ISSN 1070-3632, Russian Journal of General Chemistry, 2007, Vol. 77, No. 1, pp. 103–110. © Pleiades Publishing, Ltd., 2007.
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Molecular Structure and Mesomorphism in the Series of Discotic Esters Derived from Benzene, Triazine, Biphenyl, and Triphenylene

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Received May 23, 2006

Abstract—New mesogenic discotic triazine-based esters were synthesized and shown to form a low-temperature monotropic columnar mesophase. Using as examples the newly synthesized compounds and previously prepared discotic esters on the basis of benzene, biphenyl, and triphenylene, the effects of the nature and size of the central fragment and the number, position, length, and rigidity of peripheral substituents therein on the mesomorphic properties (in particular, the ability to form columnar and nematic mesophases) were examined. The main structural factors stabilizing columnar and nematic mesophases were revealed. Nematic mesomorphism was shown to be favored by increased size of the central fragment of the discogen molecule and by the presence of cyclohexane fragments in peripheral substituents.

DOI: 10.1134/S1070363207010148

Discotic mesogens (DM) attract researchers' attention due to their prospective use as organic materials for new generation of electronic devices [1]. Studies on these compounds as self-organized systems are also interesting from the viewpoint of supramolecular chemistry and nanotechnology [2]. Search for qualitative and quantitative relations between their molecular structure and mesomorphic properties is important for purposeful synthesis of such mesogens. The principal aspects of studies on discotic mesogens were formulated in [3]. In the present work we examined a new series of compounds having structures of type **I**–**V** and analyzed the effects of such structural factors as the nature and size of the central fragment and the position, length, number, and rigidity of peripheral substituents on their mesomorphic properties.

Discotic mesogens **I**–**V** contain bridging ester moieties and central fragments having different sizes. Compounds **I** and **III** (Table 1) were synthesized previously [4–6] taking into account the results of preliminary prediction of a definite type of mesomorphic properties on the basis of molecular parameters [10, 11]. The data on mesomorphic properties of triphenylene derivatives **IV** and **V** (Table 1) were taken from [7, 9]. 1,3,5-Triazine derivatives **II** were synthesized according to Scheme 1.

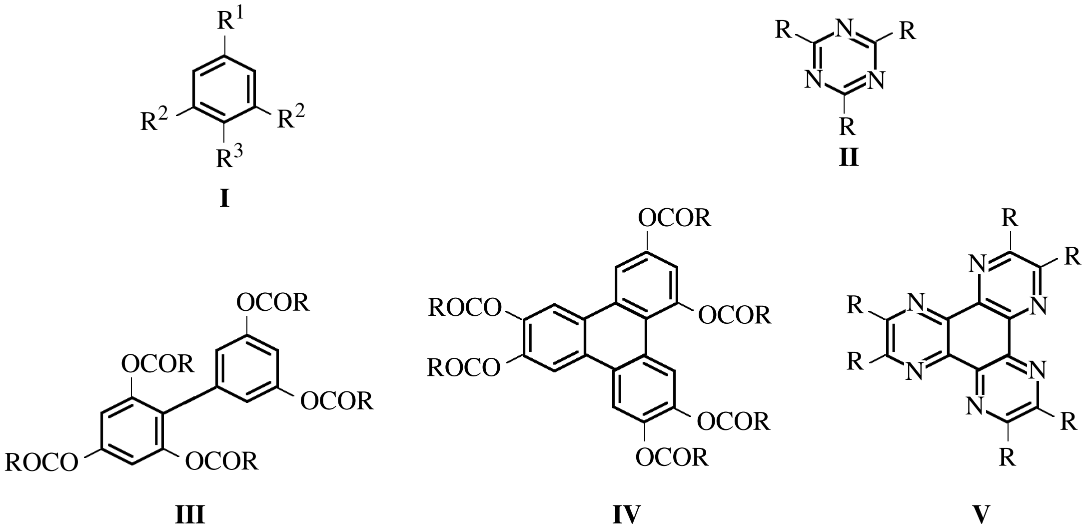
In keeping with the preliminary predictions made in [12], liquid crystalline properties in this series of compounds can be found starting from $n = 10$. A detailed procedure for the synthesis of the corresponding derivative and its identification data are given in Experimental.

