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Effect of the Nature of Chiral Fragment on Mesomorphic Properties of Triphenylene Ethers

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Abstract—A number of new 2,3,6,7,10,11-hexakis(heptyloxy)triphenylenes possessing a chiral substituent in position *1* of the triphenylene ring system and characterized by predictable mesomorphism were synthesized. The effect of the nature of the chiral fragment (dehydrocholic, lipoic, and *d*-tartaric acid residues) on the mesomorphic properties of triphenlylene ethers was studied. *N*-[2,3,6,7,10,11-hexakis(heptylozy)triphenylen-1-yl]amides derived from the above acids, unlike initial monomesomorphic amine, exhibit enantiotropic columnar polymorphism and give rise to helical columnar structure at reduced temperature. The newly synthesized amides are characterized by lower thermal stability and broader temperature range of the mesophase. Introduction of two chiral dehydrocholic acid fragments into the triphenylene core favors columnar (including chiral) mesomorphism.

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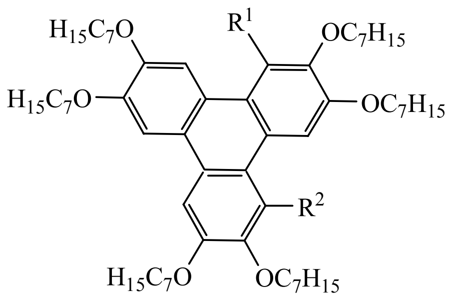
Discotic mesogens (DM) have been extensively studied over a period of more than 30 years [1–9]. A considerable number of publications were concerned with the synthesis of triphenylene derivatives which formed columnar and nematic mesophases [10–13], in particular, their photoconducting properties in the mesophase were studied [14]. Network-like polymeric films were recently synthesized from monomeric nematic triphenylene derivatives. The obtained films showed a relatively high charge mobility and were used in an electroluminescent device [15]. Ogoshi et al. [16] synthesized star-shaped polyoxazoline liquid crystals on the basis of triphenylene derivatives. Discotic mesogens are promising materials for the design of molecular conductors in various optical and electronic devices, such as pattern-tracing machines, laser printers, photoelectric elements, light-emitting diodes, field transistors, and holographic data storage systems [4]. Search for new chiral discotic mesogens and study on the relations between their structure and chiral mesomorphism remain so far important problems [17], taking into account poor information on such compounds and their potential applications in practice.

We previously reported [18] on the design of a series of triphenylene derivatives containing chiral fragments, which were predicted to form columnar and chiral mesophases, and synthesized two derivatives with predictable columnar chiral mesomorphism. In continuation of our studies on new discotic chiral mesogens on the basis of substituted triphenylene derivatives containing various chiral substituents in position *1*, in the present work we made an attempt to synthesize new triphenylene derivatives **I–III**. Compounds **I–III** were predicted to display columnar (Col) and chiral types of mesophases using a procedure which was developed by us previously by analysis of almost 3000 disc-shaped compounds with a known type of mesomorphism and tested by the synthesis of more than 100 new discotic mesogens [19].

Using a series of chiral and achiral mesogenic and non-mesogenic disc-shaped compounds we previously showed [17] that the procedure for prediction of mesogenic properties [4, 19–26] on the basis of the structure of particular molecules and calculation and analysis of their molecular parameters is generally applicable to disc-shaped structures. However, to



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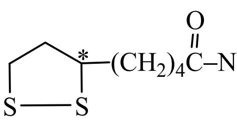
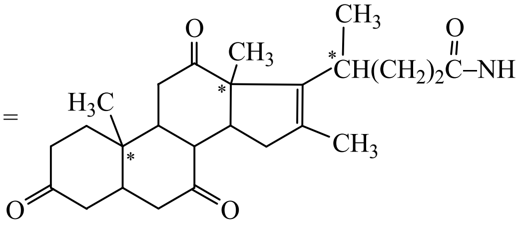
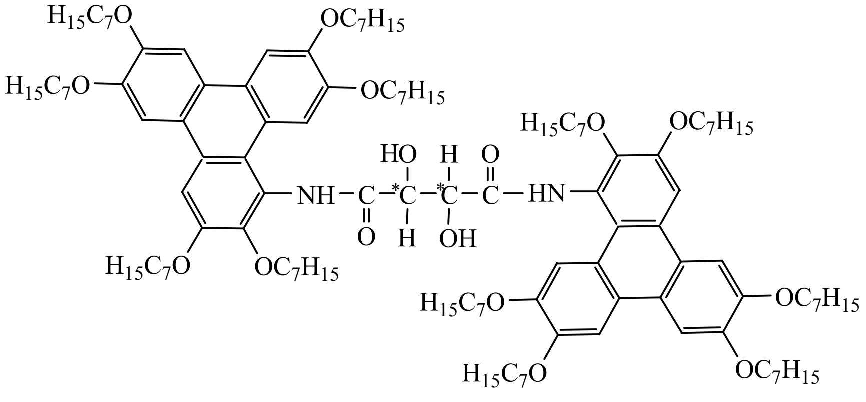
I, R^1 = H, R^2 =  ; **II**, R^1 = R^2 = 



