

Molecular Parameters and Mesomorphism of Discotic Polysubstituted Benzene Derivatives

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Abstract—The previously developed approach to prediction of columnar and nematic mesophases for compounds with disc-shaped molecules was applied to low-molecular-weight polysubstituted benzene derivatives. The molecular parameters were calculated for 39 new compounds: derivatives of 1,3,5-tri(4-hydroxybenzoyloxy)benzene, 1,3,5-tri(4-cyclohexylbenzoyloxy)benzene, 1,3,5-tri(4-hydroxyphenylcarboxy)benzene, 1,2,3-tri(4-cyclohexylbenzoyloxy)benzene, etc. The prediction results were checked by the synthesis of certain compounds from the test sample and examination of their mesomorphic behavior. The prediction of the mesomorphism is negative for only three of the 39 compounds considered. The majority of the molecules under consideration tend to form columnar supramolecular packings, and the capability to form a nematic phase is predicted for only eight compounds. For a number of structures, the capability and incapability to form a nematic phase are equally probable. Synthesis and examination of the mesomorphic behavior of seven representatives of the series considered showed good agreement with the prediction results. 1,3,5-Tri(4-heptyloxybenzoyloxy)benzene, 1,3,5-tri(4-cyclohexylbenzoyloxy)benzene, 1,3,5-tri(4-nonyloxyphenylcarboxy)benzene, and 1,2,3-tri(4-cyclohexylbenzoyloxy)benzene pass into the liquid crystal state forming mainly columnar supramolecular packings. 1,3,5-Tri(4-octyloxybenzoyloxy)benzene and 1,3,5-tri(4-nonyloxybenzoyloxy)benzene melt at 55 and 48°C, respectively, without forming a mesophase, which is also well consistent with the prediction results.

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Thermotropic liquid crystals are used in gas chromatography as stationary phases of enhanced selectivity [1, 2]. However, discotic mesogens [3–7], especially those with nematic phases (N_D , N_{Col}) are still poorly studied in this respect. The interaction of non-mesogenic compounds with a solid adsorption film of a mesogenic modifier, high-temperature disco-nematic liquid crystal (a hexahydroxytriphenylene derivative) was studied only relatively recently [8]. Somewhat later, the sorption and selective properties of a binary high-temperature liquid crystal sorbent based on nematic calamitic and discotic mesogens were reported [9].

Further studies of the chromatographic behavior of liquid crystals as stationary phases require nematic discogens of low molecular weights and with low temperatures of phase transitions. To search for such compounds, we used the approach we are developing for the choice of potential discotic mesogens, based on quantitative molecular parameters (K , K_c , K_p , K_s , K_{ar} , M_m , M_r) and their classification series (Ia, Ib) [10–12]. The molecular parameters can be conventionally subdivided into five groups.

(1) Parameters taking into account the anisometry of a molecule or its parts:

$$K = L_{\max}/s, K_c = l_c/b_c, K_p = l_c/2l_p,$$

where L_{\max} is the maximal molecular length; s , molecular thickness; l_c and b_c , length and width of the central part of the molecule, respectively; and l_p , length of the periphery taking into account the *trans* conformation of the hydrocarbon substituents.

(2) Parameter taking into account the degree of substitution of the central molecular fragment with peripheral substituents. It is defined as the ratio of the number of substituents in the molecule N to the maximum possible number of substituents N_{\max} :

$$K_s = N/N_{\max}.$$

(3) Molecular-weight parameter taking into account the ratio of the weights of the central (M_c) and peripheral (M_p) parts of the molecule:

$$M_m = M_c/M_p.$$

(4) Reduced molecular-weight parameter:

$$M_r = M_m K_s.$$

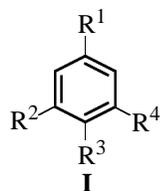
(5) Parameter describing the conventional packing density of the peripheral substituents in mesogenic and nonmesogenic structures. It is calculated as the ratio of total number of the peripheral C, H, and other atoms (N_p) from the empirical formula of a discotic molecule to the area of the molecular cross section taken across the molecular core minus the area of the central core with the adjacent heteroatom:

