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Synthesis and Properties of [4-(Triphenylmethyl)phenoxy]acetic and 3-[4-(Triphenylmethyl)phenoxy]propionic Acids and Their Condensation with Phthalimide Leading to *meso*-Substituted Tetrabenzoporphyrins

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Abstract—Condensation of 4-(triphenylmethyl)phenol with monochloroacetic and 3-bromopropionic acids gave, respectively, [4-(triphenylmethyl)phenoxy]acetic and 3-[4-(triphenylmethyl)phenoxy]propionic acids which reacted with phthalimide in the presence of zinc acetate to produce *meso*-substituted zinc tetrabenzoporphyrins having bulky triphenylmethyl groups in the substituent. The corresponding free ligands were obtained by demetalation of the zinc complexes. Spectral properties of the synthesized compounds were studied.

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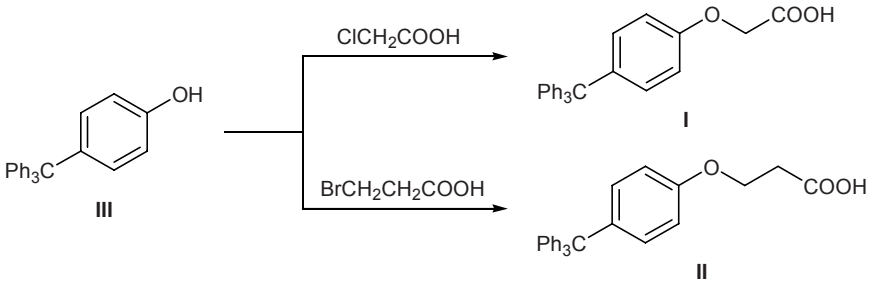
Unlike phthalocyanine, tetrabenzoporphyrin molecule may be modified via introduction of substituents into not only isoindole fragments but also *meso* positions of the macroring. *meso*-Substituted tetrabenzoporphyrins possess interesting nonlinear optical [1, 2], acid–base [3], and electrochemical properties [4]. They may be used in the diagnostics and photodynamic therapy of cancer [5–7], as well as catalysts [8]. Such properties of *meso*-substituted tetrabenzoporphyrins determine importance of their further detailed studies.

Up to now, three main methods for the synthesis of *meso*-substituted tetrabenzoporphyrins have been reported. The first of these includes high-temperature template condensation of substituted acetic acids with phthalimide or its derivatives in the presence of bivalent metal (as a rule, zinc or cadmium) salts or hydroxides [9, 10]. The second method is based on direct introduction of substituents into the *meso* positions of already generated macroring [11]. Finally, the third, most advanced approach is based on condensation of aromatic aldehydes with saturated isoindole derivatives, such as 4,5,6,7-tetrahydroisoindole [12, 13] or 4,7-dihydroisoindole [14, 15], followed by mild oxidation of hydrogenated tetrabenzoporphyrins thus formed. Although the first method is not free from some serious disadvantages related to thermal instability of many substituted phthalimides and arylacetic acids, its undoubted merit is that it includes only one step. Therefore, just the first method was used in the present work to synthesize *meso*-substituted tetrabenzoporphyrins.

An important group of tetrapyrrole macroheterocycles consists of compounds having bulky substituents in their molecules. Phthalocyanines containing triphenylmethyl groups possess quite valuable properties from the applied viewpoint [16–18]. However, we have found no published data on the synthesis and properties of *meso*-substituted tetrabenzoporphyrins having triphenylmethyl groups in the *meso* substituents, though such derivatives should also be interesting from the viewpoint of practice. Presumably, the reason is the lack of preparative procedures for the synthesis of acetic acid derivatives containing triphenylmethyl groups, which could be used as initial compounds for the synthesis of *meso*-substituted tetrabenzoporphyrins. Taking the above stated into account, the present work was aimed at developing procedures for the preparation of [4-(triphenylmethyl)phenoxy]acetic acid (**I**) and 3-[4-(triphenylmethyl)phenoxy]propionic acid (**II**) and synthesizing *meso*-substituted tetrabenzoporphyrins via condensation of acids **I** and **II** with phthalimide.

Compounds **I** and **II** were obtained by condensation of 4-triphenylmethylphenol (**III**) with chloroacetic and

Scheme 1.