III

distinguish a subclass consisting of chiral discotic mesogens it was necessary to use other informative molecular parameters. Such parameters were proposed by us [17] and tested on a series of triphenylene derivatives [18]. Following the developed procedures, we calculated and analyzed molecular parameters of compounds **I**–**III** with a view to determine the possibility for these compounds to form columnar and chiral mesophases (Tables 1, 2).

Taking into account that the calculated molecular parameters of the designed triphenylene derivatives **I**–**III** (Table 1) fall into the ranges typical of generic series (*1*) [2, 4, 17–20], they should exhibit meso-

Table 1. Molecular parameters and prediction of mesomorphic properties of triphenylene derivatives **I**–**III**[a]

Comp. no.	E, kcal mol^{-1}	M_m	M_r	K_s	K_{ar}	K_p	K	P_{Col+N}
I[b]	87.96	0.46	0.27	1.05	0.244	0.47	2.56	+
II	170.31	0.30	0.18	1.10	0.245	0.36	4.02	+
III[c]	118.05	0.66	0.38	1.39	0.340	0.79	2.98	+
III[d]	133.43	0.66	0.38	2.28	0.127	1.16	2.16	–

[a] K_s = 0.58 is the substitution parameter; P_{Col+N} stands for prediction of mesomorphism intrinsic to discotic mesogens; M_m is the molecular-weight parameter; M_r is the reduced molecular-weight parameter; K, K_c, and K_p are parameters characterizing anisometry of the whole molecule and its particular fragments; K_{ar} is the parameter characterizing the density of surrounding by peripheral substituents. For more detail, see [2, 4, 17–20]. [b] Data of [18]. [c] *cis* Conformer. [d] *trans* Conformer.

Table 2. Prediction of chiral mesomorphism for triphenylene derivatives **I**–**III**[a]

Comp. no.	$K_{\text{ch.c}}$	$K_{\text{ch.l}}$	$K_{\text{ch.p}}$	P_{Ch}
I	0.14	0.23	0.66	–
II[b]	0.75[c]	0.13	0.29	±
II[d]	0.75[c]	0.22	0.48	±
II[e]	0.75[c]	0.36	0.75	±
III	0.14	0.045[c]	0.39	±

[a] $K_{\text{ch.c}}$ is the parameter reflecting the number of chiral centers in the molecule; $K_{\text{ch.l}}$ characterizes remoteness of chiral center from the central fragment of the molecule; $K_{\text{ch.p}}$ is the parameter taking into account the length of the hydrocarbon radical containing chiral center; P_{Ch} stands for prediction of chiral mesomorphism. [b] The parameters were calculated assuming the presence of one chiral center in one dehydrocholic acid fragment. [c] The parameters falls into the range typical of generic series (*2*). [d] The parameters were calculated assuming the presence of two chiral center in two substituents. [e] The parameters were calculated assuming the presence of three chiral center in three substituents. The formulas for calculation of these parameters were given in [17–19].

morphic properties typical of discotic mesogens. Two conformations were considered for triphenylene derivative **III** having a tartaric acid fragment. The first of these is characterized by maximally extended structure (*trans*, Fig. 1a), and in the second conformation the triphenylene fragments are spatially as close as possible (*cis*, Fig. 1b). As follows from the calculated parameters (Table 1), the *cis* conformer of **III** is preferred for the formation of columnar order in mesophase.

$$K = 2.0\text{–}8.5;\ K_c = 1.0\text{–}2.6;\ K_p = 0.2\text{–}0.7;\ K_s = 0.25\text{–}1.0;$$
$$K_{\text{ar}} = 0.08\text{–}0.45;\ M_m = 0.3\text{–}0.8;\ M_r = 0.15\text{–}0.8. \qquad (1)$$

