6.84 (1H, q, J_{39} = 2.2 Hz, 3-H); 8.10 (1H, s, 4-H); 8.95 (1H, s, 9-H); 8.02 and 7.98 (2H, m, 5-H and 8-H); 7.46 ppm (2H, m, 6-H and 7-H). Found, %: C 80.5, H 5.4, N 6.4. $C_{14}H_{11}NO$. Calculated, %: C 80.4, H 5.3, N 6.7.

5,6-Benzindole (VI). 25 ml of a 5% methanol solution of KOH was added to 0.1 gram (0.478 mmole) of compound V. The mixture was kept at 40-50°C for 15 min, then at 20-22°C for 24 h. The methanol was evaporated and the residue extracted with ether. The ether layer was washed with water and dried over $MgSO_4$. After evaporation of ether, 5,6-benzindole was obtained in the form of white or light pinkish flakes, yield 0.07 g (88%), R_f 0.80, mp 180-181°C. IR spectrum in $CHCl_3$: 3490 (NH); in KBr: 3415 (NH), 1610 (w), 1580 (w), 1560 (w), 1518 (m), 1492 (m) (skeletal vibrations with primary participation of multiple bonds), 860 (s), 736, 703 cm^{-1} (s) (nonplanar vibrations of CH and NH). PMR spectrum ($CDCl_3$): 7.16 (1H, m, J_{23} = 3.17, $J_{2'}$ = 2.68 Hz, 2-H); 6.48 (1H, m, J_{31} = 1.95, J_{39} = 0.98 Hz, 3-H); 7.95 (1H, s, 4-H); 7.76 and 7.69 (2H, m, 5-H and 8-H); 7.22 and 7.12 (2H, q, 6-H and 7-H); 7.72 ppm (1H, s, NH).

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SYNTHESIS OF TETRAPHENYLPORPHINES WITH ACTIVE GROUPS IN THE PHENYL RINGS. 4.* FUNCTIONALLY SUBSTITUTED MONOHYDROXY DERIVATIVES OF TETRAPHENYLPORPHINE

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Monosubstituted porphyrins bonded to 8-hydroxyquinoline, α-naphthalene, anthraquinone, and naphthoquinone residues, as well as dimeric porphyrins, were obtained by alkylation of monohydroxyphenyltriphenylporphines.

In nature metalloporphyrins function in the composition of protein formations, the active groups of which have a pronounced effect on the properties of the metal complexes [2]. The synthesis and study of porphyrins having on the periphery of their molecules active functional groups that are capable of interacting with the central metal atom of the porphyrin ring are therefore of great value.

The synthesis of such compounds, which is based on modification of natural porphyrins, is quite complex and includes a large number of steps [3]. In addition, the bonds formed after "tying" of residues with active groups are not very strong (this is primarily the case for amide bonds [4]).

For the synthesis of such compounds it is interesting to use synthetic porphyrins with hydroxy groups that are capable of forming ether bonds. For this, we selected tetraphenylporphine derivatives that have high stabilities and are readily formed in the condensation of pyrrole with benzaldehydes [5].

*See [1] for communication 3.

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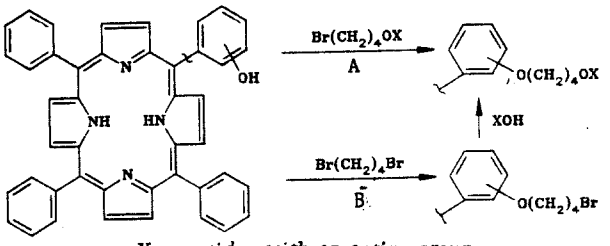
The goal of the research was to obtain monosubstituted tetraphenylporphines with active groups by alkylation of monohydroxyphenyltriphenylporphines I-III (Scheme I). The latter can be readily obtained by condensation of pyrrole with a mixture of the hydroxybenzaldehyde and benzaldehyde as in [6] and purification by column chromatography on silica gel.

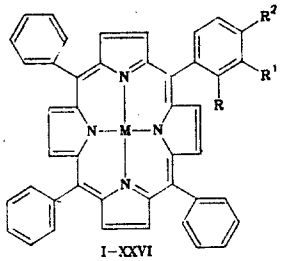
The alkylation of hydroxy-substituted tetraphenylporphines with alkyl halides can be carried out readily and in high yields in DMF in the presence of an alkaline agent (usually K_2CO_3) [7], We used a similar method and obtained monohexadecyloxyphenyltriphenylporphines VII-IX, as well as tetraphenylporphines monosubstituted with 8-hydroxyquinoline (X-XII), α-naphthalene (XIII-XV), anthraquinone (XVI), and naphthoquinone (XVII) residues attached to the benzene ring through a tetramethylene fragment. In addition, dimeric porphyrins XXIV-XXVI were obtained.