3-bromopropionic acids, respectively, in dimethylformamide in the presence of potassium hydroxide (Scheme 1). Acids **I** and **II** were isolated as colorless powders which are soluble in benzene, chloroform, and acetone. Their structure was confirmed by the data of elemental analysis and IR and ^{1}H NMR spectra. The IR spectrum of **I** contains an absorption band at 3552 cm^{-1} due to stretching vibrations of O–H bond in the carboxy group, absorption bands at 3083 and 3027 cm^{-1} correspond to vibrations of C–H bonds in the benzene rings. The band at 2932 cm^{-1} was assigned to C–H vibrations of the methylene group. The carboxy group gave rise to carbonyl absorption band at 1699 cm^{-1}, and bands at 1262 and 1159 cm^{-1} belonged to stretching vibrations of the ether C–O bonds. A strong band at 698 cm^{-1} is typical of bending vibrations of aromatic rings in the triphenylmethyl group. The IR spectrum of acid **II** resembled that of com-

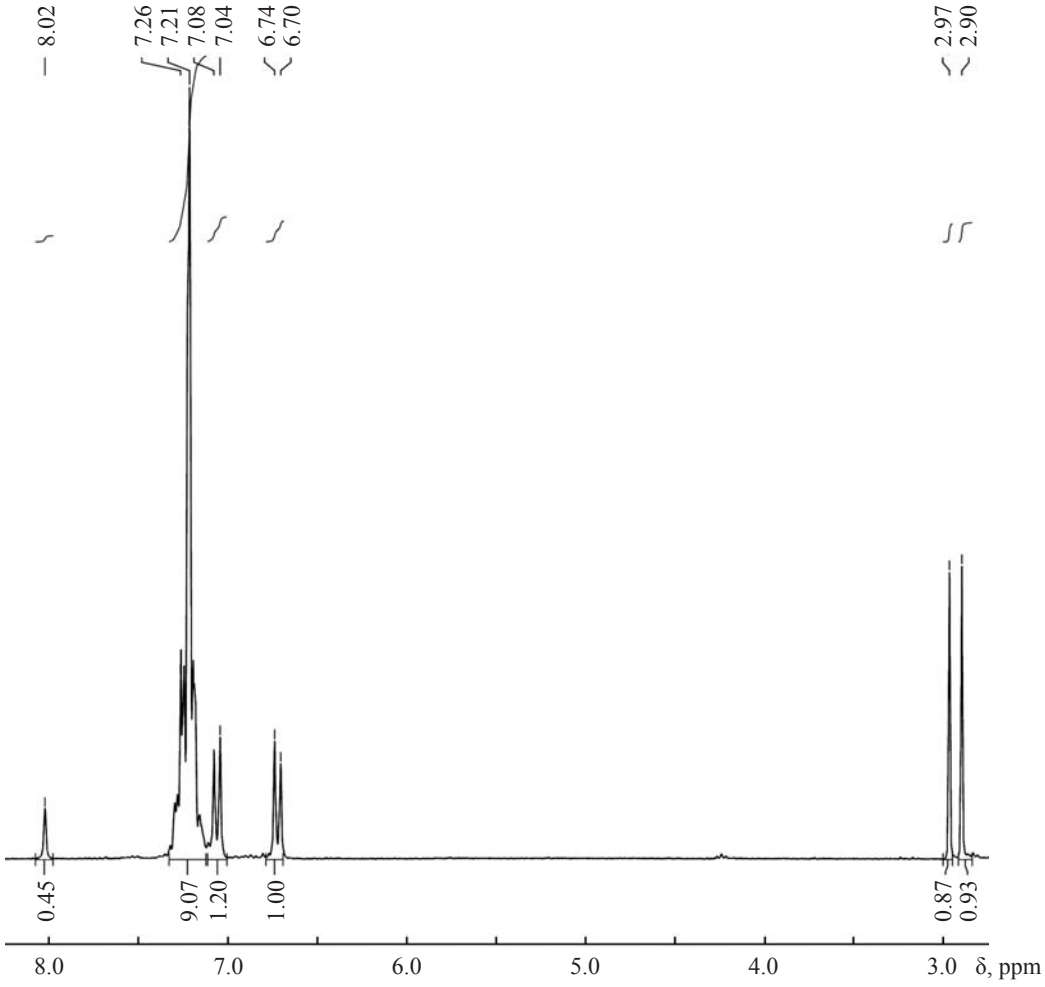
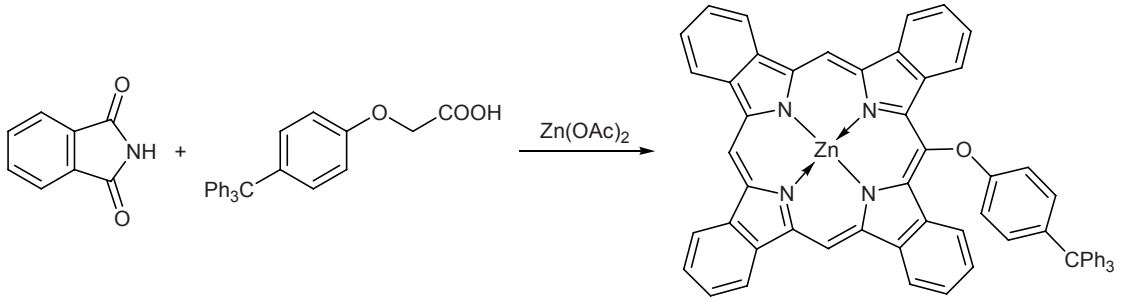


