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Nucleophilic Substitution in 4-Bromo-5-nitrophthalodinitrile: XI.[1] Preparation, Properties, and Prediction of Mesomorphism in Mixed-Substituted Phthalocyanines Containing Aryloxy and Benzotriazole Fragments

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Abstract—Stepwise nucleophilic substitution of bromine and nitro group in 4-bromo-5-nitrophthalodinitrile has led to a series of phthalonitriles containing benzotriazole and aryloxy fragments; basing on them, the mixed-substituted phthalocyanines have been prepared. The spectral properties of products have been studied. According to simulation of columnar mesomorphism only one of the products is not capable of mesomorphism characteristic of discotic mesogens; the result has been confirmed with the experiment.

Keywords: phthalocyanine, simulation, mesomorphism, molecular descriptor

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Phthalocyanines, synthetic analogs of natural heterocyclic porphyrins, are attractive as promising dyes, catalysts, and sensor materials [2–4]. Moreover, phthalocyanines and their derivatives, basing on the physicochemical properties, can be applied as photoconductive materials in laser printers, LCDs, optical recording devices, optical light diodes, and as liquid crystals with one-dimensional conductivity [4].

In this work we report on preparation, spectral properties, and mesomorphism of a series of new benzotriazolyl-substituted phthalocyanines.

Phthalonitriles **I–V** were prepared from 4-bromo-5-nitrophthalonitrile via nucleophilic substitution of bromine with *ortho*-phenylenediamine with subsequent cyclization into benzotriazole fragment and nucleophilic substitution of nitro group with substituted phenols using the procedures reported elsewhere [5, 6]. The products **I–V** are white or light-beige powders, insoluble in water and soluble in DMF, chloroform, and acetone. Their melting points are above 200°C.

Following the procedure described in [7], benzotriazolyl-substituted phthalocyanines **VI–X** were prepared by heating of phthalonitriles **I–V** with urea at 180–220°C. Urea was introduced into the reaction mixture in order to decrease its melting point and increase the products yield (Scheme 1).

The products were further extracted from the reaction mixtures with chloroform and purified by column chromatography on alumina (chloroform as eluent).

Yields of the purified derivatives **VI–X** were of 70–80%. The products were dark-green, insoluble in water and readily soluble in benzene, chloroform, acetone, and DMF. Their structures were confirmed by

