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Nucleophilic Substitution in 4-Bromo-5-nitrophthalonitrile: XIV.[1] Synthesis and Properties of 4,5-Bis[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile and Phthalocyanines Therefrom

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Abstract—4,5-Bis[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile was prepared by the nucleophilic substitution of the nitro group and bromine in 4-bromo-5-nitrophathalonitrile. The product was used to synthesize the corresponding octasubstituted phthalocyanine, as well as its magnesium, copper, zinc, cobalt, and nickel complexes. The spectral properties of the synthesized phthalocyanines were studied.

Keywords: 4-bromo-5-nitrophthalonitrile, nucleophilic substitution, metal phthalocyanines, electronic absorption spectra

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At present phthalocyanines and complexes are actively studied due to their wide practical use [2, 3]. Special attention is attached to liquid crystalline phthalocyanines [4–6] as new materials for nanotechnology [7–9]. As both tetrasubstituted [10] and bifunctionally substituted phthalocyanines [2] are synthesized from the corresponding unsymmetrically substituted phthalonitriles [2, 11, 12], such syntheses may form mixtures of randomers of various structures [10, 13, 14], which are extremely difficult to separate because of the close physicochemical characteristics of these compounds. At the same time, the physicochemical [15] and, in particular, liquid crystalline properties of phthalocyanines [3, 7] are quite sensitive to how uniform is the composition of the studied substance. For instance, the presence of randomers may affect both the temperature parameters of the mesophase and the very ability of the compound mesophase formation [3, 7]. The formation of randomer mixtures is commonly prevented by prepacycle highly symmetric phthalocyanines on the basis of phthalonitriles having the same substituents in the 3- and 6- or 4- and 5-positions of the benzene cycle [11, 15–17].

We previously found that copper tetra-4-(benzotriazol-1-yl)tetra-5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanine [18], which contains no bulky peripheral aliphatic substituents, forms an enantiotropic mesophase and shows the ability to retain the mesophase structure on glass transition [19]. Therefore, for the research on the influence of the chemical structure of substituted phthalocyanines on their physicochemical properties of interest is to vary the nature of the substituents *ortho* to the 4-(1-methyl-1-phenylethyl)phenoxy group by prepacycle highly symmetrical phthalocyanines derived from 4-(1-methyl-1-phenylethyl)phenol.

The aim of the present work was to synthesize octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]pthalocyanine and its metal complexes and to study the influence of the structure of the synthesized phthalocyanine derivatives on their physicochemical properties.

First we synthesized 4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile (**2**) starting from 4-bromo-5-nitrophthalonitrile (**1**) (Scheme 1). The reaction was