$$K_{ar} = N_p / (\pi l_p^2 + \pi l_p l_c).$$

For the class of discotic mesogens **Ia**, K 2.0–8.5, K_c 1.0–2.6, K_p 0.2–0.7, K_s 0.25–1.00, K_{ar} 0.08–0.30, M_m 0.3–0.8, and M_r 0.15–0.80; for the subclass N_D or N_{Col} in the class of discotic mesogens (**Ib**), M_m 0.9–2.3.

The molecular-weight parameter M'_m for **Ib** was introduced in [12] to distinguish the subclass of discotic mesogens (N_D) and columnar-nematic (N_{Col}) mesogens in the class of discotic mesogens; it is based on the analysis of molecular parameters of 136 compounds with the discotic molecular shape, exhibiting mainly the nematic type of the mesomorphism.

Using this approach and aiming to find new discotic mesogens with columnar or nematic mesophases and a low molecular weight as promising materials for gas chromatography [8, 9], we considered a new series of polysubstituted benzene derivatives **Ia–If**



$R^1 = R^2 = R^4 = \text{OC(O)C}_6\text{H}_4(4\text{-OC}_n\text{H}_{2n+1})$, $R^3 = \text{H}$, $n = 1\text{--}10$ (**a-1–a-10**); $R^1 = R^2 = R^4 = \text{OC(O)C}_6\text{H}_4$ , $R^3 = \text{H}$ (**b**); $R^1 = R^2 = R^4 = \text{C(O)OC}_6\text{H}_4(4\text{-OC}_n\text{H}_{2n+1})$, $R^3 = \text{H}$, $n = 1\text{--}10$ (**c-1–c-10**); $R^1 = \text{H}$, $R^2 = R^3 = R^4 = \text{OC(O)C}_6\text{H}_4(4\text{-OC}_n\text{H}_{2n+1})$, $n = 1\text{--}12$ (**d-1–d-12**); $R^1 = \text{H}$, $R^2 = R^3 = R^4 = \text{OC(O)C}_6\text{H}_4$  (**e**); $R^2 = R^4 = \text{H}$, $R^1 = R^3 = \text{CHR}'_2$, $R' = \text{OC(O)C}_6\text{H}_4(4\text{-OC}_n\text{H}_{2n+1})$, $n = 1\text{--}10$ (**f-1–f-10**).

The choice of polysubstituted benzenes **I** was governed by the demand for discotic mesogens of low

molecular weight, forming mainly nematic mesogenic structures.

For the whole series of compounds **Ia–If**, using the method of molecular mechanics (MM+) and the HyperChem program (Ver. Pro 6.0), we constructed and optimized the models of molecular structures, e.g., **Ia-7**, **Ic-7**, and **Id-4** (Fig. 1) and determined their geometric dimensions required for calculating the molecular parameters.

