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Synthesis of Chiral Aza Crown Ethers Having Exocyclic Hydroxy Groups and Their Use in Asymmetric Reduction of Ketones with Sodium Tetrahydridoborate

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Abstract—New chiral diaza crown ethers with exocyclic hydroxy groups were synthesized by reactions of (2*S*,3*S*)-1,4-dibenzyloxy-2,3-bis(2-oxiranylmethoxy)butane with *N*,*N*′-dibenzyl-1,2-ethanediamine and (4*S*,5*S*)-4,5-bis(benzylaminomethyl)-2,2-dimethyl-1,3-dioxolane. Catalytic debenzylation of the products gave the corresponding derivatives having secondary amino groups. The obtained diaza crown ethers, as well as some known crown ethers, were used as asymmetric catalysts in the reduction of pinacolone and acetophenone with sodium tetrahydridoborate in methylene chloride. Depending on the catalyst structure, the optical yield of the reduction products ranged from 5 to 90%.

One of the simplest procedures for the preparation of chiral secondary alcohols is asymmetric hydride reduction of unsymmetrical ketones in the presence of chiral catalysts. This gave an impetus to extensive studies on the enantioselectivity of ketone reduction in two-phase systems involving various chiral phase-transfer catalysts [1]. Such reactions were studied most thoroughly in liquid–liquid and liquid–solid systems with chiral quaternary ammonium salts and conformationally rigid 1,1′-binaphthalene derivatives [1–3]. Only a few examples have been reported on the use of chiral crown ethers [4, 5] and aza crown ethers [6, 7] as phase-transfer catalysts. In most cases, secondary alcoholos were obtained with high chemical yields (60–100%), but enantiomeric excess was as a rule small (1–8% [4], 20–48% [7]) or absent [6]. Greater optical yields (25–65%) were achieved only in the reduction of sterically hindered ketones in the presence of aminoborane (NH_3BH_3) complexes with chiral tetraphenyl-18-crown-6 [5].

In the reduction of ketones with sodium tetrahydridoborate in the presence of chiral ligands, preferential formation of one enantiomer is determined by the presence of a chiral barrier in the ligand. This leads to steric and hence energetic nonequivalence of intermediate complexes formed with participation of the ligand, reagent, and substrate. Therefore, efficient enantiodifferentiation requires that the reagent (in our case, tetrahydridoborate ion) sufferred the effect of the ligand chiral barrier to the maximum possible extent.

Crown ethers tend to complex formation with metal cations. Here, tetrahydridoborate ion resides above the macroring plane and does not form short contacts with the macroring [8]. Therefore, it is difficult to expect efficient enantiodifferentiation, for sodium cation is in fact replaced by a bulkier crown ether–sodium cation complex, which should not affect appreciably steric requirements of the reactive species, tetrahydridoborate ion. Thus, a necessary condition for enantioselective reduction is the presence in a crown ether molecule of additional groups which are capable of binding tetrahydridoborate ion [2]. Such groups may by hydroxy and amino [1, 2, 4, 7, 9, 10]. According to most published data, such groups were usually located in the side chain of a chiral crown ether, so that they were remote from the macroring to a longer or shorter distance. As a result, the enantioselectivity in reduction of ketones was poor.

In order to check out whether the above statement is true and to search for new catalysts for asymmetric hydride reduction of carbonyl compounds we synthesized a series of new chiral diaza crown ethers **IV**–**VI** in which exocyclic hydroxy groups are attached directly to the macrocyclic framework. The resulting compounds were then tested for enantio-

