of undeformed planar layers, which have the aspect of a system of concentric interference rings. Each dark interference band corresponds to the difference between beam paths δ equal to the whole number of wavelengths $\delta = m\lambda$

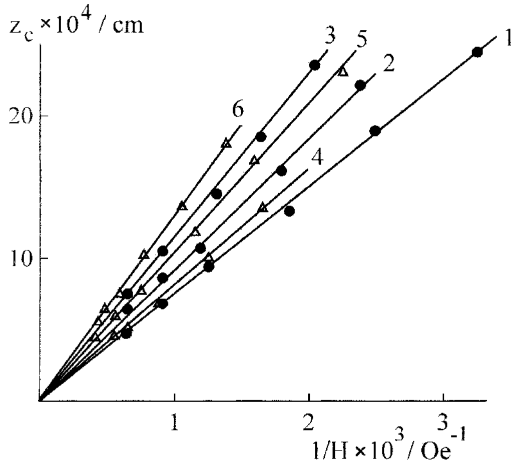

Figure 1. Critical thickness z_c versus inverse of the magnetic field strength—for the D-5-7 dimer for deformations of planar (1–3) and homeotropic (4–6) layers at temperatures $\Delta T = -1$ (1, 4), -13 (2, 5), and -59°C (3, 6).

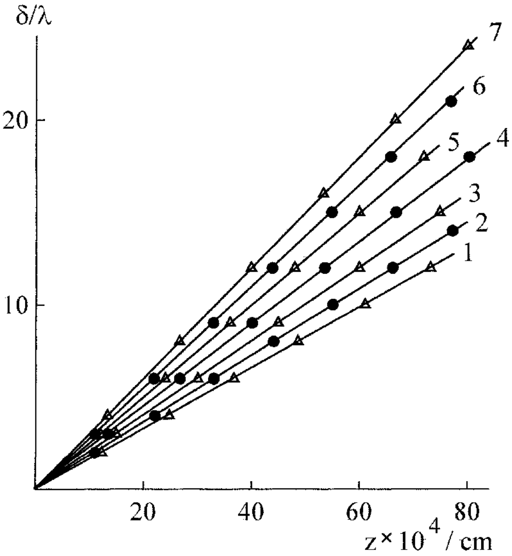

Figure 2. Relative path difference δ/λ versus layer thickness z for the D-5-7 dimer at temperatures $\Delta T = -0{,}5$ (1), $-2{,}5$ (2) -4 (3), -9 (4), -20 (5), -35 (6), and -58°C (7).

where $m = 1, 2, 3, \ldots$ Figure 2 shows the dependences of the relative path difference δ/λ on layer thickness z. It is easy to determine from the slopes of these straight lines the value of the birefringence Δn of the nematic at a given temperature ΔT

$$\Delta n = (\delta/\lambda) \times (\lambda/z) = m\lambda/z. \qquad (2)$$

In the case of an initial homeotropic orientation, the system of interference rings appears in deformed parts of the sample, i.e. in regions of sample with $z > z_c$. For each layer thickness z, the value of the birefringence $\Delta n^* = n_e(H) - n_0$ depends on the magnetic field strength H [22]. This is due to the change of director orientation with changing magnetic field. This dependence may be used to determine the value of the ratio of the elastic constants K_3/K_1. According to Saupe theory [22], variation of the effective birefringence Δn^* of a nematic layer of thickness z with increase in the magnetic field H from a critical value H_c to $H \to \infty$ is described by the relation

$$\frac{\Delta n^*}{n_0} = 1 - \frac{2H_c}{\pi H} \int_0^{\pi/2} \left[\frac{1 + (K_1/K_3 - 1)\sin^2\varphi_m \sin^2\psi}{(1 - \sin^2\varphi_m \sin^2\psi) \times (1 + \nu \sin^2\varphi_m \sin^2\psi)} \right]^{1/2} d\psi \qquad (3)$$

where $\nu = (n_0^2 - n_e^2)/n_e^2$, $\sin\psi \equiv \sin\varphi/\sin\varphi_m$, φ is the director reorientational angle (i.e. the angle between the orientations of the director in the presence and in the absence of the magnetic field H), and φ_m is the value of φ in the middle of the nematic layer, where the deformation is a maximum. The angle φ_m is defined by the relation

$$\frac{Hz}{2}\left(\frac{\Delta\chi}{K_3}\right)^{1/2} = \int_0^{\pi/2} \left[\frac{1 + (K_1/K_3 - 1)\sin^2\varphi_m \sin^2\psi}{1 - \sin^2\varphi_m \sin^2\psi} \right]^{1/2} d\psi. \qquad (4)$$