Tables 1 and 2 contain the molecular parameters and predictions of general and chiral mesomorphism for amide **I** according to the data of [18]. Calculation and analysis of molecular parameters [generic series (*2*) corresponding to chiral mesogen subclass among discotic mesogens] showed (Table 2) that it is impossible to unambiguously predict the ability of compounds **I**–**III** to form chiral mesophase. Though some parameters taken separately suggest that the formation of chiral supramolecular structures is possible, the three parameters in total indicate considerably lower probability for chiral mesomorphism of compounds **I**–**III**. Therefore, equally probable predictions are given for triphenylene derivatives **II** and **III** with dehydrocholic and tartaric acids (Table 2).

$$K_{\text{ch.c}} = 0.20\text{–}1.33;\ K_{\text{ch.l}} = 0.010\text{–}0.100;$$
$$K_{\text{ch.p}} = 0.03\text{–}0.25. \qquad (2)$$

In order to verify the above predictions and estimate the effect of the nature of the chiral fragment on liquid crystalline properties of discotic mesogens, triphenylene derivatives **I**–**III** were synthesized according to Scheme 1. In the nitration step (*iii*) we obtained a mixture of mono- and dinitrotriphenylene derivatives **VII** and **VIII** which were separated by column chromatography on γ-alumina. Dinitro compound **VII** was isolated from the first fraction, and the second fraction contained mononitro derivative **VIII**. The separation procedure was analogous to that reported in [20]. The target products, *N*-acyl-2,3,6,7,10,11-hexakis(heptyloxy)triphenylen-1-amines **I**–**III**, were synthesized according to modified procedure [20] and were purified by column chromatography on silica gel (gradient elution with hexane–ethyl acetate mixtures), followed by recrystallization from ethanol. The products were isolated as beige–