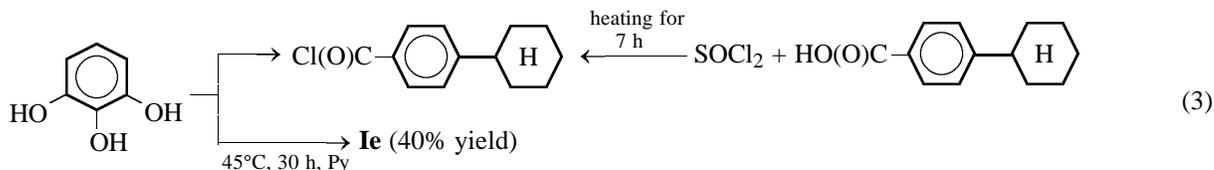
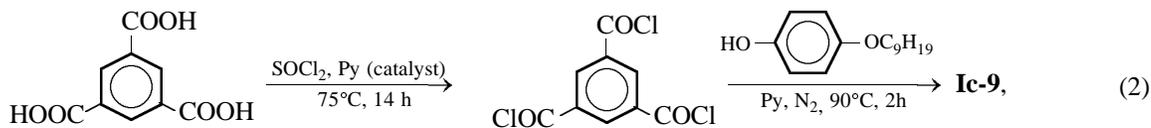
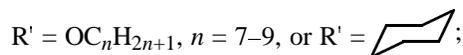
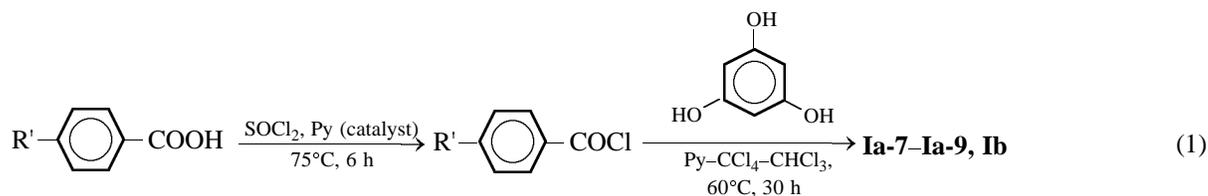
In Table 1 we give the calculated molecular parameters mainly for those representatives of series **I** for which the columnar and nematic mesomorphism is the most probable according to the prediction, for compounds for which the presence or lack of mesomorphism is equally probable, and for some nonmesogenic homologs with boundary values of molecular parameters. Also, we give the molecular parameters of compounds **Ia** and **Ic** for which the published data on the mesomorphic behavior are contradictory [13–15].

Table 1 shows that the majority of the molecular structures given in it tend to form columnar (Col) supramolecular packings according to the prediction (see classification series **Ia** and **Ib**). Only for eight compounds of this series, a nematic phase can also be expected along with a columnar phase. For a series of molecular structures, the positive or negative prediction is equally probable (Table 1).

As already noted, published data on the mesomorphism of **Ia-7**, **Ia-8**, and **Ic-9** are contradictory [13–15]. For example, Aver'yanov [13] reported on the nematic mesomorphic behavior of phloroglucinol derivative **Ia-8**, whereas, according to [14], its nearest homolog **Ia-7** has a well-defined melting point, i.e., is nonmesomorphic. The columnar ordering in the mesophase of one of **Ic** homologs was reported in [15]. Our analysis of the molecular parameters of **Ia-7** and **Ia-8** shows that the columnar or nematic mesomorphism of **Ia-8** is improbable. For **Ia-7**, the presence and lack of the mesomorphism are equally probable; the same is true for the ninth homolog of trimesic acid, **Ic-9**.

To check our predictions, we prepared by schemes (1)–(3) seven compounds of series **I**, **Ia-7–Ia-9**, **Ib**, **Ic-9**, **Id-7**, and **Ie**.

Procedures for preparing **Ia-7–Ia-9**, **Ib**, **Ic-9**, **Id-7**, and **Ie** were similar to those described to [16]. All the compounds were prepared by acylation of the starting phenols with acid chlorides in pyridine or in mixtures of pyridine with organic diluents, to reduce tarring. For the same reason, the synthesis temperature did not exceed 60°C. Only in the synthesis of trimesic acid ester [scheme (2)], the temperature was elevated to



90°C and the reaction was performed in a nitrogen flow. The yields of the final reaction products after chromatographic purification on alumina or on silica gel (eluent ethyl acetate, benzene, or their mixture)

and after additional recrystallization from ethanol were 20–40%. As an example, we describe in detail in the Experimental the procedure for preparing 1,3,5-tri(4-nonyloxybenzoyloxy)benzene **Ia-9**.