Comparison of the experimental dependence of Δn^* on H/H_c with the theoretical relations (3) and (4) allows us to determine K_3/K_1 by studying only the deformation of homeotropic layers. As an example, in figure 3 we present a plot of Δn^* against H/H_c for dimer D-5-7 at $\Delta T = -27.5$°C. The best fit of the theoretical dependence to the experimental points is obtained for $K_3/K_1 = 1.25$ (curve 2). For the purpose of comparison, we also plot the theoretical curves corresponding to $K_3/K_1 = 1.1$ and 1.4. As can be seen, all experimental points lie between these two curves, i.e. the experimental error in the determination of K_3/K_1 by this method is about 10–15%.

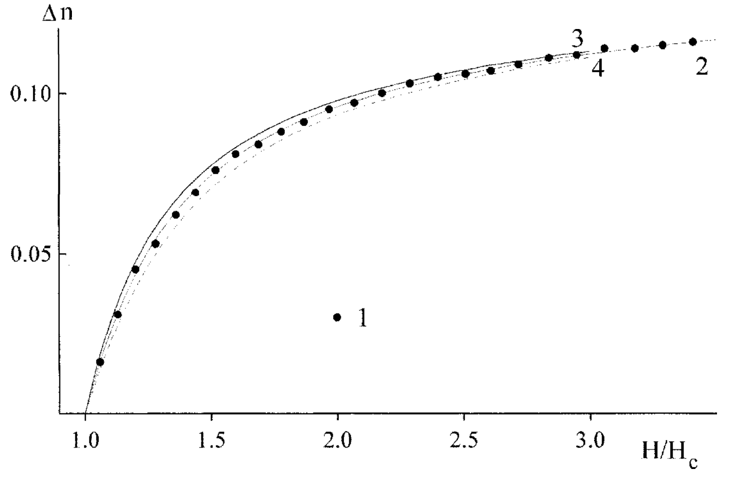

Figure 3. The plot of effective birefringence Δn^* versus relative magnetic field strength H/H_c for D-5-7 at $\Delta T = -27.5$°C (1). The theoretical curves correspond to $K_3/K_1 = 1.25$ (2), 1.4 (3), and 1.1 (4).

3. Results and discussion

3.1. *Optical anisotropy*

Figure 4 shows the temperature dependence of the optical anisotropy values obtained for the dimers being investigated. The shape of these dependences is typical of a nematic LC: the values of Δn increase monotonically with decreasing sample temperature. When the values of Δn are not very high ($\Delta n < 0.2$), these dependences may be described by an approximate equation [23]

$$\Delta n = (2\pi/3n)(n^2 + 2)N_A(\Delta\alpha/M_0)\rho S \qquad (5)$$

where S is the degree of orientational order, n is the mean refractive index, ρ is the density of the nematic, M_0 is the molecular mass, $\Delta\alpha$ is the polarizability of the nematic molecules and N_A is Avogadro's number.

It is known that even over a wide temperature range the values of the mean refractive index n and the density ρ for a nematic LC change only slightly, within a few % (see for example [24]). In other words, the main contribution to the increase in Δn with decreasing temperature is provided by changes in the parameter S. Hence, to a first approximation, the dependence of Δn on ΔT reflects the temperature dependence of the degree of orientational order S of the nematic.

As can be seen from figure 4, the value of Δn changes when the alkylene spacer binding the rigid anisotropic groups becomes longer. According to equation (5), this experimental fact may be caused by changes in both the order parameter S and the specific polarizability $\Delta\alpha/M_0$ of the dimer molecules.

