Synthesis of Magnesium Octa-4,5-phenoxyphthalocyanine and Sulfo- and Alkylsulfamoyl Derivatives on Its Basis

S. A. Znoiko^a*, A. I. Petlina^a, G. P. Shaposhnikov^a, and N. N. Smirnov^a

^a Ivanovo State Chemical Technological University, Institute of Macroheterocyclic Compounds, Sheremetevskii pr. 7, Ivanono, 153000 Russia *e-mail: znoykosa@yandex.ru

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Abstract—By the reaction of nucleophilic substitution of bromine atom and the nitro group of 4-bromo-5nitrophthalonitrile 4,5-diphenoxyphthalonitrile was obtained and on its basis new derivatives of octa-4,5phenoxyphthalocyanine were synthesized soluble in water- and organic solvents. The spectral properties and thermodestruction of the products were investigated.

Keywords: 4-bromo-5-nitrophthalonitrile, octa-4,5-phenoxyphthalocyanine, phenoxy- and sulfophenoxysubstituted phthalocyanines, Mg-phthalocyanine

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Phthalocyanines, whose key structural motif is porphyrin chromophore modified with aza-bridges [1], remain the objects of constant intense studies [1–3]. Particular interest attract aryloxy substituted phthalocyanines [4–6] as well as their water-soluble derivatives, as new materials for nanotechnology [7–9].

Phenoxy group is the simplest aroxy substituent. At the same time the phenoxy and sulfophenoxy substituted phthalocyanines possess a number of useful properties. Thus, water-soluble metal complex of tetra-4-(4-sulfophenoxy)phthalocyanine with cobalt(II) exhibits an activity in oxidation of mercaptans in the process of catalytic purification of liquified casinghead gas, and its activity is higher than that of commercial catalyst, cobalt disulfophthalocyanine, and the degree of conversion, by an example of propanethiol, is higher [10]. The metal complex with zinc(II) of similar structure demonstrated luminescent properties with the quantum yield of luminescence and the vield of singlet oxygen being one of the best among the investigated compounds [11]; it also shows antimicotic and antiviral activity [12].

Since tetrasubstituted phthalocyanines are prepared on the basis of the corresponding unsymmetrically substituted phthalonitriles, the formation of inseparable mixtures of positional structural isomers is possible [2, 13-15]. It is noteworthy that physicochemical properties of organic compounds are very sensitive to homogeneity of the studied compound [16, 17]. Hence, to avoid the formation of a mixture of randomers, it is better to prepare highly symmetrical derivatives, that is, octaphenoxysubstituted phthalocyanines [18–21], which also possess a number of useful properties. However, there are no data in the literature on sulfonic acids and sulfonamides of octaphenoxysubstituted phthalocyanines, so it was of interest to synthesize new water- and organosoluble derivatives of octaphenoxy substituted phthalocyanines.

With the aforesaid in mind, the goal of the present work was the synthesis and investigation of some physicochemical properties of sulfo and alkylsulfamoyl derivatives of octa-4,5-phenoxyphthalocyanine. In the first stage the initial 4,5-diphenoxyphthalonitrile was synthesized (Scheme 1).

The method is known of the synthesis of 4,5-diphenoxyphthalonitrile **3** from 4,5-dichlorophthalonitrile in nonaqueous DMSO with addition by portions of potassium carbonate [22–24]. The reaction time did not exceed 2 h, the yield of product **3** reached 82%. The replacement of DMSO by DMF and the increase in the time of heating did not increase the yield.

There was also an attempt to synthesize compound **3** basing on 4-bromo-5-nitrophthalonitrile **1** in aqueous DMF in the presence of potassium carbonate as



deprotonating agent [25] with the isolation of the intermediate nitrosubstituted phthalonitrile **2**. This led to an increase in the reaction time, complicated the synthetic procedure, and decreased the yield of the target product to 78%. In simultaneous substitution of bromine and nitro group in aqueous DMF in the presence of potassium carbonate at 80–90°C in the course of 8 h (Scheme 1) compound **3** is formed in a higher yield (85%). Therefore, the use of 4-bromo-5-nitrophthalonitrile as a precursor allows preparing 4,5-diphenoxyphthalonitrile **3** in a higher yield than when using commercial 4,5-dichlorophthalonitrile.

Then, based on compound **3**, magnesium octa-4,5phenoxyphthalocyanine **4** was obtained by the nitrile method in the presence of magnesium acetate by heating for 2 h at $180-185^{\circ}C$ (Scheme 2).

Compound **4** was extracted from the reaction mixture with chloroform and purified by column chromatography with chloroform as an eluent with subsequent removal of the solvent. The synthesized compound is dark-green solid insoluble in water, readily soluble in organic solvents: chloroform, acetone, and DMF.