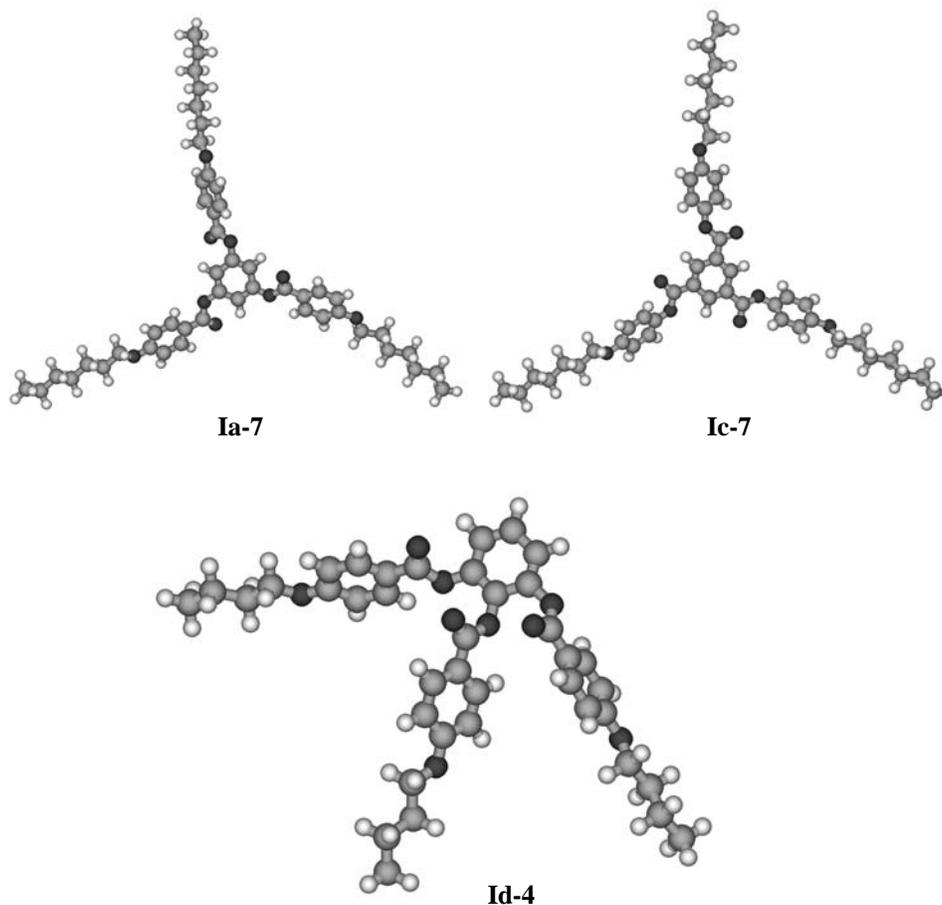


Fig. 1. Examples of optimized molecular structures of compounds of series **I**: **Ia-7**, E 56.74 kcal mol⁻¹; **Ic-7**, E 57.05 kcal mol⁻¹; **Id-4**, E 44.44 kcal mol⁻¹ (E is the energy of the optimized molecular model).

Table 1. Molecular parameters and prediction of the mesomorphism of compounds of series **Ia–If**^a

