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Nucleophilic Substitution in 4-Bromo-5-nitrophthalodinitrile: XVI.[1] 4-(1*H*-Benzotriazol-1-yl)-5-[(4-carboxyphenyl)oxy/sulfanyl]-phthalonitriles and Cobalt Phthalocyanines Thereof

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Abstract—A method of synthesis of 4-(1*H*-benzotriazol-1-yl)-5-[(4-carboxyphenyl)oxy]- and -5-[(4-carboxyphenyl)sulfanyl]phthalonitriles starting with 4-bromo-5-nitrophthalonitriles was developed. The synthesized phthalonitriles were used to prepare cobalt tetra-4-(1*H*-benzotriazol-1-yl)-tetra-5-[(4-carboxyphenyl)oxy/sulfanyl]phthalocyanines. The spectral and catalytic properties of the resulting octasubstituted phthalocyanines were studied.

Keywords: 4-bromo-5-nitrophthalodinitrile, cobalt phthalocyanine, octasubstituted phthalocyanines, phthalonitriles

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Metal complexes of substituted phthalocyanines containing peripheral functional groups exhibit unique physicochemical properties due to which they have been the subject of enduring interest of researchers and found application in diverse fields ranging from nanoelectronics to photovoltaics [2]. The enhancement of water solubility of phthalocyanines, most commonly through the introduction of peripheral sulfo or carboxy groups, facilitates their study and practical application.

Carboxylic acids, where the carboxy groups are linked to the phthalocyanine core by spacer bridges occupy special place among phthalocyanine derivatives. Such spacers impart additional chemical activity to the macrocycle, which can be used to design practically valuable materials [2–5]. Over the past few years phthalocyanines with oxy- [2, 6, 9–11], amino- [8, 10], or mercaptophenylene bridges [7, 10] have attracted increased attention. Such substituted macroheterocyclic compounds have been found to be promising candidates for application as medical materials [12], photosensitizers [7, 8, 13, 14], and catalysts [15, 16]. At the same time, there have been only two groups of substituted phthalocyanines mentioned in this context: tetrasubstituted phthalocyanines [2, 6–9, 15–16] and symmetric octasubstituted phthalocyanines [3–5, 17].

Bifunctionally substituted phthalocyanines [10] containing, along with carboxy and nitro groups, some other substituents like small nitrogenous heterocycles whose water-soluble derivatives may be interesting for drug design have scarcely been reported in the literature [18].

The present paper reports the synthesis and physicochemical properties of 4,5-octasubstituted cobalt phthalocyanines containing the fragments of *p*-hydroxybenzoic and *p*-mercaptobenzoic acid and 1-benzotriazole in the substituents.

At the first stage of the study we have developed a method for the synthesis of 4-(1*H*-benzotriazol-1-yl)-5-[(4-carboxyphenyl)oxy]phthalonitriles **5a** and **5b** required to obtain the corresponding bifunctionally substituted cobalt phthalocyanines **6a** and **6b**. As the starting compound we chose 4-bromo-5-nitrophthalonitrile **1** which allows 4,5-substituted phthalonitriles with different substituents with high yields [2] (Scheme 1).

