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Synthesis and Properties of Dimeric Octaalkylporphyrins with a Polyether Linker

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Abstract—Octaalkylporphyrin dimers with a polyether linker were synthesized, and their physicochemical properties were studied. Specific response of the tetrapyrrole chromophore to complexation with potassium cation at the polyether moiety was examined.

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Development of effective methods for detection of chemical compounds, including biologically important ones, with the use of sensors is an essential problem of modern science [1–6]. Progress in the development of methods of organic synthesis stimulated considerable interest in nanosized molecular sensors in which a specific receptor giving a response to the presence of a definite substrate and molecular signaling fragment that transforms primary response into analytical signal are chemically linked to each other in a single molecule [7–9]. Substrate recognition generally involves a chemical reaction, i.e., a system produces primary response as a result of chemical interaction between receptor and substrate.

Tetrapyrrole compounds possess unique electronic properties (i.e., absorption and fluorescence and/or phosphorescence spectra), and they may be optimal transducers of primary analytical signal into optical response of a sensor. On the other hand, these compounds may be also used as source of primary analytical signal, for they are capable of forming complexes with various ionic species [10–13]. Thus a single molecule may act as both receptor and transducer, which makes it possible to simplify the synthetic procedure and reduce the size of molecular sensor.

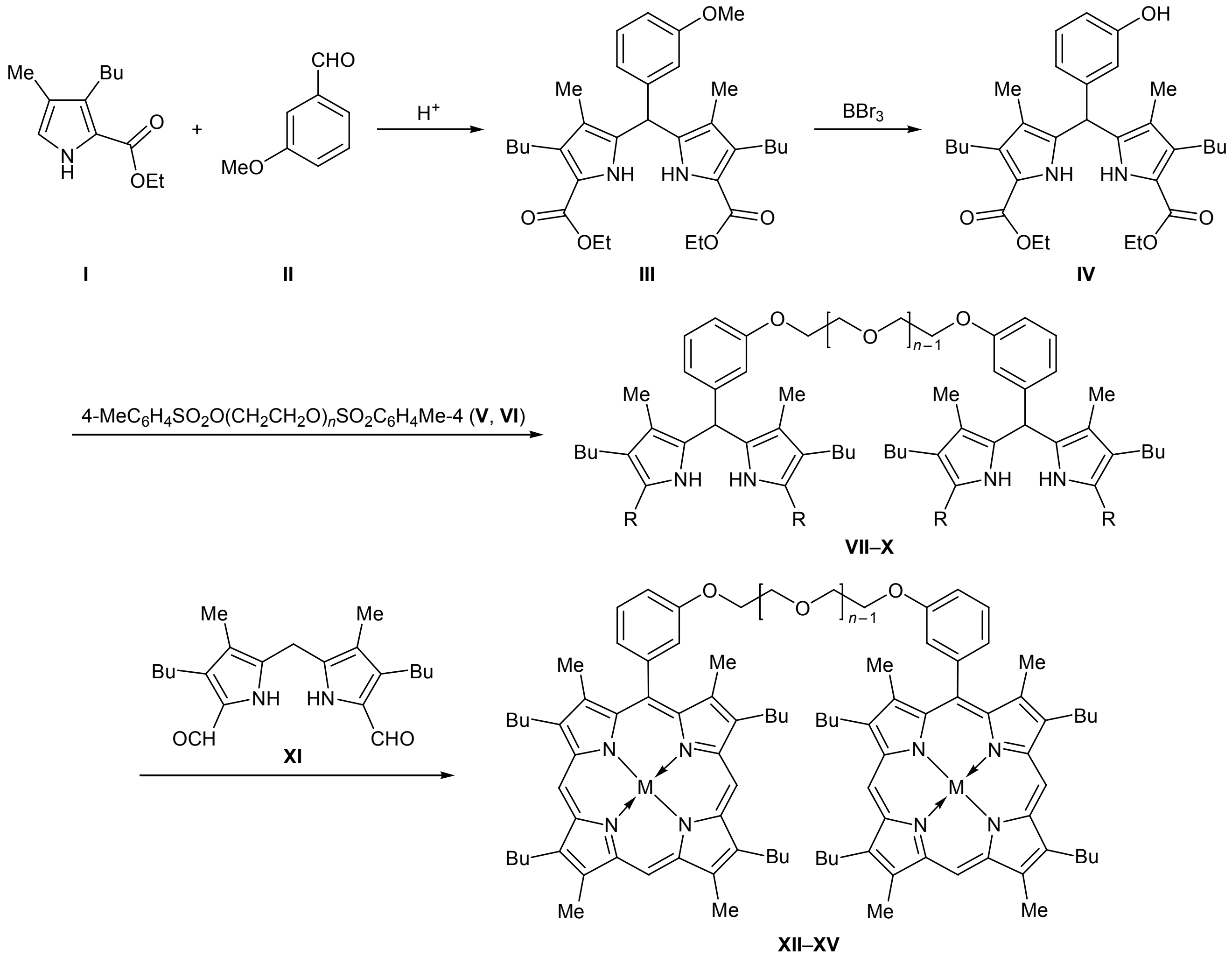
From the above viewpoint, undoubted interest is attracted by tetrapyrrole chromophores chemically modified with polyether fragments which possess their own complexing ability toward cations [6, 14, 15]. Polyether fragments can also be used as building blocks in the design of preorganized three-dimensional structures via covalent bonding with porphyrin macrorings.