Fig. 1. ^{1}H NMR spectrum of 3-[4-(triphenylmethyl)phenoxy]propionic acid (**II**) in $CDCl_3$.

Scheme 2.



IV **I** **V**

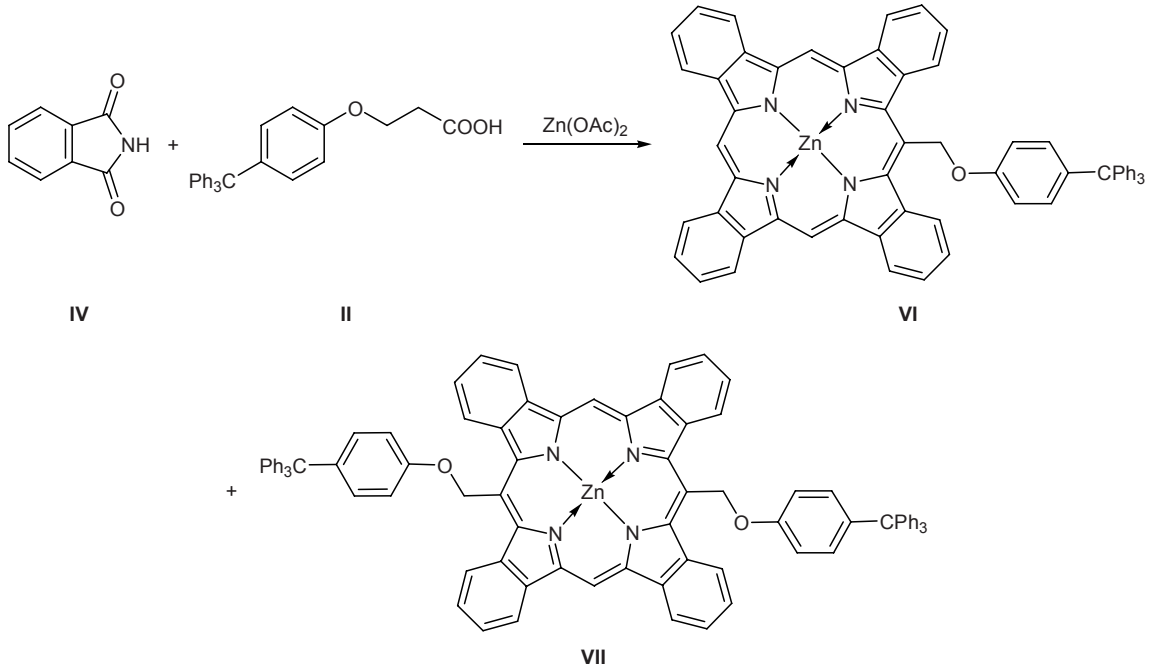
pound **I** with the difference that the absorption band at 2939 cm^{-1} had higher intensity due to the presence of two methylene groups.

In the ^1H NMR spectrum of acid **I** we observed a singlet at δ 8.10 ppm due to proton in the carboxy group, and a 15-proton multiplet was present in the region δ 7.40–7.11 ppm from aromatic protons in the triphenylmethyl group. Protons in the *para*-substituted benzene ring resonated as doublets of doublets at δ 7.10–7.05 and 6.79–6.73 ppm, and the singlet at δ 2.99 ppm was assigned to protons in the methylene group. The COOH proton signal in the spectrum of substituted propionic acid **II** (Fig. 1) was observed in a stronger field, at δ 8.02 ppm, whereas signals from 15 protons in the triphenylmethyl group and 4 protons in the phenoxy group were located in the same regions as in the spectrum of **I**. Protons in the two methylene groups of molecule **II** resonated as two singlets at δ 2.97 and 2.90 ppm.