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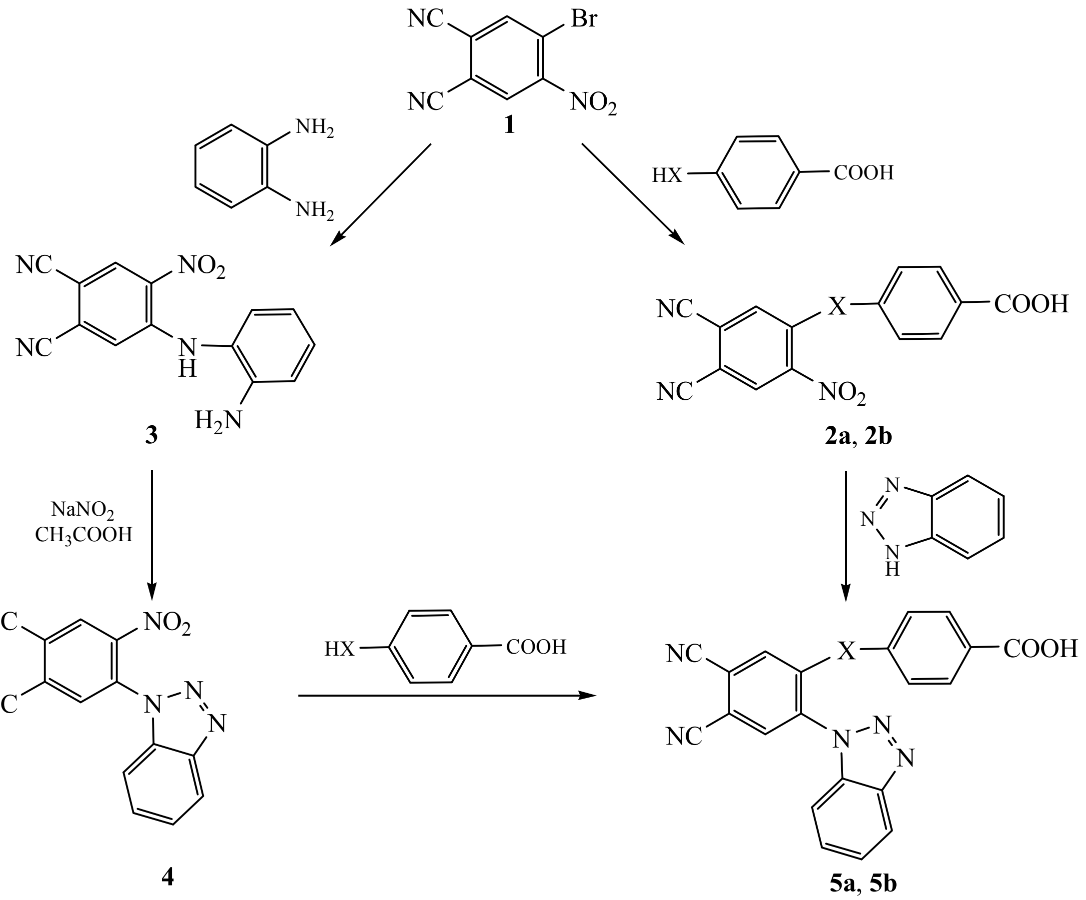
[1] For communication XV, see [1].

Scheme 1.



X = O (**2a**, **5a**), S (**2b**, **5b**).

Earlier we developed a two-stage procedure for the synthesis of benzotriazolyl-substituted phthalonitriles, with the first stage involving the substitution of the bromine atom in compound **1** by phenoxy and naphthyloxy groups [19]. Further on 4-aryloxy-5-nitrophthalonitriles was reacted with 1*H*-benzotriazole to obtain the corresponding 4-aryloxy-5-(1*H*-benzotriazol-1-yl)phthalonitriles in high yields. However, we found that the substitution of bromine by *p*-carboxyphenyloxy- or *p*-carboxyphenylsulfanyl groups followed by the substitution of the nitro groups by benzotriazol residue gave the target phthalonitiles **5a** and **5b** in yields as low as 38 and 5%, respectively. Apparently, the low yields in this case are explained by the fact that the water-soluble target products could not be isolated completely when synthesized from substrates **2a** and **2b** which, too, are water soluble. Higher yields of compounds **5a** (up to 78%) and **5b** (up to 84%) were attained in the synthesis from 4-(1*H*-benzotriazol-1-yl)-5-ntrophthalonitrile **4** (Scheme 1) preliminarily synthesized by a known procedure [20]. Such an essential increase of the yields of compounds **5** can be explained by that phthalonitrile **4** is insoluble in water. Therefore, we made use of the latter approach in the synthesis of the target phthalonitriles.