Scheme 1



X — residue with an active group

It is apparent from Scheme 1 that "tying" of residues with active groups to the porphyrin can be carried out via two pathways: by alkylation of the monohydroxyphenyltriphenylporphine with an alkyl halide with an active group (A) or by prior alkylation of the hydroxyporphyrin with excess 1,4-dibromobutane with the formation of an ω-bromobutyloxyphenyltriphenylporphine with its subsequent reaction under similar conditions with the hydroxy derivative (B).



I-XXVI

Pathway A is preferable, since it gives a higher yield of the desired product (based on the starting porphyrin). For example, for porphyrin XIII the yields from I are 91% (pathway A) and 37% (pathway B). However, one cannot always obtain a haloalkane that has a second active functional goup. In this connection we used primarily pathway B.

By varying the alkylation conditions we observed that a higher yield (91% for XIII) can be obtained at 20°C. In refluxing DMF the yield decreases (74% for XIII); however, the reaction time can be decreased significantly (from 24 h to 2 h). Thus in the case of alkylation of active compounds it is better to carry out the reaction at 20°C (in the preparation of IV-XV and XXI-XXIII), whereas it is better to carry out the reaction at the boiling point of DMF (in the preparation of XVI and XVII) for compounds that are difficult to alkylate.

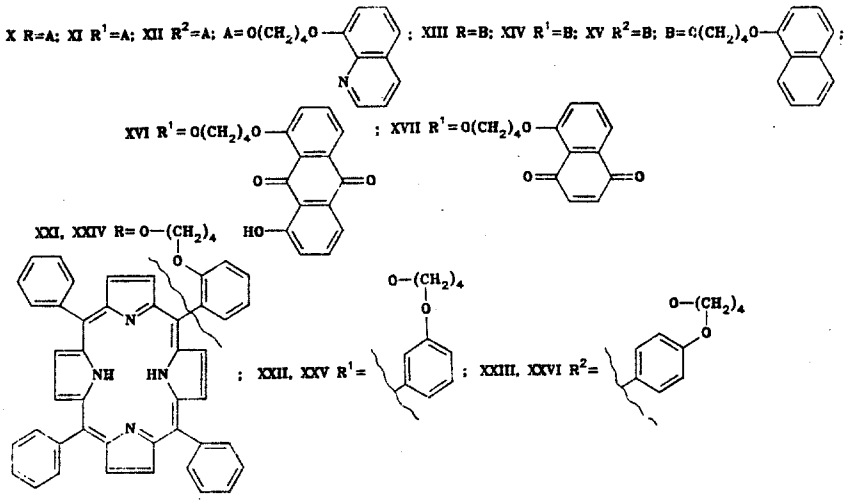
The reaction of zinc complexes (XVIII-XX) of ω-bromobutoxyphenyltriphenylporphines with monohydroxyphenyltriphenylporphines I-III gave dimeric porphyrins XXI-XXIII, which have electronic absorption spectra that are intermediate between the spectra of the starting free porphyrins and the metal complexes (Table 1).

The electronic absorption spectra of monosubstituted tetraphenylporphines IV-XVII, XXIV, and XXVI (Table 1) display a significant similarity. The small bathochromic shift of the absorption bands of porphyrins that have substituents in the para position (VI, IX, XII, XV, and XXVI) is evidently due to partial transmission of the electronic effect of the substituent to the porphyrin macroring; this is impossible in the case of ortho and meta substituents because of steric hindrance.