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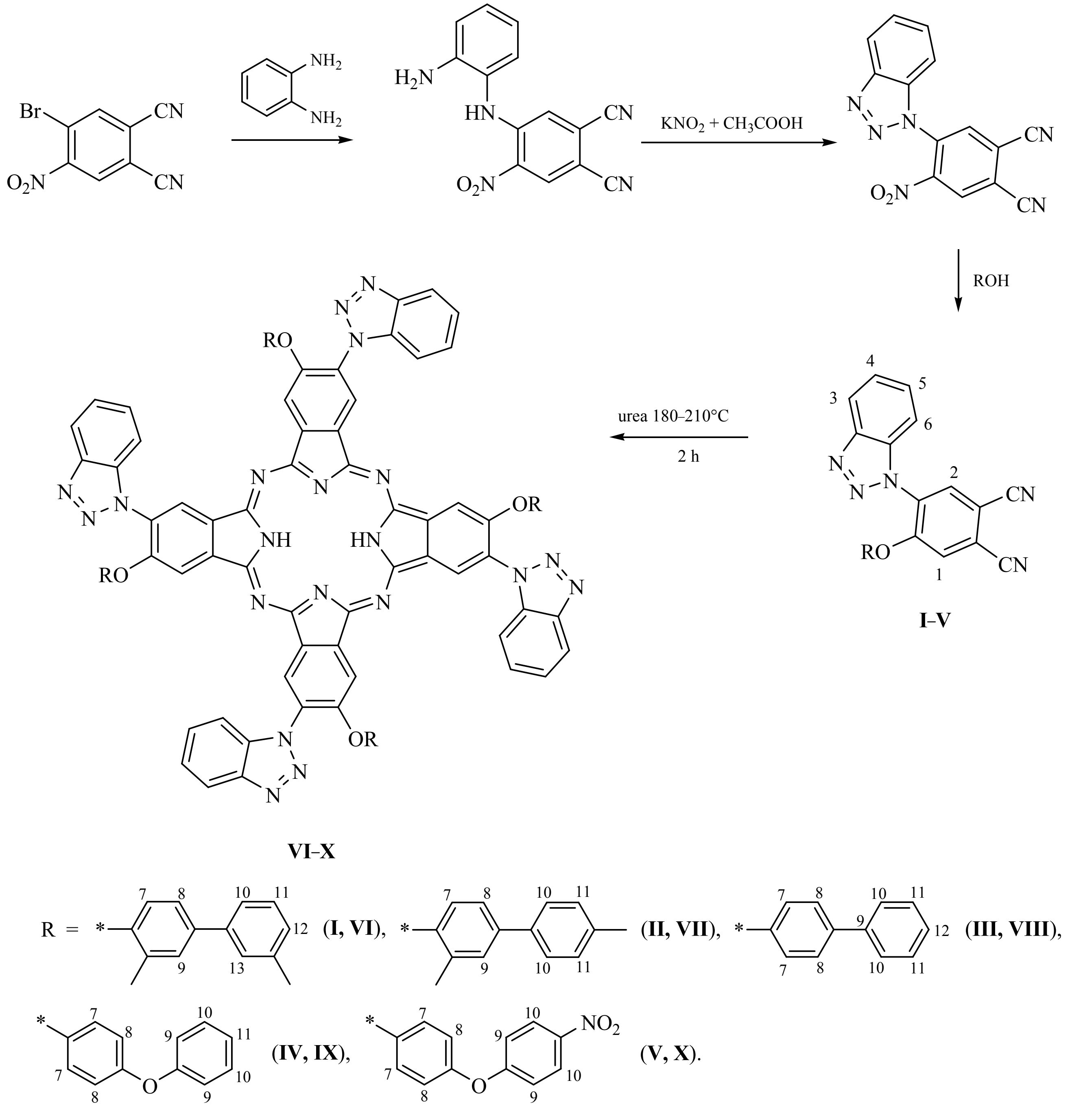
[1] For communication X, see [1].

Scheme 1.



elemental analysis, ^1H NMR, IR, and electron absorption spectra.

By the number and position of the signals ^1H NMR spectra of phthalocyanines **VI–X** were quite similar to those of phthalonitriles **I–V**. The main difference was as follows: In the spectra of phthalocyanines the signal of protons in position 1 was shifted upfield, the most upfield signals at –2.10 to –1.85 ppm were singlets of the transannular imino groups; most of the signals appearing as singlets in the phthalonitriles spectra were broadened and split due likely to the formation of mixture of isomers.

In the IR spectra of compounds **VI–X** no absorption bands were found at 2230–2240 cm^{-1} (range of

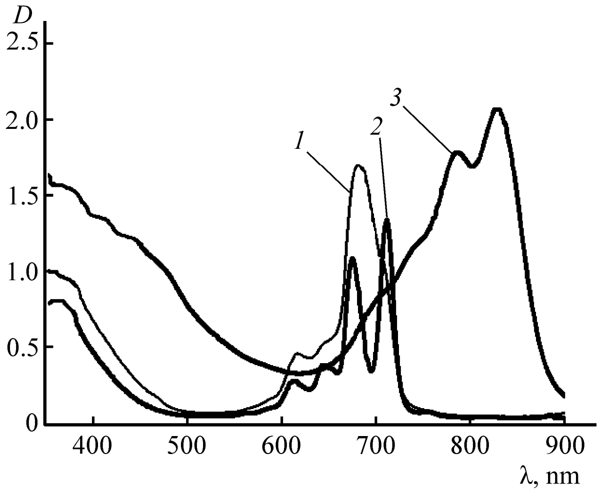


Fig. 1. Electron absorption spectra of solutions of compound **IX** in DMF (*1*), chloroform (*2*), and concentrated sulfuric acid (*3*).

nitrile group stretching vibrations) thus confirming that the products were free of the staring materials.