4-(1*H*-Benzotriazol-1-yl)-5-[(4-carboxyphenyl)oxy/sulfanyl]phthalonitriles **5** were identified by elemental analysis, MALDI–TOF mass spectrometry, and IR spectroscopy. All physicochemical characteristics of the samples of compounds **5a** and **5b**, synthesized by two routes, are fully identical.

The IR spectra of phthalonitriles **5** show no bands of symmetrical and antisymmetrical vibrations of the nitro N–O bonds and contain vibration bands of the benzotriazolyl fragment at 743–744 and 1050 cm^{-1}, as well as bands at 1245 (Ar–O–Ar, **5a**) [21] and 696 cm^{-1} (C–S–C, **5b**).

Further on, to find out whether phthalonitriles **5** can be used in the synthesis of the corresponding phthalocyanines, we evaluated the thermal stability of com-

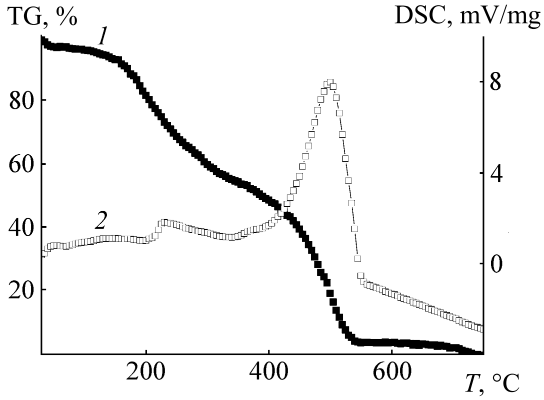


Fig. 1. Thermoanalytical curves for compound **2a:** (*1*) TG and (*2*) DSC.

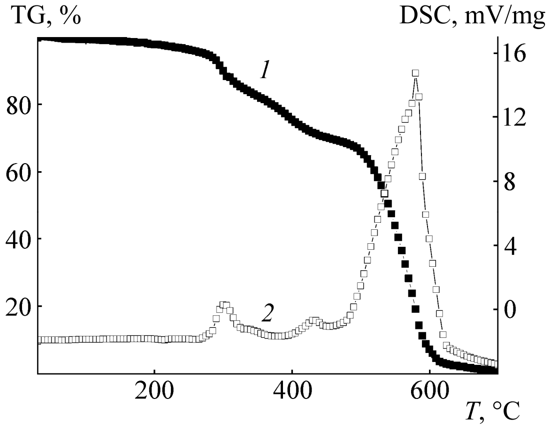


Fig. 2. Thermoanalytical curves for compound **5a:** (*1*) TG and (*2*) DSC.

pounds **2a**, **2b**, and **5a**. As seen from Figs. 1–3, the thermal degradation of the synthesized phthalonitriles involves a few steps. Thus, the DSC curve of compound **2a** displays two exothermic peaks at 228 and 500°C (Fig. 1). The weight loss in the range 175–315°C is 42%, which is associated with a loss of the *p*-carboxyphenyloxy group. The next step initiates at 400°C and corresponds to complete degradation of compound **2a**.

The first step of thermal degradation of compound **5a** containing the benzotriazolyl substituent instead of nitro groups is shifted to higher temperatures (Fig. 2, Table 1). The weight loss at this step corresponds to a loss of the benzotriazol fragment. The second step, too, is shifted to higher temperatures.

The thermal degradation of phthalonitrile **5b** having the nitro and *p*-carboxyphenylsulfanyl substituents ortho to each other occurs in an even more complicated way (Fig. 3), and weight loss initiates at a higher temperature than in compound **5a**.

Therefore, it was concluded that the studied phthalonitriles all preserve their composition and structure during the synthesis of the corresponding carboxy-substituted phthalocyanines, which occurs at 170–200°C.