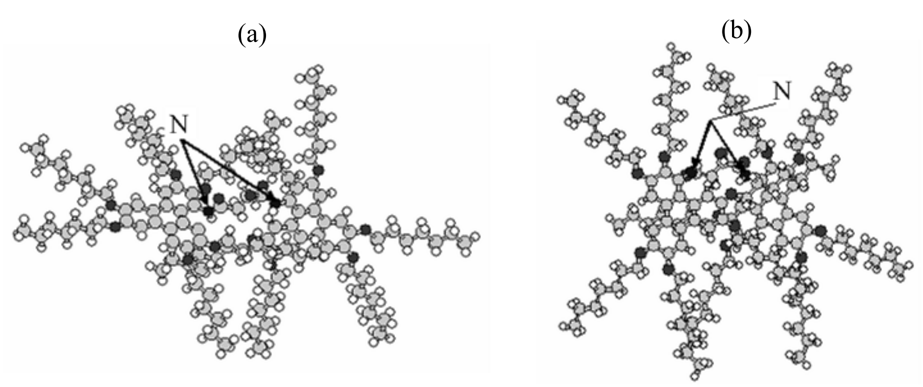
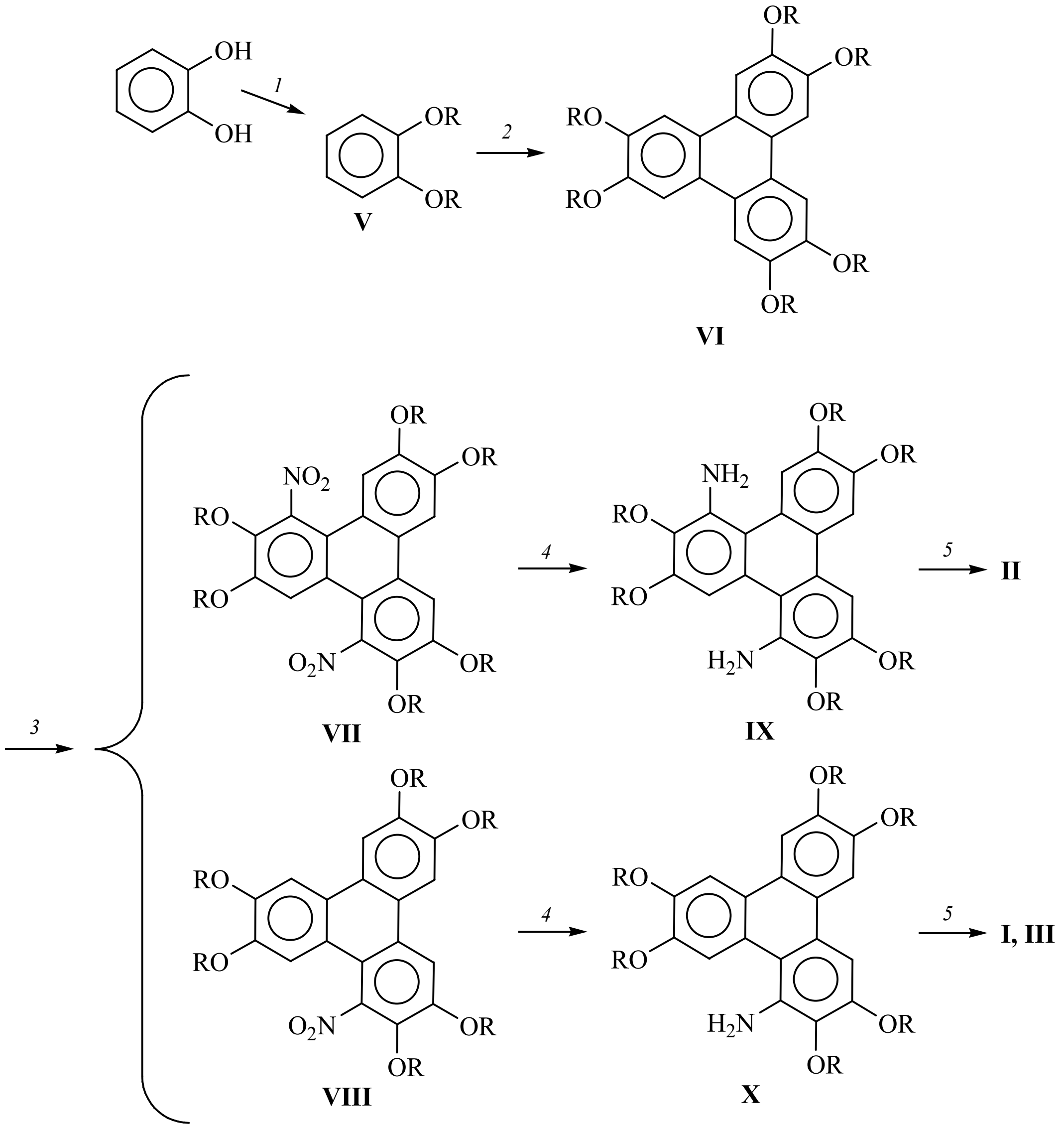


Fig. 1. Structures of the molecule of chiral triphenylene derivative **III** optimized by the MM$^+$ method (HyperChemPro 6.0): (a) *trans* conformer and (b) *cis* conformer. White circles correspond to hydrogen atoms, gray circles, to carbon, and dark circles, to oxygen.

Scheme 1.



R = C_7H_{15}, *i*: $C_7H_{15}I$, KF, KOH, DMF, phase-transfer catalyst [21]; *ii*: tetrachloro-1,4-benzoquinone, 81% H_2SO_4, hexane, phase-transfer catalyst, 25°C, 5–7 days [22]; *iii*: HNO_3–AcOH (1 : 7.5), diethyl ether, 20 min [23]; *iv*: Sn, AcOH, CH_2Cl_2, 15 h [23–25]; *v*: R^1COOH, CH_2Cl_2, Et_3N, *N*,*N*′-dicyclohexylcarbodiimide (DCC), 4-dimethylaminopyridine (DMAP), 5–7 h, 25–30°C [20].

pinkish substances. The detailed procedures for the synthesis and purification of amides **I**–**III** are given in Experimental. Their purity was checked by HPLC, and the structure was confirmed by the electronic absorption, ^{1}H NMR, and IR spectra.

Samples of compounds **I**–**III** were examined by thermal polarizing microscopy (Fig. 2) and differential thermal analysis (DTA, Table 3). The results of thermal microscopic studies showed that amides **I**–**III** exhibit low-temperature enantiotropic dimesomorphism. No characteristic textures were revealed during the first heating run, so that the type of mesophase was not identified. Furthermore, we failed to observe polymorphic transitions; these transitions were revealed only upon repeated heating and cooling (Fig. 2), as well as by differential scanning calorimetry (DSC, Table 3).