Comp. no.	E , kcal mol ⁻¹	k	K_p	K_{ar}	m_m	M_r	$P_{Col\ and\ N}$	M'_m	P_N
Ia-1	41.90	4.15	0.43	0.133	0.64	0.32	+	9.65	–
Ia-2	42.64	4.92	0.37	0.127	0.57	0.28	+	5.54	–
Ia-3	45.36	4.91	0.33	0.118	0.51	0.26	+	3.74	–
Ia-4	48.15	5.16	0.29	0.113	0.46	0.23	+	2.82	–
Ia-5	50.97	4.90	0.26	0.106	0.42	0.21	+	2.26	+
Ia-6	53.81	5.34	0.24	0.101	0.39	0.20	±	1.89	+
<i>Ia-7</i>	<i>56.74</i>	<i>5.20</i>	<i>0.22</i>	<i>0.095</i>	<i>0.36</i>	<i>0.18</i>	<i>±</i>	<i>1.62</i>	<i>±</i>
<i>Ia-8</i>	<i>59.48</i>	<i>5.62</i>	<i>0.20</i>	<i>0.091</i>	<i>0.34</i>	<i>0.17</i>	–		
<i>Ia-9</i>	<i>62.24</i>	<i>5.73</i>	<i>0.17</i>	<i>0.086</i>	<i>0.32</i>	<i>0.16</i>	–		
<i>Ib</i>	<i>64.49</i>	<i>4.09</i>	<i>0.37</i>	<i>0.156</i>	<i>0.43</i>	<i>0.22</i>	+	1.75	+
Ic-1	42.73	5.27	0.48	0.130	0.64	0.32	+	9.65	–
Ic-2	43.42	5.69	0.42	0.145	0.57	0.28	+	5.54	–
Ic-3	46.10	5.40	0.36	0.116	0.51	0.26	+	3.74	–
Ic-4	48.82	5.94	0.32	0.110	0.46	0.23	+	2.82	–
Ic-5	51.55	5.62	0.29	0.103	0.42	0.21	+	2.26	+
Ic-6	54.33	5.30	0.26	0.098	0.39	0.20	+	1.89	+
Ic-7	57.05	5.64	0.24	0.093	0.36	0.18	±	1.62	+
Ic-8	59.84	6.03	0.22	0.089	0.34	0.17	±	1.42	+
<i>Ic-9</i>	<i>62.71</i>	<i>6.37</i>	<i>0.22</i>	<i>0.097</i>	<i>0.32</i>	<i>0.16</i>	<i>±</i>	<i>1.27</i>	<i>±</i>
Ic-10	65.24	7.79	0.19	0.081	0.30	0.15	–		
Id-1	40.22	4.37	0.42	0.135	0.64	0.32	+	10.70	–
Id-2	41.71	4.30	0.35	0.104	0.57	0.28	+	5.54	–
Id-3	44.44	4.69	0.31	0.121	0.51	0.26	+	3.37	–
Id-4	47.26	3.75	0.27	0.115	0.46	0.23	+	2.82	–
Id-5	50.23	3.42	0.24	0.108	0.42	0.21	±	2.26	±
Id-6	53.02	3.67	0.22	0.103	0.39	0.20	±	1.89	±
<i>Id-7</i>	<i>55.40</i>	<i>4.63</i>	<i>0.20</i>	<i>0.097</i>	<i>0.36</i>	<i>0.18</i>	<i>±</i>	<i>1.62</i>	<i>±</i>
Id-8	58.59	4.91	0.23	0.087	0.34	0.17	±	1.42	±
Id-9	61.29	4.76	0.20	0.084	0.32	0.16	±	1.27	±
<i>Ie</i>	<i>64.49</i>	<i>4.09</i>	<i>0.40</i>	<i>0.150</i>	<i>0.43</i>	<i>0.22</i>	+	0.09	–
If-1	27.12	3.11	0.64	0.204	0.64	0.43	+	0.18	–
If-2	30.52	2.44	0.52	0.182	0.57	0.38	+	0.26	–
If-3	33.45	3.69	0.53	0.162	0.51	0.34	+	0.35	–
If-4	38.59	3.38	0.42	0.156	0.47	0.31	+	0.44	–
If-5	39.47	4.35	0.37	0.150	0.43	0.28	+	0.53	–
If-6	41.88	5.45	0.32	0.142	0.39	0.26	+	0.61	–
If-7	43.41	4.83	0.26	0.140	0.36	0.24	+	0.69	–
If-8	46.07	4.61	0.27	0.126	0.34	0.22	+	0.75	–
If-9	49.62	3.77	0.22	0.124	0.32	0.21	±	0.79	–

^a K_c 1.01, K_s 0.50 for **Ia–Ie**; K_c 1.66–2.46, K_s 0.67 for **If**. Data for the compounds that we synthesized to check the prediction are printed italic. Empty cells mean that M'_m was not calculated and prediction of the nematic mesomorphism was not made because of the negative general prediction of the mesomorphism. E is the energy of the optimized molecular model; $P_{col\ and\ N}$, prediction of the mesomorphism; and P_N , prediction of mainly nematic mesomorphism.