Further on we employed the nitrile method [2] to synthesize octasubstituted cobalt phthalocyanines **6a** and **6b** by heating phthalonitriles **5a** and **5b** at 190–195°C with cobalt acetate for 2 h in yields of 49 and 52%, respectively (Scheme 2). No urea was required for successful synthesis.

Phthalocyanines **6** were identified by elemental analysis, MALDI–TOF mass spectrometry, and electronic absorption (UV-Vis) and IR spectroscopy. The UV-Vis spectrum (in DMF) of complex **6b** containing mercaptobenzoic acid fragments over the periphery shows a broadened visible band, which is likely to suggest that the compound exists in an associated form. Moreover, dilution of the solution to a concentration of 0.2×10^{-6} M does not destroy associates completely (Fig. 4). By contrast, 4-carboxyphenoxy derivative **6a** exists, judging from its UV-Vis spectrum in DMF, as a monomer in this solvent [10].

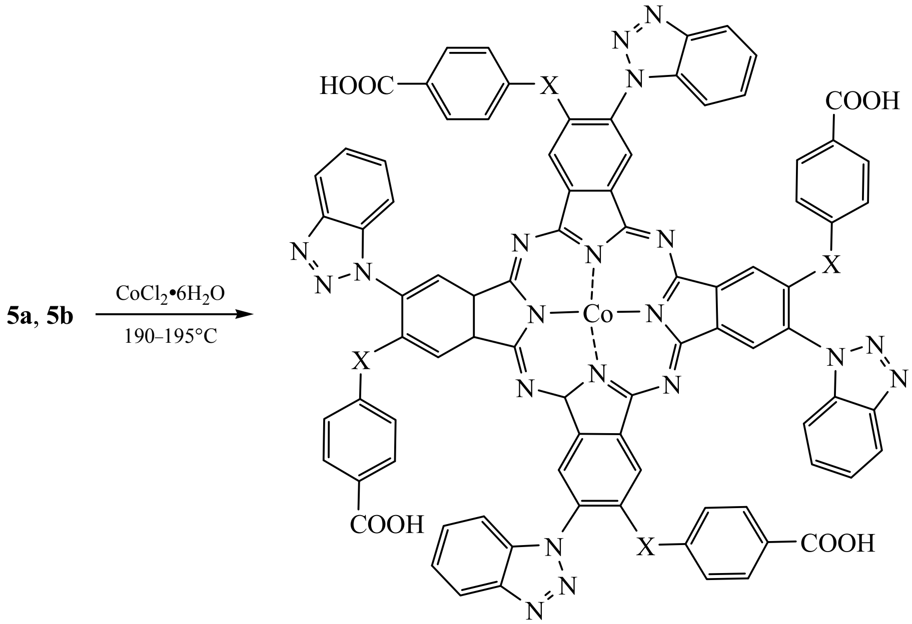
Comparing the UV-Vis spectra of compounds containing both the benzotriazol-1-yl and *p*-carboxyphenoxy (**6a**) or *p*-carboxyphenylsulfanyl substituents (**6b**) in conc. H_2SO_4 with the spectra of complexes the same peripheral benzoic acid fragments but nitro- instead of benzotriazolyl substituents, we revealed a hyproschromic shift of the long-wave absorption bands in going from the benzotriazolyl- to nitro-substituted compounds (Table 2). A hypsochromic shift is also observed in going from 4-carboxyphenylsulfanylphhtalocynaine to the respective 4-carboxyphenoxy-substituted analog (Table 2).

Finally, we studied the catalytic activity of the synthesized cobalt phthalocyanines in the homo-

Table 1. Thermogravimetry data for phthalonitriles **2a**, **2b**, and **5a**

Comp. no.	Peak weight loss temperature, °C	Peak exothermic effect temperature, °C
2b	490–600	497, 552
2a	400–560	500
5a	500–630	583

Scheme 2.



