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New phosphorus-containing metal phthalocyanine complexes. Synthesis and spectral and electrochemical studies

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Hitherto unknown phosphorus-containing 4,5-bis(diethoxyphosphorylmethyl)- and 4-methyl-5-diethoxyphosphorylmethylphthalonitriles were synthesized starting from *o*-xylene. Their tetramerization afforded free phthalocyanine ligands and their complexes with Zn, Ni, Co, and a number of rare-earth metals. The spectral and electrochemical properties of the phthalocyanines synthesized were studied.

Key words: phthalocyanines, phthalonitrile, Arbuzov reaction.

Active search for water-soluble compounds having intense absorption in the region >600 nm is being carried out, because they can be used as photosensitizers for photodynamic therapy. Water-soluble phthalocyanine complexes can be regarded as representatives of such compounds.[1,2] Their ability to selective accumulation in tumors with production of photoexcited forms of oxygen, first of all, singlet oxygen, was established.

In addition, these complexes can be of interest as catalysts, dyes, or activators of textile bleaching and can be used to enhance the sensitivity of ion-selective electrodes.[3]

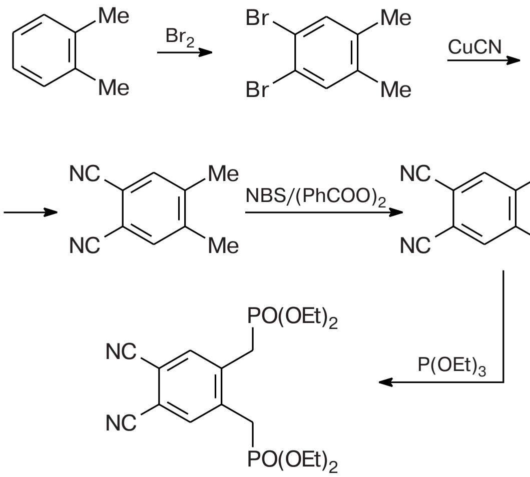
Sulfo derivatives are most widely used as water-soluble phthalocyanines. They are formed as mixtures with variable compositions and structures upon direct sulfonation of phthalocyanines, which decreases their value.

The present work is aimed at synthesizing new water-soluble metal phthalocyanine complexes based on 4,5-bis(diethoxyphosphorylmethyl)phthalonitrile (**1**) and 4-methyl-5-diethoxyphosphorylmethylphthalonitrile (**2**). Tetrakis(phosphorylmethyl)phthalocyanine complexes containing four phosphorylmethyl groups have been synthesized previously.[4]

Nitrile **1** was prepared from *o*-xylene as shown in Scheme 1.

The first two steps in the presented scheme are straightforward.[5,6] The methyl groups in dimethylphthalonitrile were brominated with *N*-bromosuccinimide in boiling carbon tetrachloride using a known procedure.[7] The bromomethyl derivative was refluxed with an excess of triethyl phosphite to give the target nitrile **1** in good yield (40—60%). The use of acetonitrile as a solvent (by analogy with the synthesis of the 4-phosphorylmethyl derivative[4]) decreased the yield of the target nitrile to 10—17%. Nitrile **1** was characterized by ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy, and its composition was confirmed by the data from elemental analysis.

Scheme 1



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In the chromatographic purification of phthalonitrile **1**, a compound was isolated, which was identified as 4-methyl-5-diethoxyphosphorylmethylphthalonitrile (**2**) on the basis of ^{1}H, ^{13}C, and ^{31}P NMR spectroscopy, mass spectrometry, and elemental analysis data. This compound is formed, most likely, due to incomplete benzylic bromination of dimethylphthalonitrile and difficulties in separation of 4-bromomethyl-5-methylphthalonitrile from 4,5-bis(bromomethyl)phthalonitrile (Scheme 2).