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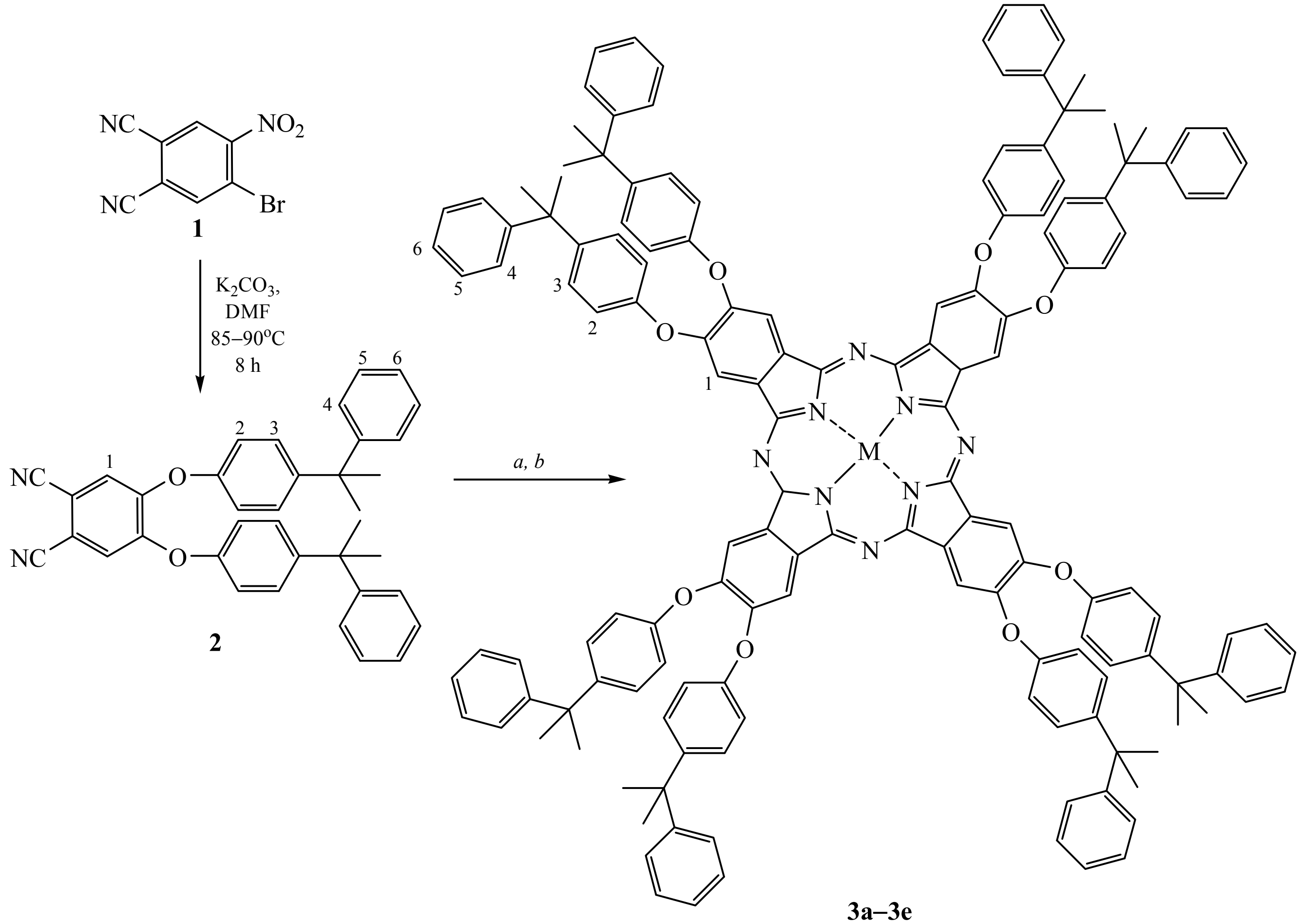
[1] For communication XIII, see [1].

Scheme 1.



3a–3e

a, $M(CH_3COO)_2 \cdot nH_2O$, 160–170°C; *b*, $CoCl_2 \cdot 6H_2O$, 155–160°C; M = Mg (**a**, *n* = 4), Cu (**b**, *n* = 2), Co (**c**), Ni (**d**, *n* = 4), Zn (**e**, *n* = 2).

performed in aqueous DMF at 80–90°C in the presence of an equimolar amount of potassium carbonate which acts as a deprotonating agent [20, 21], for 8 h. When the reaction time was decreased to 4 h, only bromine substitution was observed, and no target product formed.

The composition and structure of compound **2** were confirmed by elemental analysis, MALDI–TOF mass spectrometry, ^1H NMR and IR spectroscopy. The mass spectrum of compound **2** shows signals at *m*/*z* 549 and 588 corresponding to the $[M + H]^+$ and $[M + K]^+$ ions.