TABLE 1. Characteristics of Monosubstituted Tetraphenylporphines IV-XXVI

Porphyrin	R_f	λ_{max}, nm (lg ε)					Found, %			Empirical formula	Calculated, %			Yield, %
		I	II	III	IV	Soret	C	H	N		C	H	N	
IV	0,26 (A)	647 (3,76)	590 (3,82)	550 (3,91)	515 (4,29)	419 (5,88)	83,9	7,6	6,8	$C_{60}H_{62}N_4O$	84,3	7,3	6,6	62
V	0,53 (A)	648 (3,77)	590 (3,81)	550 (3,94)	515 (4,31)	419 (5,74)	83,7	7,5	6,8	$C_{60}H_{62}N_4O$	84,3	7,3	6,6	51
VI	0,68 (A)	648 (3,82)	591 (3,79)	552 (3,98)	516 (4,29)	420 (5,77)	84,0	7,5	6,5	$C_{60}H_{62}N_4O$	84,3	7,3	6,6	44
VII	0,85 (B)	648 (3,83)	590 (3,92)	550 (4,03)	515 (4,40)	419 (5,79)	75,1	4,3	7,6	$C_{48}H_{37}BrN_4O$	75,3	4,8	7,3	58
VIII	0,82 (B)	648 (3,76)	590 (3,80)	550 (3,91)	516 (4,27)	419 (5,65)	75,6	4,9	7,8	$C_{48}H_{37}BrN_4O$	75,3	4,8	7,3	56
IX	0,75 (B)	649 (3,75)	592 (3,66)	552 (3,84)	517 (4,13)	421 (5,55)	75,6	4,5	7,7	$C_{48}H_{37}BrN_4O$	75,3	4,8	7,3	45
X	0,37 (C)	648 (3,75)	590 (3,82)	550 (3,94)	517 (4,30)	420 (5,66)	81,9	5,6	8,7	$C_{57}H_{43}N_5O_2$	82,5	5,2	8,4	34
XI	0,40 (C)	648 (3,79)	590 (3,81)	550 (3,94)	516 (4,30)	419 (5,70)	82,8	4,9	8,3	$C_{57}H_{43}N_5O_2$	82,5	5,2	8,4	36
XII	0,42 (C)	649 (3,82)	589 (4,02)	552 (3,99)	516 (4,27)	420 (5,66)	82,1	5,3	8,6	$C_{57}H_{43}N_5O_2$	82,5	5,2	8,4	33
XIII	0,88 (B)	652 (3,52)	592 (3,60)	550 (3,68)	516 (3,93)	420 (5,16)	84,5	4,8	7,2	$C_{58}H_{44}N_4O_2$	84,1	5,3	6,8	91*
XIV	0,90 (B)	647 (3,70)	590 (3,78)	550 (3,86)	516 (4,27)	419 (5,66)	84,6	5,7	6,3	$C_{58}H_{44}N_4O_2$	84,1	5,3	6,8	43
XV	0,93 (B)	648 (3,80)	592 (3,77)	552 (3,96)	517 (4,25)	420 (5,67)	83,7	4,9	6,1	$C_{58}H_{44}N_4O_2$	84,1	5,3	6,8	15
XVI	0,99 (B)	647 (3,72)	590 (3,84)	551 (3,95)	515 (4,81)	420 (5,85)	81,2	4,7	6,1	$C_{66}H_{42}N_4O_5$	81,6	4,3	5,8	48
XVII	0,52 (B)	645 (3,76)	590 (3,86)	550 (3,94)	516 (4,16)	419 (5,57)	82,3	4,6	6,2	$C_{62}H_{42}N_4O_4$	82,1	4,6	6,2	15
XVIII	0,62 (B)		595 (3,67)	554 (4,20)		424 (5,54)	69,8	4,3	6,9	$C_{48}H_{35}BrN_4OZn$	69,6	4,2	6,8	92
XIX	0,75 (B)		594 (3,80)	553 (4,28)		424 (5,53)	69,1	4,2	6,4	$C_{48}H_{35}BrN_4OZn$	69,6	4,2	6,8	92
XX	0,76 (B)		596 (3,82)	554 (4,22)		424 (5,63)	69,7	4,6	6,3	$C_{48}H_{35}BrN_4OZn$	69,6	4,2	6,8	79
XXI	0,32 (B)	648 (3,32)	595 (3,69)	552 (4,17)	516 (3,81)	422 (5,57)	79,9	4,6	7,8	$C_{92}H_{66}N_8O_2Zn$	80,1	4,8	8,1	7
XXII	0,63 (B)	648 (3,63)	593 (3,85)	551 (4,23)	516 (4,08)	422 (5,67)	79,7	5,2	8,4	$C_{92}H_{66}N_8O_2Zn$	80,1	4,8	8,1	73
XXIII	0,90 (B)	648 (3,78)	595 (3,94)	552 (4,24)	518 (4,11)	422 (5,69)	80,4	4,6	7,9	$C_{92}H_{66}N_8O_2Zn$	80,1	4,8	8,1	24
XXIV	0,24 (B)	645 (3,83)	589 (4,02)	550 (4,09)	515 (4,30)	419 (5,86)	83,7	4,8	8,8	$C_{92}H_{68}N_8O_2$	83,9	5,0	8,5	74
XXV	0,84 (B)	648 (3,87)	590 (3,93)	550 (4,06)	516 (4,40)	419 (5,80)	83,6	5,2	8,9	$C_{92}H_{68}N_8O_2$	83,9	5,0	8,5	93
XXVI	0,75 (B)	648 (3,69)	591 (3,76)	552 (3,93)	517 (4,22)	418 (5,71)	84,2	5,3	8,6	$C_{92}H_{68}N_8O_2$	83,3	5,0	8,5	89