To confirm the structures of the compounds prepared, we recorded their electronic and IR absorption spectra (Table 2) and performed the elemental analysis (Table 3). The electronic absorption spectra of **Ia-7–Ia-9** and **Ic-9** exhibit two main bands of benzene absorption in the UV range (Table 2). The UV spectra of

these compounds in ethanol are similar in shape to those of tetraalkanoyloxyhydroquinones whose structure was determined in [17]. In the UV spectra of compounds **Ib** and **Ie** containing cyclohexyloxybenzyloxy groups, compared to phloroglucinol esters **Ia-7–Ia-9**, the first absorption band is shifted toward shorter

Table 2. UV and IR absorption spectra of compounds of series **Ia–Ic** and **Ie**^a

Comp. no.	λ_1 , nm ($\log \varepsilon_1$)	λ_2 , nm ($\log \varepsilon_2$)	λ_3 , nm ($\log \varepsilon_3$)	λ_4 , nm ($\log \varepsilon_4$)	$\nu(\text{C}=\text{O})$ of ester	$\nu(\text{C}-\text{O})$ of ester	$\delta(\text{CH})$
Ia-7	274 sh (4.98)	270 (5.00)	264 sh (4.96)	212 (4.85)	1733	1251, 1166	842, 691
Ia-8	277 sh (4.79)	270 (4.81)	263 sh (4.75)	213 (4.75)	1732	1251, 1166	850, 695
Ia-9	275 (4.88)	–	–	212 (4.80)	1731	1250, 1160	842, 688
Ib	278 sh (3.88)	270 (4.90)	245 (4.93)	238 sh (4.82)	1740	1256, 1176	832, 664
Ic-9	279 sh (3.54)	264 sh (4.35)	–	221 (4.85)	1746	1222, 1183	865, 693
Id	251 sh (4.78)	247 (4.86)	242 sh (4.78)	208 (4.74)	1749	1231, 1178	845, 685 ^b

^a The UV spectra were taken in ethanol, and the IR spectra, in KBr pellets; sh, shoulder. ^b 1,2,3-Substitution of the benzene ring (1,3,5-substitution in the other cases).

Table 3. Analytical data and phase transition temperatures for compounds of series **Ia–Ic** and **Ie**

Comp. no.	T_m , °C	T_{cl} , °C	ΔT , °C	Found, %		Formula	Calculated, %		M
				C	H		C	H	
Ia-7	54	58	4 ^a	74.16	7.57	$\text{C}_{48}\text{H}_{60}\text{O}_9$	73.81	7.76	781.08
Ia-8	–	55	0	75.76	8.90	$\text{C}_{51}\text{H}_{66}\text{O}_9$	74.41	8.10	823.17
Ia-9	–	48	0	75.90	7.19	$\text{C}_{54}\text{H}_{72}\text{O}_9$	74.95	8.40	865.26
Ib	133	137	4 ^b	79.91	7.00	$\text{C}_{45}\text{H}_{48}\text{O}_6$	78.91	7.08	684.93
Ic-9	47	75	28	74.55	8.22	$\text{C}_{54}\text{H}_{72}\text{O}_9$	74.95	8.40	865.26
Id	134	147	10	79.89	6.88	$\text{C}_{45}\text{H}_{48}\text{O}_9$	78.91	7.08	684.93

^a The mesophase is observed only in the course of initial heating. ^b On cooling, the sample forms a glass from the mesophase. T_m is the melting point; T_{cl} , clarification point; ΔT , range of existence of the mesophase.

wavelengths, probably because of the absence in **Ib** and **Ie** of ether fragments. The UV spectrum of **Ic-9** (nonyloxyhydroquinone ester of trimesic acid) has two absorption bands: a weak band at 221 nm and a stronger band at 279 nm; this pattern is characteristic of benzene derivatives with isolated chromophores.