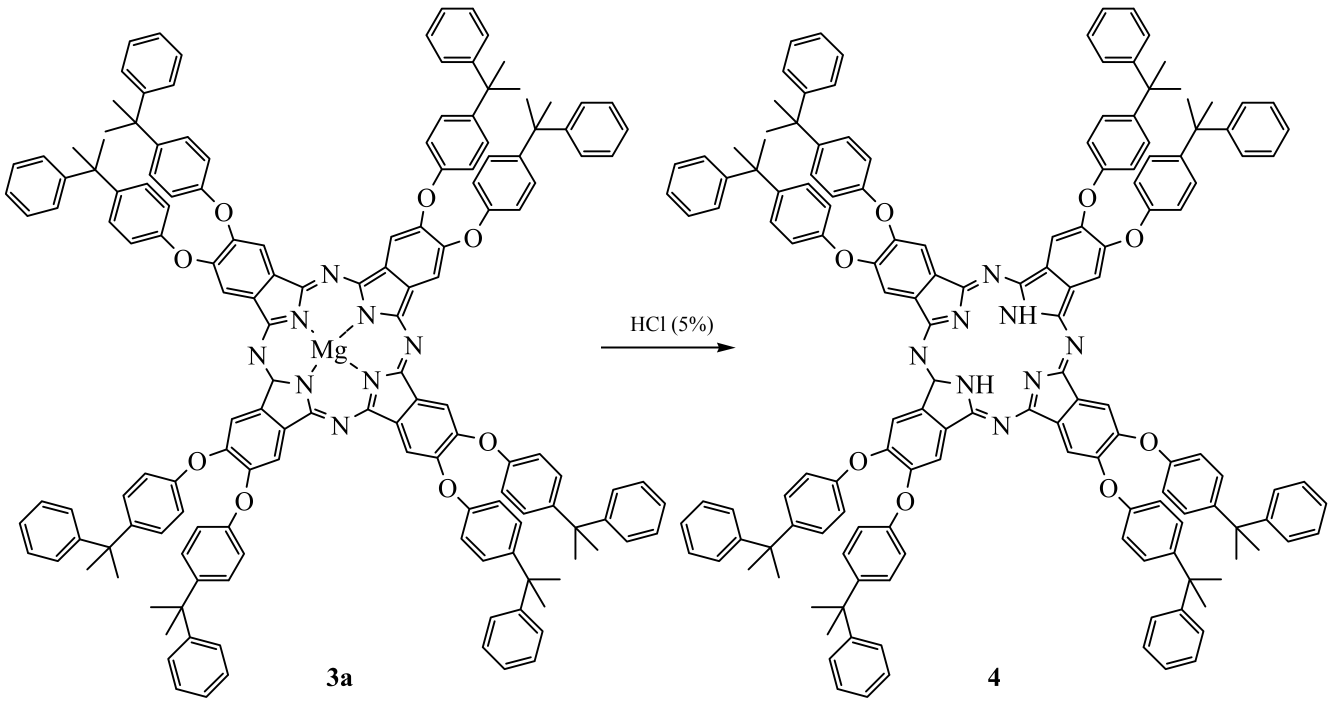
The IR spectrum of compound **2** does not contain the stretching and deformation vibration bands of the nitro group, which are present in the spectrum of the starting 4-bromo-5-nitrophthalonitrile (**1**) [22], and contains the stretching vibration bands of the cyano (2233 cm^{-1}) and Ar–O–Ar groups (1218 cm^{-1}) [23].

The ^1H NMR spectrum of compound **2** displays an upfield (2.19 ppm) 12-proton strong singlet signal of the methyl groups of the 4-(1-methyl-1-phenylethyl)-phenoxy substituents. Downfield benzene proton signals of the phthalonitrile molecule (7.33 ppm) and phenoxy (6.96–6.98 ppm) and oxyaryl groups (7.09–7.17 ppm) are also observed.

By melting compound **2** together with magnesium, copper, nickel, and zinc acetates, as well as with cobalt chloride for 1.5 h we synthesized the corresponding octasubstituted metal phthalocyanines **3a**–**3d** (Scheme 1). Treatment of compound **3a** with 5% aqueous HCl gave phthalocyanine **4** (Scheme 2).

The synthesized compounds are dark green solids insoluble in water and readily soluble in chloroform, acetone, and DMF. It should be noted that compounds **3a**–**3d** are quite poorly soluble in conc. H_2SO_4.

Scheme 2.



Phthalocyanine **4** does not dissolve in sulfuric acid even on prolonged standing and decomposes on heating.

As known [24, 25], the dissolution of phthalocyanine and its derivatives in conc. H_2SO_4 is associated with the protonation of the *meso*-nitrogen atoms of the phthalocyanine macrocycle. Probably, in our case, the bulky and branched 4-(1-methyl-1-phenylethyl)phenoxy groups prevent efficient interaction of H_2SO_4 with the macrocyclic core of the phthalocyanine molecule, which is responsible for the low solubility of compounds **3** and **4** in sulfuric acid.

The synthesized phthalocyanine derivatives were identified by elemental analysis, IR and ^{1}H NMR spectroscopy, and MALDI–TOF mass spectrometry.