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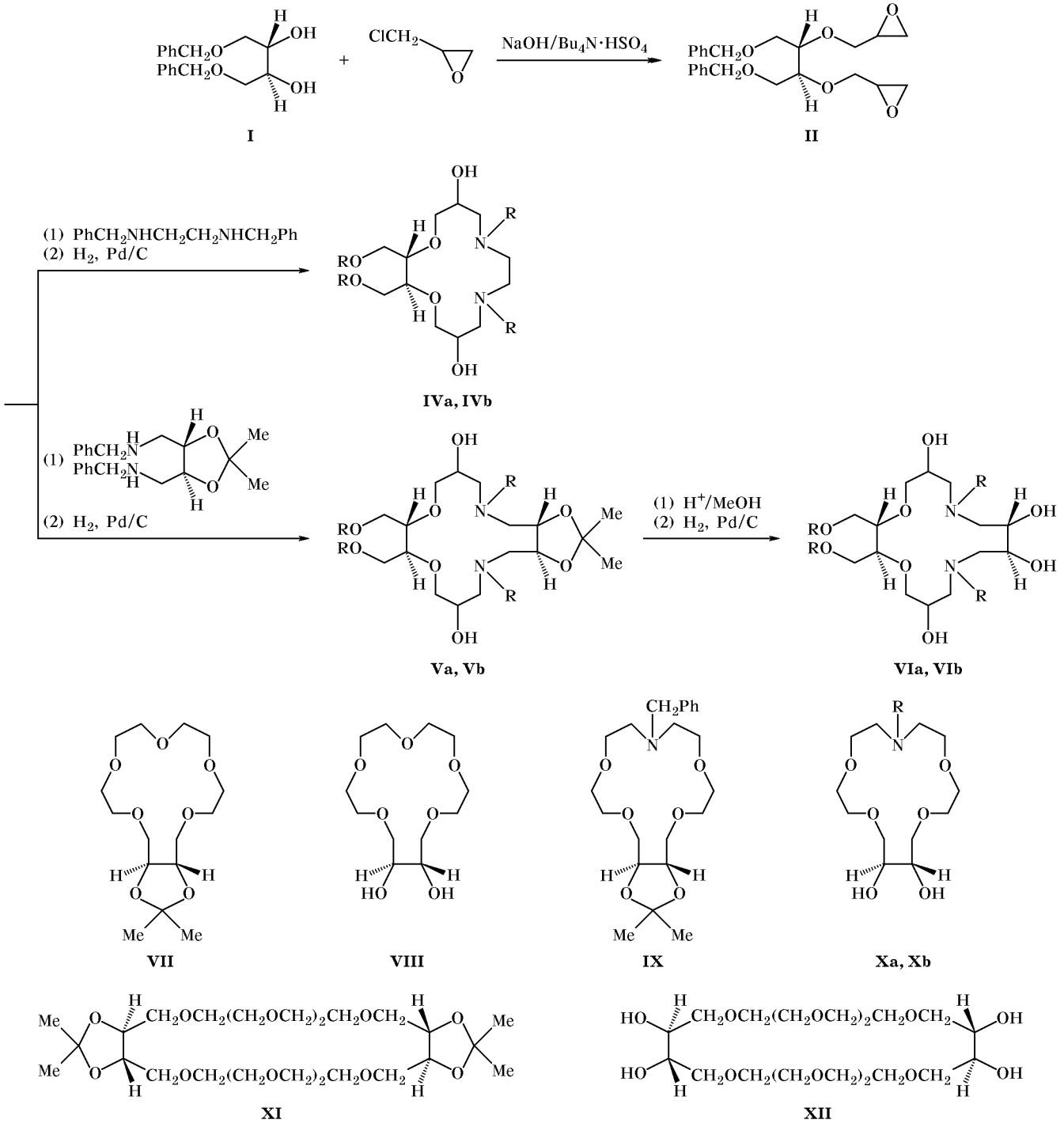
Scheme 1.



IV–**VI**, **X**, R = $PhCH_2$ (**a**), H (**b**).

selectivity in the reduction of some ketones with sodium tetrahydridoborate. For comparison, we also examined enantiodifferentiating properties in the same reactions of chiral podands **I** and **III** and previously described crown ethers **VII**, **VIII**, **XI**, and **XII** [11] and aza crown ethers **IX**, **Xa**, and **Xb** [12] (Scheme 1). The initial chiral compounds for the synthesis of aza crown ethers **IV**–**VI** were diol **I** [13] and diamine **III**. Alkylation of diol **I** with 1-chloro-2,3-epoxypropane under conditions of phase-transfer catalysis gave