We made an attempt to synthesize {tetra-*meso*-[4-(triphenylmethyl)phenoxy]tetrabenzoporphyrinato}-zinc(II) by reaction of phthalimide (**IV**) with excess acid **I** in the presence of zinc(II) oxide. However, heating of the reactants at 350°C over a period of 2 h resulted in the formation of a brown melt which did not contain even traces of the target compound. Raising the temperature or increasing the reaction time led to charring. We succeeded in obtaining tetrabenzoporphyrins by replacing zinc(II) oxide by acetate. In this case, the reaction occurred at 310°C and was complete in 40–50 min (the reaction mixture turned green).

We expected formation of a mixture of zinc complexes of *meso*-[4-(triphenylmethyl)phenoxy]-substituted tetrabenzoporphyrins with different numbers of

Scheme 3.



IV **II** **VI** **VII**

meso substituents, taking into account that acetate ion present in the reaction mixture can act as source of methylene group. However, chromatographic separation of the reaction mixture gave only unsubstituted tetrabenzoporphyrinatozinc and one more porphyrin derivative, the latter being characterized by better solubility in organic solvents. This product was identified as {*meso*-[4-(triphenylmethyl)phenoxy]tetrabenzoporphyrinato}zinc(II) (**V**) (Scheme 2). The absence of tetrabenzoporphyrins with a larger number of *meso* substituents may be rationalized in terms of steric hindrances. Analogous pattern was observed previously, e.g., in the reaction of 4-phenylphthalimide with (naphthalen-1-yl)acetic acid in the presence of zinc acetate, which also produced only the corresponding mono-*meso*-substituted derivative [19].

Likewise, the reaction of phthalimide (**IV**) with acid **II** in the presence of zinc(II) oxide gave no desired *meso*-substituted tetrabenzoporphyrin. However, the reaction performed in the presence of zinc(II) acetate resulted in the formation of two readily soluble tetrabenzoporphyrins which were isolated by column chromatography and identified as {5-[4-(triphenylmethyl)phenoxymethyl]tetrabenzoporphyrinato}zinc(II) (**VI**) and {5,15-bis[4-(triphenylmethyl)phenoxymethyl]tetrabenzoporphyrinato}zinc(II) (**VII**) (Scheme 3). Obviously, the presence of an extra methylene unit in molecule **II** reduces steric hindrances to the formation of *meso*-substituted tetrabenzoporphyrins.

We believe that just *trans*-disubstituted tetrabenzoporphyrin **VII** is formed for steric reasons. Quantum-chemical calculations (AM1) of compound **VII** and its *cis*-substituted isomer, {5,10-bis[4-(triphenylmethyl)phenoxymethyl]tetrabenzoporphyrinato}zinc(II), showed that the former is more energetically favorable by 62.13 kJ/mol. In addition, molecule **VII** is more planar.

Treatment of compounds **V**–**VII** in chloroform with concentrated hydrochloric acid gave, respectively, 5-[4-(triphenylmethyl)phenoxy]tetrabenzoporphyrin (**VIII**), 5-[4-(triphenylmethyl)phenoxymethyl]tetrabenzoporphyrin (**IX**), and 5,15-bis[4-(triphenylmethyl)phenoxymethyl]tetrabenzoporphyrin (**X**) which were isolated and purified by column chromatography.

Porphyrins **V**–**X** are dark green crystalline substances which are readily soluble in a number of organic solvents. Their structure was confirmed by elemental analyses and electronic, vibrational, and NMR spectra. The electronic absorption spectra of **V** and

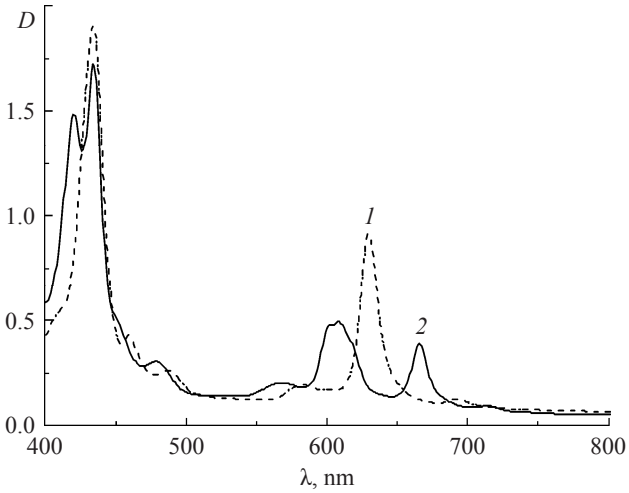


Fig. 2. Electronic absorption spectra of compounds (*1*) **V** and (*2*) **VIII** in benzene.