The present work was aimed at developing new molecular sensors for ion recognition on the basis of porphyrin dimers in which mutual arrangement of the tetrapyrrole chromophores can be controlled via complex formation of the polyether linker with alkali metal cations. The results of studying the response of tetrapyrrole chromophore to complex formation with alkali metal cations indicated that polyether-bridged bis-porphyrins are promising as base structures for the design of molecular devices with useful functionalities.

By reaction of ethyl 3-butyl-4-methylpyrrole-2-carboxylate (**I**) with 3-methoxybenzaldehyde (**II**) in ethanol in the presence of a catalytic amount of hydrochloric acid we obtained (3-methoxyphenyl)dipyrrolylmethane **III**. Compound **III** was subjected to demethylation by treatment with BBr_3, and (3-hydroxyphenyl)-dipyrrolylmethane **IV** thus obtained was brought into condensation with polyethylene glycol bis(4-toluenesulfonates) **V** and **VI** in the presence of cesium carbonate in a mixture of dimethylformamide with acetonitrile. The condensation products were bis-dipyrrolylmethanes **VII**–**X** in which the dipyrrolylmethane fragments were linked through conformationally labile polyether bridges (Scheme 1).

Alkaline hydrolysis and subsequent thermal decarboxylation of compounds **VII** and **VIII** gave the corresponding derivatives **IX** and **X** having no substituents on C^5 in the pyrrole rings. The condensation of **IX** and **X** with 5,5′-methylenedi(3-butyl-4-methyl-1*H*-pyrrole-2-carbaldehyde) (**XI**) in acid medium, followed by oxidation with tetrachloro-1,4-benzoquinone, afforded bis-porphyrins **XII** and **XIII**. The latter reacted with

Scheme 1.