The data of differential scanning calorimetry (DSC) for series II compounds showed the following phase transitions: crystal → isotropic liquid on heating and isotropic liquid → mesophase on third cooling. The second minimum typical of the transition mesophase → crystal was not observed on cooling, for all mesomorphic homologs crystallize below room temperature. The results of DSC are consistent with the thermomicroscopic observations (Table 2).

Examination of samples of compound **IIe** and 2,3,6,7,10,11-hexaoctyloxytriphenylene (**IX**) on slow cooling in the vicinity of the phase transition from isotropic liquid to mesophase showed that their textures are similar (Fig. 1). The supramolecular structure of triphenylene ether **IX** was reliably identified as hexagonal columnar ($\mathrm{Col_h}$) [3]. On the basis of the similarity in textures of compounds **IX** and **II** (Fig. 1) and the results of preliminary prediction related to the type of mesomorphism [12], we believe that the latter is characterized by the same packing of molecules in the mesophase as that found for **IX**, the more so both these compounds are miscible in the liquid crystalline state.

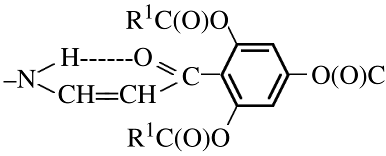
The effect of the nature of the central fragment on mesomorphic properties was examined using

Scheme 1.



I **II** **III** **IV** **V**

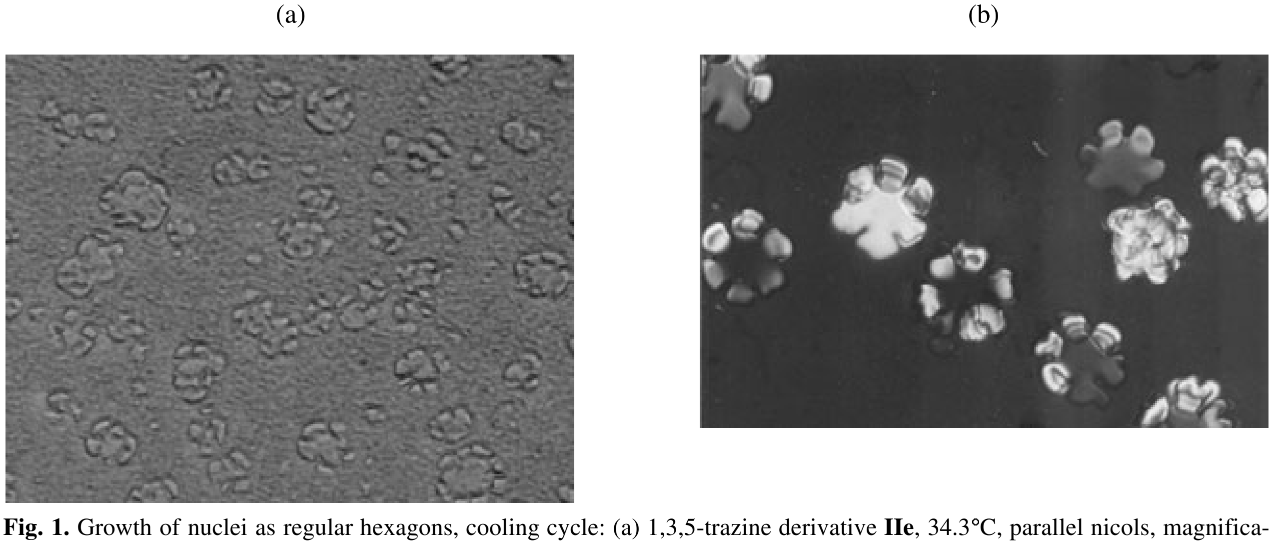
I, R^3 = H, $R^1 = R^2 = OCOC_6H_4OC_nH_{2n+1}$, n = 6 (**a**), 7 (**b**), 8 (**c**), 9 (**d**), 11 (**e**); R^3 = H, $R^1 = R^2 = C(O)OC_6H_4OC_nH_{2n+1}$, n = 9 (**f**); R^3 = H, $R^1 = R^2 = OCOC_6H_4C_6H_{11}$-*cyclo* (**g**); R^1 = H, $R^2 = R^3 = OCOC_6H_4C_6H_{11}$-*cyclo* (**h**).