In the IR spectra of **Ia-7–Ia-9**, **Ib**, **Ic-9**, and **Ie** (Table 2), the absorption bands of the starting compounds are absent, and characteristic bands of stretching and bending vibrations of ester groups appear; the spectra also contain out-of-plane bending vibration bands of the C–H groups of the benzene ring, with the pattern characteristic of 1,3,5- and 1,2,3-substitution.

Thermomicroscopic examination of compound specimens showed that only compounds **Ia-7**, **Ib**, **Ic-9**, and **Ie** passed into the mesomorphic state (Table 3), in agreement with the prediction results (Table 1). The predicted lack of the mesomorphism of **Ia-8** and **Ia-9** is also confirmed by the experimental data (Tables 1, 3). Examination of the behavior of thin specimens of **Ia-8** and **Ia-9** in polarized light in heating and cooling cycles has not revealed formation of any characteristic textures and manifestation of mesomorphic properties.

These compounds melt within a narrow temperature interval ($\leq 1^\circ\text{C}$). On cooling, crystals of dendritic (for **Ia-9**) or parallelepiped (for **Ia-8**) shape appear with a slight delay.

Compound **Ia-7** exhibits mesomorphic behavior during initial melting in the interval 54–58°C. However, in repeated heating, mesomorphic properties are no longer manifested.

Thermomicroscopic examination of a specimen of ester **Ic-9** in polarized light allows this compound to be classed with enantiotropic liquid crystals (Table 3). In the course of heating from 47 to 75°C, the specimen occurs in the anisotropic state, exhibiting flowability and forming on heating a flower-like texture (Fig. 2a) similar to that of the tenth homolog of hexahydroxytriphenylene ether and its nitro analog [7]. On cooling, the substance tends to strongly supercool, down to room temperature. Then spherulites of very regular round shape gradually grow. On pressing or shearing, a flow texture is observed in the form of columnar paths ending with regular spherulites, or of elongated strips formed from confocal domains (Fig. 2b). To identify the type of the supramolecular

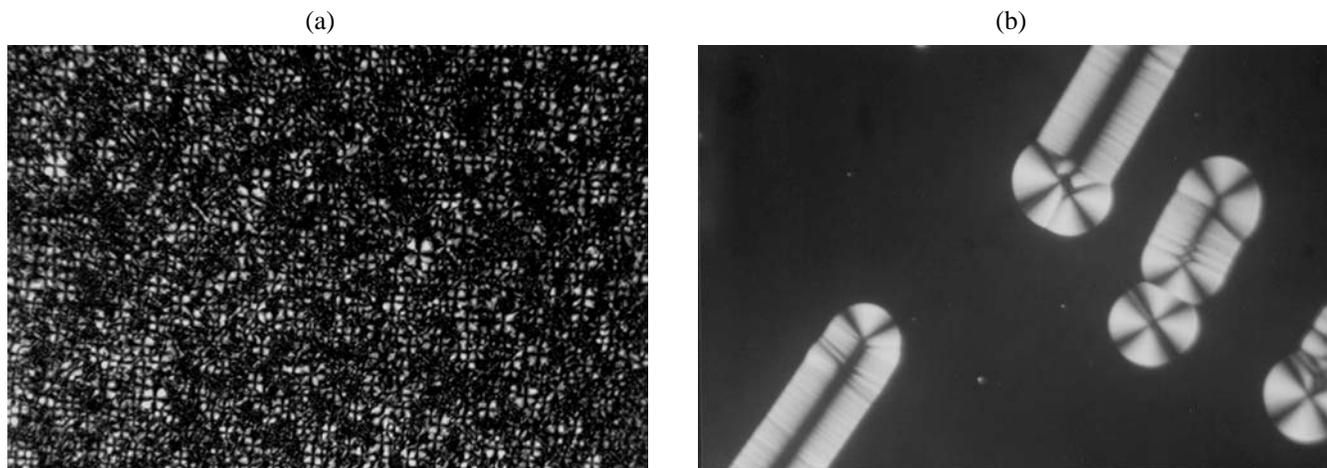


Fig. 2. Textures of trimesic acid ester **Ic-9**. Crossed nicols; $\times 160$. (a) Flower texture, 62°C, heating cycle; (b) cooling cycle, shear deformation, 20°C.

