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Synthesis and Molecular Structure of Bis(benzo)aza-14-crown-4 Ethers with 7-Azabicyclo[3.3.1]nonane and Homologous Fragments

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Abstract—By condensation of cycloalkanes with 2-{2-[2-(2-formylphenoxy)ethoxy]ethoxy}benzaldehyde and ammonium acetate by Petrenko–Kritchenko method first representatives of new heterocyclic systems, bis(benzo)-aza-14-crown-4 ethers containing a 7-aza-bicyclo[3.3.1]nonane or its homologous fragment, were obtained in 6–15% yield. With growing cycle of the initial cycloketone the yield of the azacrownophane considerably grew. By means of XRD analysis the molecular structure was established of four synthesized macrocycles, the relative configuration of asymmetrical atoms was determined, and the size of the internal cavities of the azacrown part was estimated.

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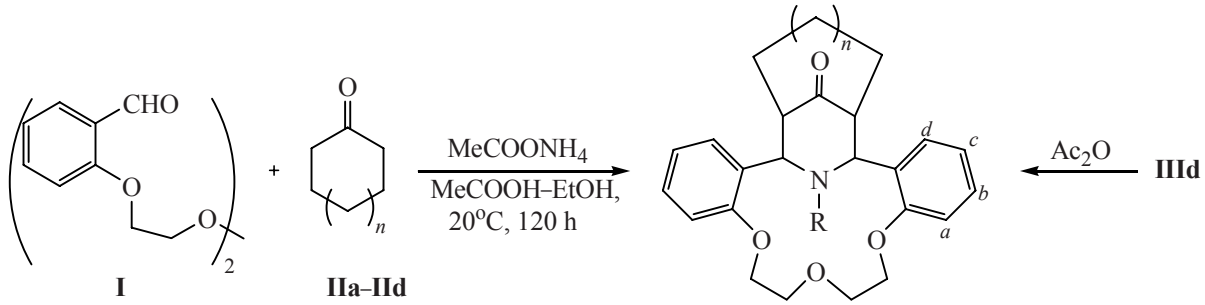
Successful syntheses of first dibenzopiperidinoaza-crown ethers by the condensation of dialkyl ketones with 2-{2-[2-(2-formylphenoxy)ethoxy]ethoxy}benzaldehyde and ammonium acetate by the method of Petrenko–Kritchenko were described in [1]. Thus obtained aza-crownophanes contained in their piperidone fragment C-methyl, C-phenyl, or C-alkoxycarbonyl substituents. In keeping with the internet program PASS they may be used as the object of studies for inhibitors of a series of enzymes [2]. Taking into consideration that the replacement of the above cited substituents by cycloalkane moieties would improve the interaction of the potential drug with the cell bioreceptors [3] we planned to check the possibility to synthesize new groups of bis(benzo)aza-14-crown-4 ethers containing in their structure 7-aza-bicyclo[3.3.1]nonane or its homologous fragments.

To this end we similarly applied in this study the Petrenko–Kritchenko method introducing into the condensation with dialdehyde **I** and ammonium acetate cycloalkanones **IIa–IId**. The reaction was carried out in a mixture of ethanol with acetic acid at 20°C for 5 days at a slight dilution. Target compounds **IIIa–IIId** were



I **IIa–IId** **IIIa–IIId, IV**

III, R = H, *n* = 1 (**a**, 6%), 2 (**b**, 7%), 3 (**c**, 9%), 9 (**d**, 15%); R = COMe, *n* = 9 (**IV**, 74%).

isolated by column chromatography in 6–15% yield with respect to the taken dialdehyde **I**.

With the growing ring in initial ketone **IIα–IId** the yield of diazacrownophanes **IIIα–IIId** increased. By treating with acetic anhydride azacrown **IIId** we obtained in good yield its N-acetyl derivative **IV**.