Electron absorption spectra of compounds **VI–X** in chloroform contained two intense long-wave absorptions at around 675 nm and 710–711 nm (Fig. 1 and Table 1) typical of phthalocyanines [8].

Spectra of the same compounds in DMF contained instead of two long-wave bands a single *Q*-band at around 680 nm (Fig. 2), confirming the formation of dianion form of phthalocyanine in the basic solvent [9].

Compounds **IX** and **X** could be re-precipitated from concentrated sulfuric acid with their properties preserved, whereas products **VI–VIII** were sulfonated under similar conditions yielding water-soluble products.

Solutions of compounds **VI–X** in concentrated sulfuric acid, as compared with those in organic solvents, revealed significant red shift of the absorption bands (Table 1); the shift was the most prominent in the cases of products **VI** and **VII** and the least significant in the case of derivative **VIII**.

In order to study theoretically the structure and properties of liquid crystals, various modeling and simulation methods are used [10–12]; mesomorphism can be predicted as well using certain molecular descriptors [13] and parameters [14–17].

Previously, we reported on mesomorphism of mixed-substituted phthalocyanines containing peripheral benzotriazolyl groups [18, 19]. An approach to predict mesomorphism applying the molecular parameters used in this work was similar to that discussed in [20].

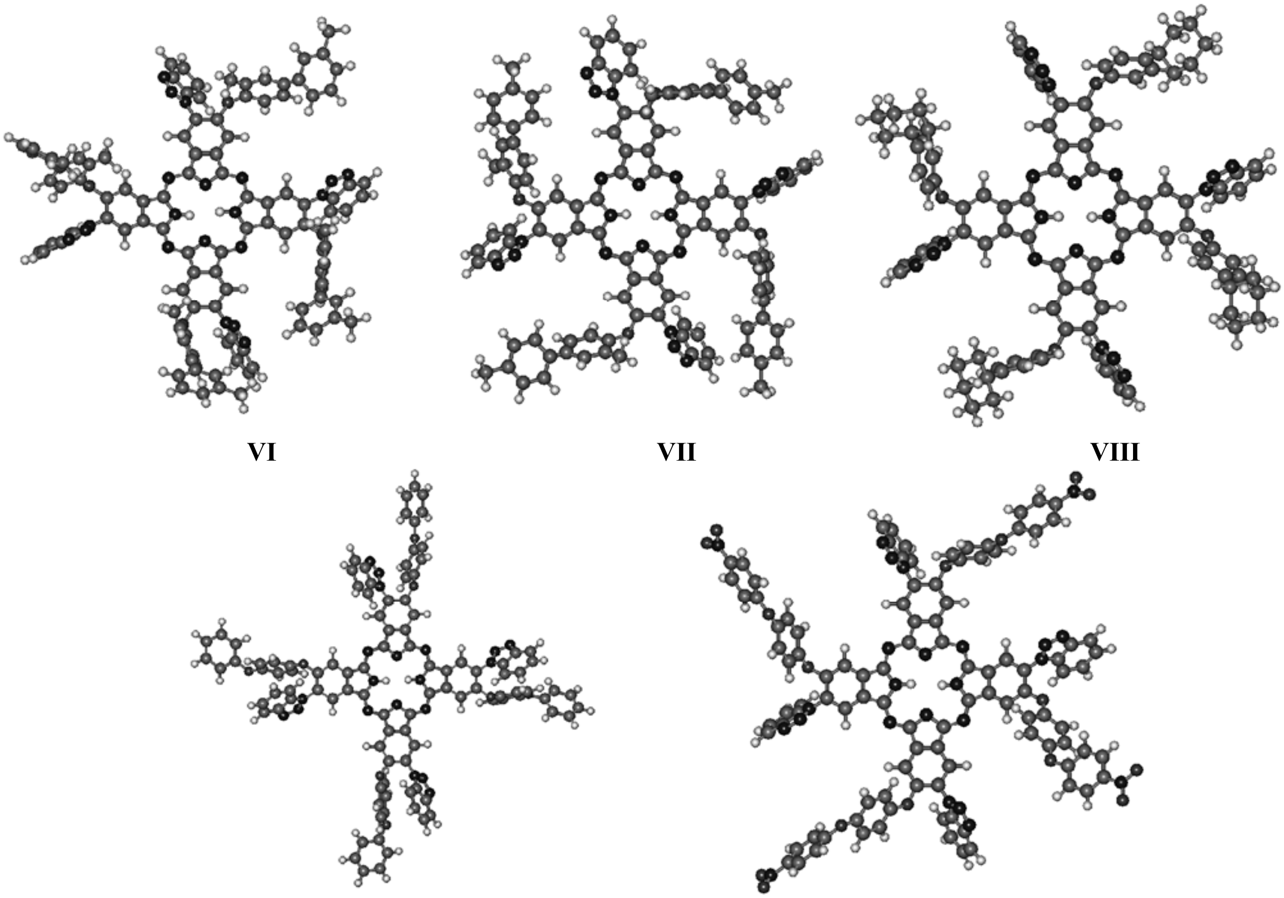
Molecular models were constructed and optimized utilizing HyperChem software package (molecular mechanics MM^+ method, see results in Fig. 2). The extracted geometry parameters formed the input of the original CMP ChemCard software.