structure of **Ic-9** in the mesophase, we assessed its miscibility with other compounds showing different types of mesomorphism. In particular, we revealed mutual solubility of 1,3,5-tri(4-nonyloxyphenylcarboxy)benzene with 1-nitro-2,3,6,7,10,11-hexa(decyloxy)triphenylene in a contact sample in the mesophase (on heating and cooling). 1-Nitro-2,3,6,7,10,11-hexa(decyloxy)triphenylene, according to [18], is a discotic mesogen and forms a columnar supramolecular structure in the mesophase in the temperature range from 28 to 115°C. Thus, our results allow compound **Ic-9** to be classed with discotic mesogens with a columnar structure of the mesophase, the more so as its miscibility test with a calamitic liquid crystal, *p*-(*n*-octyloxy)benzylidene-*p*'-cyanoaniline, forming the nematic and smectic phases in the range from 65 to 78°C (*Cr* 65 *S_A* 78 *N* 86 *I*) [19], gave a negative result.

EXPERIMENTAL

The procedure for calculating the molecular parameters was the same as that described in [11, 12]. The capability of a compound to form columnar or mainly nematic structures was predicted using classification series Ia, Ib and the ChemCard program modulus [11]. The prediction results were checked by synthesis of particular representatives of compound series **Ia–If** and examination of their mesomorphic behavior by thermopolarization microscopy (an MIN-8 microscope equipped with a specially designed heating stage) and by miscibility tests with mesogens of different structural types.

The compounds synthesized were identified by elemental analysis and by UV (Specord UV–Vis) and IR (Avatar-360 FT-IR ESP) spectroscopy.

Below is a detailed protocol of the synthesis and

purification of 1,3,5-tri(4-nonyloxybenzoyloxy)benzene **Ia-9**. The compound was prepared by acylation of 1,3,5-trihydroxybenzene with 4-nonyloxybenzoyl chloride. A solution of 3.46 g of 4-nonyloxybenzoyl chloride (prepared beforehand) in 65 ml of anhydrous pyridine was added dropwise with vigorous shaking to 0.45 g of 1,3,5-trihydroxybenzene, and ~10 ml of benzene was added. The mixture slightly warmed up, and a white precipitate formed. The mixture was gradually heated to 50°C and kept at this temperature for 38 h. Then the mixture was cooled to room temperature and allowed to stand for several days. After that, the mixture was poured into 300 ml of 5% HCl. The target product was extracted from the solution with CCl₄ and washed with water to neutral reaction. After the water separation, the solvent was distilled off, and the residue (3.3 g) was purified by chromatography on alumina (eluent ethyl acetate) and recrystallized from ethanol. Yield 65%. Ester **Ia-9** is readily soluble in acetone, benzene, and ether; in ethanol it dissolves on heating. The UV spectrum is shown in Table 2. IR spectrum (KBr), cm⁻¹: 2923 [*v*_{as}(CH₂)], 2853 [*v*_s(CH₂)], 1731 [*v*(C=O), ester carbonyl], 1509, 1468, 1422 (skeleton vibrations of benzene ring); 1250, 1160 [*v*(C–O) of ester group], 842, 688 [δ (CH), out-of-plane benzene vibrations (1,3,5-substitution)]. Found, %: C 75.90; H 7.19. C₅₄H₇₂O₉. Calculated, %: C 74.95; H 8.40.

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