The analysis of ^{1}H NMR spectra of azacrownophanes **IIIa–IIId** showed that the methylene protons of the cycloalkane fragment gave rise to three groups of multiplets located most upfield (from 1.0 to 2.1 ppm). Methylene protons of the polyether bridge appeared as a group of multiplets in the region usual for similar crown ethers (from 3.68 to 4.1 ppm) [4]. The signals of α- and β-protons of the piperidine rings of azacrownophanes **IIIa–IIId** were shifted downfield (δ 4.12–4.26 and 2.88–3.36 ppm respectively) due to deshielding with the crown ether fragment (it had been formerly found that all analogous protons in 2,6-diarylpiperidin-4-ones resonated in the region 2.56–2.76 ppm) [5]. This effect was apparently caused by the magnetic field of the phenoxy moieties of the oligoether bridge as was also observed in the other azacrown ethers containing a piperidine ring [1]. The assignment of signals from the aromatic protons in the spectra of azacrownophanes **IIIa–IIId** was no [problem, in all events four signals appeared of integral intensity 2H each testifying to the symmetric location of two fused benzene rings. Protons H^{α} and H^{c} (the notation of aromatic protons is given on the Scheme) are subjected to the shielding effect of an oxygen and appear upfield as a doublet (δ 6.26–6.96 ppm) and a triplet (δ 6.36–6.86 ppm) respectively. The other protons H^{b} and H^{d} give signals in the region usual for aromatic protons (δ 6.82–7.26 ppm) as triplets and doublets respectively.

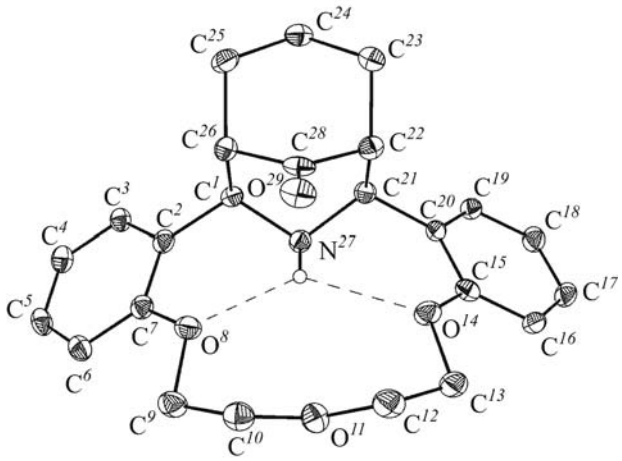


Fig. 1. Molecular structure of compound **IIIa** shown with ellipsoids of anisotropic displacements (probability 40%); only the hydrogen of amino group is shown involved into an intramolecular hydrogen bond; the hydrogen bonds are shown by dash at lines.

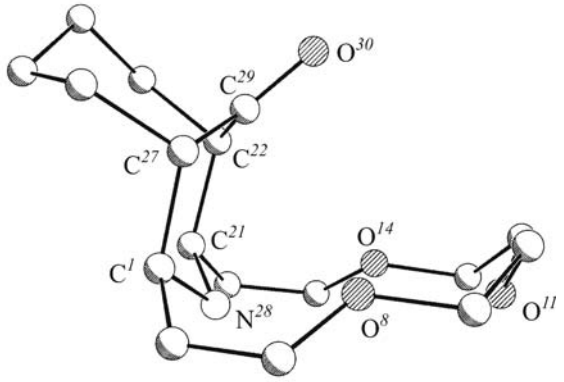


Fig. 2. Conformation of bicyclic and crown ether fragments in compound **IIIb**.

Aiming at unambiguous proof of the structure of compounds synthesized, at establishment of their stereochemical characteristic, and at the estimation of the size of the intramolecular cavity in the azacrown ether part we subjected aza-14-crown-4 **IIIa–IIId** to XRD analysis. The obtained molecular structures of compounds obtained are presented on Figs. 1–4.

Compounds **IIIa–IIId** are constituted of two main parts: a 14-membered azacrown ether cycle containing three oxygen atoms, and a bicycle composed of a tetrahydropyridine and a cyclohexanone (**IIIa**) or its homologs cycloheptanone (**IIIb**), cyclooctanone (**IIIc**), and cyclododecanone (**IIId**) fragments.