6a, 6b

X = O (**5a**, **6a**), S (**5b**, **6b**).

geneous liquid-phase oxidation of sodium *N,N*-diethylcarbamodithioate (Scheme 3). The reaction was performed in an aqueous alkaline medium at 298 K, and the pH of the reaction mixture was varied from 8 to 12. The choice of this specific model reaction was motivated, first of all, by its high practical significance, because it is used to produce Thiuram E, the key component of drugs against chronic alcoholism [22–24]. The oxidation of sodium *N,N*-diethylcarbamodithiolate in the presence of phthalocyanine catalysts occurs in mild conditions, which prevents further oxidation of the formed disulfide. The advantages of the latter reaction also include a higher quality of the final product, as well as a more facile isolation of the product from the reaction mixture and a lack of necessity of its further purification. Moreover, this reaction is a convenient model reaction for studies on the catalytic activity of metal phthalocyaninates in oxidative processes involving thiolate ions [25], because all its kinetic parameters are easily traced by

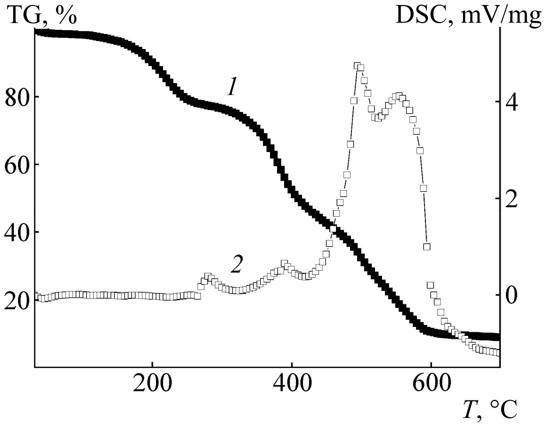


Fig. 3. Thermoanalytical curves for compound **2b:** (*1*) TG and (*2*) DSC.

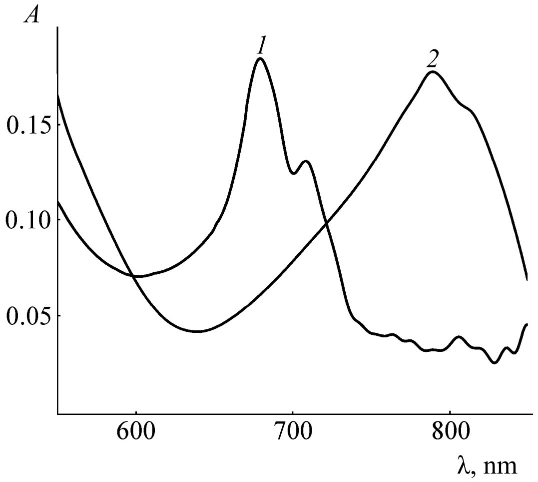
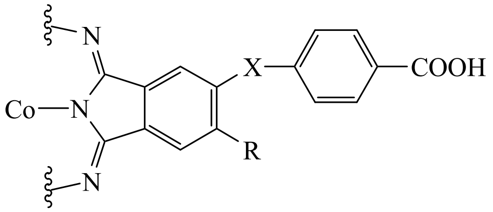


Fig. 4. Electronic absorption spectra of complex **6b** (c = 0.2×10^{-6} M) in (*1*) DMF and (*2*) sulfuric acid.

Table 2. Absorption maxima and apparent rate constants of the oxidation of sodium *N,N*-diethylcarbamodithiolate phthalocyanine with cobal phthalocyanines containing benzoic acid fragments



Comp. no.	X	R	λ, nm			$k_{app}\times10^5$, s^{-1}
			DMF	H_2O	H_2SO_4	
	O	NO_2	675	705	813	7.0
	S	NO_2	608, 680	700	807	26.9
6a	O	1*H*-Benzotriazol-1-yl	609, 679	678	728	5.9
6b	S	1*H*- Benzotriazol-1-yl	677, 710	680	789	1.5

spectral methods. The catalytic activity of cobalt phthalocyanines was estimated by the procedure in [26, 27].