The DSC curve of lipoic acid derivative **I** upon first heating run contained three well resolved peaks which, according to the thermal microscopic data at 13.5°C,

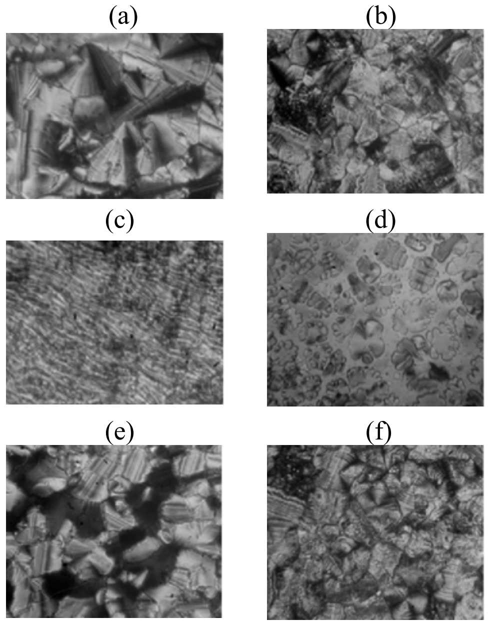


Fig. 2. Textures of chiral triphenylene derivatives (a–c) with lipoic (**I**) and (d–f) *d*-tartaric acids (**III**); magnification ×200; (a–c, e, f) crossed nicols, (d) parallel nicols: (a) pseudofocal–conical texture in the cooling run, 20°C; (b) growth of flower-shaped domains with circular hatching during the transition from hexagonal columnar mesophase to twisted columnar (chiral) mesophase upon cooling (19°C); (c) fingerprint texture at 15°C on cooling; (d) rod-like domains in the Col_h mesophase; (e) mosaic texture with longitudinal hatching; (f) helical domains together with those characterized by cross hatching at 20°C on heating.

corresponded to the transition from the crystalline phase to chiral columnar mesophase (Figs. 2b, 2c; Table 3). Further heating (27.9°C) revealed transition to columnar hexagonal mesophase which persisted up to 42.6°C (Fig. 2a). During the cooling run, the samples were considerably overcooled, and the first phase transition for compound **I** was observed at 2.8° C, i.e., at a temperature lower by almost 40°C than on heating. Transition into chiral mesophase was observed at –10°C, and this phase persisted down to –43.7°C. According to the DSC data, analogous transformations occurred with dehydrocholic and *d*-tartaric acid derivatives **II** and **III**. Figure 2 shows characteristic textures of samples of **I** and **III**, which indicated two phase transitions with columnar hexagonal and possible helical columnar ordering. Examination of bis-amide **II** revealed two typical textures: rod-like domains were observed in the high-temperature region near the transition from isotropic liquid to mesophase, and at lower temperature (20°C and below) banana-shaped domains appeared with cross hatching which characterized twisting of columns in the mesophase.

Thus the experimental data are very consistent with the predictions made for general mesomorphism typical of discotic mesogens (Table 3). However, some differences exist between the experimental data and predictions for chiral mesomorphism (Table 3), which are likely to be related to insufficiently informative parameters and small number chiral mesogens of that type synthesized up to now.

Our results allowed us to class amides **I**–**III** with discotic dimesomorphic mesogens which give rise to low-temperature chiral columnar mesophase (Col_{Ch}). The broadest mesophase temperature range was observed for bis-dehydrocholic acid amide **II** both on

Table 3. Phase transition temperatures (°C) and enthalpies[a] (Δ*H*, J/g) of chiral triphenylene derivatives **I**–**III** according to the DSC data, predictions of their mesomorphic properties, and experimental mesomorphism[b]

Comp. no.	Cr		Col_{x1}		Col_h		I	P_{KM+N}	E_{KM+N}	P_{Ch}	E_{Ch}
I, heating	•	13.5 (29.3)	•	27.9	•	42.6	•	+	+	–	+
cooling	•	–43.7	•	–8.2	•	2.8 (16.8)	•				
II, heating	•	12.2	•	42.0 (24.1)	•	59.6	•	+	+	±	+
cooling	•	–60.6 (–0.48)	•	–25.6	•	–4.7	•				
III, heating	•	13.2 (10.8)	•	25.5	•	44.0 (28.0)	•	+	+	±	+
cooling	•	–50.5[c]			•	0.24 (–12.6)	•				

[a] In parentheses. [b] P_{Ch} stands for prediction of chiral mesomorphism, and E_{Ch} stands for experimental mesomorphism. [c] Vitrification.

heating and on cooling. The same mesogen is also characterized by the broadest temperature range for chiral supramolecular structure. Thus introduction of two additional chiral fragments into triphenylene core favors both general and chiral mesomorphic properties.