The ability of the studied compounds to form columnar and nematic mesophases was described by the calculated molecular parameters with the typical threshold values: K 2–8.5, K_c 1–2.6, K_p 0.2–0.7, K_s 0.25–1.00, M_m 0.2–0.8, M_r 0.15–0.80, and K_{ar} 0.08–0.30 (Table 2). Even a single parameter outside the desired range indicated that the compound could not form the mesophases typical of discotic mesogens. Data in Table 2 show that four of the studied phthalocyanines (**VI**, **VII**, **IX**, and **X**) can form the above-mentioned mesophases.

In order to confirm the theoretical findings, the prepared phthalocyanines **VI–X** were studied with thermal polarization microscopy (Figs. 3 and 4). Indeed, four of the products were transformed into anisotropic state upon vitrification, thus revealing their hidden (latent) thermotropic mesomorphism and confirming the theoretical predictions (Table 2).

Table 1. Absorption bands in electronic spectra of compounds **VI–X**

Comp. no.	λ_{max}, nm (log ε) (intensity ratio)		
	DMF	chloroform	conc. H_2SO_4
VI	681	675 (5.06), 711 (5.14)	792, 831 [0.92 : 1]
VII	679	675 (4.93), 711 (5.02)	795, 836 [0.87 : 1]
VIII	680 (4.82)	675 (4.92), 710 (5.00)	805
IX	681	675 (4.99), 710 (5.08)	785, 827 [0.86 : 1]
X	678	677 (4.90), 711 (4.98)	781, 826 [0.87 : 1]



VI VII VIII

IX X

Fig. 2. Optimized geometry of molecules of compounds **VI–X**.

In particular, phthalocyanine **VI** became isotropic liquid at heating to 150°C. During cooling, the molten sample vitrified at 150–149°C as seen from the cracks net appeared at 50°C (Fig. 3a). Upon further cooling, the anisotropic rainbow broad pattern and dark blurs were seen (the so called “leopard spots” [20]) (Fig. 3b).

Compound **VII** could be molten into isotropic liquid as well at 180–185°C, being partially decomposed. Its cooling to 100°C revealed the cracks net showing vitrification, upon further cooling the well defined anisotropic domains were seen (Figs. 4a, 4b).

Table 2. Calculated molecular parameters of compounds **VI–X** and mesomorphism prediction[a]

Comp. no.	E, kcal/mol	M_m	M_r	K	K_p	K_{ar}	P_{Col+N}
VI	159.87	0.48	0.24	2.49	0.59	0.25	+
VII	165.14	0.48	0.24	2.32	0.57	0.29	+
VIII	140.16	0.51	0.26	1.89	0.62	0.26	–
IX	135.43	0.50	0.25	2.59	0.58	0.21	+
X	133.13	0.43	0.22	2.52	0.51	0.18	+

[a] For all the studied compounds, K_s = 0.5 and K_c = 1.33. P_{Col+N} points at possibility of mesomorphism typical of discotic mesogens (“–” impossible, ”+” possible).