It was found that the benzotriazolyl-substituted phthalocyanines exhibited a much lower catalytic activity compared to their nitro-substituted analogs (Table 2).

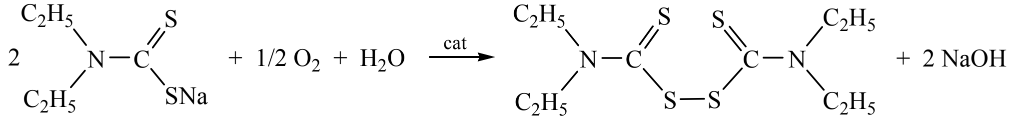
Thus, we developed a method of synthesis of 4-(1*H*-benzotriazol-1-yl)-5-[(4-carboxyphenyl)oxy]- and -5-[(4-carboxyphenyl)sulfanyl]phthalonitriles. It was found that these compounds preserve their composition and structure under the conditions of synthesis of the corresponding cobalt phthalocyanines, i.e. at 170–200°C. The synthesized phthalonitriles were used to prepare cobalt tetra-4-(1*H*-benzotriazol-1-yl)-tetra-5-[(4-carboxyphenyl)oxy/sulfanyl]phthalocyanines. It was established that the benzothiazolyl-substituted phthalocyanines exhibited a lower catalytic activity compared to their nitro-substituted analogs in the homogeneous liquid-phase oxidation of sodium *N,N*-diethylcarbamodithioate under mild conditions.

EXPERIMENTAL

The UV-Vis spectra were recorded in organic solvents (DMF and chloroform), aqueous alkaline media, and concentrated sulfuric acid on a HITACHI U-2001 spectrophotometer at room temperature in the range 325–900 nm. The IR spectra were run on an Avatar 360 FTIR ESP instrument in the range 400−4000 cm^{-1} in KBr pellets. Elemental analysis was per-formed on a Thermo FlashEA 1112 CHNSO analyzer. The MALDI−TOF mass spectra were measured on a Shimadzu Biotech Axima Confidence in the positive ion mode, using 2,5-dihydroxybenzoic acid as a matrix. The samples were prepared mixing equal volumes of THF or aqueous acetonitrile solutions of the analytes (10^{-4}–10^{-5} M) with a THF solution of the matrix (30 mg/mL). The thermooxidative degradation of the synthesized phthalonitriles was studied on Jupiter Netzsch STA 449 F3 synchronous thermal analysis instrument in an oxygen–argon atmosphere using a platinum crucible. Before elemental and thermogravimetric analysis the samples were thermally treated at 110°C for 2 h.

4-[(4,5-Dicyano-2-nitrophenyl)oxy]benzoic acid (2) was synthesized as described in [10]. Yield 75%. IR spectrum, ν, cm^{-1}: 2231 (C≡N), 1719 (C=O), 1558 [$\nu_{as}(NO_2)$], 1380 [$\nu_s(NO_2)$], 1254 (Ar–O–Ar). Mass spectrum, *m*/*z*: 309.31 $[M]^-$. Found, %: C 59.02; H 2.32; N 13.30. $C_{15}H_7N_3O_5$. Calculated, %: C 58.26; H 2.28; N 13.59. *M* 309.66.

Scheme 3.



4-[(4,5-Dicyano-2-nitrophenyl)sulfanyl]benzoic acid (3) was synthesized as described in [10]. Yield 78%. IR spectrum, ν, cm^{-1}: 2232 (C≡N), 1725 (C=O), 1550 [$\nu_{as}(NO_2)$], 1383 [$\nu_s(NO_2)$], 1117 (Ar–S–Ar). Mass spectrum, *m*/*z*: 324.71 [*M* – H]$^-$. Found, %: C 55.25; H 2.02; N 13.00. $C_{15}H_7N_3O_4S$. Calculated, %: C 55.38; H 2.17; N 12.92. *M* 325.65.