*This is the yield by method A; the yield of XIII by method B was 37%.



I R=OH; II R¹=OH; III R²=OH; IV R=$OC_{16}H_{33}$; V R¹=$OC_{16}H_{33}$; VI R²=$OC_{16}H_{33}$; VII, XVIII R=$O(CH_2)_4Br$; VIII, XIX R¹=$O(CH_2)_4Br$; IX, XX R²=$O(CH_2)_4Br$; unindicated R, R¹, R²=H; I—XVII, XXIV—XXVI M=2H; XVIII—XXIII M=Zn

EXPERIMENTAL

The electronic absorption spectra of solutions of the compounds in chloroform were recorded with a Specord M40 spectrophotometer. The NMR spectra of solutions in deuterochloroform were obtained with a Tesla BS-497 spectrometer (100 MHz) with hexamethyldisiloxane (HMDS) as the internal standard. The IR spectra of suspensions in mineral oil, which were placed between potassium bromide plates, were recorded with an IKS-29 spectrometer. The individuality and purity of the compounds were established by thin-layer chromatography (TLC) of Silufol in chloroform–hexane (1:1) (A), chloroform (B), and ether (C) systems and on the basis of the results of elementary analysis.

5-(2-Hexadecyloxyphenyl)-10,15,20-triphenylporphine (IV). A mixture of 0.5 g (0.81 mmole of I, 0.6 g (4.03 mmole) of n-hexadecyl bromide, 0.6 g (4.03 mmole) of anhydrous potassium carbonate, and 30 ml of DMF was stirred at 20°C for 24 h, after which it was poured into 150 ml of water, and the aqueous mixture was heated to the boiling point and filtered with a Büchner funnel. The precipitate was dried to constant mass at 20°C, dissolved in 50 ml of chloroform, and chromatographed with a column (2.5 × 60 cm) packed with silica gel (L 100/250) with elution with chloroform. The first dark-red zone of porphyrin IV was collected. The solvent was concentrated to a volume of 5 ml, and the product was precipitated with 30 ml of methanol. The yield was 0.42 g (62%). IR spectrum: 1250 (ν_{as} C–O–C), 1075 cm^{-1} (ν_s C–O–C).

A similar procedure was used to obtain 0.35 g (51%) of 5-(3-hexadecyloxyphenyl)-10,15,20-triphenylporphine (V) from II and n-hexadecyl bromide, as well as 0.28 g (44%) of 5-(4-hexadecyloxyphenyl)-10,15,20-triphenylporphine (VI) from III and n-hexadecyl bromide.

5-(2-Bromotetramethyleneoxyphenyl)-10,15,20-triphenylporphine (VII). A mixture of 0.5 g (0.81 mmole) of I, 0.9 g (4.03 mmole) of 1,4-dibromobutane, 0.6 g (4.03 mmole) of anhydrous potassium carbonate, and 30 ml of DMF was stirred at 20°C for 24 h, after which 0.36 g (58%) of porphyrin VII was isolated as in the preparation of IV.

A similar procedure was used to obtain 0.34 g (56%) of 5-(3-bromotetramethyleneoxyphenyl) 10,15,20-triphenylporphine (VIII) from II and 1,4-dibromobutane. NMR spectrum: 8.75 (t, β protons of the porphyrin ring), 8.22 (s, ortho protons of the phenyl rings), 7.62 (t, meta and para protons of the phenyl rings), 4.43 (t, OCH_2 protons of the alkyl chain), 4.13 (t, CH_2 protons of the alkyl chain), 2.78 (t, CH_2 protons of the alkyl chain), and −2.76 ppm (s, NH protons).