Reduction of pinacolone and acetophenone with sodium tetrahydridoborate in methylene chloride in the presence of chiral ligands **I** and **III**–**XII**

Ligand no.	Reaction time, h		$[\alpha]_D^{20}$, deg		Optical yield,[a] %		Predominant enantiomer	
	pinacolone	acetophenone	3,3-dimethyl-2-butanol	1-phenylethanol	3,3-dimethyl-2-butanol	1-phenylethanol	3,3-dimethyl-2-butanol	1-phenylethanol
I	68	57	0.5	–2.1	6.5	4.9	*S*	*S*
III	74	66	–0.8	3.0	10.4	7.0	*R*	*R*
IVa	14	13	–0.5	–25.3	64.9	59.0	*R*	*S*
IVb	8	6	6.2	31.3	80.5	73.0	*S*	*R*
Va	11	10	–5.4	–27.0	70.1	62.9	*R*	*S*
Vb	8	7	6.9	–34.7	89.6	80.9	*S*	*S*
VIa	10	9	6.3	–32.2	81.8	75.0	*S*	*S*
VIb	5	4	–6.5	–34.3	84.4	79.9	*R*	*S*
VII	26	23	1.3	–5.1	16.9	11.9	*S*	*S*
VIII	18	16	2.1	–7.7	27.3	17.9	*S*	*S*
IX	20	18	–3.2	14.6	41.6	34.0	*R*	*R*
Xa	14	12	–4.2	20.2	54.5	47.1	*R*	*R*
Xb	10	9	4.8	23.6	62.3	55.0	*S*	*R*
XI	20	17	–1.5	7.3	19.5	17.0	*R*	*R*
XII	12	10	–2.5	10.7	32.5	24.9	*R*	*R*

[a] Ratio (in percent) of the specific rotation of the product to the specific rotation of the pure enantiomer; $[\alpha]_D^{20}$ values of the pure enantiomers of 3,3-dimethyl-2-butanol and 1-phenylethanol were assumed to be equal to ±7.7° [16] and ±42.9° [17], respectively.

diepoxy derivative **II** which reacted with *N*,*N'*-dibenzyl-1,2-ethanediamine or diamine **III** to afford the corresponding diaza crown ethers **IVa** and **Va**. By debenzylation of **IVa** and **Va** we obtained diaza crown ethers **IVb** and **Vb** having secondary amino groups. Deacetalization of **Va** yielded diaza crown ether **VIa**, and its subsequent debenzylation afforded compound **VIb**.

The catalytic activity and enantiodifferentiating properties of compounds **I** and **III**–**XII** were estimated in the reduction of pinacolone and acetophenone with sodium tetrahydridoborate in methylene chloride at 0°C. The amount of the catalyst was 10 mol % with respect to the substrate. The time necessary for complete conversion of the initial ketone was taken as a measure of catalytic activity. In all cases, the yields of 3,3-dimethyl-2-butanol and 1-phenylethanol were no less than 97% (according to the GLC data). In the absence of a catalyst, the yield of the alcohols did not exceed 1%; therefore, the contribution of noncatalytic process was neglected.

All the examined crown and aza crown ethers **IV**–**XII** turned out to be superior to acyclic podands **I** and **III** in the catalytic activity. This means that cyclic structure is the determining factor in the formation of soluble complexes with the reagent (see table). In the series of compounds **IV**–**XII**, the lowest catalytic activity was observed for crown ethers **VII** and **XI** and aza crown ether **IX** which contain no hydroxy groups. The presence of hydroxy groups in the macroring considerably increases the reaction rate. In this respect, illustrative are the data for compounds **VII** and **VIII**, **IX** and **Xa**, and **XI** and **XII**. Aza crown ethers are more efficient catalysts than nitrogen-free analogs. Crown ethers **IVb**, **Vb**, **VIb**, and **Xb** with secondary amino groups exhibit a higher catalytic activity than those having tertiary nitrogen atoms (**IVa**, **Va**, **VIa**, **Xa**). These data indicate participation of the hydroxy and amino groups in the reduction process. As shown previously, successful reduction of carbonyl compounds with sodium tetrahydridoborate in aprotic solvents in the presence of crown ethers requires catalysis by proton-donor compounds (e.g., water or alcohols) [14]. Presumably, a similar catalytic effect is produced by the hydroxy and amino groups present in the aza crown ether molecule. An alternative explanation for the observed acceleration of the reduction process in the presence of hydroxy or amino macrocyclic compounds may be change of the reagent nature as a result of the reaction of crown ether with tetrahydridoborate ion, which could lead to trihydroalkoxyborate or trihydroaminoborate ions.