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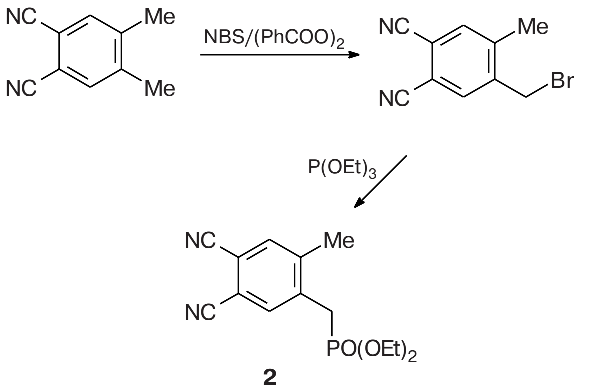
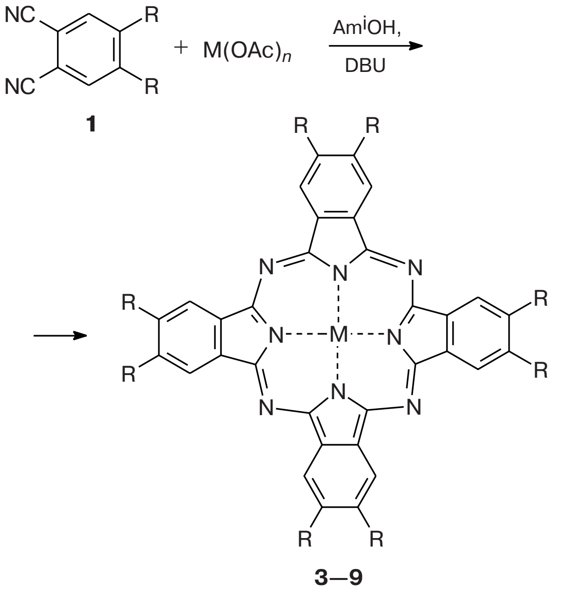
Scheme 2



The tetramerization of nitrile **1** with magnesium, copper, or zinc acetates in boiling isoamyl alcohol (Am^{i}OH) in the presence of 1,8-diaza[5.4.0]bicycloundec-7-ene (DBU) affords symmetric octakis(diethoxyphosphorylmethyl)phthalocyanines of the corresponding metals in 34—95% yields (Scheme 3).

Scheme 3

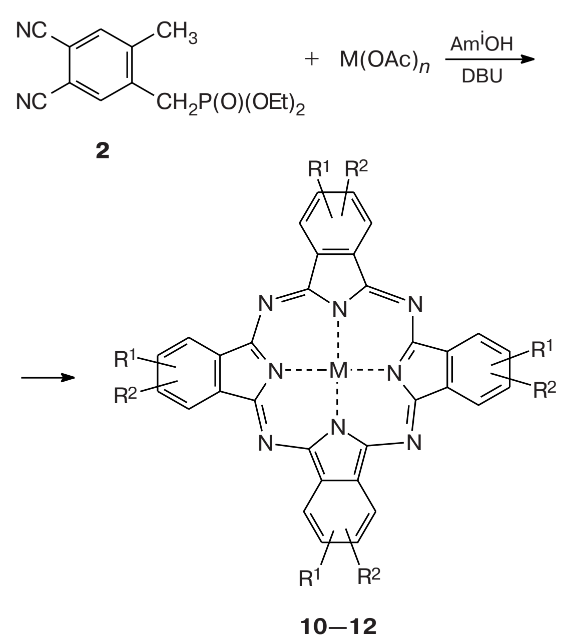


n = 2, M = Mg (**3**), Zn (**4**), Cu (**5**),
n = 3, M = Nd(OAc) (**6**), Eu(OAc) (**7**), Tb(OAc) (**8**), Lu(OAc) (**9**).
R = $CH_2P(O)(OEt)_2$

With rare-earth metal acetates, the corresponding phthalocyanine complexes are obtained in 40—85% yields.