For the dimers investigated, the $\Delta\alpha$ value is determined to a considerable extent by the anisotropy of polarizability of the *para*-substituted aromatic mesogenic groups. The contribution provided by alkyl chains to the difference between the main polarizabilities of the molecules does not exceed 10%. Therefore, when the length of the flexible groups in the molecules of these dimers increases, the $\Delta\alpha$ value remains virtually unchanged, whereas the molecular mass M_0 increases slightly. As a result, the $\Delta\alpha/M_0$ ratio decreases. However, even on passing from D-3-6 to D-4-7, when the number of $-CH_2-$ groups in the dimer molecules increases by five, the decrease in $\Delta\alpha/M_0$ does not exceed 5%. For these dimers at temperatures near the phase transition point, I → N, the experimental values of Δn differ by more than 20% (figure 4). This difference in Δn values may be explained only if it is assumed that for D-3-6 the parameters of the nematic orientational order S are much greater than those for D-4-7.

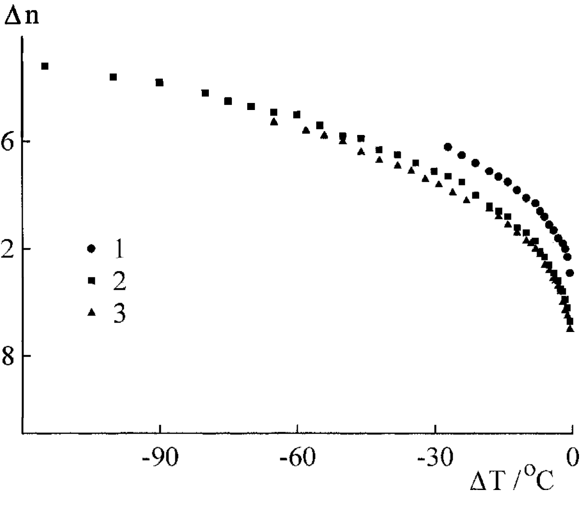

Figure 4. Temperature dependences of the optical anisotropy Δn for D-3-6 (1), D-4-7 (2), and D-5-7 (3).

On further increase in spacer length on passing from D-4-7 to D-5-7, no marked changes in Δn were observed (figure 4). Throughout the temperature range investigated, the values of Δn for D-4-7 are slightly higher than those for D-5-7, but this difference is within the experimental error of Δn. The molecular masses of these dimers are close (table). Therefore, it is not a great assumption to consider that the $\Delta\alpha/M_0$ ratios for them coincide. Consequently, it may be concluded that for dimers D-4-7 and D-5-7 the values of S and its temperature dependence virtually coincide.

In order to explain the discussed changes in the values of Δn and S with increasing spacer length, let us analyse the chemical structure of these nematogens. The rigid aromatic groups in the molecules of these lateral dimers retain a considerable freedom of rotation around their short axes near the point of binding of these groups to the flexible fragment. The change in mutual orientation of the *para*-aromatic axes of the mesogenic groups as a result of these rotations can lead to considerable changes in the values of the order parameter S and the optical anisotropy Δn of these nematics.

On passing from the $-(CH_2)_3-$ spacer to the $-(CH_2)_4-$ spacer, the increase in Δn can be explained if it is assumed that the mesogenic cores linked by these quite short spacers do *not* rotate markedly about their short axes. In fact, let us assume that the spacer chain is mainly in the *trans*-conformation, just as in the molecules of linear dimers. Then the increase in spacer length by one $-CH_2-$ group will lead to a sharp change in the angle formed by the *para*-aromatic axes of the mesogenic groups. If on the whole this does not involve any marked rotation of the anisotropic groups about their short axes, the degree of orientational order S, and hence the birefringence Δn, should decrease drastically. This situation is probably observed on passing from the $-(CH_2)_3-$ spacer to the $-(CH_2)_4-$ spacer. However, when the spacer chain increases by another $-CH_2-$ group, reorientation of the mesogenic cores as a result of their rotation about their short axes may take place in a considerable number of molecules. This reorientation compensates for the jump in the angle between the long axes of the mesogenic groups when the spacer becomes longer. Therefore, on passing from D-4-7 to D-5-7 the value of Δn could virtually remain unchanged. However, for a rigorous quantitative verification of these assumptions, it is necessary to study the properties of the mesophases for a larger number of homologous lateral dimers.