II, R = $-N(H\text{------}O{=})\text{-}CH{=}CH\text{-}C\text{-}C_6H_2[OC(O)R^1]_2\text{-}O(O)CR^1$, $R^1 = C_nC_{2n+1}$, n = 8 (**a**), 9 (**b**), 10 (**c**), 11 (**d**), 12 (**e**), 14 (**f**), 16 (**g**).

III, R = $-C_6H_4OC_nH_{2n+1}$, n = 6 (**a**), 8 (**b**), 11 (**c**), R = $-C_6H_4-C_6H_{11}$-*cyclo* (**d**). **IV**, R = $-C_6H_4OC_nH_{2n+1}$, n = 6 (**a**), 8 (**b**), 10 (**c**), 11 (**d**), R = $-C_6H_4-C_6H_{11}$-*cyclo* (**e**). **V**, R = $-C_6H_4-H_{19}$.

the series including polysubstituted benzene **Ie**, 1,3,5-triazine **IId**, 1,3,3'5,5'-pentahydroxybiphenyl derivative **IIIc**, triphenylene **IVd**, and compound **V** (Table 3). Replacement of carbon atoms in the central fragment of 1,3,5-trihydroxybenzene ester **Ie** by nitrogen and of the ester groups by enamino (1,3,5-triazine **IId**) gives rise to monotropic columnar mesophase, whereas 1,3,5-tris(4-undecyloxybenzoyloxy)benzene (**Ie**) possesses no liquid crystalline properties (Table 1). We believe that the appearance of columnar mesophase is favored by the presence of intermolecular hydrogen bonds between the enamino groups of



(a) (b)

Fig. 1. Growth of nuclei as regular hexagons, cooling cycle: (a) 1,3,5-trazine derivative **IIe**, 34.3°C, parallel nicols, magnification ×400; (b) 2,3,6,7,10,11-hexaoctyloxytriphenylene (**IX**), 78°C, crossed nicols, magnification ×250 [3].

Table 1. Phase transition temperatures (°C) of discotic mesogens **I** and **III–V**[a]

Compound no.	n	Cr→M$_1$	M$_1$→M$_2$	M$_2$→N$_D$	N$_D$→I	Reference
Ia	6	–	60^b	66	–	[4]
Ib	7	–	54^b	58	–	[5]
Ic	8	–	54	–	–	[5]
Id	9	–	48	–	–	[5]
Ie	11	^{c}				[5]
If	9	–	47^d	75	–	[5]
Ig	6-*cyclo*	–	–	133	137	[5]
Ih	6-*cyclo*	102	140	–	157	[4]
IIIa	6	<20^d	–	93	212	[4]
IIIb	8	<20^d	–	74	214	[6]
IIIc	11	<20^d	–	79	174	[6]
IIId	6-*cyclo*	–	–	240	258	[4]
IVa	6	–	186^e	193	274	[7]
IVb	8	–	152^f	168	244	[7]
IVc	10	–	142^g	191	212	[7]
IVd	11	–	145^g	179	185	[7]
IVe	6-*cyclo*	–	–	288	340	[9]
V	9	–	81	–	–	[8]

[a] Hereinafter, Cr stands for crystalline phase, M for mesophase, N$_D$ for nematic discotic, and I for isotropic liquid. [b] The structure of the mesophase was not reliably determined. [c] Predicted as nonmesogen. [d] Columnar mesophase (Col). [e] Columnar tilted mesophase (Col$_t$). [f] Columnar rectangular mesophase (Col$_r$). [g] Columnar rectangular disordered mesophase (Col$_{rd}$).

neighboring triazine **II** molecules. This assumption is indirectly supported by the results reported in [13–16]. It was noted in these publications that hydrogen bonding between neighboring molecules in a stack favors ordering of mesophase formed by compounds having similar groups at the central fragment. By contrast, hexakis(4-nonylphenyl)dipyrazino[2,3-*f*:2',3'-*h*]quinoxaline (**V**) [9] which, like triazines **II**, contain nitrogen atoms in the central fragment but no enamine spacers does not form mesophase (Table 3). Thus, the presence of bridging enamino group in the series of triazine derivatives **II** is a factor stabilizing columnar mesophase. On the other hand, increase in the electron density on the central fragment in the series of benzene, biphenyl, and triphenylene derivatives: –0.118 (**I**) < –0.175 (**III**) < –0.607 (**IV**) stabilizes nematic mesophase.