EXPERIMENTAL

The molecular parameters were calculated according to the procedure described in [4, 18–26]. Predictions for compounds **I**–**III** to form columnar structures were made in terms of generic series (*1*) with the aid of ChemCard program [21]. Mesomorphic properties of compounds **I**–**III** were studied by thermal polarizing microscopy using an MIN-8 microscope equipped with a custom-made hot stage and a photo attachment.

HPLC analysis was performed on a Liquochrom Model 2010 liquid chromatograph equipped with a Beckman Model 110A pump and a UV-Detector 308 spectrophotometric detector (spectral range 180–440 nm). The optical density was measured at λ 328 nm. A 250×4-mm column was packed with Separon SGX C18 (average grain size 7 μm); the column was eluted at 25°C with *n*-hexane at a flow rate of 0.5 ml/min. The chromatograms were processed using Mul'tikhrom 1.5 program.

The electronic absorption spectra were recorded on a Varian Cary-100 spectrophotometer; samples were placed into rectangular quartz cells with a cell path length of 10 mm. The IR transmission spectra were measured on an Avatar 360 FT-IR ESP instrument from samples prepared as KBr pellets. The ^{1}H NMR spectra were obtained on a Bruker AC-200 spectrometer at 200.13 MHz using carbon tetrachloride as solvent and tetramethylsilane as internal reference.

Phase transitions were analyzed by differential scanning calorimetry using a Netzch DSC 204 F1 instrument; aluminum cell, sample weight ~20 mg, heating under nitrogen from –110 to 100°C at a rate of 10 deg/min.

The procedures for the synthesis and purification of pyrocatechol and triphenylene derivatives **V**–**X** were given in [18–25].

(*R*)-5-(1,2-Dithiolan-3-yl)-*N*-[2,3,6,7,10,11-hexakis(heptyloxy)triphenylen-1-yl]pentanamide (I). A solution of 0.1 g of lipoic acid, 0.5 g of compound **X**, 0.13 g of *N*,*N'*-dicyclohexylcarbodiimide, and 0.073 g of 4-dimethylaminopyridine in 30 ml of a mixture of anhydrous methylene chloride and triethylamine (1:1) was stirred for 7 h at room temperature. The mixture was diluted with a threefold volume of methanol–water (20:1), and the slightly greenish precipitate was filtered off, washed with alcohol, dried, and subjected to chromatography on silica gel (5–40 μm) using hexane–ethyl acetate (50:1) as eluent. The eluate was evaporated under reduced pressure (water-jet pump) to isolate 0.48 g (80–83%) of a beige–pinkish substance which was soluble in hexane, chloroform, acetone, and ethyl acetate. The product was additionally purified by recrystallization from ethanol. UV spectrum (petroleum ether), λ, nm (logε): 370 (3.59), 350 (3.68), 272 (4.52), 215 (4.29). IR spectrum (KBr), ν, cm^{-1}: 3324–3398 (NH); 1740 (C=O); 1611, 1517, 1429 (C=C$_{\text{arom}}$); 1650 (C=O, amid); 1545 (δNH); 1263 (C–N). ^{1}H NMR spectrum (CDCl$_3$), δ, ppm: 0.98 s (21H, CH$_3$), 1.34 m (14H, C**H**$_2$CH$_3$), 1.56 t (48H, CH$_2$), 1.92 m (6H, CH$_2$C**H**), 2.05 d (2H, CH), 4.13 m (2H, OCH$_2$), 4.21 m (12H, OCH$_2$), 7.37 s (1H, H$_{\text{arom}}$), 7.78 s (1H, H$_{\text{arom}}$), 7.794 s (1H, H$_{\text{arom}}$), 7.827 s (1H, H$_{\text{arom}}$), 8.81 s (1H, NHCO). Found, %: C 73.11; H 10.53; N 1.56. $C_{68}H_{109}O_7N_1S_2$. Calculated, %: C 73.13; H 9.86; N 1.25.