3.2. *Orientational elastic constants*

Figure 5 shows the temperature dependences of $K_1/\Delta\chi$ and $K_3/\Delta\chi$ for the nematic dimers investigated. It should first be noted that for D-4-7 and D-5-7, over the entire temperature range, the points corresponding to the bend elastic constants are slightly higher than those for splay elastic constants, i.e. $K_3/K_1 > 1$. The dependences of K_3/K_1 on temperature, ΔT, for these dimers are shown in figure 6. The points for these dependences were obtained by two methods. First, the values of $K_1/\Delta\chi$ and $K_3/\Delta\chi$ were determined from independent experimental investigations on the deformations of planar and homeotropic layers, and subsequently the K_3/K_1 ratios were calculated. Second, the K_3/K_1 ratio was determined from the results of investigations of only homeotropic texture deformations using Saupe theory [22]. The values of K_3/K_1 obtained by these two methods coincide within experimental error (figure 6), which again indicates that these LCs are rigidly bound to the glass support.

The ratio of the elastic bend and splay constants K_3/K_1 is known to be very sensitive to changes in the structure and geometrical dimensions of LC molecules. According to theoretical concepts, K_3/K_1 must increase with the ratio $W = L/D$, where L and D are the effective length and thickness, respectively, of the nematic molecules (cf. [11] and references therein). Experimental investigations have shown that the behaviour of nematics—the molecules of which may be regarded as rigid rods—corresponds qualitatively to theoretical predictions. When these molecules become longer, K_3/K_1 increases, whereas when their thickness increases as a result of inserting bulky substituents into the phenylene rings, K_3/K_1 decreases [12, 13]. For nematics with flexible end groups in their molecules, even qualitative agreement between experiment and theory is not observed. In this case when the lengths of end groups increase, i.e. when W increases, the K_3/K_1 ratio decreases relatively rapidly [13–16]. This fact is usually attributed to the difference between the real shape of the molecule and the model rod-like shape, a difference which is caused by the 'coiling' of end groups or by the deviation of their long axes from the direction of the rigid group of the molecule. The possibility of the existence in nematics with this molecular structure not only of long range orientational order, but also of short range translational order has also been suggested [11].

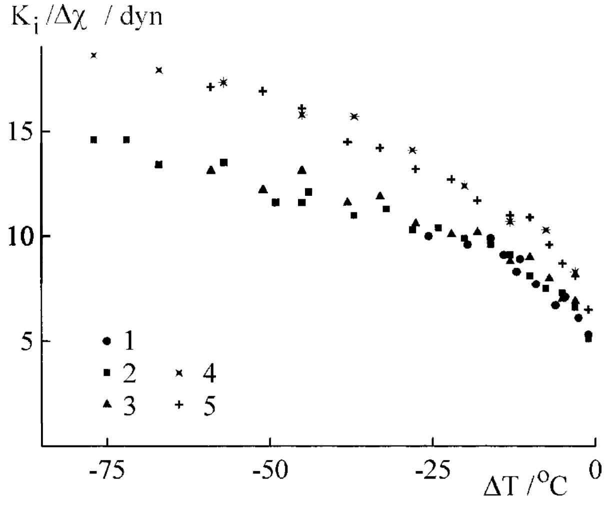

Figure 5. Temperature dependences of the $K_1/\Delta\chi$ (1–3) and $K_3/\Delta\chi$ (4, 5) ratios for D-3-6 (1), D-4-7 (2, 4), and D-5-7 (3, 5).

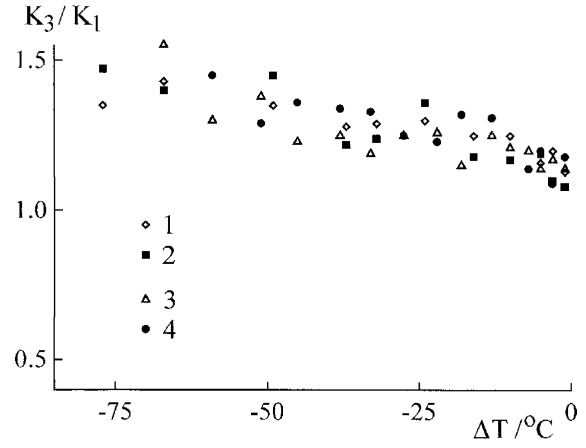

Figure 6. Temperature dependences of the K_3/K_1 ratios for D-4-7 (1, 2) and D-5-7 (3, 4). The points defined by 2 and 4 were obtained from the investigations of homeotropic texture deformations using Saupe theory.