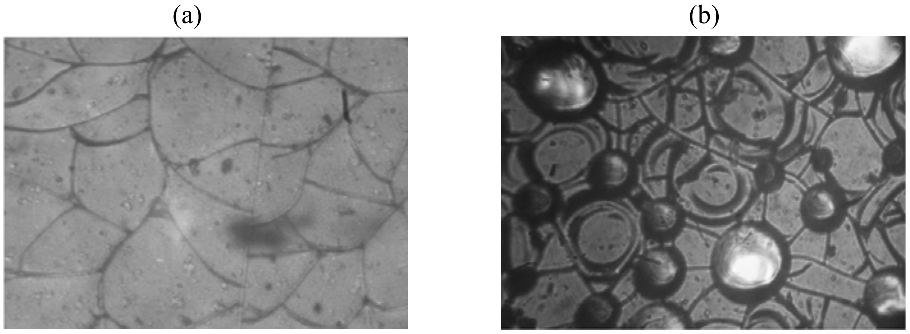


Fig. 3. Texture of glassy phthalocyanine **VI** upon cooling cycle: (a) is network of cracks at 50ºC; (b) are traces of stress (“leopard spots”) at 25ºC. Nicol prisms are parallel.

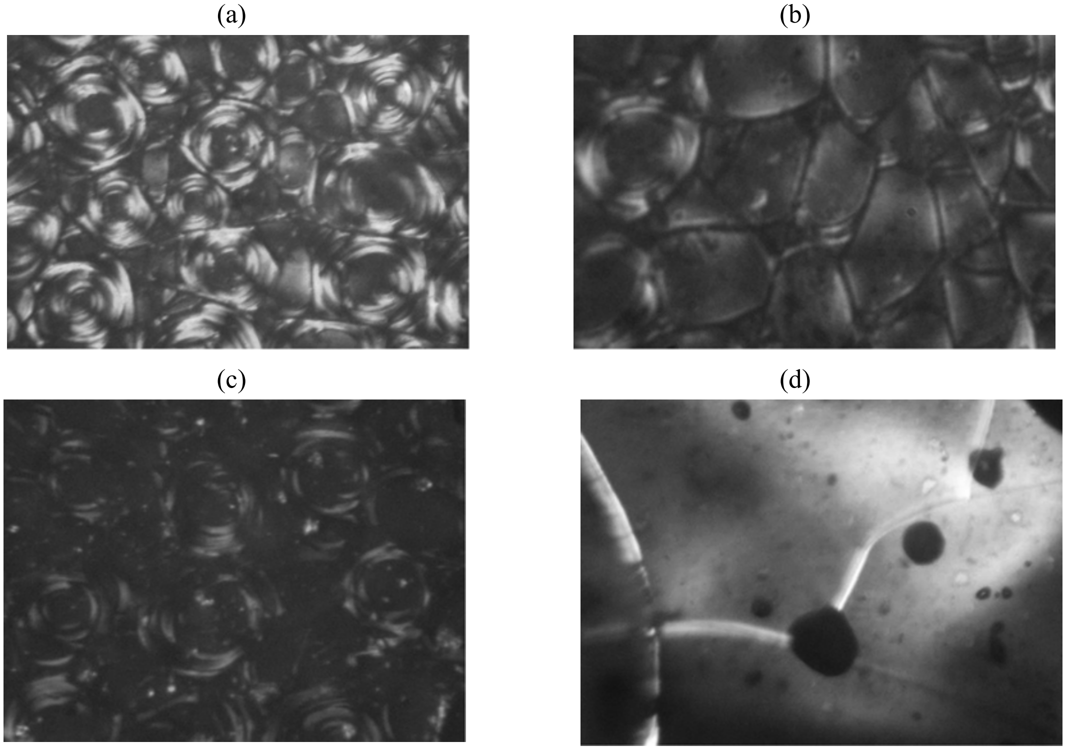


Fig. 4. Texture of glassy phthalocyanines (a, b) **VII**, (c) **IX**, and (d) (**X**) upon cooling cycle. Observation conditions: (a) Nicol prisms parallel, (b–d) Nicol prisms transposed; temperature, ºC: (a) 85, (b) 65, (c) 25, and (d) 28.

