

Nucleophilic Substitution in 4-Bromo-5-nitrophthalodinitrile: IX.¹ Synthesis of 4-(Morpholin-4-yl)-5-aryloxyphthalodinitriles and Copper Phthalocyanines Based Thereon

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Received June 19, 2008

Abstract—Nucleophilic aromatic substitution of bromine in 4-bromo-5-nitrophthalodinitrile with the morpholine residue and the subsequent substitution of nitro group with the aryloxy one yielded 4-morpholyl-5-aryloxyphthalodinitriles. From these substances octasubstituted copper phthalocyanines were synthesized, and their physicochemical properties were studied.

DOI: 10.1134/S1070363209050260

Metal phthalocyanines and their derivatives are intensely studied during the last fifty years. They are widely used as pigments and dyes, catalysts, chemical sensors, nonlinear optic materials, photosensitizers, and liquid crystalline materials. They also find their use in medicine and some other branches of science and technology [2–11].

Up to now a wide range of substituted metal phthalocyanines is synthesized, but the reports dealing with such compounds containing heterocyclic residues as substituents are scarce [1, 11, 12]. Data on metal phthalocyanines containing saturated heterocyclic residues are practically absent. We believe that it is connected with the absence or difficult availability of corresponding precursors, in particular of heteryl-substituted phthalodinitriles. As known, “small” (three-seven-membered) heterocycles are present in the structure of the majority of highly effective pharmaceuticals, pesticides, dyes, luminophors, and some other substances of practical use [13]. It was reported in [8] that aryloxy-substituted zinc phthalocyanines exhibit the activity in oxidation reactions. Hence, considering the importance of phthalocyanines as well as of the “small” heterocyclic compounds, the

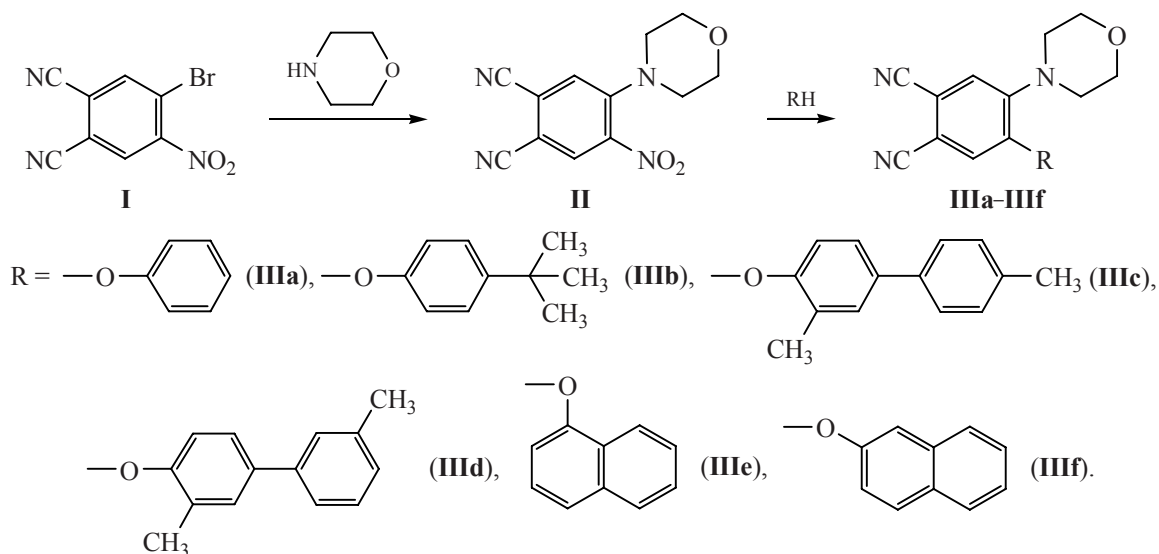
extension of the of range of phthalocyanine complexes by compounds containing aryl and heterocyclic fragments in their structure is urgent.