The forteen-membered azacrown ether rings in all compounds investigated have nearly the same conformation. The sizes of the internal cavities of the crown ethers estimated as a double average distance between the *n*-electron-donor atoms and their centroid (where the centroid is the center of the tetragon $NO^{8}O^{11}O^{14}$) equal 4.02, 4.01, 3.97, and 4.04 Å respectively. The conformations of the polyether fragments are $C^{7}O^{8}C^{9}C^{10}O^{11}C^{12}C^{13}O^{14}C^{15}$ *t*-$g^{(-)}$-*t*-*t*-$g^{(+)}$-*t* (*t* is transoid, ±180°; *g* is gauche, ±60°). This structure of the crown rings in compounds **IIIa–IIId** was determined in particular by nearly symmetric bifurcate intramolecular hydrogen bonds N–H···O^{8} and N–H···O^{14} (Table 1). Owing to these intramolecular hydrogen bonds the donor atoms of the crown rings N, O^{8}, O^{11}, and H^{14} are not located in one plane (the mean square deviations of these atoms from the plane are 0.158, 0.147, 0.142, and 0.104 Å respectively). The angles between the planes of the benzene rings of

the molecules equal 58.4, 66.2, 55.9, and 59.8° respectively.

The conformation of the bicyclic fragments in compounds **IIIa–IIId** possesses an interesting feature: with the growing length of the polymethylene bridge of the cycloalkanone fragment the tetrahydropyridine ring alters its conformation from a slightly distorted *boat* [in compound **IIIa** atoms C^{28} and N^{27} deviate from the plane of the other atoms of the ring by –0.677 and –0.659 Årespectively (Fig. 1); in compound **IIIb** atoms C^{29} and N^{28} deviate from the plane of the other atoms of the ring by 0.473 and 0.678 Å, respectively (Fig. 2)] to a nearly ideal *chair* [in compound **IIId** atoms C^{34} and N^{33} deviate from the plane of the other atoms of the ring by –0.738 and 0.627 Å, respectively (Fig. 4)], therewith going through a *semiboat* conformation [in compound **IIIc** atoms C^{30} and N^{29} deviate from the plane of the other atoms of the ring by 0.225 and 0.712 Å, respectively (Fig. 3)]. The polymethylene bridges have here the following conformations $g^{(-)}$-$cl^{(+)}$-$g^{(-)}$ (σl is *clinal*, ±90°) (compounds **IIIa** and **IIIb**), $g^{(+)}$-$cl^{(-)}$-$cl^{(+)}$-$g^{(-)}$ (compound **IIIc**), and $g^{(+)}$-$cl^{(-)}$-$g^{(-)}$-t-$g^{(-)}$-$g^{(-)}$-t-$g^{(-)}$ (compound **IIId**). It is presumable that at intermediate dimensions of the cycloalkane fragment the tetrahydropyridine ring would take also conformations of sofa and semichair.

Compounds **IIIa–IIId** are diastereomers with four asymmetric centers: C^{1}, C^{21}, C^{22}, and C^{26} (compounds **IIIa** and **IIIb**); C^{1}, C^{21}, C^{22}, and C^{28} (compound **IIIc**); C^{1}, C^{21}, C^{22}, and C^{32} (compound **IIId**). All azacrownophanes **IIIa–IIId** are racemates with the identical relative configuration of these centers: *rac*-[1*R**, 21*S**, 22*R**, 26(27, 28, 32)*S**]. The cyclohexane, cycloheptane, and cycldodecane fragments of compounds **IIIa, IIIb**, and **IIId** are fused to the piperidine ring by the 1,3-*cis*-diequatorial junction, and in compound **IIIc** the cyclooctanone fragment is joined in 1,3-*cis*-pseudoequatorial mode. The analogous type of the junction to the piperidine ring is observed in the crown ether bridges of all the four azacrownophanes. Evidently the relative configuration of the chiral atoms and the junction type of the cycloalkane and crown ether fragments to the piperidine ring is governed by the method of the synthesis of compounds under investigation that possesses a high selectivity.