Similar behavior was observed in the cases of phthalocyanines **IX** and **X**. Compound **IX** formed the isotropic liquid at 140°C, the cracks network upon cooling appeared at 40°C, and the “leopard spots” were seen at room temperature (Fig. 4c). Compound **X** turned into isotropic melt at 187°C, and vitrified to form the anisotropic texture upon cooling (Fig. 4d).

Compound **VIII** softened at 150°C and at higher temperature could be molten into isotropic liquid (236°C). Its cooling to below 170°C caused vitrification, but no anisotropic mesophase was formed.

Hence, using the mixed-substituted phthalocyanine bearing benzotriazolyl and aryloxy groups as an example, we demonstrated the prediction of mesomorphism from theoretical modeling.

EXPERIMENTAL

Electron absorption spectra (325–900 nm, room temperature) were recorded using the HitachiU-2001 spectrophotometer. IR spectra (thin films from evaporated chloroform solutions, 400–4000 cm^{-1}) were registered using the Avatar 360 FT-IR ESP spectrometer.

^{1}H NMR spectra (5 wt % in $CDCl_3$, TMS as internal reference) were obtained on a Bruker DRX-500 spectrometer. Elemental analysis was performed on a CHNS-O FlashEA, 1112 series elemental analyzer.

Mesomorphic properties of the prepared compounds were observed with the MIN-8 optical polarization microscope equipped with specially designed temperature-controlled stage.

4-Bromo-5-nitrophthalonitrile was obtained as described in [21] and was used for further synthesis of 4-(1-benzotriazolyl)-5-nitrophthalodinitrile [5].

4-(1-Benzotriazolyl)-5-(4-cyclohexylphenoxy)-phthalonitrile (III) was prepared as described in [22].

Preparation of phthalonitriles (I, II, IV, V). A mixture of 1 mmol of the corresponding substituted phenol and 1 mmol (0.290 g) of 4-(1-benzotriazolyl)-5-nitrophthalonitrile was dissolved in 5 mL of DMF, then 0.138 g (1 mmol) of potassium carbonate in 2 mL of water was added to the solution. The mixture was maintained at room temperature during 1 h. The precipitate was filtered off, washed with water and isopropanol, and dried at 60°C.

4-(1-Benzotriazolyl)-5-[4-(3-methylphenyl)-2-methylphenoxy]phthalonitrile (I) was prepared from 0.198 g of 4-(3-methylphenyl)-2-methylphenol. Yield 0.39 g (88%), mp 210–212°C. IR spectrum, ν, cm^{-1}: 1221 (Ar–O–Ar), 2228 (C≡N), 1045 (N=N), 745 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.71 s (H^1, 1H), 8.13 s (H^3, 1H), 8.25 s (H^2, 1H), 8.05 s (H^6, 1H), 7.78–7.80 m (H^4, 1H), 7.50 s (H^5, 1H), 7.33 s (H^8, 1H), 7.38 m (H^{7,10,11}, 3H), 7.12 s (H^9, 1H), 7.29 m (H^{12,13}, 2H), 2.24–2.30 m (CH_3, 6H). Found, %: C 75.60; H 4.31; N 15.80. $C_{28}H_{19}N_5O$. Calculated, %: C 76.18; H 4.34; N 15.86.