This report deals with the synthesis of 4-morpholyl-5-aryloxyphthalodinitriles and copper phthalocyanines obtained therefrom. As known, numerous *o*-disubstituted phthalodinitriles can be prepared on the basis of highly reactive 4-bromo-5-nitrophthalodinitrile **I** [1, 14–23].

The nucleophilic substitution of bromine in 4-bromo-5-nitrophthalodinitrile **I** under the action of morpholine yielded 4-(morpholin-4-yl)-5-nitrophthalodinitrile **II** [21]. The subsequent nucleophilic substitution of the nitro group by means of substituted phenols or 1-(2)-naphthol led to the target phthalodinitriles **IIIa–IIIf**.

The composition and structure of substituted phthalodinitriles **IIIa–IIIe** were confirmed by elemental analysis and the IR and ¹H NMR spectroscopy. In the IR spectra of compounds **IIIa–IIIf** the majority of bands characteristic of substituted phthalodinitriles appeared [24]. Strong band at 2223–2227 cm^{−1} corresponds to the nitrile group vibrations. The absorption observed at 2852–2933 cm^{−1} relates to the C–H bond vibrations of the methylene groups of the saturated

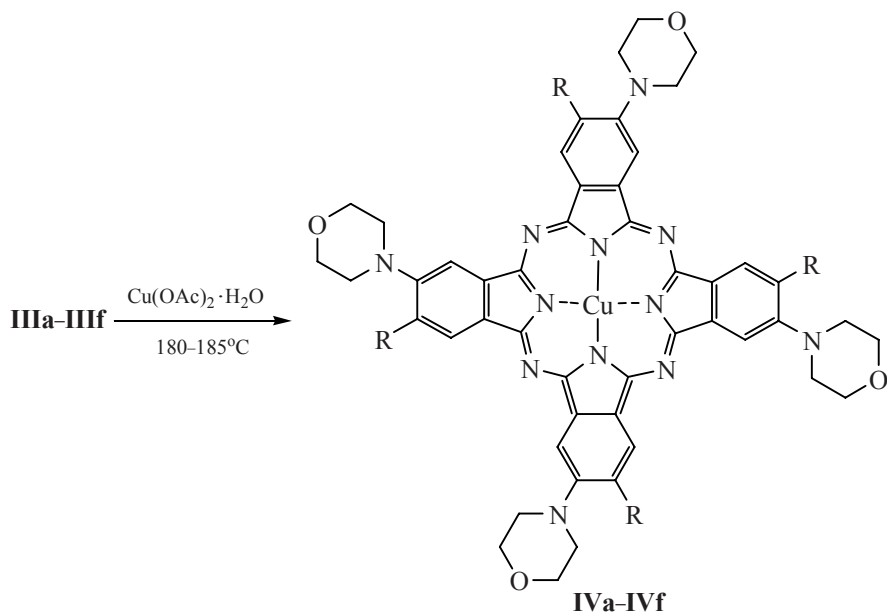
¹ For communication VIII, see [1].



monoazocycle and the alkyl fragments. These bands increase in intensity for compounds **IIIb–III d** containing the methyl groups. Absorption bands at $1214\text{--}1217\text{ cm}^{-1}$ related to the Ar–O–Ar bond vibrations were also observed [25]. ^1H NMR spectra of the compounds obtained contain several types of signals. The protons of the morpholine cycle appeared at δ 3.30–3.33 and 3.64–3.68 ppm, the protons of the aromatic rings gave signals at δ 6.85–7.66 ppm, and the signals of protons belonging to methyl and *tert*-butyl groups were observed at 2.24–2.25, 2.35–2.38, and 1.15 ppm. As expected, the protons of the aliphatic ring form two groups of signals at 3.30–3.33 ppm and 3.64–3.68 ppm related to the CH_2N and CH_2O groups respectively.