The cyclization of nitrile **2** proceeds similarly giving a mixture of isomeric tetramethyl-tetrakis(diethoxyphosphorylmethyl)phthalocyanines in 40—72% yields (Scheme 4). The reaction of nitriles **1** and **2** with lithium isopentoxide in Am^{i}OH results in the corresponding free phthalocyanine ligands **13** and **14** in 40% yield.

Scheme 4



R^1 = $CH_2P(O)(OEt)_2$, R^2 = Me; M = Zn (**10**), Co (**11**), Ni (**12**).

Note that fusion of 4-diethoxyphosphorylmethylphthalonitrile with metal salts produced the phthalocyanine complexes in yields not exceeding 25% (see Ref. 4).

All the synthesized phthalocyanines are water-soluble. The bivalent metal complexes and free ligands are also soluble in chloroform and ethyl acetate. Rare-earth metal complexes **6—9** with acetate as the counterion are insoluble in these organic solvents but form solutions in alcohols (methanol, ethanol).

Hydrolysis of zinc octakis(diethoxyphosphorylmethyl)phthalocyanine with aqueous NaOH followed by neutralization with hydrochloric acid affords zinc octakis(phosphonomethyl)phthalocyanine **15**. This complex is insoluble in organic solvents but soluble in 10% aqueous alkali.

The mass spectra of complexes **3—5** and **10—12** obtained by different methods (MALDI-TOF, chemical ionization) exhibit the molecular ion peaks with the characteristic isotope distribution of the signals. The typical molecular ion peak is shown in Fig. 1.

The mass spectra of complexes **7** and **8** contain no peaks of molecular ions. In this case, the spectra exhibit the peaks characteristic of free ligand **13**.

The ^{1}H and ^{31}P NMR spectra of zinc (**4**) and lutetium (**9**) octakis(diethoxyphosphorylmethyl)phthalocyanines (**4** and **9**) confirm the structures of the obtained compounds.

We studied the absorption spectra of the complexes in water and organic solvents. The data obtained suggest

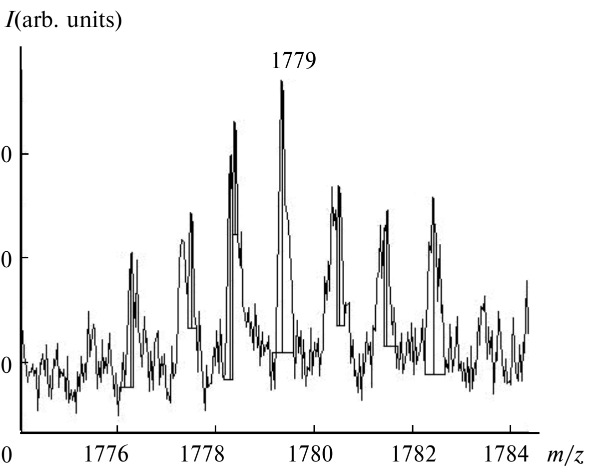


Fig. 1. Mass spectrum of complex **4**. Molecular ion peak.

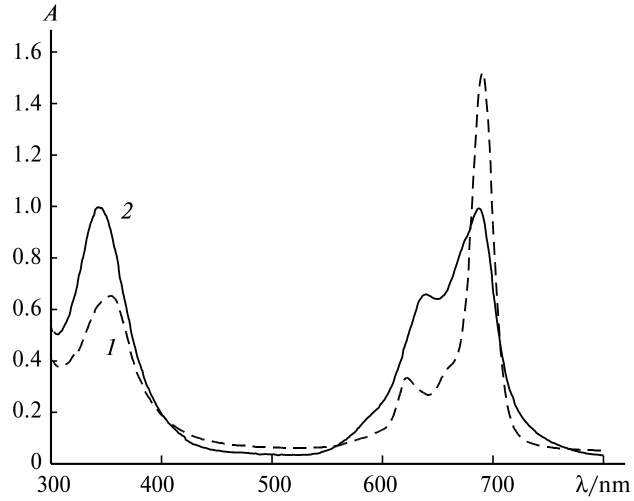


Fig. 2 Absorption spectra of complex **4** in water in the presence of NaOH at pH 13.5 (*1*) and $(Bu_4N)OH$ at pH 10 (*2*).

that these compounds are strongly aggregated in solutions. Aggregation in aqueous solutions can be suppressed by the addition of tetrabutylammonium hydroxide. The typical spectra of complex **4** in aqueous solutions are shown in Fig. 2. The absorption maxima for compounds **3—14** are listed in Table 1.