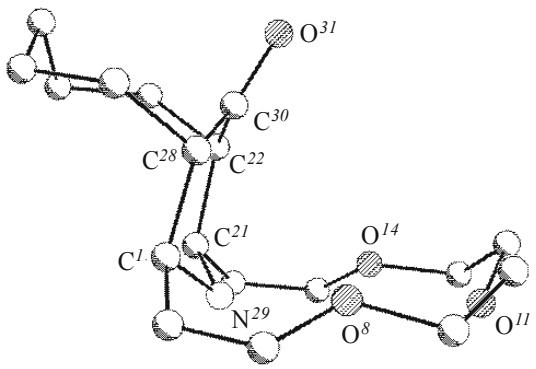


Fig. 3. Conformation of bicyclic and crown ether fragments in compound **IIIc**.

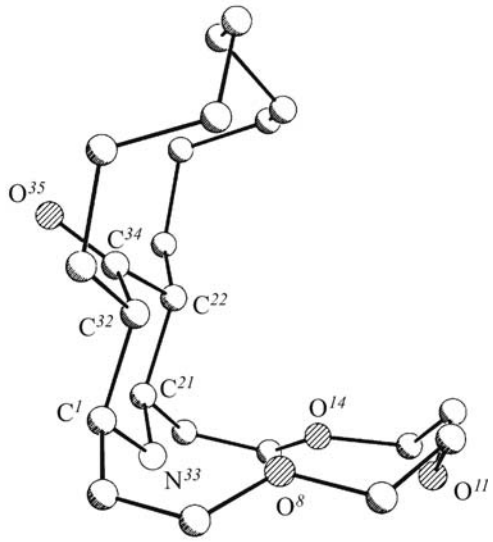


Fig. 4. Conformation of bicyclic and crown ether fragments in compound **IIId**.

Table 1. Parameters of intramolecular hydrogen bonds in azacrownophanes **IIIa–IIId**

Compound no.	Fragment	*d*(N–H), Å	*d*(H···O), Å	*d*(N···O), Å	ω(N–H···O), deg
IIIa	N^{27}–H^{27N}···O^{8}	0.88	2.33	2.931(3)	126
	N^{27}–H^{27N}···O^{14}	0.88	2.48	2.992(3)	117
IIIb	N^{28}–H^{28N}···O^{8}	0.86	2.44	2.961(2)	120
	N^{28}–H^{28N}···O^{14}	0.86	2.41	2.950(2)	121
IIIc	N^{29}–H^{29N}···O^{8}	0.92	2.42	2.962(2)	117
	N^{29}–H^{29N}···O^{14}	0.92	2.31	2.880(2)	120
IIId	N^{33}–H^{33N}···O^{8}	0.89	2.49	3.060(1)	122
	N^{33}–H^{33N}···O^{14}	0.89	2.28	2.868(1)	124

Table 2. Main crystallographic data and refinement parameters for azacrownophanes **IIIa–IIId**

Compound no.	**IIIa**	**IIIb**	**IIIc**	**IIId**
Empirical formula	$C_{24}H_{27}NO_4$	$C_{25}H_{29}NO_4$	$C_{26}H_{21}NO_4$	$C_{30}H_{39}NO_4$
M	393.47	407.49	421.52	477.62
T, K	120	100	100	100
Crystal system	monoclinic	rhombic	monoclinic	monoclinic
Space group	$P2_1/c$	*Pbca*	$P2_1/n$	$P2_1/c$
a, Å	17.691(3)	7.7423(8)	10.0529(7)	11.6532(5)
b, Å	7.1368(13)	17.4337(17)	11.6510(8)	7.2690(4)
c, Å	17.264(3)	30.538(3)	18.6481(13)	29.768(2)
α, deg	90	90	90	90
β, deg	117.104(3)	90	93.714(5)	96.005(5)
γ, deg	90	90	90	90
V, Å^3	1940.4(6)	4121.9(7)	2179.6(3)	2507.7(2)
Z	4	8	4	4
d_c, g cm^{-3}	1.347	1.313	1.285	1.265
F(000)	840	1744	904	1032
μ, mm^{-1}	0.091	0.088	0.086	0.083
$2\theta_{max.}$, deg	52	56	58	54
Number of measured reflections	15051	44288	20934	25667
Number of independent reflections	2505	4915	5583	5431
Number of observed reflections with	3536	2747	4105	4652
$I>2\sigma(I)$	188	275	280	316
Number of refined parameters	0.0793	0.0562	0.0478	0.0363
$R_1[I>2\sigma(I)]$	0.1783	0.1056	0.1150	0.0922
wR_2 (all data)	1.005	1.000	1.005	1.001
GOF				

Thus we prepared first representatives of the new class of dibenzoazacrownophanes joined to cycloalkane fragments, and established their tree-dimensional molecular structures.