It should be emphasized that the relation between the efficiency of asymmetric induction (optical yield) and structural parameters of the compounds under study is almost analogous to the relation observed for their catalytic activity: (1) asymmetric induction of cyclic compounds **IV**–**XII** is considerably greater than that observed for acyclic compounds **I** and **III**; (2) in the series of cyclic compounds, asymmetric induction caused by crown ethers **VII**, **VIII**, **XI**, and **XII** is considerably less effective than that typical of aza crown ethers **IV**–**VI**, **IX**, and **X**; (3) the presence of exocyclic hydroxy groups increases the enantioselectivity of the reaction (cf. **VII** and **VIII**; **IX** and **Xa**; **XI** and **XII**); (4) aza crown ethers **IVa**, **Va**, **VIa**, and **Xa** with tertiary amino groups give rise to a lower asymmetric induction than those having secondary amino groups (compounds **IVb**, **Vb**, **VIb**, and **Xb**; see table).

It may be seen that the catalytic activity and the magnitude of asymmetric induction for the examined compounds is determined by the same or similar factors. The most probable is formation of alkoxytrihydridoborate anions which are fixed in the close vicinity to the chiral barrier of the macroring or participation of hydroxy groups at asymmetric centers of the molecule in hydride ion transfer. Nitrogen atoms could stabilize the structure of intermediate complex or transition state [1].

Some of the examined diaza crown ethers having exocyclic hydroxy groups, e.g., compounds **IVb**, **Vb**, **VIa**, and **VIb**, showed a strong asymmetric induction in the reduction of ketones with sodium tetrahydridoborate; therefore, they are promising as catalysts for asymmetric synthesis of alcohols.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker AM-250 spectrometer (250 MHz) using $CDCl_3$ as solvent and HMDS as internal reference. The molecular weights were determined from the mass spectra which were obtained on Varian MAT-112 and MKh-1321 instruments with direct sample admission into the ion source (electron impact, 20–70 eV). The optical rotations ($[\alpha]_D^{20}$) were measured on a Perkin–Elmer 241MC polarimeter. GLC analysis was performed on a Chrom-5 chromatograph equipped with a flame-ionization detector; glass column, 1200 × 3 mm; stationary phase 5% of SE-30 on Inerton AW-DMCS (0.100–0.125 mm); carrier gas helium. Thin-layer chromatography was performed on Silufol plates (development with a solution of ninhydrin in diethyl ether) and glass plates with a fixed layer of aluminum oxide (L 5/40 μm, neutral; development with iodine vapor). Glass columns charged with silica gel (L 40/100 μm) or Al_2O_3 (L 40/250 μm, neutral) were used for preparative liquid chromatography.