Table 2. Phase transition temperatures of homologous 1,3,5-triazine-based discogens **II**

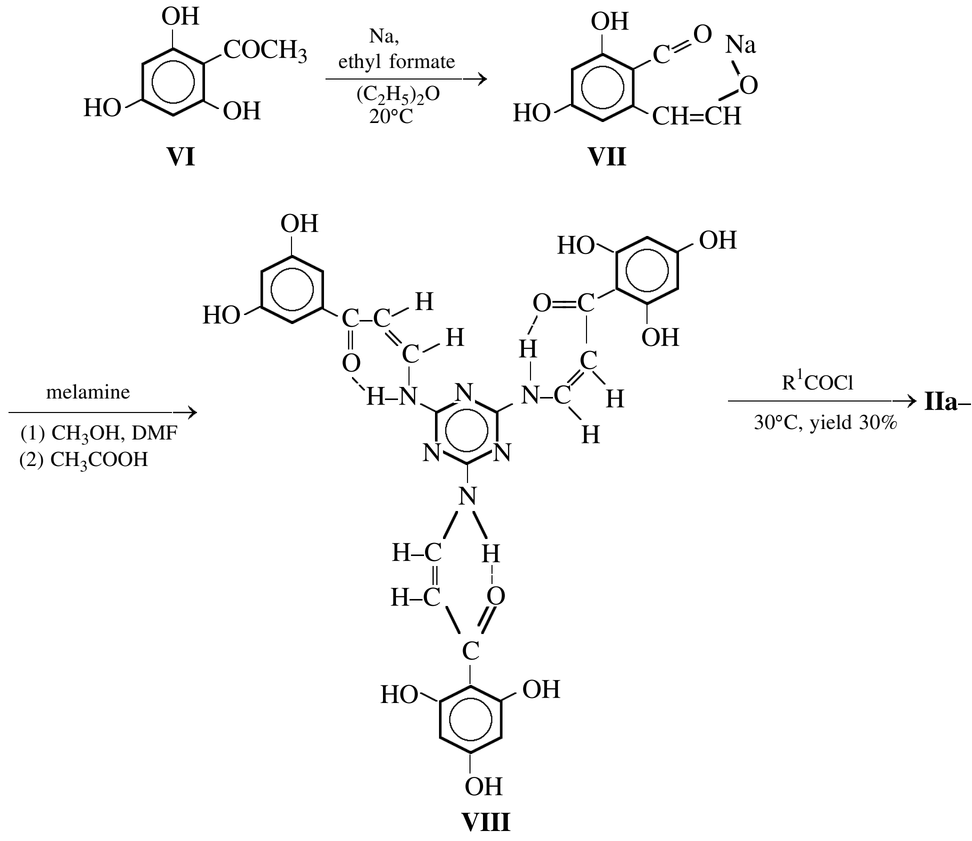
Comp. no.	n	Cr→M[a]	M→I
IIa	8	–	25.0
IIb	9	–	54.0
IIc	10	(29.0); (29.5)^b	33.0; 33.6^b
IId	11	(25.0)	28
IIe	12	(35.0); (35.5)^b	48.3; 42.5^b
IIf	14	(47)	52.6
IIg	16	(54.0); (56.1)^b	62.3; 61.7^b

[a] The temperature of monotropic phase transition is given in parentheses. [b] DSC data.

Effect of the number of benzene rings in a disc-like molecule and the size of its central fragment. Table 4 compares polysubstituted derivatives of benzene (**Ic**), biphenyl (**IIIb**), and triphenylene (**IVb**). It

Table 3. Temperature ranges of mesophases and inematic phase of compounds **I–V**

Comp. no.	Mesophase	Temperature range, °C	
		mesophase	nematic phase
Ie	Nonmesogen	0	0
IId	Col	>25	0
IIIc	Col, N$_D$	>154	95
IVd	Col$_r$, N$_D$	40	6
V	Nonmesogen	0	0



(1)

$R^1 = C_8H_{17}$ (**a**), C_9H_{19} (**b**), $C_{10}H_{21}$ (**c**), $C_{11}H_{23}$ (**d**), $C_{12}H_{25}$ (**e**), $C_{14}H_{29}$ (**f**), $C_{16}H_{32}$ (**g**).

is seen that increase in the number of benzene rings in the central fragment in going from benzene derivatives **Ia**–**Ie** to substituted biphenyls **IIIa**–**IIIc** and triphenylenes **IVa**–**IVd** leads to appearance of two-dimensional nematic-ordered phase (N_D) in addition to columnar mesophase (Col), i.e., esters like **IIIa**–**IIIc** and **IVa**–**IVd** exhibit polymorphism. On the other hand, the greater the cross-sectional area of the central fragment in the *XY* plane (S_{cf}^{XY}) of a discogen molecule, the broader the mesophase temperature range (ΔT_{mes}) and the higher the clearing temperature, including that of N_D mesophase (Table 4).