The IR spectra of octasubstituted phthalocyanines **3** and **4** display vibration bands of the oxyaryl groups (1213–1216 cm^{-1}), and well as skeletal vibration bands (1320–1340, 1560–1605 cm^{-1}) of the macrocylic core of the phathalocyanine molecule [26]. At the same time, the band at 2233 cm^{-1}, characteristic of the cyano groups [23] in phthalonitrile **2** is no longer observed, which points to a lack of admixture of the parent compound in the products. The spectrum of phthalocyanine **4** shows bands at 3157 and 1016 cm^{-1} assignable to endocyclic imino groups [26]. These bands are absent from the spectrum of the parent magnesium phthalocyanine **3a**.

Since compounds **3a**–**3e** and **4** contain 8 bulky 4-(1-methyl-1-phenylethyl)phenoxy substituents that shield the chelated metal atom (cobalt or copper) from от RF radiation (like it was previously observed with other phthalocyanine derivatives with bulky and long-chain substituents [27, 28]) and enhance the solubility of the compounds in chloroform, we could obtain the ^{1}H NMR spectra of the synthesized copper, nickel, and cobalt phthalocyanines to find that they are almost completely coincide with the spectra of the parent phthalonitrile **2**.

The electronic absorption (UV-Vis) spectra of octasubstituted phthalocyanines **3** and **4** gave evidence showing that the synthesized compounds all are unassociated in organic solutions. Therefore, we could measure their extinction coefficients (see table). A solvatochromic effect that revealed itself in the bathochromic shift of the *Q*-band by 3–8 nm for all the studied compounds (Fig. 1), as well as in the increase in the molar extinction coefficient in going from DMF to chloroform, was observed. Furthermore, the position of the longwave absorption band was affected by the nature of the complexing metal, as evidenced by the fact that the bathochromic shift of this band changes with metal in the following order: **3c** (Co) < **3d** (Ni) < **3a** (Mg), **3b** (Cu), **3e** (Zn).

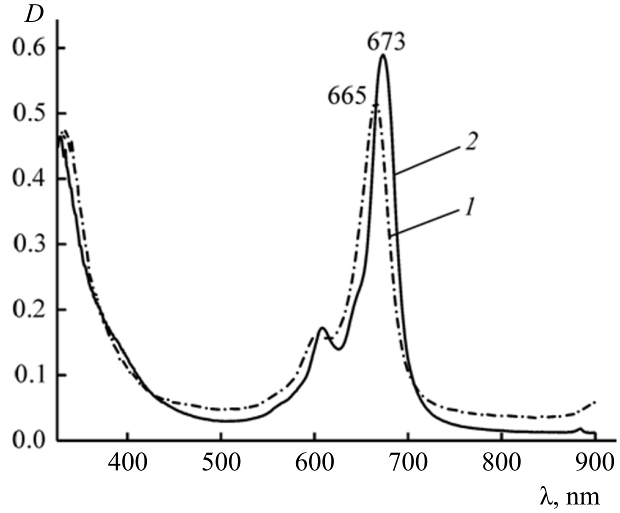


Fig. 1. Electronic absorption spectra of compound **3c** in (*1*) DMF and (*2*) chloroform ($c = 0.45\times10^{-5}$ M).

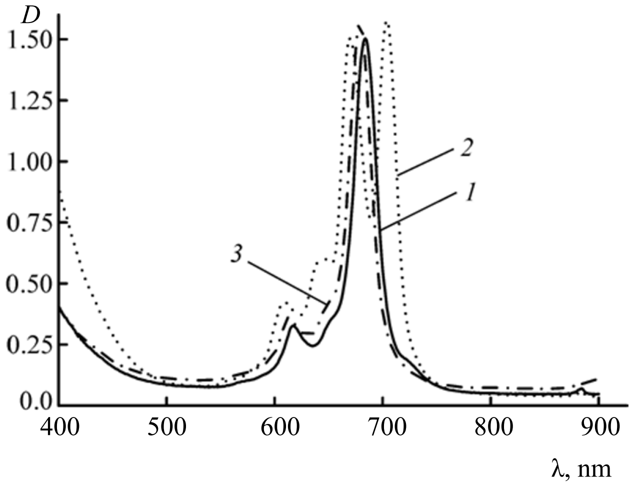


Fig. 2. Electronic absorption spectra of compounds **3a** and **4** in different solvents ($c = 0.85\times10^{-5}$ M): (*1*) **3a** ($CHCl_3$); (*2*) **4** ($CHCl_3$); and (*3*) **4** (DMF).