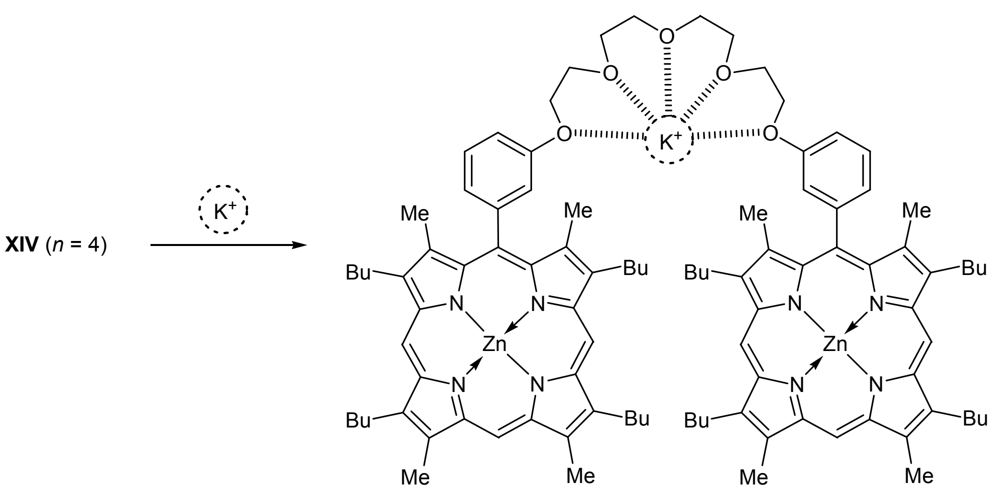
XII, **XIII**, M = H_2; **XIV**, **XV**, M = Zn; **VII**, **VIII**: R = COOEt; **IX**, **X,** R = H; **V**, **VII**, **XII**, **XIV**, $n = 4$; **VI**, **VIII**, **X**, **XIII**, **XV**, $n = 5$.

zinc acetate in boiling dimethylformamide to produce bis-porphyrin zinc complexes **XIV** and **XV**. The assumed structures of compounds **XII**–**XV** were consistent with their elemental compositions and electronic absorption, ^{1}H NMR, and mass spectra.

We examined how complex formation of porphyrins **XIV** and **XV** with potassium cation (K^+) in toluene–methanol (5 : 1) affects the properties of the tetrapyrrole chromophores. Compound **XIV** was found to bind K^+ ion yielding complex **XVI** (Scheme 2) due to correspondence of geometric parameters of the polyether cavity to those of the substrate. The complexation is accompanied by change of conformation of the molecule so that the porphyrin fragments in the dimer become closer to each other, which is reflected in the electronic absorption and ^{1}H NMR spectra. In particular, absorption bands in the electronic spectrum of **XVI** were displaced and broadened, and their intensity was lower, as compared to porphyrin **XIV** (see figure). Thus there are all characteristic features indicating exciton interaction between π-electron systems of closely located (oriented face-to-face) tetrapyrrole chromophores [6]. Mutual shielding of ring currents in the neighboring tetrapyrrole macrorings gives rise to upfield shift of proton signals in the ^{1}H NMR spectrum of complex **XVI** relative to the corresponding signals of porphyrin **XIV**.

According to the spectrophotometric titration data, the composition of complex **XVI** is 1 : 1: only one jump is observed on the titration curve (see figure), and the electronic absorption spectra revealed a family of isosbestic points. The stability constant of the complex ($K_s = 0.081 \times 10^6$ l mol^{-1}) was calculated according to standard procedure [16] from the spectrophoto-

Scheme 2.



XVI

metric data obtained at two wavelengths (corresponding to ascending and descending parts of the graph) using the following formula:

$$K_s = \frac{[\mathrm{ZnP}\cdot\mathrm{K}^+]}{[\mathrm{ZnP}]\cdot[\mathrm{K}^+]} = \frac{1}{[\mathrm{K}^+]}\left[\frac{\Delta A_{i,\lambda_1}}{\Delta A_{0,\lambda_1}}\,\frac{\Delta A_{0,\lambda_2}}{\Delta A_{i,\lambda_2}}\right]\mathrm{mol}^{-1}.$$

Here, λ_1 is the descending wavelength, λ_2 is the ascending wavelength, $[\mathrm{K}^+]$ is the concentration of potassium ions, ΔA_0 is the maximal variation of the optical density at the given wavelength, and ΔA_i is variation of the optical density at a given wavelength and at a given concentration [6, 16]. The error in the determination of K_s was 7–10%.