The signals of aromatic protons from the phthalodinitrile fragment are observed in the ranges 7.67–7.69 ppm and 7.24–7.26 ppm. These signals correspond to the protons adjacent to the nitrogen and oxygen atoms respectively. Signals of the methyl group protons neighboring to the phenol oxygen atom in the compounds **IIIc**, **IIId** are observed at 2.24–2.35 ppm, while the methyl groups belonging to the benzene part of biphenyl fragment give signals at 2.35 ppm in the case of *p*-located hydrogen and at 2.38 ppm for the *m*-located one. The protons of *tert*-butyl group in compound **IIIb** give rise to a singlet at 1.15 ppm.

Compounds **IIIa–III f** are crystalline substances of the color varying from light yellow to bright yellow.



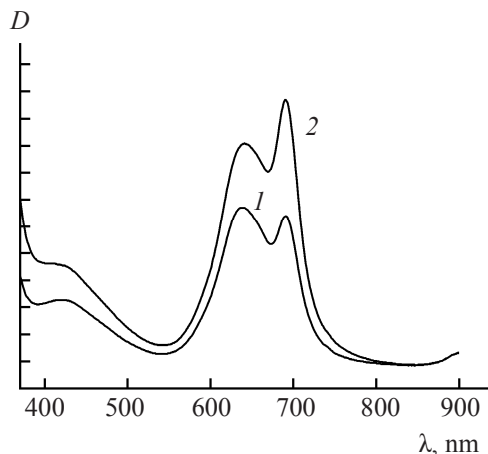


Fig. 1. The electron absorption spectra in DMF, $c\ 2.2 \times 10^{-5}$ M, (1) compound **IVa** and (2) compound **IVe**.

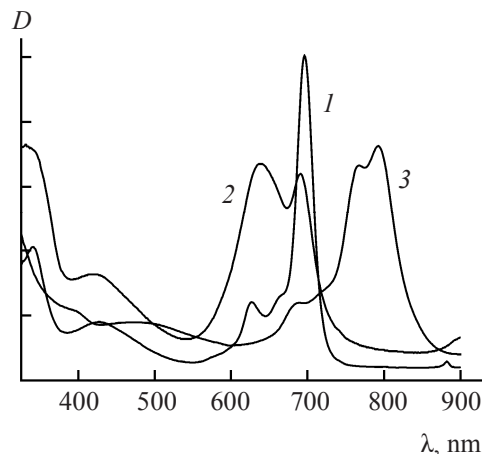


Fig. 2. Electron absorption spectra of complex **IVa**: (1) in chloroform, (2) in DMF, and (3) in the concentrated sulfuric acid.

They are soluble in the organic solvents like acetone, chloroform, DMF, ethanol, and their melting points lie in the range 148–223°C.

Metal complexes **IVa–IVf** were synthesized by the reaction of corresponding substituted phthalodinitriles **IIIa–IIIf** with copper acetate in the presence of carbamide at 180–185°C for 2 h.

The melt obtained was ground and washed successively with 5% hydrochloric acid until the decoloration of filtrate, and then with water until the neutral reaction. The target products were extracted with chloroform. Final purification was carried out by liquid chromatography on Al_2O_3 , elution with chloroform. Solvent was removed in a vacuum, and the target product was dried at 80°C.

The complexes obtained are dark green powders insoluble in water and well soluble in organic solvents (benzene, chloroform, DMF, acetone) and in the concentrated hydrochloric (excluding compounds **IVc**,

IVd) and sulfuric acids. Note that after precipitation of complexes **IVe**, **IVf** from the concentrated sulfuric acid sulfonic acids are formed. They become well soluble in the alkaline water solutions and do not dissolve in the organic solvents.

The structure and individuality of the compounds **IVa–IVf** were confirmed by elemental analysis, the electronic and IR spectroscopy. IR spectra of the compounds synthesized contain the absorption bands at 2953–2958, 2852–2856, 1736–1739, 1613–1617, 1592–1595, 1491–1493, 1452–1454, 1413–1414, 1250–1253, 1212–1219, 1116–1119, 1102–1108, and 744–746 cm^{-1} characteristic of substituted metal phthalocyanines [26]. Absorption bands belonging to the substituents also are retained. The analysis of the electron absorption spectra shows that the nature of solvent influences the character of spectrum and the location of absorption bands (see Figs. 1, 2, and the table). It can be presumed that complexes **IVa–IVg** in DMF exist mainly in the associated form. The long-