4-(1*H*-Benzotriazol-1-yl)-5-[(4-carboxyphenyl)-oxy/sulfanyl]phthalonitriles. *a*. A solution of 0.68 g (5 mmol) of anhydrous KCO_3 in 4 mL of water was added to a mixture of 0.5 mmol of compound **2** and 0.06 g (0.5 mmol) of 1*H*-benzotriazole в 25 mL of DMF. The reaction mixture was stirred at 70°C for 1 h. The precipitate that formed was filtered off, washed with 5% aqueous HCl, and dried.

b. A solution of 0.07 g (0.5 mmol) of anhydrous KCO_3 in 4 mL of water was added to a mixture of 0.15 g (0.5 mmol) of 4-(1*H*-benzotriazol-1-yl)-5-nitrophthalonitrile and 0.5 mmol of the corresponding benzoic acid in 25 mL of DMF. The reaction mixture was stirred at 25°C for 0.5 H. The resulting precipitate was filtered off, washed with 5% aqueous HCl, and dried.

4-[2-(1*H*-Benzotriazol-1-yl)-4,5-dicyano-2-phenoxy)]-benzoic acid (5a). Yield 0.07 g (38%, route *a*), 0.16 g (83%, route *b*). IR spectrum, ν, cm^{-1}: 2227 (C≡N), 1714 (C=O), 1245 (Ar–O–Ar), 1050 (N=N), 744 (C–N). Mass spectrum, *m*/*z*: 383.37 [*M* + 2H]$^+$. Found, %: C 65.95; H 3.02; N 18.30. $C_{21}H_{11}N_5O_3$. Calculated, %: C 66.14; H 2.91; N 18.36. *M* 381.66.

4-[2-(1*H*-Benzotriazol-1-yl)-4,5-dicyano-2-phenylsulfanyl)]benzoic acid (5b). Yield 0.01 g (5%, route *a*), 0.17 g (85%, route *b*). Found, %: C 63.35; H 3.00; N 17.56. $C_{21}H_{11}N_5O_2S$. Calculated, %: C 63.47; H 2.79; N 17.62.

Octasubstituted cobalt phthalocyanines. Urea, 60 mg (1 mmol), was added to thoroughly mixed 0.33 mmol of the corresponding substituted phthalonitrile and 54 mg (0.20 mmol) of cobal chloride hexahydrate, and the mixture was triturated, placed into a molybdenum glass tube, and heated at 190–195°C until solidified. After cooling, the resulting solid was thoroughly triturated, successively washed with 5% aqueous HCl and acetone, and subjected to column chromatography on Silica gel (eluent DMF). The solvent was removed by distillation, and the residue was dried at 80–90°C.

Cobalt tetra-1-(1*H*-benzotriazol-1-yl)tetra-5-[(4-carboxyphenyl)oxy]phthalocyanine (6a) was synthesized from 127 mg of phthalonitrile **5a**. Yield 63 mg (49%). IR spectrum, ν, cm^{-1}: 1709 (C=O), 1246 (Ar–O–Ar), 1052 (N=N), 744 (C–N). Mass spectrum, *m*/*z*: 1585.38 [*M* + H]$^+$; calculated *M* 1584.61. Found, %: C 64.05; H 3.05; N 17.53. $C_{84}H_{47}N_{20}O_{12}Co$. Calculated, %: C 63.68; H 2.8; N 17.68.

Cobalt tetra-1-(1*H*-benzotriazol-1-yl)tetra-5-[(4-carboxyphenyl)sulfanyl]phthalocyanine (6b) was synthesized from 131 mg of phthalonitrile **5b**. Yield 72 mg (52%). IR spectrum, ν, cm^{-1}: 1711 (C=O), 971 (Ar–S–Ar), 1047 (N=N), 745 (C–N). Found, %: C 63.85; H 3.11; N 17.02; S 7.66. $C_{84}H_{44}N_{20}O_8S_4Co$. Calculated, %: C 61.09; H 2.87; N 16.96; S 7.76.

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