The spectral data obtained by us differ somewhat from the published[8] spectra of the phosphorus-containing complexes with analogous composition, whose structures remain obscure from the data presented in Ref. 8.

The molar absorption coefficients of the Q band in chloroform ($\varepsilon = 2.34 \cdot 10^5$) and in aqueous NaOH (pH = 13, $\varepsilon = 4.25 \cdot 10^4$) were determined for complex **4**. It is shown that the Bouguer—Lambert—Beer law is fulfilled down to concentrations of $1 \cdot 10^{-5}$ ($CHCl_3$) and $4 \cdot 10^{-5}$ (water, pH = 13).

Complexes **6—9** are unstable in acidic aqueous solutions and decompose completely within 1 h at concentrations of 10^{-6}—10^{-5} mol L^{-1}. In alkaline solutions and in organic solvents, the complexes retain their spectral characteristics for 2—3 days.

The electrochemical properties of zinc complex **5** were studied in aqueous and acetonitrile solutions. In the case of the aqueous solution, tetra-*n*-butylammonium hydroxide, which suppresses, as mentioned above, phthalocyanine aggregation, was used as a supporting electrolyte. The cyclic voltammogram of the aqueous solution in the potential region from +0.9 to −1.0 V is presented in Fig. 3.

In this region we detected two redox transitions: one oxidation transition (0.63 V) and one reduction transition (−0.87 V). The both transitions are quasi-reversible (the difference between the forward and backward peaks in the negative potential region is 119 mV, and that in the positive region is 104 mV). An analogous situation is observed for a solution of complex **4** in acetonitrile ($Bu^n_4NBF_4$ as a

Table 1. Absorption maxima in the absorption spectra (λ/nm) of complexes **3—14** in various solvents

Complex	$CHCl_3$	EtOAc	DMSO	DMF	H_2O, Bu_4NOH (pH = 10)	H_2O, NaOH (pH = 13)
3	344, 621, 698	—	—	—	—	—
4	350, 621, 686	346, 617, 681	350,620, 688	352, 618, 685	354, 622, 691	—
5	347, 617, 685	—	—	—	—	—
6	—	—	—	—	352, 621, 689	352, 623, 692
7	—	—	—	—	351, 621, 690	352, 623, 692
8	—	—	—	—	351, 622, 691	352, 623, 692
9	—	—	—	—	352, 623, 692	351, 624, 693
10	354, 614, 682	349, 611, 677	353, 618, 685	—	—	—
11	613, 675	616, 672	613, 678	—	—	—
12	333, 616, 675	625, 671	628, 682	—	—	—
13	342, 616, 651, 674, 707	342, 609, 647, 667, 701	—	347, 613, 650, 672, 703	—	—
14	344, 612, 647, 669, 704	339, 643, 664, 699	—	341, 613, 643, 670, 702	—	—