Location of the absorption bands in the electron absorption spectra of complexes

Comp. no.	λ_{max} , nm (log ϵ , ratio of intensities)		
	DMF ($c = 2.2 \times 10^{-5}$ M)	chloroform	conc. H_2SO_4
IVa	691, 639, 422, 331 (1:1.05:0.51:1.14)	697 (5.11), 628 (4.49), 428 (4.36), 341 (4.72)	793, 768 (1:0.92)
IVb	694, 651, 433, 332 (1:0.61:0.34:1.08)	698 (4.97), 633 (4.15), 430 (4.05), 343 (4.70)	794, 768 (1:0.93)
IVc	694, 651, 432, 337 (1:0.63:0.36:1.05)	698 (4.93), 628 (4.25), 425 (4.22), 347 (4.80)	792, 771 (1:0.96)
IVd	693, 645, 433, 338 (1:0.60:0.35:1.05)	698 (4.93), 627 (4.24), 430 (4.21), 335 (4.81)	792, 770 (1:0.96)
IVe	691, 641, 410, 333 (1:0.85:0.42:1.59)	698 (4.98), 628 (4.34), 426 (4.23), 329 (4.76)	785, 768 (1:0.98)
IVf	691, 641, 413, 334 (1:0.85:0.41:1.57)	697 (4.98), 627 (4.35), 427 (4.23), 331 (4.75)	788, 769 (1:0.97)

wave part of the spectrum contains two intense bands at 691–694 (the monomeric form) and 639–651 ppm (the associated form). From the ratio of the absorption band intensities at the equal concentrations of complexes it can be considered qualitatively which of the compounds under investigation is more prone to association in DMF. It was found that compound **IVa** was more inclined to association than compounds **IVb–IVf** (see Fig. 1).

The complexes obtained exist in chloroform solution in the monomeric form. Their long-wave absorption band undergoes small red shift as compared to DMF solutions (about 5 nm). Nature of the aryl substituent does not affect practically either the character of the spectrum or the location of the absorption bands (see the table).

Using concentrated sulfuric acid instead of the organic solvent causes the significant red shift of the absorption bands. Its value is comparable with the analogous effect on the unsubstituted copper phthalocyanine. Under these conditions *Q*-band is split in two components of the approximately equal intensity (see Fig. 2 and the table). This effect was observed previously for the other octasubstituted copper phthalocyanines [28].

Testing of complexes obtained as dyes soluble in the organic media showed that they paint the polymers and waxes in the intense green color.

EXPERIMENTAL

The electron absorption spectra were obtained on a Hithachi U-2001 spectrophotometer in DMF, in chloroform, and in the concentrated sulfuric acid at room temperature in the range 325–900 nm. IR spectra were registered on an Avatar 360 FT-IR ESP spectrometer in the range 400–4000 cm^{-1} in thin layer (chloroform). ^1H NMR spectra were taken on a Bruker DPX-500 spectrometer (500.17 MHz) in $\text{DMSO}-d_6$. Chemical shifts are presented in the δ -scale against TMS. Elemental analyses of the compounds obtained were carried out on the CHNS-O FlashEA 1112 series analyzer.

4-Bromo-5-nitro (I) and 4-(morpholin-4-yl)-5-nitrophthalodinitrile (II) were obtained according to [29] and [21] respectively.

4-(Morpholin-4-yl)-5-aryloxyphthalodinitriles (IIIa–IIIf) (general procedure). A mixture of 0.01 mol of compound **II**, 0.02 mol of the anhydrous potassium

carbonate, 0.01 mol of substituted phenol or naphthol, and 30 ml of DMF was stirred at 130–140°C for 3 h. After cooling to room temperature the reaction mixture was poured in 100 ml of water, the precipitate obtained was filtered off, washed with 50 ml of water, and crystallized from DMF.