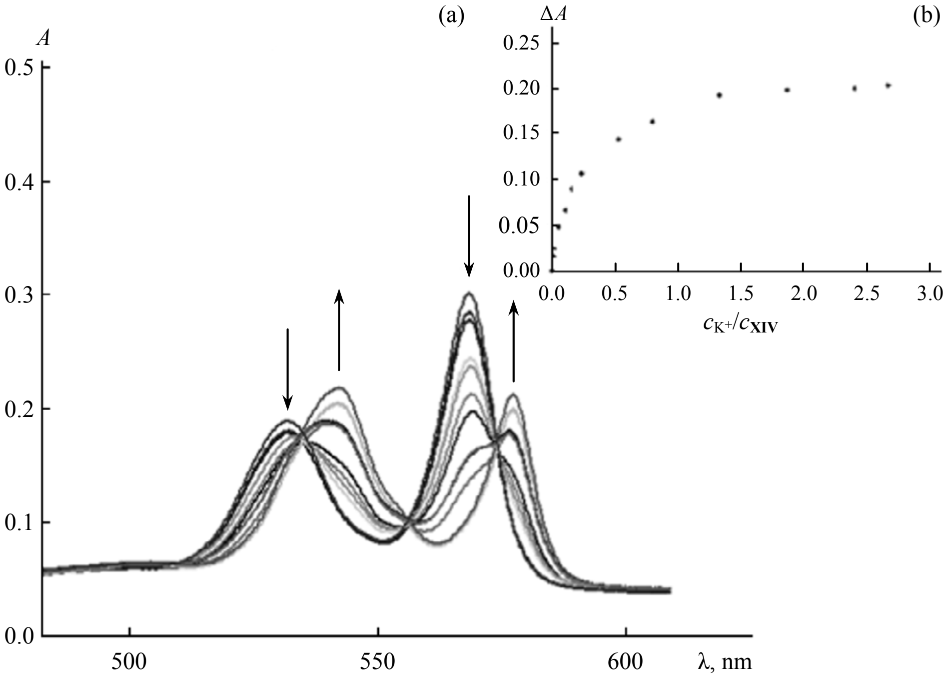
Our results showed that complex formation involving the polyether fragment in polyether-bridged porphyrin dimer gives a distinct response in the electronic absorption spectrum. Receptors like **XIV** are more advantageous than crown ethers that are traditionally used to bind alkali metal cations. The presence of a tetrapyrrole chromophore in the receptor supramolecule makes it possible to use spectrophotometric method (which is traditionally used in the chemistry of porphyrins) to study selective binding of cations. Such tetrapyrrole compounds may be promising as cation-controlled molecular switchers in extraction and membrane transport processes.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker VC-200 spectrometer at 200 MHz using benzene-d_6 as solvent and tetramethylsilane as internal reference. The mass spectra (electron impact, 70 eV) were obtained on an MKh-1310 mass spectrometer (ion source temperature 150–200°C). The electronic absorption spectra were measured from solutions in toluene on a Varian Cary 100 spectrophotometer. Tetraethylene glycol bis-(4-toluenesulfonate) (**V**) and pentaethylene glycol bis-(4-toluenesulfonate) (**VI**) were commercial products (from Sigma). Ethyl 3-butyl-4-methyl-1*H*-pyrrole-2-carboxylate (**I**) and 5,5′-methylenedi(3-butyl-4-methyl-1*H*-pyrrole-2-carbaldehyde) (**XI**) were synthesized as described in [13]. The products were isolated by column chromatography on aluminum oxide using toluene–hexane (1:2) as eluent. Organic solvents were purified by standard procedures [17]. The progress of reactions was monitored by TLC on Silufol UV-254 plates.