***N*,*N'*-2,3,6,7,10,11-Hexakis(heptyloxy)triphenylene-1,5-diyl]bis(16-methyl-3,7,12-trioxochol-16-en-24-amide) (II).** A solution of 0.44 g of dehydrocholic acid, 0.50 g of compound **IX**, 0.25 g of *N*,*N'*-dicyclohexylcarbodiimide, and 0.15 g of 4-dimethylaminopyridine in 25 ml of anhydrous methylene chloride was stirred for 8 h at room temperature. The mixture was then diluted with ethanol–water (15:1) until it became opalescent. After cooling, the light beige precipitate was filtered off, washed with cold alcohol, dried, and subjected to chromatography on silica gel (5–40 μm) using hexane–ethyl acetate (50:1) as eluent. Yield 0.55 g (60–65%); the product was soluble in hexane, chloroform, acetone, and ethyl acetate. It was additionally purified by recrystallization from ethanol. UV spectrum (petroleum ether), λ, nm (logε): 370 (4.31), 353 (4.37), 304 (4.94), 277 (5.41), 213 (4.98). IR spectrum (KBr), ν, cm^{-1}: 3317–3404 br (NH), 1771 (C=O, ketone), 1733 (C=O), 1700 (C=C), 1650 (C=O), 1544 (δ$_{\text{NH}}$), 1261 (C–N), 1250 (C–C=). ^{1}H NMR spectrum (CDCl$_3$), δ, ppm: 0.71 s (24H, CH$_3$), 0.91 s (18H, CH$_3$), 1.25 m (4H, CH$_2$), 1.35 m (14H, C**H**$_2$CH$_3$), 1.56 t (36H, CH$_2$), 1.92 m (12H, CH$_2$), 4.21 m (12H, OCH$_2$), 5.16 s (1H, CH), 5.89 q (C**H**$_2$CH), 7.37 s (1H, H$_{\text{arom}}$), 7.75 s (3H, H$_{\text{arom}}$), 7.70 s (3H, H$_{\text{arom}}$), 7.79 s (3H, H$_{\text{arom}}$), 7.83 s (3H, H$_{\text{arom}}$), 8.81

s (2H, NHCO), 8.92 s (2H, H_{arom}). Found, %: C 76.61; H 10.09; N 1.44. $C_{110}H_{162}O_{14}N_2$. Calculated, %: C 76.08; H 9.42; N 1.61.

***N,N′*-Bis[2,3,6,7,10,11-hexakis(heptyloxy)triphenylen-1-yl]-2,3-dihydroxybutanediamide (III).** A solution of 0.075 g of tartaric acid, 1.02 g of compound **X**, 0.25 g of *N,N′*-dicyclohexylcarbodiimide, and 0.073 g (0.06 mmol) of 4-dimethylaminopyridine in 30 ml of a 1:1 mixture of methylene chloride and triethylaminewas stirred for 7 h at 30–35°C. The mixture was then diluted with a threefold volume of ethanol–water (20:1), and the precipitate (cherry tinted) was filtered off, washed with alcohol, dried, and subjected to chromatography on silica gel (5–40 μm) using hexane–ethyl acetate (50:1) as eluent. Yield 0.90 g (80–84%). The product was soluble in hexane, chloroform, acetone, ethyl acetate, and ethanol on heating. It was additionally purified by recrystallization from ethanol. UV spectrum (petroleum ether), λ, nm (logε): 371 (3.96), 354 (4.01), 305 (4.54), 277 (4.95), 215 (4.47). IR spectrum (KBr), ν, cm^{-1}: 3307–3393 (NH, OH); 1768 (C=O); 1615, 1517, 1430 ($C=C_{arom}$); 1652 (C=O); 1547 (δNH); 1262 (C–N). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.98 s (21H, CH_3), 1.34 m (14H, $C\mathbf{H}_2CH_3$), 1.56 t (48H, CH_2), 1.92 m (6H, $C\mathbf{H}_2CH$), 2.05 d (2H, CH), 4.13 m (2H, OCH_2), 4.21 m (12H, OCH_2), 7.37 s (1H, H_{arom}), 7.78 s (1H, H_{arom}), 7.794 s (1H, H_{arom}), 7.827 s (1H, H_{arom}), 8.81 s (1H, NHCO). Found, %: C 76.37; H 10.37; N 1.68. $C_{124}H_{196}O_{16}N_2$. Calculated, %: C 75.56; H 10.04; N 1.42.

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