(2*S*,3*S*)-1,4-Bis(benzyloxy)-2,3-bis(2,3-epoxypropyloxy)butane (II). A solution of 6.0 g (20 mmol) of (2*S*,3*S*)-1,4-bis(benzyloxy)-2,3-butanediol (**I**) [13] in 5.9 g (64 mmol) of 1-chloro-2,3-epoxypropane was added dropwise under vigorous stirring at 45°C to a mixture of 9.4 g (102 mmol) of 1-chloro-2,3-epoxypropane, 4.8 g (120 mmol) of sodium hydroxide, 0.5 g (28 mmol) of distilled water, and 0.14 g (0.4 mmol) of tetrabutylammonium hydrogen sulfate. The mixture was then stirred for 1.5 h at 40°C, cooled, and diluted with 25 ml of benzene, and the precipitate was filtered off and washed with benzene (2 × 5 ml). The filtrate was combined with the washings and dried over Na_2SO_4. The solvent and excess 1-chloro-2,3-epoxypropane were distilled off under reduced pressure, and the residue was subjected to column chromatography on silica gel (eluent hexane–acetone, 3 : 1) to isolate 5.6 g (68%) of the product as a colorless oily substance. $[\alpha]_D^{20}$ = +3.4°C (*c* = 2.5, CH_3OH). ^{1}H NMR spectrum, δ, ppm: 2.59–2.81 m (4H, CH_2, ring), 3.51–3.92 m (12H, CH_2, CH), 4.55 s (4H, C**H**$_2$Ph), 7.33 s (10H, Ph). Found, %: C 69.47; H 7.21. M^+ 414. $C_{24}H_{30}O_6$. Calculated, %: C 69.55; H 7.29. *M* 414.49.

***N*-Benzyl[(4*S*,5*S*)-5-benzylaminomethyl-2,2-dimethyl-1,3-dioxolan-4-yl]methanamine (III).** Diethyl (4*R*,5*R*)-2,2-dimethyl-1,3-dioxolane-4,5-dicarboxylate [15], 5.0 g (20 mmol), and benzylamine, 4.5 g (42 mmol), were added to a solution of 0.05 g (2.2 mmol) of metallic sodium in 20 ml of anhydrous methanol. The mixture was stirred for 3 h at room temperature and was left overnight. The mixture was then evaporated by half, and the precipitate was filtered off. An equal volume of anhydrous diethyl ether was added to the filtrate, and the precipitate was filtered off and recrystallized from 8 ml of anhydrous methanol to obtain 6.5 g (87%) of *N*,*N*′-dibenzyl-(4*R*,5*R*)-2,2-dimethyl-1,3-dioxolane-4,5-dicarboxamide. Colorless crystals, mp 167–168°C. $[\alpha]_D^{20}$ = +6.7° (*c* = 1.0, CH_3OH). ^{1}H NMR spectrum, δ, ppm: 1.32 s (6H, CH_3), 3.96 t (4H, C**H**$_2$Ph), 4.20–4.42 m (2H, CH), 7.11–7.43 m (12H, NH, Ph). Found, %: C 68.34; H 6.45; N 7.71. M^+ 368. $C_{21}H_{24}N_2O_4$. Calculated, %: C 68.46; H 6.57; N 7.60. *M* 368.43.

A 6.2-g (17-mmol) amount of *N*,*N*′-dibenzyl-(4*R*,5*R*)-2,2-dimethyl-1,3-dioxolane-4,5-dicarboxamide was added in small portions under vigorous stirring in an argon atmosphere to a suspension of 1.4 g (37 mmol) of lithium aluminum hydride in

25 ml of anhydrous THF. The mixture was stirred for 4 h, heated for 4 h under reflux, and left overnight. It was then cooled with ice, 1.4 ml of ice water was added, the precipitate was filtered off and was shaken with THF (2 × 10 ml), and the suspension was filtered. The filtrates were combined and dried over NaOH, the solvent was distilled off under reduced pressure, and the residue was subjected to column chromatography on silica gel (eluent chloroform–methanol, 10:1) to isolate 4.2 g (73%) of compound **III** as a colorless oily substance. $[\alpha]_D^{20}$ = +12.6° (*c* = 2.5, CH_3OH). ^1H NMR spectrum, δ, ppm: 1.30 s (6H, CH_3), 2.72–2.90 m (4H, NCH_2), 3.21 br.s (2H, NH), 3.90 t (4H, C**H**$_2$Ph), 4.10–4.22 m (2H, CH), 7.09 s (10H, Ph). Found, %: C 74.21; H 8.20; N 8.16. M^+ 340. $C_{21}H_{28}N_2O_2$. Calculated, %: C 74.08; H 8.29; N 8.23. *M* 340.46.