It follows from the data in figure 6 that for the dimers under investigation the K_3/K_1 ratio does not change when the flexible fragment binding the rigid mesogenic groups becomes longer. In fact, the experimental points for D-4-7 and D-5-7 fall near the same curve for the temperature dependence of K_3/K_1. This behaviour is qualitatively similar to that observed for linear aromatic polyesters [21]. A two-fold increase in the length of flexible diethyleneglycol spacers binding *para*-substituted aromatic mesogenic groups in their molecules did not influence the K_3/K_1 ratio.

In the 80°C temperature range investigated, the K_3/K_1 ratio changes very slightly. When the temperature increases, the K_3/K_1 ratios do not increase by more than 15% (figure 6). This change is virtually completely within the experimental error of the determination of the K_3/K_1 value. Hence, just as for polymer nematics [21], it may be assumed that the K_3/K_1 ratio does not depend on temperature. Strictly speaking, it must be borne in mind that for low molecular mass nematics with K_3/K_1 values close to unity the temperature dependence of K_3/K_1 is slight.

As can be seen from figure 5, the values of $K_1/\Delta\chi$ and $K_3/\Delta\chi$ do not change much when the flexible fragment length increases. However, this does not imply that the values of the elastic constants K_1 and K_3 also do not change. In order to follow the dependence of K_1 and K_3 on spacer length, it is necessary to know the value and temperature dependence of the specific diamagnetic anisotropy $\Delta\chi$. The character of the dependence of $\Delta\chi$ is in complete agreement with the curve Δn versus ΔT in figure 4, because the value of the diamagnetic anisotropy $\Delta\chi$ is a unique function of the degree of orientational order S, just like the optical anisotropy Δn of nematics [25]. The relation between the $\Delta\chi$ values for the nematics investigated can be estimated by using the conclusions in [26]. It has been shown in this work that molar magnetic anisotropies $\Delta\chi_\mu$ of nematics are proportional to the number of benzene rings in their molecules. From the chemical structure of dimers, it can be considered that at the same values of the order parameter S, the values of the specific diamagnetic anisotropy for the dimers investigated is practically equal (the molecular masses of the dimers under investigation differ by only 5%). Consequently the possible difference in values of $\Delta\chi$ for the nematics investigated is mainly due to the difference in the S values. Taking into account the analysis of the dependences of the optical anisotropy Δn and the parameter S on temperature carried out above, we can assume that at the same temperature ΔT, the $\Delta\chi$ values for D-4-7 and D-5-7 are equal and are a little less than that for D-3-6. This difference does not exceed 15%.

Therefore from the data shown in figure 5 it follows that for dimers D-4-7 and D-5-7, like the values of the splay elastic constants K_1, so the values of the bend constants K_3 are practically equal at all temperatures ΔT. The constants K_1 for D-3-6 are 15% higher than those for D-4-7 and D-5-7. One probably must not attach much importance to this difference because the experimental error in the determination of K_1 attains 10%. It must be noted that for both low molecular mass and polymer nematics the change in the length of flexible fragments is usually accompanied by changes in the orientational elastic constant.

4. Conclusions

Orientational elastic deformations in a magnetic field have been investigated for the nematic phases of lateral dimers differing mainly in the length of the flexible fragment binding the rigid mesogenic groups. Temperature dependences of the optical anisotropy and elastic constants were determined. It has been shown that the degree of orientational order S depends on spacer length. The possibility of rotation of mesogenic groups about their short axes influences the character of this dependence.

In contrast to common low molecular mass nematics, for lateral dimers the ratio of the bend and splay elastic constants, K_3/K_1, does not change when flexible fragments become longer. From this standpoint, the properties of the dimers investigated approach those of the nematic phases of linear polymers. On the other hand, for lateral dimers the constants K_1 and K_3 do not depend on spacer length, whereas for polymer and common low molecular mass LCs, the values of K_i are very sensitive to changes in the length of flexible groups in their molecules.

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