Magnesium phthalocyanine **4** was treated with excess of chlorosulfonic acid and thionyl chloride to obtain sulfochloride **5**. The ¹H NMR spectrum of compound **5** contains the signals of protons of the phenoxy

group in the *ortho*-positions to oxygen at 7.67 ppm and to the sulfochloride group at 8.29 ppm, and the signal of protons of the benzene rings of the phthalocyanine molecule at 7.63 ppm. Sulfonic acid **6** was synthesized by hydrolysis of sulfochloride **5** in boiling water. In the ¹H NMR spectrum of sulfonic acid **6** the signals are broader as compared to those in the spectrum of sulfochloride **5**, the signal of SO₃H groups appears at 8.94 ppm, and a slight shift was observed for the signal of proton in the position *3* of the phenoxy group. Alkylsulfamoyl derivatives of phthalocyanines **7**, **8** were obtained by the reaction of sulfochloride **5** with diethyl- and octadecylamine in boiling acetone (Scheme 2).

The analysis of electron absorption spectra (UV-Vis specta) of octaphenoxy substituted phthalocyanines in DMF revealed that the introduction of substituents in the *para*-position of the phenoxy groups results in a small red shift of the longwave absorption band, the shift being the larger the bulkier and/or longer is the introduced substituent (Table 1).

The spectrum of magnesium octa-4,5-phenoxyphthalocyanine 4, originally containing a single Qband, suffers notable changes after sulfochlorination and subsequent hydrolysis. Due to high lability of magnesium complexes of phthalocyanine in acid media, not only the sulfonic group is introduced but







 $X = N(Et)_2$ (7), $NHC_{18}H_{37}$ (8).

also the metal atom is removed, as proved by the presence of two longwave absorption bands in the UV-Vis specta of compounds 6-8 (Table 1).

Sulfonic acid 6 is soluble in water and aqueous alkaline media. UV-Vis specta of this compound in water is characterized by the presence of broadened

Table 1. Electron absorption spectra of octaphenoxysubstituted

 phthalocyanines

Comp. no.	λ_{max}, nm			
	DMF	CHCl ₃	H_2SO_4	
4	678	683	_	
6	669, 696	_	767, 804	
7	648, 698	664, 700	770, 804	
8	668, 701	663, 701	813, 874	

Table 2. Temperature parameters of thermooxidative destruction of octaphenoxy substituted phthalocyanines

Comp. no.	$T_{\rm exo/endo}, ^{\circ}{ m C}$		
	I stage	II stage	$\Delta I_{\rm exo}, C$
4	290	570	500-650
6	264 ^a	492	475–540
7	260 ^a	571	475-620
8	326	484	480–600

^a Temperature at which the weight loss is maximal.

Q bands at 670 and 701 nm as well as by intense diffuse absorption band at 640 nm. The shape of the spectrum suggests that compound **6** exists in water in an associated form [26].

UV-Vis specta of the synthesized compounds in conc. sulfuric acid are characterized by a red shift of the Q band with respect to the spectra in organic solvents (Table 1) by more than 100 nm. In the case of compound **8**, an additional intense absorption band appears in the long-wave region of the spectrum at 874 nm; the same band was fixed in the spectra of **6**, **7** but only as an inflection.

Note the poor solubility of all synthesized compounds in sulfuric acid, connected, apparently, with the presence of eight bulky phenoxy groups on the periphery of the phthalocyanine molecule that, in addition, contain branched alkyl substituents (in the case of compounds 6-8) [26], which hinder protonation of the *meso*-nitrogen atoms of the phthalocyanine macroring.

Phthalocyanine derivatives containing long and/or branched alkyl fragments on the periphery of the molecule are known to be of interest from the view-point of manifesting liquid crystal properties [27–29]. Therefore, we have studied the behavior of compounds **4**, **6–8** at heating in the presence of air oxygen (Table 2).

The effect of the nature of the substituents in the *para*-position of the phenoxy group on the behavior of the synthesized compounds at heating was found (Table 2). It was noted, that the process of thermodestruction of sulfo (6) or diethylsulfamoyl derivatives (7) in the first stage is followed by intense loss of the mass of the studied sample approximately by 55–60%. Therewith, on the differential scanning calorimetry (DSC) curve an intense endo peak was fixed at 260–264°C. The temperature of maximal exo effect for the sulfoderivative 6 is by 79°C lower than that of the starting phthalocyanine 4. The presence of bulky diethylsulfamoyl fragments in the molecule of phthalocyanine 7 causes the increase in the temperature of maximal exo effect to 570°C (Table 2).

The process of decomposition of octadecylsulfamoyl derivative **8** proceeds also in two steps, the temperature of maximal exo effect of compound **8** being by 86°C lower than that of the initial magnesium octa-4,5-phenoxyphthalocyanine **4**.