The effect of bridging groups on liquid crystalline properties was analyzed using as examples esters **Ia**–**If** with different oxygen–carbon sequences in the ester groups (the role of the bridging group in enaminoketone triazine derivative **II** was discussed above). Not all esters **Ia**–**Ie** are mesomorphic. Two-dimensional columnar mesophase was observed only for two members of this homologous series on heating in a narrow temperature range [5]. Trimesinic acid derivatives **If** with inverted ester linkers give two-dimensional columnar structures both on heating and on cooling, and the corresponding temperature ranges

Table 4. Effects of the number of benzene rings and the size of the central fragment on the mesomorphic properties in the series of compounds **I, III**, and **IV**

Comp. no.	Mesophase	Number of benzene rings	Temperature range, °C		S_{cf}^{XY},[a] Å
			mesophase	nematic phase	
Ic	Nonmesogen	1	0	0	62.23
IIIb	Col, N_D	2	>214	140	102.42
IVb	Colr, N_D	3	92	76	85.25

[a] S_{cf}^{XY} is the cross-sectional area of the central fragment in the *XY* plane.

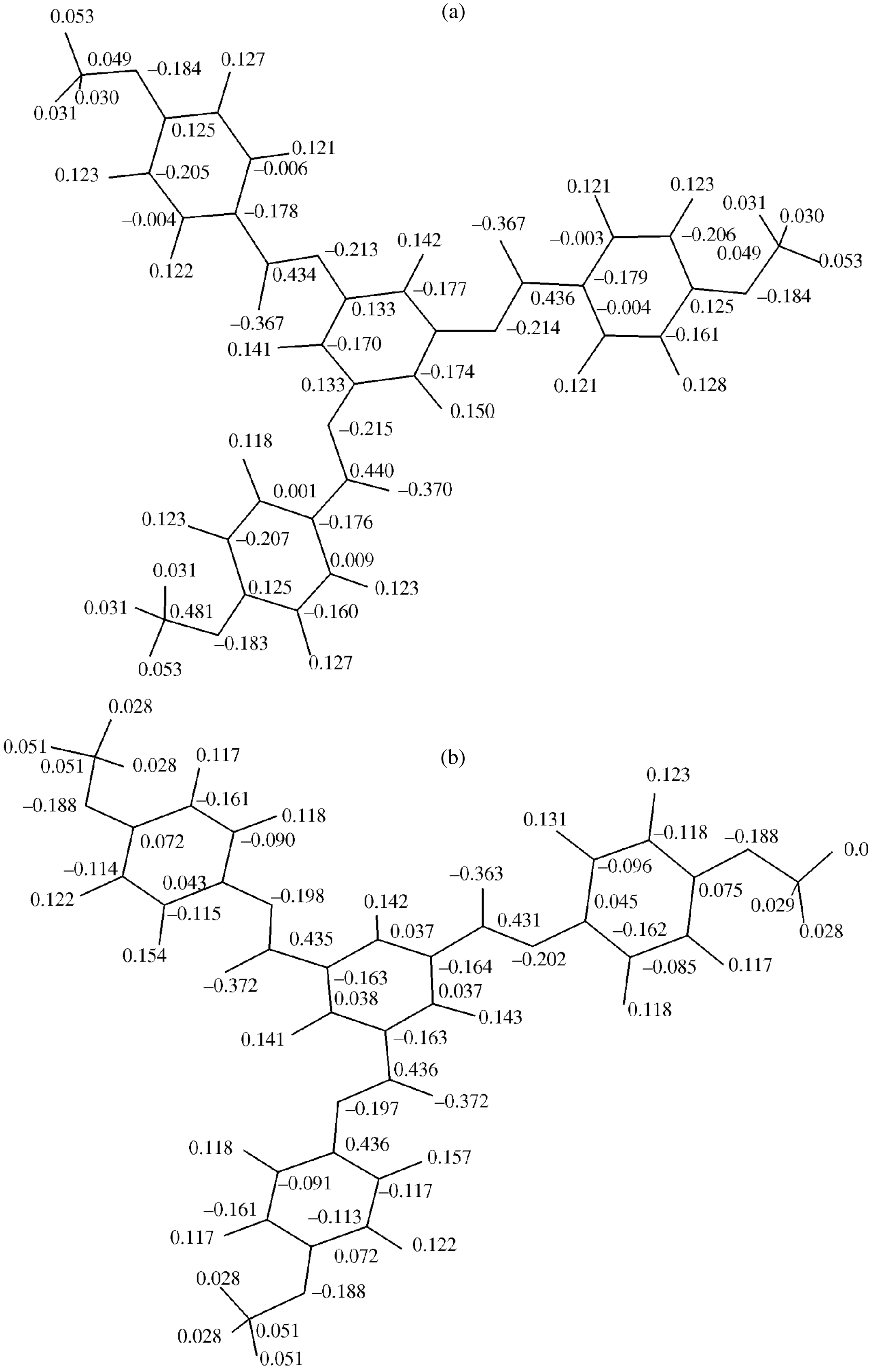


Fig. 2. Electron density distribution over the central fragments of ester molecules (a) **Ia**–**Ie** and (b) **If**; averaged electron densities on the central fragment $\bar{e}_{\mathrm{cf}}$ = –1.97 and –0.34 eV, respectively.

Table 5. Effect of the nature of the lateral substituent on the mesomorphic properties in the series of compounds **I**, **III**, and **IV**

Comp. no.	Mesophase	Temperature range, °C	
		mesophase	nematic phase
Ia	N_D	6	6
Ig	N_D	4	4
Ih	M_1, M_2	55	17
IIIa	Col_r, N_D	>192	>119
IIId	N_D	12	12
IVa	Col_t, N_D	88	81
IVe	N_D	52	52