EXPERIMENTAL

^{1}H NMR spectra were registered on a spectrometer Bruker WP-400 at the operating frequency 400 MHz from solutions in $CDCl_3$. Mass spectra were measured on a mass spectrometer Finnigan MAT INCOS-50 with a direct sample admission into the ion source, ionizing electrons energy 70 eV. IR spectra were recorded in KBr matrix on a spectrophotometer Specord 75IR. The preparative column chromatography was carried out on aluminum oxide (100/160). The completion of reactions and the purity of compounds synthesized were checked by TLC (Silufol UV-254), eluent ethyl acetate. Ketones **IIa** and **IIb** were commercial products, dialdehyde **I** was obtained as described in [1, 6].

X-ray diffraction analysis of compounds IIIa–IIId. The parameters of unit cells and the intensities of reflections were measured on an automatic three-circle diffractometer Bruker X8-APEX-II CCD equipped with a low-temperature probe KryoFlex (*T* 100 K, λMoK_α-radiation, graphite monochromator, φ and ω-scanning). The main crystallographic data and refinement parameters are compiled in Table 2. The structures of all compounds were solved by the direct method and refined by the full-matrix least-squares method in the anisotropic approximation of nonhydrogen atoms. Hydrogen atoms of amino groups were localized objectively from the difference Fourier synthesis and were included into the refinement in isotropic approximation with fixed position and thermal [U_{iso}(H) = 1.2U_{eq}(N)] parameters. The positions of other hydrogen atoms were calculated from geometry and refined in the isotropic approximation with fixed position (*rider* model) and thermal [U_{iso}(H) = 1.2U_{eq}(C)] parameters. All calculations were performed using program package SHELXTL PLUS (Version 6.12) [7]. The tables of atomic coordinates, bond lengths, bond angles, and anisotropic thermal parameters of compounds **IIIa–IIId** are deposited into Cambridge Structural Database.

Azacrownophanes IIIa–IIId. A solution of 7.0 g (22 mmol) of dialdehyde **I**, 22 mmol of cycloalkanone **IIa–IId**, and 3.4 g (44 mmol) of ammonium acetate in a mixture of 50 ml of EtOH and 2 ml MeCOOH was kept for 5 days at 20°C. The solvent was evaporated in a vacuum. To the residue 50 ml of saturated sodium carbonate solution was added, and the reaction product was extracted into chloroform (3 × 70 ml). The combined extracts were dried with anhydrous $MgSO_4$ and evaporated in a vacuum. The residue was subjected to column chromatography on aluminum oxide eluting with a mixture ethyl acetate–hexane, 1:1. The isolated fraction was purified by recrystallization from ethanol to obtain the reaction product as colorless crystals.

8,11,14-Trioxa-27-azapentacyclo-[19.5.1.1^{22,26}.0^{2,7}.0^{15,20}]-octacosa-2,4,6,15(20),16,18-hexaen-28-one (IIIa). Yield 0.54 g (6%), mp 236–237°C, R_f 0.37 (ethyl acetate). IR spectrum, ν, cm^{-1}: 3298 (NH), 1720 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.64 m (1H, H_{aliph}), 1.96 m (3H, H_{aliph}), 2.07 m (2H, H_{aliph}), 2.88 br.s (2H, HCC=O), 4.24 br.d (2H, HC–N, *J* 11.5 Hz), 3.96–4.10 m (8H, OCH_2CH_2O), 3.46 br.s (1H, NH), 6.78 d (2H, H$^{\alpha}$, *J* 8.0 Hz), 7.21 t (2H, H^{b}, *J* 8.0 and 7.4 Hz), 6.84 t (2H, H^{c}, *J* 8.0 and 7.4 Hz), 7.26 d (2H, H^{d}, *J* 8.0 Hz). Mass spectrum, *m/z* (I_{rel}, %): 393 [*M*]$^+$ (100), 392 (21), 365 (14), 339 (16), 297 (28), 296 (55), 131 (18), 121 (13), 91 (10), 77 (7). Found, %: C 73.21; H 6.80; N 3.42. $C_{24}H_{27}NO_4$. Calculated, %: C 73.28; H 6.87; N 3.56.