4-(Morpholin-4-yl)-5-phenoxyphthalodinitrile (IIIa). This compound was obtained according to the general procedure from 0.94 g of phenol. Yield 2.63 g (86%), mp 174–176°C. ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm (*J*, Hz): 7.67 s (1H), 7.24 s (1H), 7.15 m (5H, H–Ar), 3.65 t (4H, CH_2O , *J* 4.0), 3.30 t (4H, CH_2N , *J* 4.0). Found, %: C 70.57; H 5.23; N 13.56. $\text{C}_{18}\text{H}_{15}\text{N}_3\text{O}_2$. Calculated, %: C 70.81; H 4.95; N 13.76.

4-(Morpholin-4-yl)-5-(4-*tert*-butylphenoxy)phthalodinitrile (IIIb). This compound was obtained according to the general procedure from 1.50 g of 4-*tert*-butylphenol. Yield 2.66 g (79%), mp 148–150°C. ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm (*J*, Hz): 7.67 s (1H), 7.34 d (2H, *J* 8.3), 7.24 s (1H), 6.85 d (2H, *J* 8.3), 3.64 br.s (4H, CH_2O), 3.33 br.s (4H, CH_2N), 1.15 s (9H, *t*-Bu). Found, %: C 73.64, H 6.34, N 11.42. $\text{C}_{22}\text{H}_{23}\text{N}_3\text{O}_2$. Calculated, %: C 73.11, H 6.41, N 11.63.

4-(Morpholin-4-yl)-5-([3,4'-dimethyl(1,1'-biphenyl)-4-yl]oxy)phthalodinitrile (IIIc). This compound was obtained according to the general procedure from 1.98 g of 3,4'-dimethyl-(1,1'-biphenyl)-4-ol. Yield 3.19 g (78%), mp 221–223°C. ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm (*J*, Hz): 7.69 c (1H, H^3), 7.66 d (1H, H^2 , *J* 1.0), 7.57 d (2H, *J* 7.9), 7.51 d.d (1H, *J* 1.0, 8.5), 7.26 d (2H, *J* 7.9), 7.25 s (1H), 7.00 d (1H, *J* 8.5), 3.68 t (4H, CH_2O , *J* 4.2), 3.31 t (4H, CH_2N , *J* 4.2), 2.35 s (3H, Me), 2.24 s (3H, Me). Found, %: C 76.42, H 5.45, N 10.50. $\text{C}_{26}\text{H}_{23}\text{N}_3\text{O}_2$. Calculated, %: C 76.26, H 5.56, N 10.26.

4-(Morpholin-4-yl)-5-([3,3'-dimethyl(1,1'-biphenyl)-4-yl]oxy)phthalodinitrile (IIId). This compound was prepared according to the general procedure from 1.98 g of 3,3'-dimethyl-(1,1'-biphenyl)-4-ol. Yield 3.32 g (81%), mp 207–209°C. ^1H NMR spectrum ($\text{DMSO}-d_6$), δ , ppm: 7.69 s (1H), 7.67 d (1H, *J* 1.2), 7.52 d.d (1H, *J* 1.2, 8.5), 7.49 s (1H), 7.45 d (1H, *J* 7.9), 7.38 t (1H, *J* 7.9), 7.26 s (1H), 7.17 d (1H, *J* 7.9), 7.01 d (1H, *J* 8.5), 3.68 t (4H, CH_2O , *J* 4.3), 3.30 t (4H, CH_2N , *J* 4.3), 2.38 s (3H), 2.25 s (3H). Found, %: C 76.35, H 5.72, N 10.52. $\text{C}_{26}\text{H}_{23}\text{N}_3\text{O}_2$. Calculated, %: C 76.26, H 5.66, N 10.26.

4-(Morpholin-4-yl)-5-(1-naphthyloxy)phthalodinitrile (IIIe). This compound was obtained according

to the general procedure from 1.98 g of 1-naphthol. Yield 2.58 g (78%), mp 161–162°C. Found, %: C 72.78, H 5.21, N 12.60. $C_{20}H_{17}N_3O_2$. Calculated, %: C 72.49, H 5.17, N 12.68.