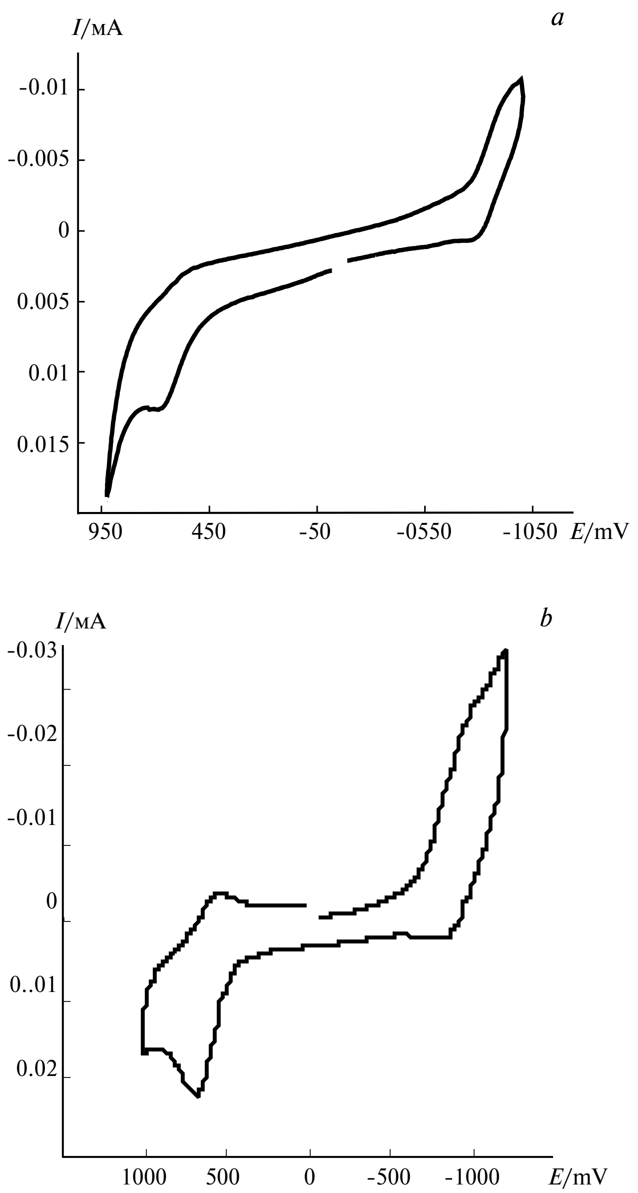


Fig. 3. Cyclic voltammograms of complex **4** in H_2O (0.1 *M* $Bu^n{}_4NOH$; Ag|AgCl|KCl; ν = 200 mV s^{-1}; 20 °C) (*a*) and in CH_3CN (0.05 *M* $Bu^n{}_4NBF_4$; Ag|AgCl|KCl; ν = 200 mV s^{-1}; 20 °C) (*b*).

supporting electrolyte). In this case, we also detected two peaks in the CV with potential values of 0.61 V for oxidation and –0.88 V for reduction. The both transitions are also quasi-reversible (the differences between the forward and backward peaks are 132 and 121 mV, respectively). Only insignificant differences in the voltammograms are observed (see Fig. 3). They are due to a decrease in the backward oxidation peak in water, which is probably caused by the partial destruction of the oxidized form of phthalocyanine.

Experimental

Absorption spectra in the visible region were recorded on a Helios-α spectrometer in quartz cells with the layer thickness of 1, 0.5, and 0.1 cm. ^1H, ^{13}C, and ^{31}P NMR spectra were measured on a Bruker AC-200 instrument in $CDCl_3$ and CD_3OD. The chemical shifts are given in the δ scale relative to $SiMe_4$. Thin layer chromatography was carried out on the plates Merck Silica gel 60 F_{256} and Merck Aluminium Oxide F_{256} neutral. Column chromatography was performed on silica gel Merck (70—230 mesh). Mass spectra were measured on Finnigan MAT INCOS-50 (EI 70 eV, chemical ionization) and VISION-2000 (MALDI TOF) instruments. IR spectra were recorded with a UR-20 instrument for suspensions in Nujol.

All solvents were purified prior to use by standard procedures.

Metal salts were kept before syntheses in a vacuum drying oven for 4 h at 110 °C.

4,5-Dibromo-*o*-xylene. Molecular bromine (42.5 mL, 0.82 mol) was added to *o*-xylene (50 mL) during 5 h with stirring and cooling. The reaction mixture was stirred for 4 h, then diluted with $CHCl_3$ (50 mL), and washed with a solution of $NaHSO_3$. The organic layer was washed with water, and the solvent was evaporated *in vacuo*. The residue was recrystallized from methanol. The product was obtained in a yield of 60.1 g (48%), m.p. 87—89 °C (Ref. 5: 88—89 °C).