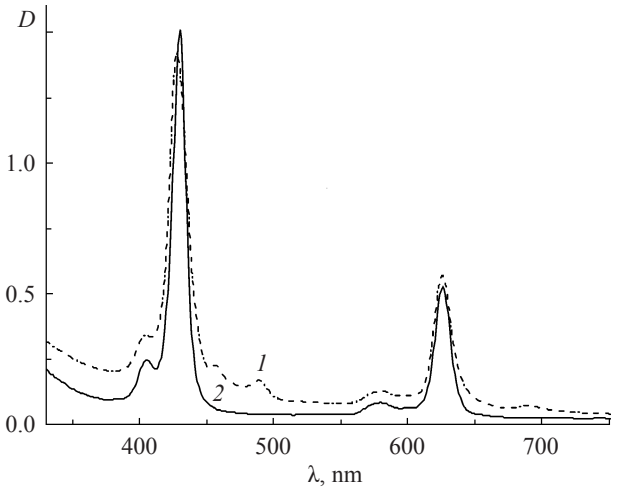


Fig. 3. Electronic absorption spectra of compounds (*1*) **VI** and (*2*) **VII** in benzene.

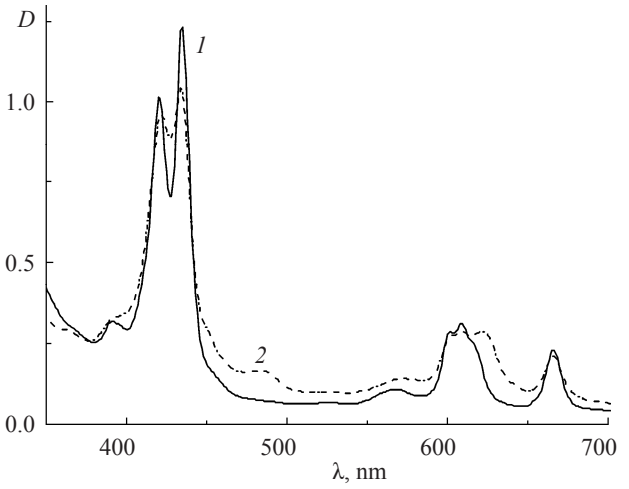


Fig. 4. Electronic absorption spectra of compounds (*1*) **IX** and (*2*) **X** in benzene.

VIII (Fig. 2) are typical of tetrabenzoporphyrins. They contained a strong Soret band and less intense *Q* band in the visible region. In the spectrum of porphyrin **VIII** (Fig. 2, curve *2*), these bands were split into two components due to lower symmetry of the macroring. The absorption maxima in the spectrum of complex **V** (Fig. 2, *1*) are slightly displaced to the red region relative to the corresponding absorption maxima of unsubstituted zinc tetrabenzoporphyrin [20] (by 4 and 10 nm for the *Q* band and Soret band, respectively), which may be due to some deviation of the macroring from planar structure and its polarization. In fact, in the spectrum of **V** we also observed two weak bands in the region λ 488–460 nm, which can be assigned to charge transfer from the electron-donating substituent to the macroring. The relative intensity of the *Q* band also slightly increases. In the electronic absorption spectrum of (tetrabenzoporphyrinato)zinc(II), the intensity ratio of the Soret and *Q* bands is equal to 1:0.34 [21] against 1:0.49 in the spectrum of complex **V**. The red shift of the main absorption maxima in the spectrum of **VIII** as compared to its unsubstituted analog amounts to 4–9 nm, and the charge-transfer band has its maximum at λ 479 nm.

The electronic absorption spectra of zinc complexes **VI** and **VII** (Fig. 3) are characterized by insignificant blue shift of the main maxima relative to those observed in the spectrum of **V**. However, the positions of absorption maxima in the spectrum of **VI** (Fig. 3, *1*) are almost the same as in the spectrum of unsubstituted (tetrabenzoporphyrinato)zinc, whereas the Soret band in the spectrum of **VII** (Fig. 3, *2*) is displaced by 6 nm to longer wavelengths. No charge-transfer bands were observed in the electronic absorption spectrum of complex **VII**.