4-(Morpholin-4-yl)-5-(2-naphthoxy)phthalodinitrile (III_f). This compound was obtained according to the general procedure from 1.44 g of 2-naphthol. Yield 2.74 (83%), mp 190–192°C. Found, %: C 72.70, H 5.21, N 12.55. $C_{20}H_{17}N_3O_2$. Calculated, %: C 72.49, H 5.17, N 12.68.

Copper tetra-4-(morpholin-4-yl)-tetra-5-aryloxyphthalocyanines (IV_a–IV_e) (general procedure). A batch of substituted phthalodinitrile, 0.5 mmol, was thoroughly ground with 0.14 mmol of copper acetate dihydrate and 1 mmol of carbamide. The mixture obtained was heated to 180–185°C and kept for 2 h. The reaction mixture was washed successively with 5% hydrochloric acid until the decoloration of the filtrate, and then with water until the neutral reaction of the medium. The target products were extracted with chloroform. Final purification was carried out by column chromatography on Al_2O_3 , elution with chloroform. Solvent was distilled off in a vacuum, and the target product was dried at 80°C. The complexes obtained are dark green powders insoluble in water and soluble in the organic solvents.

Copper tetra-4-(morpholin-4-yl)-tetra-5-phenoxyphthalocyanine (IV_a). This compound was prepared according to the general procedure from 153 mg of the compound III_a. Yield 83.4 mg (52%). Found, %: C 67.16, H 4.67, N 13.28. $C_{72}H_{60}N_{12}O_8Cu$. Calculated, %: C 67.30, H 4.71, N 13.08.

Copper tetra-4-(4-*tert*-butylphenoxy)-tetra-5-(morpholin-4-yl)phthalocyanine (IV_b). This compound was prepared according to the general procedure from 181 mg of compound III_b. Yield 78.6 mg (47%). Found, %: C 67.72, H 6.71, N 12.04. $C_{80}H_{92}N_{12}O_8Cu$. Calculated, %: C 67.99, H 6.56, N 11.89.

Copper tetra-4-(morpholin-4-yl)-tetra-5-{[3,4'-dimethyl(1,1'-biphenyl)-4-yl]oxy}phthalocyanine (IV_c). This compound was prepared according to the general procedure from 205 mg of compound III_c. Yield 89.1 mg (43%). Found, %: C 73.85, H 5.58, N 10.04. $Cu_{104}H_{92}N_{12}O_8Cu$. Calculated, %: C 73.41, H 5.45, N 9.88.

Copper tetra-4-(morpholin-4-yl)-tetra-5-{[3,3'-dimethyl(1,1'-biphenyl)-4-yl]oxy}phthalocyanine

(IV_d). This compound was prepared from 205 mg of compound III_d. Yield 87.6 mg (41%). Found, %: C 73.69, H 5.56, N 10.90. $C_{104}H_{92}N_{12}O_8Cu$. Calculated, %: C 73.41, H 5.45, N 9.88.

Copper tetra-4-(morpholin-4-yl)-tetra-5-(1-naphthoxy)phthalocyanine (IV_e). This compound was prepared according to the general procedure from 118 mg of the compound III_e. Yield 60.1 mg (49%). Found, %: C 68.98, H 5.01, N 11.89. $C_{80}H_{68}N_{12}O_8Cu$. Calculated, %: C 69.18, H 4.93, N 12.10.

Copper tetra-4-(morpholin-4-yl)-tetra-5-(2-naphthoxy)phthalocyanine (IV_f). This compound was prepared according to the general procedure from 118 mg of the compound III_f. Yield 57.5 mg (47%). Found, %: C 69.37, H 5.09, N 11.89. $C_{80}H_{68}N_{12}O_8Cu$. Calculated, %: C 69.18, H 4.93, N 12.10.

ACKNOWLEDGMENTS

The work was carried out with the financial support of the Ministry of Science and Education of the Russian Federation (no. RNP 2.2.1.1.7280, no. RNP 2.1.1.1180).

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