UV-Vis spectrum of phthalocyanine **4** in chloroform contains two strong longwave bands, which is characteristic of phthalocyanine ligands [24, 25]. In DMF no splitting of the *Q*-band takes place (Fig. 2). The lacking *Q*-band splitting in the basic medium is explained by an increase in the symmetry of the phthalocyanine molecule on the deprotonation of the endocyclic nitrogen atoms in the organic base medium to form the dianionic form of the phthalocyanine, which, like metal phthalocyanines, has a D_{4h} symmetry [29].

The transition from organic solvents to conc. H_2SO_4 is accompanied by a bathochromic shift of the longwave absorption bands by more than 100 nm (see table, Fig. 3). This shift depends on the nature of the complexing metal and increases in the order: **3a** (Mg) < **3d** (Ni) < **3e** (Zn) < **3c** (Co) < **3b** (Cu). Compound **3a** in conc. H_2SO_4 decomposes even on short standing, which is associated with demetalation and subsequent decomposition of the macroheterocycle.

Comparison of the UV-Vis spectra of copper phthalocyanine **3b** and phthalocyanine **4** with those of the previously studied tetra-4-(benzotriazol-1-yl)tetra-5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanine and its copper complex reveals hypsochromic shift of *Q*-band by 4 nm on the replacement of the benzotriazole fragment by 4-(1-methyl-1-phenylethyl)phenoxy group in organic solvents [18] and a bathochromic shift of the longwave band by 28 nm for compound **3b** in conc. H_2SO_4 (Fig. 3).

Thus, the nucleophilic substitution of the nitro group and bromine by the 4-(1-methyl-1-phenyl-ethyl)-phenoxy group was used to obtain 4,5-bis[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile. Melting the latter compound together with some *d*-metal salts gave previously unknown metal phthalocyanines. Demetalation of the magnesium complex of octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanine gave the corresponding phthalocyanine. The position of the

Electronic absorption spectra of octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanines **3** and **4**

Comp. no.	M	ESP, λ, nm (log ε)		
		DMF	$CHCl_3$	H_2SO_4
3a	Mg	361 (4.48), 613 (4.06), 679 (4.76)	383 (4.25), 618 (4.15), 685 (4.82)	805
3b	Cu	344(4.92), 612 (4.71), 679 (5.15)	340 (5.00), 615 (4.74), 684 (5.22)	838
3c	Co	333(4.63), 600 (4.16), 665 (4.65)	326(4.72), 610 (4.27), 673 (4.82)	823
3d	Ni	371(4.41), 605 (4.34), 673 (4.84)	382 (4.39), 609 (4.33), 675 (4.95)	812
3e	Zn	365 (4.85), 612 (4.75), 679 (5.00)	354 (4.74), 616 (4.34), 683 (5.08)	814
4	HH	367 (4.44), 603(4.31), 678 (4.84)	610 (4.32), 647 (4.33), 670 (4.86), 704 (4.88)	–

Q-band is affected by the nature of the complexing metal, as evidenced by the bathochromic shifts.

EXPERIMENTAL

UV-Vis spectra were recorded in organic solvents (DMF and chloroform), aqueous alkalis, and concentrated sulfuric acid on a HITACHI U-2001 spectrophotometer at room temperature in the range 325–900 nm. The IR spectra were registered on a Nicolet Avatar 360 FTIR ESP spectrometer in the range 400–4000 cm^{-1} in thin films (chloroform) and KBr pellets. The ^{1}H NMR spectra ($CDCl_3$) were registered on a Bruker DRX-500 instrument. Elemental analysis was performed on a FlashEA 1112 CHNS-O analyzer. The MALDI–TOF mass spectra were obtained on a Shimadzu Biotech Axima Confidence instrument in the positive ion mode, matrix 2,5-dihydroxybenzoic acid.

4-Bromo-5-nitrophthalonitrile (1) was synthesized by the known procedure [30], mp 140–142°C. Found, %: C 38.10; H 0.76; N 16.50. $C_8H_2BrN_3O_2$. Calculated, %: C 38.16; H 0.80; N 16.67.