4,5-Dimethylphthalonitrile. A mixture of 4,5-dibromoxylene (**1**) (20 g, 0.07 mol) and CuCN (37 g, 0.4 mol) in DMF (230 mL) was refluxed for 5 h. After cooling, the solution was poured into 1.5 L of a solution of $FeCl_3$. The precipitate was filtered off, washed with water, and extracted with $CHCl_3$ (600 mL). The extract was dried with $CaCl_2$. The solvent was distilled off on a rotary evaporator, and the resulting product was purified by recrystallization (twice) from benzene. The target phthalonitrile was obtained in a yield of 5.73 g (0.035 mol, 50%), m.p. 170—172 °C (literature data[6]: m.p. 171—173 °C).

4,5-Bis(bromomethyl)phthalonitrile. A mixture of 4,5-dimethylphthalonitrile (1.43 g, 9.17 mmol), *N*-bromosuccine imide (4.90 g, 27.7 mol), and benzoyl peroxide (10 mg) in CCl_4 (13 mL) was refluxed with vigorous stirring for 12 h. After completion of the reaction, the solution was washed with water (300 mL), and the solvent was evaporated. The residue was purified by column chromatography (silica gel, eluent benzene). The yield was 1.67 g (58%), m.p. 115—117 °C (literature data[7]: m.p. 110—122 °C). Found (%): C, 38.23; H, 1.87; N, 8.99. $C_{10}H_6N_2Br_2$. Calculated (%): C, 38.25; H, 1.93; N, 8.92. ^1H NMR, δ: 7.84 (s, 2 H, Ar), 4.61 (s, 4 H, CH_2).

4,5-Bis(diethoxyphosphorylmethyl)phthalonitrile (1). A mixture of 4,5-bis(bromomethyl)phthalonitrile (4 g, 12.78 mmol) and triethyl phosphite (9 mL, 8.72 g, 52.54 mmol) was heated at 140 °C until the evolution of ethyl bromide ceased completely (4 h). After completion of the reaction, the excess of triethyl phosphite was removed *in vacuo*. The residue was purified by column chromatography, admixtures were eluted with ethyl acetate, and the target compound was eluted with ethanol. The yield was 2.1 g (38%), m.p. 110—112 °C. MS, *m/z*: 428 $[M]^+$; 413 $[M - CH_3]^+$; 154 $[M - 2P(O)(OEt)_2]^+$. ^1H NMR, δ: 7.66 (s, 2 H, Ar); 3.51, 3,44 (d, 4 H, Ar—$C\underline{H}_2$); 4.05 (q, 8 H, —$C\underline{H}_2CH_3$); 1.26 (t, 12 H, —$CH_2C\underline{H}_3$). ^{13}C NMR ($CDCl_3$), δ: 16.3 (—$CH_2\underline{C}H_3$); 62.5 (—$\underline{C}H_2CH_3$); 115 (CN); 32.8 (Ar—$\underline{C}H_2$—P); 113.9 (NC—$\underline{C}_{Ar}$); 138.6 (P—$CH_2\underline{C}_{Ar}$); 136 (CH_{Ar}). ^{31}P NMR: 23.52 ppm (s).

Found (%): C, 50.26, 50.72; H, 6.20, 5.99; N, 6.40, 6.19. $C_{18}H_{26}N_2O_6P_2$. Calculated (%): C, 50.47; H, 6.12; N, 6.54. IR (KBr): 2233 (C≡N); 1240 (P=O); 1053, 1035 (P—O—C); 3031, 1603, 1506, 866 (arom.).