(2*S*,3*S*)-8,11-Dibenzyl-2,3-bis(benzyloxymethyl)-1,4-dioxa-8,11-diazacyclotetradecane-6,13-diol (IVa). A solution of 1.8 g (4.3 mmol) of compound **II** and 1.0 g (4.3 mmol) of *N*,*N*′-dibenzyl-1,2-ethanediamine in a mixture of 22 ml of THF and 22 ml of ethanol was heated for 8 h under reflux. The solvents were distilled off under reduced pressure, and the residue was subjected to column chromatography on neutral Al_2O_3 (eluent chloroform–benzene–2-propanol–methanol, 8:3:0.3:0.3) to isolate 1.9 g (68%) of compound **IVa** as a colorless oily substance. $[\alpha]_D^{20}$ = +12.2° (*c* = 1.0, CH_3OH). ^1H NMR spectrum, δ, ppm: 2.18–2.82 m (8H, NCH_2), 3.08–3.85 m (18H, OCH_2, OCH, NC**H**$_2$Ph, OH), 4.36 s (4H, OC**H**$_2$Ph), 7.03–7.29 m (20H, Ph). Found, %: C 73.25; H 7.59; N 4.20. M^+ 654. $C_{40}H_{50}N_2O_6$. Calculated, %: C 73.37; H 7.70; N 4.28. *M* 654.84.

(2*S*,3*S*)-2,3-Bis(hydroxymethyl)-1,4-dioxa-8,11-diazacyclotetradecane-6,13-diol (IVb). A stream of hydrogen was passed over a period of 2 h through a suspension of 225 mg of 10% Pd/C in 15 ml of anhydrous methanol. Crown ether **IVa**, 1.2 g (1.8 mmol), was then added, and the mixture was stirred for 16 h in a stream of hydrogen. The catalyst was filtered and washed with methanol (2 × 5 ml), and the solvent (methanol and toluene formed in the reaction) was distilled off under reduced pressure. Diaza crown ether **IVb** was isolated as a colorless oily substance. Yield 523 mg (97%). $[\alpha]_D^{20}$ = +7.3° (*c* = 1.0, CH_3OH). ^1H NMR spectrum, δ, ppm: 2.25–2.98 m (10H, NCH_2, NH), 3.18–3.90 m (16H, CH_2, OCH, OH). Found, %: C 49.15; H 8.79; N 9.43. M^+ 294. $C_{12}H_{26}N_2O_6$. Calculated, %: C 48.97; H 8.90; N 9.52. *M* 294.34.

(3a*S*,10*S*,11*S*,17a*S*)-5,16-Dibenzyl-10,11-bis(benzyloxymethyl)-2,2-dimethylperhydro[1,3]dioxolo[4,5-*j*][1,4,8,13]dioxadiazacyclohexadecane-7,14-diol (Va) was synthesized as described above for diaza crown ether **IVa** from 3.8 g (9.2 mmol) of compound **II** and 3.1 g (9.2 mmol) of diamine **III** in a mixture of 46 ml of THF and 46 ml of ethanol. Yield 4.9 g (71%). Colorless oily substance. $[\alpha]_D^{20}$ = +19.6° (*c* = 1.0, CH_3OH). ^1H NMR spectrum, δ, ppm: 1.25 s (6H, CH_3), 2.32–2.85 m (8H, NCH_2), 3.24–4.10 m (20H, OCH_2, OCH, NC**H**$_2$Ph), OH), 4.40 s (4H, OC**H**$_2$Ph), 7.10–7.32 m (20H, Ph). Found, %: C 71.42; H 7.89; N 3.65. M^+ 754. $C_{45}H_{58}N_2O_8$. Calculated, %: C 71.59; H 7.74; N 3.71. *M* 754.95.