A similar procedure was used to obtain 0.28 g (45%) of 5-(4-bromotetramethyleneoxyphenyl) 10,15,20-triphenylporphine (IX) from III and 1,4-dibromobutane.

5-[2-(8-Quinolyloxy)tetramethyleneoxyphenyl]-10,15,20-triphenylporphine (X). A mixture of 0.4 g (0.81 mmole) of VII, 0.5 g (4.03 mmole) of 8-hydroxyquinoline, 0.6 g (4.03 mmole) of anhydrous potassium carbonate, and 30 ml of DMF was stirred at 20°C for 24 h, after which 0.21 g (34%) of porphyrin X was isolated as in the preparation of IV.

A similar procedure was used to obtain 0.22 g (36%) of 5-[3-(8-quinolyloxy)tetramethyleneoxyphenyl]-10,15,20-triphenylporphine (XI) from VIII and 8-hydroxyquinoline, as well as 0.20 g (33%) of 5-[4-(8-quinolyloxy)tetramethyleneoxyphenyl]-10,15,20-triphenylporphine (XII) from (IX) and 8-hydroxyquinoline.

5-[2-(1-Naphthyloxy)tetramethyleneoxyphenyl]-10,15,20-triphenylporphine (XIII). A) A mixture of 0.5 g (0.81 mmole) of I, 1.4 g (4.03 mmole) of 1-naphthyloxytetramethylene bromide, 0.6 g (4.03 mmole) of anhydrous potassium carbonate, and 30 ml of DMF was stirred at 20°C for 24 h, after which 0.55 g (91%) of porphyrin XIII was isolated as in the preparation of porphyrin IV.

B) A mixture of 0.4 g (0.81 mmole) of VII, 0.7 g (4.03 mmole) of α-naphthol, 0.6 g (4.03 mmole) of anhydrous K_2CO_3, and 30 ml of DMF was stirred at 20°C for 24 h, after which 0.22 g (37%) of porphyrin XIII was isolated as in the preparation of IV. NMR spectrum: 8.75 (t, β protons of the porphyrin ring), 8.11 (s, ortho protons of the phenyl rings), 7.66 (t, meta and para protons of the phenyl rings), 6.44-7.44 (m, protons of the naphthalene residue), 5.00 (t, OCH_2 protons of the alkyl chain), 2.30 (t, CH_2 protons of the alkyl chain), and −2.78 ppm (s, NH protons).

5-[3-(1-Naphthyloxy)tetramethyleneoxyphenyl]-10,15,20-triphenylporphine (XIV). This compound [0.26 g (43%)] was obtained by method B from VIII and α-naphthol as in the preparation of XIII. NMR spectrum: 8.80 (t, β protons of the porphyrin ring), 8.15 (s, ortho protons of the phenyl rings), 7.73 (t, meta and para protons of the phenyl rings), 7.22-7.58 (m, protons of the naphthalene ring), 4.17 (OCH_2 protons of the alkyl chain), 2.12 (t, CH_2 protons of the alkyl chain), and −2.82 ppm (s, NH protons).

5-[4-(1-Naphthyloxy)tetramethyleneoxyphenyl]-10,15,20-triphenylporphine (XV). This compound [0.10 g (15%)] was obtained from IX and α-naphthol by method B as in the preparation of XIII.

5-{3-[1-(8-Hydroxy-9,10-anthraquinonyl)oxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine (XVI). A mixture of 0.4 g (0.81 mmole) of VIII, 1 g (4.03 mmole) of 1,8-diohydroxyanthraquinone, 0.6 g (4.03 mmole) of anhydrous potassium carbonate, and 30 ml of DMF was refluxed for 2 h, after which it was cooled, and XVI [0.36 g (48%)] was isolated as in the preparation of porphyrin IV. NMR spectrum: 8.80 (t, β protons of the porphyrin ring), 8.18 (s, ortho protons of the phenyl rings), 7.70 (t, meta and para protons of the phenyl rings), 7.29-7.85 (m, protons of the anthraquinone residue), 4.15 (t, OCH_2 protons of the alkyl chain), 2.0 (t, CH_2 protons of the alkyl chain), and −2.84 ppm (s, NH protons). IR spectrum: 1675 cm^{-1} ($\nu_{C=O}$).