4-(1-Benzotriazolyl)-5-[4-(4-methylphenyl)-2-methylphenoxy]phthalonitrile (II) was prepared from 0.200 g of 4-(4-methylphenyl)-2-methylphenol. Yield 0.38 g (86%), mp 210–212°C. IR spectrum, ν, cm^{-1}: 1217 (Ar–O–Ar), 1045 (N=N), 746 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.71 s (H^1, 1H), 8.23 m (H^3, 1H), 8.13 s (H^2, 1H), 8.00 m (H^{6,9}, 2H), 7.79 s (H^4, 2H), 7.53 m (H$^{5, 10}$, 2H), 7.40 s (H^7, 1H), 7.20 s (H^{11}, 1H), 2.24–2.34 m (CH_3, 6H). Found, %: C 76.50; H 4.13; N 16.08. $C_{28}H_{19}N_5O$. Calculated, %: C 76.18; H 4.34; N 15.86.

4-(1-Benzotriazolyl)-5-(4-oxyphenylphenoxy)-phthalonitrile (III) was prepared from 0.185 g of 4-oxyphenylphenol. Yield 0.37 g (86%), mp 205–208°C. IR spectrum, ν, cm^{-1}: 1220 (Ar–O–Ar), 1045 (N=N), 745 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.73 s (H^1, 1H), 8.23 s (H^3, 1H), 8.17 s (H^2, 1H), 8.05 s (H^6, 1H), 7.78–7.81 m (H^4, 1H), 7.55–7.51 s (H^5, 1H), 6.92–6.95 m (H^{7,8}, 2H), 7.36 m (H^{9,10}, 2H), 7.28 s (H^{11}, 2H). Found, %: C 72.65; H 3.71; N 15.50. $C_{27}H_{17}N_5O$. Calculated, %: C 73.13; H 3.86; N 15.79.

4-(1-Benzotriazolyl)-5-[4-(4-nitrophanoxy)phenoxy]phthalonitrile (IV) was prepared from 0.230 g of 4-(4-nitrophenoxy)phenol. Yield 0.43 g (91%), mp 220–222°C. IR spectrum, ν, cm^{-1}: 1560 [$\nu_{as}(NO_2)$], 1354 [$\nu_s(NO_2)$], 1220 (Ar–O–Ar), 1045 (N=N), 743 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.97 s (H^{10}, 1H), 8.70 s (H^1, 1H), 8.30 s (H^3, 1H), 8.18 s (H^2, 1H), 8.04 s (H^6, 1H), 7.80 s (H^4, 1H), 7.40–7.51 m (H^5, 1H), 6.98 s (H^7, 1H), 7.04 s (H^8, 1H), 6.83–7.00 m (H^9, 1H). Found, %: C 65.60; H 3.30; N 17.80. $C_{26}H_{14}N_6O$. Calculated, %: C 65.80; H 2.97; N 17.71.

Phthalocyanines VI–X were prepared by heating of 0.45 mmol of the corresponding substituted phthalonitrile **I–V** in the presence 10 mg (0.166 mmol) of urea during 2 h. The target products were extracted with chloroform and then purified by column chromatography (alumina, chloroform as eluent). After the solvent evaporation the products were dried at 60–80°C.

Tetra-4-(1-benzotriazolyl)tetra-5-[4-(3-methylphenyl)-2-methylphenoxy]phthalocyanine (VI) was prepared from 200 mg of **I** at 185°C. Yield 156 mg (79%). IR spectrum, ν, cm^{-1}: 3071 (N–H), 2830, 2845 (CH_3), 1210 (Ar–O–Ar), 1047 (N=N), 1009 (H_2Pc), 745 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.51 m (H^1, 4H), 8.27 m (H^3, 4H), 8.22 m (H^2, 4H), 8.18 m (H^6, 4H), 7.71 m (H^4, 4H), 7.49–7.61 m (H^5, 4H), 7.43 m (H^8, 4H), 7.28 m (H^{7,10,11}, 12H), 7.20 s (H^9, 4H), 7.09 m (H^{12,13}, 4H), 2.31–2.44 m (CH_3, 12H), 1.85 s ($NH_{transannul}$, 2H). Found, %: C 76.46; H 4.40; N 15.58. $C_{112}H_{78}N_{20}O_4$. Calculated, %: C 76.09; H 4.45; N 15.84.