Therefore, in the present work, 4,5-diphenoxyphthalonitrile was synthesized by nucleophilic substitution of the bromine atom and the nitro group of 4-bromo-5-nitrophthalonitrile. Fusion of this compound with magnesium acetate gave magnesium octa-4,5phenoxyphthalocyanine, and on its basis new waterand organosoluble derivatives of octa-4,5-phenoxyphthalocyanine were synthesized. It was found that introduction of alkylsulfamoyl fragments causes a red shift of the longwave absorption band of the synthesized compounds. Thermogravimetric study has shown that the highest temperature of the maximum exo effect was observed for magnesium octa-4,5phenoxyphthalocyanine and for the synthesized on its basis octa-4,5-(4-diethylsulfamoylphenoxy)phthalocyanine. The lowest temperature of the maximum exo effect was observed for *para*-octadecylsulfamoylphenoxysubstituted phthalocyanine.

EXPERIMENTAL

Electron absorption spectra were recorded in organic solvents (DMF and chloroform), aqueous alkali media, and concentrated sulfuric acid on a HITACHI U-2001 spectrometer at room temperature in the range 325-900 nm. IR spectra were taken on an Avatar 360 FT-IR ESP instrument in the range 400-4000 cm⁻¹ from KBr pellets. MALDI-TOF mass spectra were obtained on a Shimadzu Biotech Axima Confidence mass spectrometer in the positive ions mode. As a matrix, 2,5-dihydroxybenzoic acid was used. Thermooxidative destruction of the synthesized phthalonitriles was studied on a STA 449 F3 Jupiter (Netzsch, Germany) instrument for synchronous thermal analysis in oxygen-argon atmosphere in platinum crucible, heating rate 5°C/min. Prior to carrying out the elemental and thermogravimetric analysis the samples of the studied compounds were heated at 110°C for 2 h.

4-Bromo-5-nitrophthalonitrile (1) was synthesized by the known procedure [30], mp 140–142°C. Mass spectrum, m/z 251.18 $[M - H]^+$; calculated 252.03. Found, %: C 38.10, H 0.76, N 16.50. C₈H₂BrN₃O₂. Calculated, %: C 38.16, H 0.80, N 16.67.

4-Nitro-5-phenoxyphthalonitrile (2). 0.25 g (1 mmol) of 4-bromo-5-nitrophthalonitrile and 0.091 g (1 mmol) of phenol preliminarily crystallized from hexane were dissolved in 50 mL of DMF, and the solution of 0.14 g (1 mmol) of potassium carbonate in 5 mL of water was added. The reaction mixture was stirred at 20–25°C for 0.5 h, then the target product was filtered off, washed with hot water till the smell of phenol ceased, and dried at 80–90°C. Yield 0.24 g

(94%). IR spectrum, v, cm⁻¹: 2234 (C=N), 1535 [v_{as}(NO₂)], 1345 [v_s(NO₂)], 1208 (ArOAr). Found, %: C 63.57, H 2.58, N 18.03. $C_{14}H_7N_3O_3$. Calculated, %: C 63.40, H 2.66, N 18.10.

4,5-Diphenoxyphthalonitrile (3). *a*. 0.30 g (1.5 mmol) of 4,5-dichlorophthalonitrile and 0.28 g (3.0 mmol) of phenol preliminarily crystallized from hexane were dissolved in 15 mL of DMF and a solution of 0.42 g (3 mmol) of potassium carbonate in 1 mL water was added. The reaction mixture was stirred at 80–90°C for 12 h, then the target product was filtered off, washed with hot water till the smell of phenol ceased, and dried at 80–90°C. Yield 0.26 g (82%). Found, %: C 76.38; H 3.98; N 8.72. $C_{20}H_{12}N_2O_2$. Calculated, %: C 76.91; H 3.87; N 8.97.

b. Compound **3** was prepared similarly from 0.26 g (1 mmol) of 4-nitro-5-phenoxyphthalonitrile, 0.096 g (1 mmol) of phenol and 0.14 g (0.1 mmol) of potassium carbonate; reaction time 9 h. The target product was filtered off, washed with 2-propanol, and dried at 80–90°C. Yield 0.21 g (78%). IR spectrum, v, cm⁻¹: 2226 (CN), 1588 (C-C_{skel}), 1561 (–N=), 1243 (ArOAr), 693 (C-H_{def}). Found, %: C 76.42; H 4.00; N 8.69. $C_{20}H_{12}N_2O_2$. Calculated, %: C 76.91; H 3.87; N 8.97.

c. Compound **3** was prepared similarly from 2.52 g (0.01 mol) of 4-bromo-5-nitrophthalonitrile, 1.82 g (0.02 mol) of phenol and 2.76 g (0.02 mol) of potassium carbonate; reaction time 9 h. in 7 mL water. The target product was filtered off, washed with hot water until the smell of phenol ceased, and dried at 80-90°C. Yield 2.65 g (85%). IR spectrum, v, cm⁻¹: 2227 (CN), 1589 (C-C_{skel}), 1282 (C_{Ar}-H), 1243 (ArOAr). Mass spectrum, *m/z*: 313 [*M* + H]⁺ (33%), 335 [*M* + Na]⁺ (97%), 350 [*M* + K]⁺ (50%); calculated 312. Found, %: C 76.55; H 4.10; N 8.78. C₂₀H₁₂N₂O₂. Calculated, %: C 76.91; H 3.87; N 8.97.