are broader than those found for alkoxybenzoates **Ia**–**Ie** [5]. The observed behavior of esters **Ia**–**Ie** and If may be rationalized in terms of different electron density distribution over the molecular framework which (with account taken of microsegregation) includes the central and ester linkers. Semiempirical MM+ and PM3 calculations (HyperChem Pro 6.0) showed that the electron density on the central fragment of molecule **I** (R^3 = H, R^1 = R^2 = $OCOC_6H_4OCH_3$) is lower than in **I** [R^3 = H, R^1 = R^2 = $C(O)OC_6H_4OCH_3$] (Fig. 2). On the other hand, the specific electron density, i.e., the electron density per unit area of the central fragment cross-section in the *XY* plane ($\bar{e}_{\mathrm{TsF}}/S^{XY}_{\mathrm{TsF}}$) is larger for esters of the second type: –C(O)O–, $\bar{e}_{\mathrm{TsF}}/S^{XY}_{\mathrm{TsF}}$ = –0.046 eV E^{-2}; –OCO–, $\bar{e}_{\mathrm{cf}}/S^{XY}_{\mathrm{cf}}$ = –0.005 eV E^{-2} [4]. Presumably, increased specific electron density on the central fragment of trimesinic acid derivatives **If** should enhance π–π interactions between molecules in a column, i.e., should favor their stacking, as is in fact observed. In this case, enantiotropic mesomorphism is characterized by higher clearing temperatures (as compared to **Ia**–**Ie**) and broader temperature ranges.

The effects of the nature, number, position, and length of substituents was traced in the series of benzene (**I**), triazine (**II**), biphenyl (**III**), and triphenylene derivatives (**IV**). Within the homologous series of polysubstituted 1,3,5-triazines **II**, the length of the hydrocarbon radical exerts an appreciable effect on the type of textures formed: they vary from rod-like (*n* = 10–12) to looped columnar–helical (*n* = 14) and nongeometric (*n* = 16). The type of texture, as well as the number of mesophases and their type, of esters **IIIa**–**IIIc** derived from 1,3,3',5,5'-pentahydroxybiphenyl does not change as the hydrocarbon radical becomes longer, but an even–odd dependence of the $N_D \rightarrow I$ transition temperature is observed [6].