Diethyl 5,5′-(3-methoxyphenylmethylene)di(3-butyl-4-methyl-1*H*-pyrrole-2-carboxylate) (III). Ester **I**, 5 g (0.01 mol), and aldehyde **II**, 0.67 ml (0.01 mol), were dissolved in 100 ml of ethanol containing 3 ml of hydrochloric acid, and the solution was stirred for 3 h at 65°C. It was then cooled and diluted with 100 ml of water, and the precipitate was filtered off, washed with water, dried at room temperature, and recrystallized from methanol–water (2:1). Yield 6.1 g (79%). ^{1}H NMR spectrum, δ, ppm: 0.79 t (6H, $\mathrm{CH_2CH_2C\mathbf{H}_3}$), 0.98 m (4H, $\mathrm{CH_2C\mathbf{H}_2CH_3}$), 1.15 m (4H, $\mathrm{C\mathbf{H}_2CH_2CH_3}$), 1.36 t (6H, $\mathrm{OCH_2C\mathbf{H}_3}$), 2.20 s (6H, 4-$\mathrm{CH_3}$), 2.28 t (4H, 3-$\mathrm{CH_2}$), 3.66 s (3H, $\mathrm{CH_3O}$), 4.24 m (4H, $\mathrm{OCH_2}$), 5.28 s (1H, CH), 6.47 d (1H, 4′-H), 6.52 t (1H, 3′-H), 6.71 d (1H, 2′-H), 6.92 s (1H, 6′-H), 9.50 s (2H, NH).

Diethyl 5,5′-(3-hydroxyphenylmethylene)di(3-butyl-4-methyl-1*H*-pyrrole-2-carboxylate) (IV). A solution of 0.19 ml (0.2 mol) of $\mathrm{BBr_3}$ in 5 ml of



(a) Variation of the electronic absorption spectra in the system **XIV**–K$^+$ (c_{XIV} = 1 × 10^{-5}, c_{K^+} = 1 × 10^{-6}–1 × 10^{-4} M) and (b) spectrophotometric titration curve of complex **XIV** with potassium ion K$^+$ in the system toluene–methanol, 5 : 1 (c_{XIV} = 1 × 10^{-5} M; λ = 568 nm; temperature 298 K).

chloroform was added to a solution of 6.0 g (0.02 mol) of dipyrrolylmethane **III** in 70 ml of chloroform, and the mixture was heated for 1 h at room temperature. Methanol, 5 ml, was then added, and the mixture was stirred for 30 min at room temperature and neutralized with aqueous ammonia. The organic phase was washed twice with water, the solvent was distilled off under reduced pressure at 35°C, and the residue was recrystallized from ethanol–water (2:1). Yield 5.5 g (96%). ^{1}H NMR spectrum, δ, ppm: 0.78 t (6H, CH$_2$CH$_2$C**H**$_3$), 0.97 m (4H, CH$_2$C**H**$_2$CH$_3$), 1.12 m (4H, C**H**$_2$CH$_2$CH$_3$), 1.33 t (6H, OCH$_2$C**H**$_3$), 2.18 s (6H, 4-CH$_3$), 2.27 t (4H, 3-CH$_2$), 4.21 m (4H, OCH$_2$), 5.23 s (1H, CH), 6.44 d (1H, 4′-H), 6.49 t (1H, 5′-H), 6.67 s (1H, 2′-H), 6.78 s (1H, 6′-H), 9.48 s (2H, NH).