Magnesium octa-4,5-(phenoxy)phthalocyanine (4). A mixture of 0.31 g (0.1 mmol) of compound **3** and 0.11 g (0.05 mmol) of magnesium acetate tetrahydrate was heated for 2 h at 180–185°C, then cooled and dissolved in chloroform. The precipitate was filtered off, the residue evaporated and chromatographed on silica using toluene as an eluent. Yield 0.27 g (82%). Mass spectrum, m/z: 1273 $[M]^+$, calculated 1273. Found, %: C 75.20; H 4.01; N 8.55. C₈₀H₄₈MgN₈O₈. Calculated, %: C 75.45; H 3.80; N 8.80.

Octa-4,5-(phenoxy)phthalocyanine sulfochloride (5). To the mixture of 2 mL (18 mmol) of chlorosulfonic acid and 2 mL (18 mmol) of thionyl chloride 0.25 g (0.2 mmol) of compound **4** was added at vigorous stirring. The reaction mixture was stirred at 20°C in the course of 0.5–1 h, then poured on ice treated with sodium chloride. The precipitate was separated using glass frit filter and dried in desiccator over sulfuric acid during 3 days. The target compounds were extracted with acetone, the solvent removed to obtain dark-green solid compound, readily soluble in acetone and DMF, moderately soluble in chloroform. Yield 0.33 g (80%). IR spectrum, v, cm⁻¹: 3563 (OH), 1614 (C–C), 1530 (N=), 1322 (S=O), 1291 (C_{Ar}-H), 1224 (ArOAr), 714 [δ (C–H). ¹H NMR spectrum, δ , ppm (CDCl₃): 8.29 d (16H, H3, J_{HH} = 8.15 Hz), 7.67 d (16H, H2, J_{HH} = 8.17 Hz), 7.63 s (16H, H1).

Octa-4,5-(4-sulfophenoxy)phthalocyanine (6). Mixture of 204 mg (0.1 mmol) of compound **5** and 10 mL of water was heated to complete dissolution, then water was removed. The obtained sulfonic acid was purified by column chromatography on silica M60 eluting with water. Yield 126 mg (67%). IR spectrum, v, cm⁻¹: 3407 (OH), 1587 (C–C), 1532 (N=), 1279 (C_{Ar}–H), 1240 (ArOAr), 1166 (S=O), 689 [δ (C–H)]. ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 9.84 s (8H, SO₃H), 8.35 s (16H, H³), 7.75 m (16H, H¹), 7.65 m (16H, H²). Found, %: C 49.75; H 3.08, N 5.77; S 12.89. C₈₀H₅₀N₈O₃₂S₈. Calculated, %: C 50.79; H 2.66; N 5.92; S 13.56.

Octa-4,5-(4-alkylsulfamoylphenoxy)phthalocyanines (7, 8). To a solution of 204 mg (0.1 mmol) of compound **5** in 30 mL of acetone the excess of diethylamine or 0.22 g of octadecylamine was added. The mixture was refluxed at 60°C for 1–1.5 h. The process was monitored by the completeness of dissolution of a sample of the reaction mixture in chloroform. After completion of the reaction, acetone was removed, the target products were extracted from the reaction mixture with chloroform. Finally, the products were purified by column chromatography on silica M60 eluting with chloroform.

Compound 7. Yield 193 mg (83%). IR spectrum, v, cm⁻¹: 3424 (OH), 3071 (NH), 2975, 2821 (CH₂, CH₃), 1575 (C–C), 1540 (N=), 1387 [δ (NH)], 1276 (C_{Ar}–H), 1240 (ArOAr), 1131 (S=O). Found, %: C 56.82; H 6.02, N 9.44; S 10.60. C₁₁₂H₁₂₂N₁₆O₂₄S₈. Calculated, %: C 57.67; H 5.27; N 9.61; S 10.99.

Compound 8. Yield 280 mg (72%). IR spectrum, v, cm⁻¹: 3421 (OH), 2921, 2843 (CH₂, CH₃), 1590 (C–C), 1532 (N=), 1340 [δ (NH)], 1270 (C_{Ar}–H), 1240

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68.27; H 9.35, N 5.33; S 6.81. $C_{224}H_{346}N_{16}O_{24}S_8$. Calculated, %: C 68.92; H 8.93; N 5.74; S 6.57.

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