(3a*S*,10*S*,11*S*,17a*S*)-10,11-Bis(hydroxymethyl)-2,2-dimethylperhydro[1,3]dioxolo[4,5-*j*][1,4,8,13]dioxadiazacyclohexadecane-7,14-diol (Vb) was synthesized as described above for diaza crown ether **IVb** by hydrogenation of 0.9 g (1.2 mmol) of compound **Va** in 10 ml of anhydrous methanol in the presence of 150 mg of 10% Pd/C. Yield 461 mg (98%). Colorless oily substance. $[\alpha]_D^{20}$ = +11.7° (*c* = 1.0, CH_3OH). ^1H NMR spectrum, δ, ppm: 1.28 s (6H, CH_3), 2.20–2.92 m (10H, NCH_2, NH), 3.15–3.96 m (18H, OCH_2, OCH, OH). Found, %: C 51.59; H 8.52; N 7.18. M^+ 394. $C_{17}H_{34}N_2O_8$. Calculated, %: C 51.76; H 8.69; N 7.10. *M* 394.46.

(2*S*,3*S*,10*S*,11*S*)-8,13-Dibenzyl-2,3-bis(benzyloxymethyl)-1,4-dioxa-8,13-diazacyclohexadecane-6,10,11,15-tetraol (VIa). Methanol and acetone were slowly distilled off (over a period of 3 h) from a solution of 3.0 g (4 mmol) of acetal **Va** in a mixture of 4.5 ml of 2 N hydrochloric acid and 20 ml of methanol. The mixture was cooled, 3.8 ml of a 20% aqueous solution of sodium hydrogen carbonate was added, and the mixture was throroughly stirred and extracted with benzene (5 × 10 ml). The extract was dried over Na_2SO_4, and the solvent was distilled off under reduced pressure to obtain almost pure compound **VIa** as a colorless oily substance. Yield 2.5 g (88%). $[\alpha]_D^{20}$ = +13.4° (*c* = 1.0, CH_3OH). ^1H NMR spectrum, δ, ppm: 2.24–2.88 m (8H, NCH_2), 3.28–4.03 m (22H, OCH_2, OCH, NC**H**$_2$Ph, OH), 4.37 s (4H, OC**H**$_2$Ph), 7.08–7.27 m (20H, Ph). Found, %: C 70.68; H 7.74; N 3.79. M^+ 714. $C_{42}H_{54}N_2O_8$. Calculated, %: C 70.56; H 7.61; N 3.92. *M* 714.89.

(2*S*,3*S*,10*S*,11*S*)-2,3-Bis(hydroxymethyl)-1,4-dioxa-8,13-diazacyclohexadecane-6,10,11,15-tetraol (VIb) was synthesized as described above for diaza crown ether **IVb** by hydrogenation of 1.7 g (2.4 mmol) of compound **VIa** in 15 ml of anhydrous methanol in the presence of 300 mg of 10% Pd/C. Yield 809 mg (96%). Colorless oily substance. $[\alpha]_D^{20}$ = +22.5° (*c* = 1.0, CH_3OH). ^1H NMR spectrum, δ, ppm:

2.15–2.79 m (10H, NCH_2, NH), 3.10–3.97 m (20H, OCH_2, OCH, OH). Found, %: C 47.34; H 8.41; N 7.99. M^+ 354. $C_{14}H_{30}N_2O_8$. Calculated, %: C 47.45; H 8.53; N 7.90. M 354.40.

Asymmetric reduction. A mixture of 0.5 mmol of appropriate ligand and 0.38 g (10 mmol) of sodium tetrahydridoborate in 50 ml of methylene chloride was cooled to 0°C under stirring, and a solution of 0.5 g (5 mmol) of pinacolone or 0.6 g (5 mmol) of acetophenone in 5 ml of methylene chloride was added. The mixture was stirred at 0°C until the initial ketone disappeared completely (GLC), unreacted sodium tetrahydridoborate was filtered off and washed with methylene chloride (2×5 ml), and the filtrate was combined with the washings, washed with water until neutral reaction, and dried over Na_2SO_4. The solvent was distilled off under atmospheric pressure, and the residue was subjected to column chromatography on silica gel using pentane–diethyl ether (5:2) as eluent to isolate enantiomeric alcohol mixture; its specific rotation $[\alpha]_D^{20}$ was measured (see table).

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