The effect of the lateral substituent nature is clearly seen while comparing compounds I, **III**, and **IV**, where R = $OCC_6H_4OC_6H_{13}$ or $OCC_6H_4C_6H_{11}$-*cyclo* (Table 5). Replacement of the alkoxy group in the series of polysubstituted 1,3,5-trihydroxybenzene derivatives **Ia**–**Ie** by cyclohexane fragment (compound **Ig**) favors formation of more thermally stable nematic phase whose temperature range approaches that typical of **Ia** (Table 5). By contrast, introduction of a cyclohexane fragment into the lateral substituent of 1,2,3-trihydroxybenzenebased ester **Ih** gives rise to bimesomorphism with simultaneous increase in the thermal stability of the mesophase (Table 5). Unlike 1,3,3',5,5'-pentakis(hexyloxybenzoyloxy)biphenyl (**IIIa**), 1,3,3',5,5'pentakis(cyclohexylbenzyl)biphenyl (**IIId**) shows only one mesomorphic transition to N_D phase which exists in a narrower temperature range but is more thermally stable. An analogous replacement in triphenylene derivative **IVe** leads to disappearance of two-dimensional columnar mesophase while nematic phase (N_D) is conserved but with a higher clearing temperature than that for alkoxy-substituted triphenylene-based benzoates **IVa**–**IVd**. Thus, the effect of the nature of lateral substituents on the mesomorphic properties of the compounds under study cannot be predicted a priori, and it strongly depends on the structure of the central fragment and position of the lateral substituent.

Increase in the number of lateral substituents and the size of the central fragment in the series of symmetrically substituted cyclohexyl derivatives of benzene (**Ig**), biphenyl (**IIId**), and triphenylene (**IVe**) leads to increase of the clearing temperature and broadening of the temperature range of nematic phase. It may be presumed that introduction of a relatively rigid cyclohexane fragment into peripheral substituents containing no flexible hydrocarbon moieties gives rise to compounds which are characterized mainly by nematic mesomorphism. The results of studies on discotic mesogens having both cyclohexane fragments and flexible hydrocarbon radicals in the lateral substituents [3] showed that such structures favor formation of only columnar mesophase. In our case, the observation of only nematic phase may be rationalized in terms of the presence of bulky cyclic fragments at the periphery of disc-shaped molecule and the absence of main factors favoring microsegregation [17] which should maintain supramolecular columnar ordering. Here, the shape anisometry is responsible for the conservation of only mutual spatial orientation of particular molecules which form nematic phase. We were thus the first to reveal a positive role of a rigid cyclohexane fragment in the lateral substituent in the formation of nematic phase.

Discotic mesogens **Ig** and **Ih** having similar lateral substituents but in different positions of the benzene ring (1, 3, 5 and 1, 2, 3, respectively) showed some differences in their mesomorphic behavior. Compound **Ig** gives nematic phase in a narrow temperature range (Table 5). Symmetry distortion in molecule **Ih** leads to bimesomorphism, some increase in the thermal stability of the mesophase, and extension of its temperature range (Table 5). The different behaviors of the above mesogens are likely to originate from specific features of their steric structure and steric factors following therefrom. Simulation and optimization of spatial models of molecules **Ig** and **Ih** suggest twinning of molecules Ih (with asymmetric structure and rotation of the benzene rings relative to the central fragment) upon stacking. As a result, molecules **Ih** are packed in columns with a shift, which should lead to twisting of columns. This assumption was confirmed by us while studying the texture of **Ih**, which appeared as helical fragments.

Analysis of the above data led us to draw the following conclusions:

(1) Enamine bridging groups stabilize columnar ordering of discotic triazine derivatives via formation of intermolecular hydrogen bonds. Increase in the electron density on the central fragment in the series of benzene (**Ia**–**Ie**), biphenyl (**IIIa**–**IIIc**), and triphenylene derivatives (**IVa**–**IVd**) stabilizes nematic phase;

(2) The mesophase temperature range for the compounds under study, including that of nematic phase, strongly depends on the size of the central fragment: the larger the latter, the wider the temperature range and the higher the thermal stability of the mesophase;

(3) Columnar ordering of molecules of benzene derivatives **Id** and **If** is favored by replacement of the –OC(O)– ester lether by –C(O)O–;

(4) The effect of lateral substituents on phase transitions in the examined series of discogens **I**, **III**, and **IV** cannot be estimated unambiguously; it is largely determined by the structure of the central fragment and the positions of the lateral substituents;

(5) Introduction of a cyclohexane fragment into peripheral substituents of a discogen molecule was shown for the first time to favor formation of nematic (N_D) mesophase. The presence of two cyclic fragments at the periphery of a discogen molecule, provided that flexible hydrocarbon radicals are absent, gives rise to bimesomorphism;

(6) As follows from a comparison of the mesomorphic properties of compounds **Ig** and **Ih**, distortion of molecular symmetry leads to expansion of the mesophase temperature range.