1,11-Bis[3-bis(4-butyl-5-ethoxycarbonyl-3-methyl-1*H*-pyrrol-2-yl)methylphenoxy]-3,6,9-trioxaundecane (VII). Dipyrrolylmethane **IV**, 60 mg (0.24 mmol), was dissolved at room temperature in 50 ml of a 1:2 dimethylformamide–acetonitrile mixture, 32.5 mg (0.10 mmol) of cesium carbonate and 42.9 mg (0.12 mmol) of tetraethylene glycol bis-(4-toluenesulfonate) were added in succession to the solution under argon, and the mixture was stirred for 24 h. The solvent was distilled off under reduced pressure, the residue was treated with a mixture of 5 ml of 10% hydrochloric acid and 50 ml of methylene chloride, and the organic phase was separated, washed twice with water, dried over sodium sulfate, and evaporated to a volume of 15 ml. The residue was diluted with 8 ml of methanol, and the precipitate was filtered off and dried at 55°C. Yield 175.9 mg (65%). ^{1}H NMR spectrum, δ, ppm: 0.77 t (12H, CH$_2$CH$_2$C**H**$_3$), 0.95 m (8H, CH$_2$C**H**$_2$CH$_3$), 1.11 m (8H, 4-CH$_2$C**H**$_2$), 1.31 t (12H, OCH$_2$C**H**$_3$), 2.16 s (12H, 3-CH$_3$), 2.25 t (8H, 4-CH$_2$), 3.68 m and 4.13 m (8H each, OCH$_2$CH$_2$O), 4.19 m (8H, OC**H**$_2$CH$_3$), 5.21 s (2H, CH), 7.36 m (4H, 2′-H, 6′-H), 7.56 t (2H, 5′-H), 7.77 d (2H, 4′-H), 9.42 s (4H, NH).

1,14-Bis[3-bis(4-butyl-5-ethoxycarbonyl-3-methyl-1*H*-pyrrol-2-yl)methylphenoxy]-3,6,9,12-tetraoxatetradecane (VIII) was synthesized in a similar way. Yield 202.5 mg (72%). ^{1}H NMR spectrum, δ, ppm: 0.79 t (12H, CH$_2$CH$_2$C**H**$_3$), 0.97 m (8H, CH$_2$C**H**$_2$CH$_3$), 1.09 m (8H, 4-CH$_2$C**H**$_2$), 1.29 t (12H, OCH$_2$C**H**$_3$), 2.13 s (12H, 3-CH$_3$), 2.23 t (8H, 4-CH$_2$), 3.68 m (16H, OCH$_2$CH$_2$O), 4.14 m (4H, OCH$_2$CH$_2$O), 4.21 m (8H, OC**H**$_2$CH$_3$), 5.17 s (2H, CH), 7.34 m (4H, 2′-H, 6′-H), 7.52 t (2H, 5′-H), 7.76 d (2H, 4′-H), 9.40 s (4H, NH).

1,11-Bis[3-bis(4-butyl-3-methyl-1*H*-pyrrol-2-yl)methylphenoxy]-3,6,9-trioxaundecane (IX). A solution of 10 mg of potassium hydroxide in 15 ml of water was added to a solution of 50 mg of dipyrrolylmethane **VII** in 75 ml of ethanol. The mixture was heated for 30 min at 90°C, the solvent was distilled under reduced pressure, and the residue, a yellow oily substance, was washed with water, dried at room temperature, and used in further syntheses without additional purification. Yield 37.9 mg (81%).

1,14-Bis[3-bis(4-butyl-3-methyl-1*H*-pyrrol-2-yl)methylphenoxy]-3,6,9,12-tetraoxatetradecane (X) was synthesized in a similar way. Yield 36.1 mg (77%).