4,5-Bis[4-(1-methyl-1-phenylethyl)phenoxy]phthalonitrile (2). A solution of 2.76 g (2 mmol) K_2CO_3 in 10 mL of water was added to a solution of 2.52 g (1 mmol) of compound **1** and 4.24 g (2 mmol) of 4-(1-methyl-1-phenylethyl)phenol in 70 mL of DMF. The mixture was stirred at 80–90°C for 8 h and then poured into water. The viscous reddish-brown liquid that formed was separated, diluted with 10 mL of aqueous isopropanol (1 :1, vol/vol), and left to stand for 3 days. A precipitate formed and was filtered off, washed with isopropanol and water, and dried in air at 80°C. Yield 2.40 g (44%), mp 123–125°C. IR spectrum, ν, cm^{-1}: 2967, 2929, 2869 (CH_3), 2233 (CN), 1589 (C–C), 1318, 1162 (CH_{Ar}), 1217 (Ar–O–Ar). ^{1}H NMR spectrum, δ, ppm: 7.33 s (2H, H^1), 7.01–7.03 m (4H, H^2), 6.96–6.98 m (4H, H^3), 7.09–7.17 m (10H, H$^{4–6}$), 2.19 s (12H, CH_3). Mass spectrum, *m*/*z* 549.19 (34.95) $[M + H]^+$, 588.13 (83.23) $[M + K]^+$. *M* 548.68. Found, %: C 82.95; H 6.02; N 5.11. $C_{38}H_{32}N_2O_2$. Calculated, %: C 83.19; H 5.88; N 4.96.

Metal octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanines 3a–3e. A thoroughly triturated mixture of 55 mg (0.1 mmol) of compound **2**, 50 mg (0.8 mmol) of urea, and 0.1 mmol of metal acetate or chloride was heated at 155–170°C for 1.5 h, after which it was dissolved in chloroform and chromatographed on alumina (eluent—chloroform).

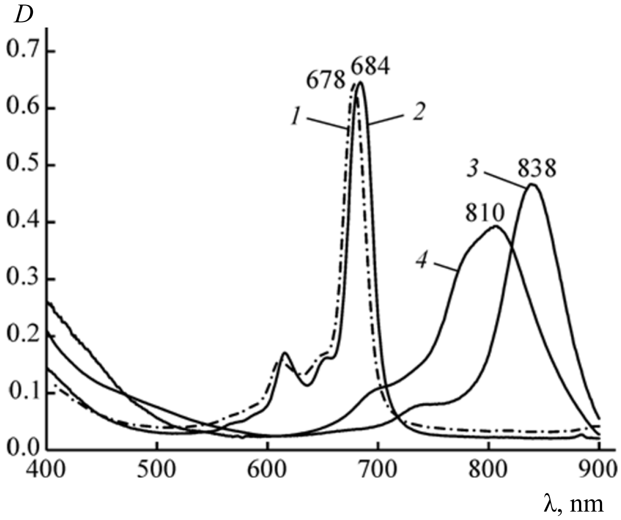


Fig. 3. Electronic absorption spectra of compound **3b** in (*1*) DMF, (*2*) chloroform, (*3*) H_2SO_4, and (*4*) of copper tetra-4-(benzotriazol-1-yl)tetra-5-[4-(1-methyl-1-phenylethyl)-phenoxy]phthalocyanine in H_2SO_4 ($c = 0.45\times10^{-5}$ M).

Magnesium octa-4,5-[4-(1-methyl-1-phenylethyl)-phenoxy]phthalocyanine (3a) was synthesized from compound **2** and 21.4 mg of magnesium acetate tetrahydrate. Yield 37.2 mg (67%). IR spectrum, ν, cm^{-1}: 2963, 2922, 2867 (CH_3), 1567 (C–C), 1322, 1165 (CH_{Ar}), 1212 (Ar–O–Ar). Mass spectrum, *m*/*z*: 2219.29 (80.57) $[M]^+$. *M* 2219.04. Found, %: C 82.05; H 6.04; N 4.94. $C_{152}H_{128}N_8O_8Mg$. Calculated, %: C 82.27; H 5.81; N 5.05.