The electronic absorption spectra of free ligands **IX** and **X** (Fig. 4) are fairly similar to the spectrum of unsubstituted tetrabenzoporphyrin. Unlike monosubstituted porphyrin **IX** (Fig. 4, *1*), compound **X** (Fig. 4, *2*) displayed a charge-transfer band with its maximum at λ 485 nm. In addition, increase in the number of *meso*-substituents is accompanied by some decrease in the degree of splitting of the main absorption bands, which is characteristic of *meso*-substituted tetrabenzoporphyrins [22].

The IR spectra of compounds **V** and **VIII** contained absorption bands typical of tetrabenzoporphyrins. In particular, bands in the regions 1650–1420 (C=C, C=N) and 3087–3044 cm^{-1} (C–H), as well as at 1244 and 1236 cm^{-1} (C–O), were present. Strong absorption bands in the region 703–694 cm^{-1} should be assigned to bending vibrations of monosubstituted benzene rings in the triphenylmethyl groups. Porphyrin **VIII** showed in the IR spectrum a broadened band at 3244 cm^{-1} due to stretching vibrations of N–H bonds. The IR spectral patterns of compounds **VI**, **VII**, **IX**, and **X** were essentially similar to those observed for porphyrins **V** and **VIII**.

Four groups of signals may be distinguished in the ^{1}H NMR spectrum of zinc complex **V**. Two most downfield singlets appeared due to resonance of three protons in the *meso* positions: δ 8.14 (15-H) and 8.07 ppm (10-H, 20-H). Multiplet signal in the region δ 7.41–7.28 ppm belongs to 16 protons in the fused benzene rings, while aromatic protons in the triphenylmethyl group give rise to a multiplet at δ 7.28–7.10 ppm. Finally, a doublet of doublets was present in the upfield region (δ 7.09–6.86 ppm) due to four protons in the *para*-substituted phenoxy group. The ^{1}H NMR spectrum of the corresponding free ligand, porphyrin **VIII**, resembles the spectrum of complex **V**, except for the signal at δ –2.23 ppm (2H, NH). Compounds **VI** and **VII** displayed in the ^{1}H NMR spectra additional signals in the region δ 4.26–4.34 ppm, which belong to methylene protons in the *meso*-substituents. The NH protons in porphyrins **IX** and **X** resonated at δ –2.34 and –2.31 ppm, respectively, indicating the absence of appreciable distortion of planar structure of their molecules.

Thus the condensation of phthalimide with [4-(triphenylmethyl)phenoxy]acetic acid in the presence of zinc(II) acetate is characterized by high selectivity, and it yields only the corresponding mono-*meso*-substituted tetrabenzoporphyrin complex. The condensation of phthalimide with 3-[4-(triphenylmethyl)phenoxy]-propionic acid gives a mixture of mono- and bis-*meso*-substituted derivatives, i.e., increase in the number of methylene groups in the acid component reduces steric hindrances to the formation of macroheterocycles.

EXPERIMENTAL

The electronic absorption spectra were measured on a Hitachi UV-2001 spectrophotometer. The ^{1}H NMR spectra were recorded from solutions in $CDCl_3$ on a Bruker WM-250 instrument (250 MHz) using tetramethylsilane as internal reference. The IR spectra (400–4000 cm^{-1}) were obtained on an Avatar 360 FT-IR spectrometer from samples applied to TlI plates. The elemental compositions were determined using a FlashEA 1112 CHNS–O Analyzer.

4-(Triphenylmethyl)phenol (**III**) was synthesized according to the procedure described in [23].

Acids I and II (general procedure**).** A mixture of 6.7 g (0.02 mol) of 4-(triphenylmethyl)phenol (**III**), 0.04 mol of chloroacetic or 3-bromopropionic acid, and 2.8 g (0.07 mol) of potassium hydroxide in 40 ml of dimethylformamide was heated fir 6 h under reflux with stirring. The mixture was cooled, diluted with 100 ml of water, and acidified with hydrochloric acid to pH 1, and the precipitate was filtered off, washed with water until neutral washings (pH 7), and dried.

[4-(Triphenylmethyl)phenoxy]acetic acid (I). Yield 6.8 g (87%), colorless powder, readily soluble in benzene, chloroform, and acetone, poorly soluble in aqueous alkali, and insoluble in water. IR spectrum, ν, cm^{-1}: 3552, 3450, 3083, 3027, 2932, 1699, 1507, 1488, 1262, 1159, 698. ^{1}H NMR spectrum, δ, ppm: 2.99 s (2H, CH_2), 6.73–6.79 d (2H, C_6H_4), 7.05–7.10 d (2H, C_6H_4), 7.11–7.40 m (15H, Ph) 8.10 s (1H, COOH). Found, %: C 81.77; H 5.71. $C_{27}H_{22}O_3$. Calculated, %: C 82.21; H 5.62.