1,11-Bis[3-(2,8,12,18-tetrabutyl-3,7,13,17-tetramethylporphyrin-5-yl)phenoxy]-3,6,9-trioxaundecane (XII). Compound **IX**, 168.9 mg (0.16 mmol), and dialdehyde **XI**, 65.8 mg (0.32 mmol), were added simultaneously to a solution of 1 ml of hydrobromic acid in 80 ml of ethanol. The mixture was stirred for 2 h at room temperature and treated with a solution of 24 mg (0.32 mmol) of tetrachloro-1,4-benzoquinone in 10 ml of chloroform. Water, 50 ml, and 15% aqueous ammonia, 5 ml, were added in succession, the organic phase was separated and evaporated under reduced pressure, and the residue was dried and subjected to chromatography on aluminum oxide using toluene–hexane (1:2) as eluent. The product was additionally recrystallized from methylene chloride–methanol (1:1). Yield 85.4 mg (35%). R_f 0.47 ($\mathrm{Al_2O_3}$, toluene–hexane, 1:2). Electronic absorption spectrum (toluene), λ_max, nm (log ε): 404.7 (5.38), 504.1 (4.39), 537.5 (4.26), 570.1 (4.17), 629.1 (4.09). ^{1}H NMR spectrum, δ, ppm: –2.95 s (4H, NH), 1.02 t (24H, $\mathrm{CH_2C\mathbf{H}_3}$), 1.14 s (12H, $\mathrm{CH_3}$), 1.22 s (12H, $\mathrm{CH_3}$), 1.51 m (16H, $\mathrm{C\mathbf{H}_2CH_3}$), 2.42 m (16H, $\mathrm{C\mathbf{H}_2CH_2CH_3}$), 3.62 m and 4.09 m (8H each, $\mathrm{OCH_2CH_2O}$), 3.80 t (16H, $\mathrm{C\mathbf{H}_2CH_2CH_2CH_3}$), 7.29 m (4H, 2′-H, 6′-H), 7.41 t (2H, 5′-H), 7.77 d (2H, 4′-H), 9.99 s (4H, *meso*-H), 10.12 s (2H, *meso*-H). Mass spectrum: *m*/*z* 1525.21 (I_rel 76%) $[M + 1]^+$. Found, %: C 77.94; H 8.70; N 7.20. $\mathrm{C_{100}H_{130}N_8O_5 \cdot CH_3OH}$. Calculated, %: C 77.99; H 8.45; N 7.21.

1,14-Bis[3-(2,8,12,18-tetrabutyl-3,7,13,17-tetramethylporphyrin-5-yl)phenoxy]-3,6,9,12-tetraoxatetradecane (XIII) was synthesized in a similar way**.** Yield 98.2 mg (41%). R_f 0.42 ($\mathrm{Al_2O_3}$, toluene–hexane, 1:2). Electronic absorption spectrum (toluene), λ_max, nm (log ε): 404.5 (4.98), 504.6 (3.59), 537.1 (3.46), 570.0 (3.32), 623.1 (3.30). ^{1}H NMR spectrum, δ, ppm: –2.97 s (4H, NH), 1.07 t (24H, $\mathrm{CH_2C\mathbf{H}_3}$), 1.16 s (12H, $\mathrm{CH_3}$), 1.21 s (12H, $\mathrm{CH_3}$), 1.53 m (16H, $\mathrm{C\mathbf{H}_2CH_3}$), 2.41 m (16H, $\mathrm{C\mathbf{H}_2CH_2CH_3}$), 3.61 m (16H, $\mathrm{OCH_2CH_2O}$), 4.11 m (4H, $\mathrm{OCH_2CH_2O}$), 3.82 t (16H, $\mathrm{C\mathbf{H}_2CH_2CH_2CH_3}$), 7.31 m (4H, 2′-H, 6′-H), 7.42 t (2H, 5′-H), 7.79 d (2H, 4′-H), 10.00 s (4H, *meso*-H), 10.13 s (2H, *meso*-H). Mass spectrum: *m*/*z* 1569.27 (I_rel 69%) $[M + 1]^+$. Found, %: C 77.22; H 8.66; N 7.06. $\mathrm{C_{102}H_{134}N_8O_6 \cdot H_2O}$. Calculated, %: C 77.25; H 8.63; N 7.01.