Copper octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy] (3b) was synthesized from compound **2** and 21.7 mg of copper acetate dihydrate. Yield 43.8 mg (80%). IR spectrum, ν, cm^{-1}: 2966, 2929, 2872 (CH_3), 1602 (C–C), 1272, 1137 (CH_{Ar}), 1214 (Ar–O–Ar). ^{1}H NMR spectrum, δ, ppm: 7.31 s (8H, H^1), 7.01–7.03 m (16H, H^2), 6.96–6.98 m (16H, H^3), 7.07–7.22 m (40H, H$^{4–6}$), 2.17 s (48H, CH_3). Mass spectrum, *m*/*z*: 2257.57 (92.85) $[M + 2H]^+$. *M* 2255.91. Found, %: C 80.22; H 5.95; N 4.90. $C_{152}H_{128}N_8O_8Cu$. Calculated, %: C 80.84; H 5.71; N 4.96.

Cobalt octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanine (3c) was synthesized from compound **2** and 23.8 mg of cobalt chloride hexahydrate. Yield 42.5 mg (78%). IR spectrum, ν, cm^{-1}: 2954, 2923, 2862 (CH_3), 1561 (C–C), 1328, 1125 (CH_{Ar}), 1213 (Ar–O–Ar). ^{1}H NMR spectrum, δ, ppm: 7.33 s (8H, H^1), 7.00–7.03 m (16H, H^2), 6.97–7.00 m (16H, H^3), 7.09–7.20 m (40H, H$^{4–6}$), 2.17 s (48H, CH_3). Mass spectrum, *m*/*z*: 2253.57 (92.83) $[M + 2H]^+$. *M* 2251.13.

Found, %: C 80.82; H 6.00; N 4.82. $C_{152}H_{128}N_8O_8Co$. Calculated, %: C 81.01; H 5.72; N 4.97.

Nickel octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanine (3d) was synthesized from compound **2** and 24.8 mg of nickel acetate tetrahydrate. Yield 23.8 mg (43%). IR spectrum, ν, cm^{-1}: 2960, 2920, 2863 (CH_3), 1562 (C–C), 1321, 1125 (CH_{Ar}), 1214 (Ar–O–Ar). ^1H NMR spectrum, δ, ppm: 7.32 s (8H, H^1), 7.01–7.03 m (16H, H^2), 6.97–6.99 m (16H, H^3), 7.07–7.18 m (40H, $H^{4–6}$), 2.19 s (48H, CH_3). Mass spectrum, *m*/*z*: 2253.06 (90.12) [*M*]$^+$. *M* 2253.43. Found, %: C 80.78; H 6.12; N 4.88. $C_{152}H_{128}N_8O_8Ni$. Calculated, %: C 81.02; H 5.73; N 4.97.

Zinc octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]phthalocyanine (3e) was synthesized from compound **2** and 21.7 mg of zinc acetate dihydrate. Yield 28.8 mg (53%). IR spectrum, ν, cm^{-1}: 2965, 2933, 2869 (CH_3), 1604 (C–C), 1270, 1131 (CH_{Ar}), 1213 (Ar–O–Ar). Mass spectrum, *m*/*z*: 2260.00 (92.75) [*M*]$^+$. *M* 2260.12. Found, %: C 80.48; H 6.05; N 4.87. $C_{152}H_{128}N_8O_8Zn$. Calculated, %: C 80.78; H 5.71; N 4.96.

Octa-4,5-[4-(1-methyl-1-phenylethyl)phenoxy]-phthalocyanine (4) was prepared by adding 4 drops of 5% aqueous HCl to a solution of 10 mg of compound **3a** in 2 mL of chloroform. The chloroform was then removed, and the residue was washed with water to neutral washings and dried in air at 70–80°C. Yield 8.2 mg (82%). IR spectrum, ν, cm^{-1}: 3056 (NH), 2965, 2923, 2860 (CH_3), 1565 (C–C), 1324, 1162 (CH_{Ar}), 1216 (Ar–O–Ar), 1016 (H_2Pc). Mass spectrum, *m*/*z*: 2196.25 (80.57) [*M*]$^+$. *M* 2196.75. Found, %: C 83.05; H 6.10; N 5.00. $C_{152}H_{130}N_8O_8$. Calculated, %: C 83.11; H 5.96; N 5.10.

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