3-[4-(Triphenylmethyl)phenoxy]propionic acid (II). Yield 5.9 g (73%), colorless powder, readily soluble in benzene, chloroform, and acetone, poorly soluble in aqueous alkali, and insoluble in water. IR spectrum, ν, cm^{-1}: 3544, 3456, 3084, 3027, 2939, 1694, 1501, 1495, 1263, 1151, 702. ^{1}H NMR spectrum, δ, ppm: 2.90 s (2H, 3-H), 2.97 s (2H, 2-H), 6.70–6.74 d (2H, C_6H_4), 7.04–7.08 d (2H, C_6H_4), 7.18–7.33 m (15H, Ph), 8.02 s (1H, COOH). Found, %: C 83.04; H 5.88. $C_{28}H_{24}O_3$. Calculated, %: C 82.33; H 5.92.

{5-[4-(Triphenylmethyl)phenoxy]tetrabenzoporphyrinato}zinc(II) (V). A mixture of 1.0 g (7 mmol) of phthalimide (**IV**), 2 g (5 mmol) of acid **I**, and 2.5 g (11 mmol) of zinc(II) acetate dihydrate was heated for 50 min at 310°C. The melt was cooled, ground, and extracted with chloroform using a Soxhlet apparatus. The solvent was distilled off, the residue was dissolved in benzene, and the solution was applied to a column charged with aluminum oxide (activity grade II according to Brockmann). The column was eluted with benzene–dioxane (20 : 1 by volume), and a green zone was collected. Yield 0.37 g (24%), dark green powder, readily soluble in benzene, chloroform, acetone, and dimethylformamide. Electronic absorption spectrum (benzene), λ_{max} (log ε): 630 (4.70), 581 (4.03), 487 (4.16), 460 (4.37), 434 (5.02). IR spectrum, ν, cm^{-1}: 3087, 3024, 1650, 1507, 1421, 1244, 703. ^{1}H NMR spectrum, δ, ppm: 7.09–6.86 d.d (4H, C_6H_4O), 7.28–7.10 m (15H, Ph), 7.41–7.28 m (16H, C_6H_4), 8.07 s (2H, 10-H, 20-H), 8.14 s (1H, 15-H). Found, %: C 81.05; H 4.64; N 5.98. $C_{61}H_{38}N_4OZn$. Calculated, %: C 80.66; H 4.22; N 6.17.

Condensation of phthalimide with 3-[4-(triphenylmethyl)phenoxy]propionic acid (II). A mixture of 1.5 g (10.5 mmol) of phthalimide (**IV**), 4.0 g (10 mmol) of acid **II**, and 3.0 g (13 mmol) of zinc(II) acetate dihydrate was heated for 1 h at 320°C. The melt was cooled, ground, and extracted with chloroform using a Soxhlet apparatus. The solvent was distilled off, the residue was dissolved in chloroform, and the solution was applied to a column charged with Al_2O_3 (activity grade II according to Brockmann). The column was eluted with benzene–chloroform (1 : 1 by volume). Two green fractions were collected, from which we isolated compounds **VI** and **VII**.

{5-[4-(Triphenylmethyl)phenoxymethyl]tetrabenzoporphyrinato}zinc(II) (VI). Yield 0.22 g (9%), dark green powder, readily soluble in benzene, chloroform, acetone, and dimethylformamide. Electronic absorption spectrum (benzene), λ_{max} (log ε): 626 (4.82), 577 (4.19), 488 (4.31), 457 (4.43), 427 (5.21), 403 (4.60). IR spectrum, ν, cm^{-1}: 3088, 3024, 2938, 1652, 1496, 1411, 701. ^{1}H NMR spectrum, δ, ppm: 4.24 s (2H, CH_2), 6.84–7.10 d.d (4H, C_6H_4O), 7.11–7.26 m (15H, Ph), 7.34–7.51 m (16H, C_6H_4), 8.12–8.22 m (3H, *meso*-H). Found, %: C 80.98; H 5.12; N 5.33. $C_{62}H_{40}N_4OZn$. Calculated, %: C 80.73; H 4.37; N 6.07.