Tetra-4-(1-benzotriazolyl)tetra-5-[4-(4-methylphenyl)-2-methylphenoxy]phthalocyanine (VII) was prepared from 198 mg of **II** at 185°C. Yield 160 mg (79%). IR spectrum, ν, cm^{-1}: 3070 (N–H), 1212 (Ar–O–Ar), 1045 (N=N), 1007 (H_2Pc), 744 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.51 s (H^1), 8.21 m (H^3, 4H), 8.18 s (H^2, 4H), 8.10 m (H^{6,9}, 8H), 7.72 m (H^4, 4H), 7.61 m (H^{10}, 4H), 7.52 m (H^5, 4H), 7.36 m (H^7, 4H), 7.21 m (H^{11}, 4H), 2.21–2.44 m (CH_3, 12H), –1.89 s ($NH_{transannul}$, 2H). Found, %: C 76.31; H 4.02; N 15.18. $C_{112}H_{78}N_{20}O_4$. Calculated, %: C 76.09; H 4.45; N 15.84.

Tetra-4-(1-benzotriazolyl)tetra-5-[4-cyclohexylphenoxy]phthalocyanine (VIII) was prepared from 185 mg of **III** at 210°C. Yield 148 mg (74%). IR spectrum, ν, cm^{-1}: 3071 (N–H), 1220 (Ar–O–Ar), 1041 (N=N), 1015 (H_2Pc), 744 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.63 m (H^1, 4H), 8.24 m (H^3, 4H), 8.16 s (H^2, 4H), 8.04 m (H^6, 4H), 7.69 m (H^4, 4H), 7.59 m (H^5, 4H), 7.21 m (H^8, 4H), 7.03 m (H^7, 4H), 2.90 m (H^9, 4H), 1.89 m (H^{10}, 4H), 1.40 s (H^{11}, 4H), –2.00 s ($NH_{transannul}$, 2H). Found, %: C 74.13; H 4.92; N 16.18. $C_{104}H_{86}N_{20}O_4$. Calculated, %: C 74.44; H 5.05; N 16.70.

Tetra-4-(1-benzotriazolyl)tetra-5-[4-oxyphenylphenoxy]phthalocyanine (IX) was prepared from 195 mg of **IV** at 205°C. Yield 153 mg (77%). IR spectrum, ν, cm^{-1}: 3071 (N–H), 1218 (Ar–O–Ar), 1045 (N=N), 1010 (H_2Pc), 746 (C–N). ^{1}H NMR spectrum, δ, ppm: 8.61 s (H^1, 4H), 8.25 m (H^3, 4H), 8.19 s (H^2, 4H), 8.04 m (H^6, 4H), 7.68 m (H^4, 4H), 7.58 m (H^5, 4H), 7.45 s (H^9, 4H), 7.38 m ($H^{10,11}$, 8H), 7.03–7.06 m ($H^{7,8}$, 8H), –1.89 s ($NH_{transannul}$, 2H). Found, %: C 67.84; H 3.67; N 14.19. $C_{104}H_{86}N_{20}O_4$. Calculated, %: C 68.52; H 3.20; N 14.81.

Tetra-4-(1-benzotriazolyl)tetra-5-[4-(4-nitrophenoxy)phenoxy]phthalocyanine (X) was prepared from 215 mg of **V** at 230°C. Yield, 140 mg (70%). IR spectrum, ν, cm^{-1}: 3071 (N–H), 1218 (Ar–O–Ar), 1546 [$\nu_{as}(NO_2)$], 1356 [$\nu_s(NO_2)$], 1045 (N=N), 1010 (H_2Pc). ^{1}H NMR spectrum, δ, ppm: 8.97 s (H^{10}, 4H), 8.57 s (H^1, 4H), 8.32 m (H^3, 4H), 8.19 s (H^2, 4H), 8.04 s (H^6, 4H), 7.62 m (H^4, 4H), 7.40–7.51 m (H^5, 4H), 6.98 m (H^7, 4H), 7.04 s (H^8, 4H), 6.73–7.06 m (H, $4H^9$), –2.09 s ($NH_{transannul}$, 2H).

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