5-{3-[5-(1,4-Naphthoquinonyl)oxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine (XVII). A mixture of 0.4 g (0.81 mmole) of porphyrin VIII, 0.7 g (4.03 mmole) of 5-hydroxy-1,4-naphthoquinone [8], 0.6 g (4.03 mmole) of anhydrous potassium carbonate, and 30 ml of DMF was refluxed for 2 h, after which the mixture was cooled, and XVII [0.10 g (15%)] was isolated as in the preparation of IV. NMR spectrum: 8.78 (t, β protons of the porphyrin ring), 8.15 (s, orthoprotons of the phenyl rings), 7.71 (t, meta and para protons of the phenyl rings), 7.30-7.69 (m, protons of the naphthoquinone residue), 4.19 (t, OCH_2 protons of the alkyl chain), 1.88 (t, CH_2 protons of the alkyl chain), and −2.84 ppm (s, NH protons). IR spectrum: 1720 cm^{-1} ($\nu_{C=O}$).

5-(2-Bromotetramethyleneoxyphenyl)-10,15,20-triphenylporphine Zinc Complex (XVIII). A mixture of 0.4 g (0.81 mmole) of VII, 0.9 g (4.03 mmole) of zinc acetate dihydrate, 10 ml of methanol, and 30 ml of chloroform was refluxed for 1 h, after which it was cooled, washed with water (two 100-ml portions), and chromatographed with a column (1.5 × 60 cm) packed with silica gel with elution by chloroform. The first red zone of XVIII was collected. The solution was concentrated to 5 ml, and 0.62 g (92%) of XVIII was precipitated with 30 ml of methanol.

A similar procedure was used to obtain 0.62 g (92%) of 5-(3-bromotetramethyleneoxyphenyl) 10,15,20-triphenylporphine zinc complex (XIX) from VIII and zinc acetate dihydrate, as well as 0.53 g (79%) of 5-(4-bromotetramethyleneoxyphenyl)-10,15,20-triphenylporphine zinc complex from porphyrin IX and zinc acetate dihydrate.

Monomeric 5-{2-[2-(10,15,20-Triphenyl-5-porphinyl)phenyloxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine Zinc Complex (XXI). A mixture of 0.7 g (0.81 mmole) of XIX, 0.5 g (0.81 mmole) of I, and 0.6 g (4.03 mmole) of anhydrous potassium carbonate in 30 ml of DMF

was stirred at 20°C for 48 h, after which 0.07 g (7%) of the product was isolated as in the preparation of IV.

A similar procedure gave 0.8 g (73%) of monomeric 5-{3-[3-(10,15,20-triphenyl-5-porphinyl)phenyloxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine zinc complex (XXII) from XX and II, as well as 0.27 g (24%) of monomeric 5-{4-[4-(10,15,20-triphenyl-5-porphinyl)phenyloxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine zinc complex (XXIII) from XXI and III.

5-{2-[2-(10,15,20-Triphenyl-5-porphinyl)phenyloxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine (XXIV). A 0.14-g (0.1 mmole) sample of porphyrin XXII was dissolved in 30 ml of chloroform, 3 ml of HCl (d = 1.36 g/cm^3) was added, and the resulting mixture was heated up to the boiling point. The solution was cooled, neutralized to pH 8 with 5 ml of 25% ammonium hydroxide (the color of the solution changed from green to dark red), and chromatographed with a column (1.5 × 60 cm) packed with silica gel (L 100/250) with elution by chloroform. The first dark-red zone of XXIV was collected. The solvent was concentrated to 5 ml, and 0.1 (74%) of XXIV was precipitated with 30 ml of methanol. NMR spectrum: 8.77 (t, β protons of the porphyrin ring), 8.13 (t, ortho protons of the phenyl rings), 7.71 (m, meta and para protons of the phenyl rings), 5.00 (t, OCH_2 protons of the alkyl chain), 1.98 (t, CH_2 protons of the alkyl chain), and −2.84 ppm (s, NH protons).

A similar procedure was used to obtain 0.12 g (93%) of 5-{3-[3-(10,15,20-triphenyl-5-porphinyl)phenyloxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine (XXV) from XXII, as well as 0.11 g (89%) of 5-{4-[4-(10,15,20-triphenyl-5-porphinyl)phenyloxy]tetramethyleneoxyphenyl}-10,15,20-triphenylporphine (XXVI) from XXIII.

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