4-Diethoxyphosphorylmethyl-5-methylphthalonitrile (2) was isolated (~0.3 g) by chromatography of the reaction mixture. MS, *m/z*: 292 $[M]^+$; 155 $[M-P(O)(OEt)_2]^+$. ^{1}H NMR, δ: 7.68, 7.67 (both s, 2 H, Ar), 2.51 (s, 3 H, CH_3); 3.26, 3.19 (d, 2 H, Ar—CH_2—P); 4.04 (q, 4 H, $C\underline{H}_2$—CH_3); 1.28 (t, 6 H, —CH_2—$C\underline{H}_3$). ^{13}C NMR ($CDCl_3$), δ: 17.2 (—$CH_2\underline{C}H_3$); 62.7 (—$\underline{C}H_2CH_3$); 115.3 (CN); 32.3 (Ar—$\underline{C}H_2$—P); 17.2 ($\underline{C}H_3$—Ar); 113.0, 133.9 (2 NC—$\underline{C}_{Ar}$); 137.1 ($CH_3\underline{C}_{Ar}$); 138.6 (P—$CH_2\underline{C}_{Ar}$); 134.0, 136.3 (2 CH_{Ar}). ^{31}P NMR: 23.47 ppm (s). Found (%): C, 57.19, 57.45; H, 6.02, 6.20; N, 9.18, 9.25. $C_{14}H_{17}N_2O_3P$. Calculated (%): C, 57.53; H, 5.82; N, 9.59.

Synthesis of metal phthalocyanine complexes 3—12 (general procedure). Method *A*. A mixture of isoamyl alcohol (5 mL), phthalonitrile **1** (1 mmol) or phthalonitrile **2** (0.5 mmol), DBU (0.15 mL), and the corresponding metal acetate (0.25 mmol) was refluxed until the phthalonitrile disappeared completely (TLC monitoring). After completion of the reaction, isoamyl alcohol was distilled off from the reaction mixture. The residue was dissolved in $CHCl_3$, and the solution was washed with dilute HCl, a solution of $NaHCO_3$, and distilled water. The solution was dried with $MgSO_4$, the solvent was evaporated, and the resulting solid residue was washed with hexane and dried in air.

Method *B*. The reaction was carried out as in method *A*. After removal of the solvent, the residue was washed with ethyl acetate and hexane and dried in air.

Magnesium 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (3) was synthesized by method *A*. The yield was 0.172 g (65%).

Zinc 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (4) was synthesized by method *A*. The yield was 0.19 g (34%). ^{1}H NMR, δ: 7.72 (s, Ar); 4.06 (q, —$C\underline{H}_2$—CH_3); 3.53 (d, Ar—$C\underline{H}_2$—P); 1.28 (t, —CH_2—$C\underline{H}_3$). MS, *m/z*: 1779 $[M]^+$.

Copper 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (5) was synthesized by method *A*. The yield was 0.172 g (95%).

Neodymium 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (6) was synthesized by method *A*. The yield was 0.248 g (60%). IR (KBr), ν/cm^{-1}: 1047 (P—O—C); 1240 (P=O).

Europium 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (7) was synthesized by method *B*. The yield was 0.172 g (40%). IR (KBr), ν/cm^{-1}: 1046 (P—O—C); 1240 (P=O).

Terbium 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (8) was synthesized by method *B*. The yield was 0.267 g (60%). IR (KBr), ν/cm^{-1}: 1047 (P—O—C); 1240 (P=O).

Lutetium 2,3,9,10,16,17,23,24-octakis(diethoxyphosphorylmethyl)phthalocyanine (9) was synthesized by method *B*. The yield was 0.364 g (85%). ^{1}H NMR, δ: 7.81 (s, 8 H, Ar); 3.85 (q, 16 H, —$C\underline{H}_2$—CH_3); 1.17 (t, 24 H, —CH_2—$C\underline{H}_3$); 3.45 (s, 16 Ar—$C\underline{H}_2$—); 2.56 (s, 3 H, —C(O)$C\underline{H}_3$). ^{31}P NMR: 19.97 ppm (s). IR (KBr), ν/cm^{-1}: 1045 (P—O—C); 1240 (P=O).