EXPERIMENTAL

The electronic absorption spectra were measured on a Specord UV-Vis spectrophotometer. The IR spectra (400–4000 cm^{-1}) were recorded in KBr on an Avatar-360 FT-IR ESP spectrometer. The ^1H NMR spectra were obtained from solutions in $CDCl_3$ on a Bruker Avance 250 spectrometer.

Mesomorphic properties were studied in the temperature range from –25 to 300°C by differential scanning calorimetry (DSC) using a Perkin–Elmer Diamond DSC instrument; in all experiments, the rate of heating/cooling was 1–5 deg min^{-1}. The textures and phase bahavior were examined by polarized microscopy using a Leitz Laborlux 12 Pol polarizing microscope coupled with a Mettler FP 82 hot stage (heating rate 2 deg min^{-1}). The texture images were obtained using a Wild MPS 51 camera equipped with 24×36-mm^2 microadapter.

The simulation and optimization of geometric and electronic models were performed by the MM+ and PM3 in methods in the semiempirical approximation in the HyperChem Pro 6.0.

1,3,5-Trihydroxyacetophenone (VI) was synthesized according to the procedure described in [18].

2,4,6-Tris{3-oxo-3-[2,4,6-tris(hydroxyphenyl)]-prop-1-en-1-ylamino}-1,3,5-triazine (VIII). Phloroacetophenone, 0.84 g, was dissolved in 50 ml of anhydrous diethyl ether, and 0.42 g of ethyl formate and 0.96 g of finely dispersed metallic sodium were added to the solution. The mixture was stirred for 20–25 h at room temperature (it gradually turned turbid and cream-colored). The solvent and excess ethyl formate were distilled off under reduced pressure (water-jet pump), the residue (compound **VII**) was dried and dissolved in 30 ml of methanol, and the solution was added to a mixture of 0.21 g of 1,3,5-triazine-2,4,6-triamine in 25 ml of dimethylformamide. The resulting orange solution was acidified with acetic acid, heated to 30–40°C over a period of 20 min, cooled to room temperature, and kept for 1–2 days in a refrigerator. The cream-colored crystals of enaminoketone **VIII** were filtered off in a refrigerator. Evaporation of the filtrate gave an additional amount of a reddish product **VIII**. Overall yield 0.5 g.

2,4,6-Tris{[3-oxo-3-[(2,4,6-tris(decanoyloxy)-phenyl]prop-1-en-1-ylamino}-1,3,5-triazine (IIc). Decanoyl chloride, 8.5 mmol, was added to a solution of 0.5 g of compound **VIII** in 50 ml of anhydrous pyridine. The mixture was stirred for 24 h at a temperature not exceeding 55°C and poured into water acidified with hydrochloric acid. The cream-colored precipitate was filtered off and dried in air. Yield 35–40%. The product was purified by repeated column chromatography on silica gel L (100–250 μm) using hexane as eluent. Yield of the purified product 25%. Compound **IIc** is readily soluble in acetone, benzene, diethyl ether, and hexane. UV spectrum (chloroform), λ_{max}, nm (log ε): 320 (5.19), 265 (4.10). IR spectrum (KBr), ν, cm^{-1}: 1711 (C=O, ester); 1606 (C=O, ketone); 1263 (–C–O); 1049 [C–C(O)–C]; 880 (δ_{CH}, out-of-plane, 1,3,5-substitution); ^1H NMR spectrum, δ, ppm: 0.85–0.87 m (9H, CH_3), 1.26 s [126H, $(CH_2)_7$], 7.26 m (2H, H_{arom}). Found, %: C 69.20; H 8.80. $C_{120}H_{186}N_6O_{21}$. Calculated, %: C 70.35; H 9.15.

ACKNOWLEDGMENTS

This study was performed under financial support by the program “Development of the Mechanisms for Integration of Educational and Research Processes in the Field of Nanomaterials” (project no. RNP 2.2.1.1. 7280).

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