5,5′-[3,6,9-Trioxaundecane-1,11-diyldioxydi-1,3-phenylene]bis[(2,8,12,18-tetrabutyl-3,7,13,17-tetramethylporphyrinato)zinc(II)] (XIV). Porphyrin **XII**, 30 mg, was dissolved in 70 ml of dimethylformamide, excess zinc(II) acetate (1:10) was added, and the mixture was heated for 30 min at the boiling point, cooled, and diluted with 50 ml of water. The precipitate was filtered off, dried, and subjected to chromatography on aluminum oxide using toluene–hexane (1:2) as eluent. The product was additionally purified by recrystallization from methylene chloride–methanol (1:1). Yield 27.2 mg (87%). R_f 0.55 ($\mathrm{Al_2O_3}$, toluene–hexane, 1:2). Electronic absorption spectrum (toluene), λ_max, nm (log ε): 403.0 (5.75), 532.5 (4.51), 568.1 (4.70). ^{1}H NMR spectrum, δ, ppm: 1.00 t (24H, $\mathrm{CH_2C\mathbf{H}_3}$), 1.13 s (12H, $\mathrm{CH_3}$), 1.21 s (12H, $\mathrm{CH_3}$), 1.49 m (16H, $\mathrm{C\mathbf{H}_2CH_3}$), 2.40 m (16H, $\mathrm{C\mathbf{H}_2CH_2CH_3}$), 3.61 m (8H, $\mathrm{OCH_2CH_2O}$), 3.79 t (16H, $\mathrm{C\mathbf{H}_2CH_2CH_2CH_3}$), 4.06 m (8H, $\mathrm{OCH_2CH_2O}$), 7.28 m (4H, 2′-H, 6′-H), 7.39 t (2H, 5′-H), 7.76 d (2H, 4′-H), 9.96 s (4H, *meso*-H), 10.10 s (2H, *meso*-H). ^{1}H NMR spectrum of **XIV**–$\mathrm{K^+}$ complex, δ, ppm: 0.67 s (12H, $\mathrm{CH_3}$), 0.91 s (12H, $\mathrm{CH_3}$), 1.09 t (24H, $\mathrm{CH_2C\mathbf{H}_3}$), 1.48 m (16H, $\mathrm{C\mathbf{H}_2CH_3}$), 2.49 m (16H, $\mathrm{C\mathbf{H}_2CH_2CH_3}$), 3.59 m (8H, $\mathrm{OCH_2CH_2O}$), 3.77 t (16H, $\mathrm{C\mathbf{H}_2CH_2CH_2CH_3}$), 4.04 m (8H, $\mathrm{OCH_2CH_2O}$), 7.26 m (4H, 2′-H, 6′-H), 7.39 t (2H, 5′-H), 7.76 d (2H, 4′-H), 9.66 s (4H, *meso*-H), 9.95 s (2H, *meso*-H).

5,5′-[3,6,9,12-Tetraoxatetradecane-1,14-diyldioxydi-1,3-phenylene]bis[(2,8,12,18-tetrabutyl-3,7,13,17-tetramethylporphyrinato)zinc(II)] (XV) was synthesized in a similar way. Yield 28.7 mg (92%). R_f 0.59 ($\mathrm{Al_2O_3}$, toluene–hexane, 1:2). Electronic absorption spectrum (toluene), λ_max, nm (log ε): 403.5 (5.75), 532.1 (4.56), 568.5 (4.76). ^{1}H NMR spectrum, δ, ppm: 1.05 t (24H, $\mathrm{CH_2C\mathbf{H}_3}$), 1.15 s (12H, $\mathrm{CH_3}$), 1.19 s (12H, $\mathrm{CH_3}$), 1.52 m (16H, $\mathrm{C\mathbf{H}_2CH_3}$), 2.39 m (16H, $\mathrm{C\mathbf{H}_2CH_2CH_3}$), 3.60 m (16H, $\mathrm{OCH_2CH_2O}$), 3.81 t (16H, $\mathrm{C\mathbf{H}_2CH_2CH_2CH_3}$), 4.09 m (4H, $\mathrm{OCH_2CH_2O}$), 7.29 m (4H, 2′-H, 6′-H), 7.41 t (2H, 3′-H), 7.76 d (2H, 4′-H) 10.11 s (2H, *meso*-H), 9.98 s (4H, *meso*-H).

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