Zinc 2,9,16,23-tetrakis(diethoxyphosphorylmethyl)-3,10,17,24-tetramethylphthalocyanine (10) was synthesized by method *A*. The yield was 0.063 g (31.8%). ^{1}H NMR, δ: 7.45 (m, 8 H, Ar); 2.98 (d, 16 H, Ar—$C\underline{H}_2$); 2.35 (m, 12 H, —$C\underline{H}_3$); 4.05 (q, 8 H, —$C\underline{H}_2$—CH_3); 1.28 (m, 12 H, —CH_2—$C\underline{H}_3$). ^{31}P NMR: 26.94 ppm (s). MS, *m/z*: 1233 $[M]^+$.

Cobalt 2,9,16,23-tetrakis(diethoxyphosphorylmethyl)-3,10,17,24-tetramethylphthalocyanine (11) was synthesized by method *A*. The yield was 0.21 g (72%).

Nickel 2,9,16,23-tetrakis(diethoxyphosphorylmethyl)-3,10,17,24-tetramethylphthalocyanine (12) was synthesized by method *A*. The yield was 0.21 g (72%).

2,3,9,10,16,17,23,24-Octakis(diethoxyphosphorylmethyl)phthalocyanine (13). Metallic lithium (0.007 g, 0.1 mmol) was dissolved on heating in 2 mL of isoamyl alcohol, then phthalonitrile **1** (0.214 g, 0.5 mmol) was added, and the mixture was refluxed for 3 h. After completion of the reaction, isoamyl alcohol was removed *in vacuo*, and the residue was dissolved in $CHCl_3$ (40 mL), washed with dilute HCl, a solution of $NaHCO_3$, and distilled water. The solvent was evaporated *in vacuo*, and the residue was washed with hexane and dried in air. The yield was 0.086 g (40%). ^{1}H NMR, δ: 7.71 (s, arom.); 4.08 (br.d, Ar—$C\underline{H}_2$—); 3.61 (br.q, —$C\underline{H}_2$—CH_3); 1.32 (br.t, —CH_2—$C\underline{H}_3$). ^{31}P NMR, δ: 23.35 ppm.

2,9,16,23-Tetrakis(diethoxyphosphorylmethyl)-3,10,17,24-tetramethylphthalocyanine (14) was synthesized similarly from phthalonitrile **2** (0.10 g, 0.34 mmol). The yield was 0.04 g (40%).

Zinc 2,3,9,10,16,17,23,24-octakis(phosphonomethyl)phthalocyanine (15). Compound **4** (0.089 g, 0.5 mmol) was refluxed for 2 h in a 10% aqueous solution of NaOH (5 mL), the solution was cooled, neutralized with concentrated HCl, and the precipitate that formed was filtered off, washed with water, and dried in air. The dark green powder was obtained in a yield of 0.052 g (78%).

Electrochemical measurements were carried out with an IPC-Pro potentiostat using a three-electrode scheme with a graphite electrode (pyrolyzed polyacrylonitrile VMN-4, T_{tr} = 2400 °C, specific surface 12 $m^2 g^{-1}$, $m \approx 2$ mg) at 20 °C *vs* 0.15 *M* $Bu^n{}_4NBF_4$ in acetonitrile and *vs* 0.1 *M* Bu^nOH in water. A Pt plate was the auxiliary electrode, and a silver chloride electrode was used as a reference electrode. Oxygen was removed from the cell by purging dry argon. Voltammetric curves were detected by cyclic voltammetry at a sweep rate of 200 mV s^{-1}. The measured peak potentials were recalculated with allowance for the resistance of the electrolyte, which were calculated from the anodic and cathodic peaks of ferrocene oxidation: $(E^c - E^a) - 0.059 = I \cdot R$, where E^c and E^a are the potentials of the cathodic and anodic oxidation peaks of ferrocene, respectively; *I* is the sum of currents of the cathodic and anodic processes; *R* is the resistance of the electrolyte. The concentration in solutions of the complexes was $2 \cdot 10^{-4}$ mol L^{-1}.

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