{5,15-Bis[4-(triphenylmethyl)phenoxymethyl]-tetrabenzoporphyrinato}zinc(II) (VII). Yield 0.35 g (11%), dark green powder, readily soluble in benzene, chloroform, acetone, and dimethylformamide. Electronic absorption spectrum (benzene), λ_{max} (log ε): 626 (4.77), 579 (4.01), 430 (5.23), 405 (4.45). IR spectrum, ν, cm^{-1}: 3087, 3033, 2954, 1650, 1502, 1420, 700. ^{1}H NMR spectrum, δ, ppm: 4.34 m (4H, CH_2), 6.77–7.09 d.d (8H, C_6H_4O), 7.10–7.28 m (30H, Ph), 7.34–7.52 m (16H, C_6H_4), 8.31 s (2H, *meso*-H). Found, %: C 83.98; H 5.01; N 4.12. $C_{88}H_{60}N_4O_2Zn$. Calculated, %: C 83.17; H 4.76; N 4.41.

Porphyrins VIII–X (general procedure**).** Zinc complex **V**–**VII**, 0.2 g, was dissolved in 20 ml of chloroform, 10 ml of concentrated hydrochloric acid was added, and the mixture was stirred for 24 h at 20°C. The organic phase was separated, washed with water, 10% aqueous ammonia, and water again until neutral washings (pH 7). The solvent was removed, the residue was dissolved in benzene, and the solution was applied to a column charged with aluminum oxide of activity grade II according to Brockmann. The column was eluted with chloroform to collect a green fraction.

5-[4-(Triphenylmethyl)phenoxy]tetrabenzoporphyrin (VIII). Yield 0.16 g (86%), dark green powder, readily soluble in benzene, chloroform, acetone, and dimethylformamide. Electronic absorption spectrum (benzene), λ_{max} (logε): 666 (4.33), 608 (4.43), 565 (4.05), 479 (4.22), 434 (4.98), 420 (4.91). IR spectrum, ν, cm^{-1}: 3244, 3044, 3025, 1644, 1506, 1420, 1236, 694. ^{1}H NMR spectrum, δ, ppm: –2.23 s (2H, NH), 6.85–7.10 d.d (4H, C_6H_4O), 7.11–7.26 m (15H, Ph), 7.29–7.40 m (16H, C_6H_4), 8.08 s (2H, 10-H, 20-H), 8.13 s (1H, 15-H). Found, %: C 86.12; H 5.22; N 6.11. $C_{61}H_{40}N_4O$. Calculated, %: C 86.71; H 4.77; N 6.63.

5-[4-(Triphenylmethyl)phenoxymethyl]tetrabenzoporphyrin (IX). Yield 0.15 g (82%), dark green powder, readily soluble in benzene, chloroform, acetone, and dimethylformamide. Electronic absorption spectrum (benzene), λ_{max} (logε): 666 (4.34), 608 (4.48), 601 (4.44), 566 (4.01), 435 (5.08), 420 (4.99), 391 (4.49). IR spectrum, ν, cm^{-1}: 3245, 3026, 2931, 1666, 1485, 1476, 703. ^{1}H NMR spectrum, δ, ppm: –2.34 s (2H, NH), 4.26 s (2H, CH_2), 6.82–7.10 d.d (4H, C_6H_4O), 7.12–7.26 m (15H, Ph), 7.33–7.50 m (16H, C_6H_4), 8.15–8.21 m (3H, *meso*-H). Found, %: C 87.67; H 5.16; N 5.88. $C_{62}H_{42}N_4O$. Calculated, %: C 86.69; H 4.93; N 6.25.

5,15-Bis[4-(triphenylmethyl)phenoxymethyl]-tetrabenzoporphyrin (X). Yield 0.17 g (88%), dark green powder, readily soluble in benzene, chloroform, acetone, and dimethylformamide. Electronic absorption spectrum (benzene), λ_{max} (logε): 665 (4.34), 620 (4.41), 608 (4.41), 601 (4.39), 571 (4.11), 485 (4.17), 434 (4.97), 421 (4.94), 392 (4.47). IR spectrum, ν, cm^{-1}: 3244, 3033, 2945, 1652, 1513, 1422, 701. ^{1}H NMR spectrum, δ, ppm: –2.31 s (2H, NH), 4.34 m (4H, CH_2), 6.72–7.08 d.d (8H, C_6H_4O), 7.12–7.27 m (30H, Ph), 7.35–7.52 m (16H, C_6H_4), 8.33 s (2H, *meso*-H). Found, %: C 87.11; H 5.24; N 3.99. $C_{88}H_{62}N_4O_2$. Calculated, %: C 87.53; H 5.18; N 4.64.

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