8,11,14-Trioxa-27-azapentacyclo-[19.6.1.1^{22,27}.0^{2,7}.0^{15,20}]nonacosa-2,4,6,15(20),16,18-hexaen-29-one (IIIb). Yield 0.65 g (7%), mp 262–263°C, R_f 0.32 (ethyl acetate). IR spectrum, ν, cm^{-1}: 3313 (NH), 1697 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.52–1.66 m (4H, H_{aliph}), 1.77 m (2H, H_{aliph}), 2.03 m (2H, H_{aliph}), 3.13 t (2H, HCC=O, *J* 7.5 Hz), 4.12 t (2H, HC–N, *J* 7.5 Hz), 3.83 m (3H, OCH_2CH_2O), 3.95–4.05 m (5H, OCH_2CH_2O), 3.95–4.05 m (1H, NH), 6.76 d (2H, H$^{\alpha}$, *J* 8.0 Hz), 7.21 t.t (2H, H^{b}, *J* 8.0 and 7.54 Hz), 6.86 t.t (2H, H^{c}, *J* 8.0 and 7.44 Hz), 7.25 d (2H, H^{d}, *J* 7.5 Hz). Mass spectrum, *m/z* (I_{rel}, %): 407 [*M*]$^+$ (72), 379 (4), 352 (9), 338 (10), 297 (31), 148 (46), 134 (64), 131 (92), 121 (81), 119 (58), 107 (58), 91 (89), 77 (95), 55 (100). Found, %: C 73.51; H 7.35; N 3.59. $C_{25}H_{29}NO_4$. Calculated, %: C 73.69; H 7.17; N 3.44.

8,11,14-Trioxa-29-azapentacyclo-[19.7.1.1^{22,28}.0^{2,7}.0^{15,20}]triaconta-2,4,6,15(20),16,18-hexaen-30-one (IIIc). Yield 0.85 g (9%), mp 250–251°C, R_f 0.2 (ethyl acetate). IR spectrum, ν, cm^{-1}: 3309 (NH), 1686 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.72 m (2H, H_{aliph}), 1.80–1.96 m (6H, H_{aliph}), 2.10 m (2H, H_{aliph}), 2.98 m (2H, HCC=O), 4.26 br.t (2H, HC–N, *J* 11.3 Hz), 3.74 t.t (2H, OCH_2CH_2O, *J* 10.5 Hz), 3.95–4.05 m (6H, OCH_2CH_2O), 3.92 br.s (2H, NH), 6.78 d (2H, H$^{\alpha}$, *J* 8.0 Hz), 7.18 t.t (2H, H^{b}, *J* 8.0 and 7.3 Hz), 6.85 t (2H, H^{c}, *J* 7.8 and 7.3 Hz), 7.22 d (2H, H^{d}, *J* 7.8 Hz). Mass spectrum, *m/z* (I_{rel}, %): 421 [*M*]$^+$ (47), 393 (3), 366 (14), 339 (7), 310 (79), 297 (12), 148 (38), 131 (62), 121 (61), 107 (45), 91 (75), 77 (79), 55 (100). Found, %: C 74.12; H 7.52; N 3.18. $C_{26}H_{31}NO_4$. Calculated, %: C 74.08; H 7.41; N 3.32.

8,11,14-Trioxa-33-azapentacyclo-[19.11.1.1^{22,32}.0^{2,7}0^{15,20}]tetratriaconta-2,4,6,15(20),16,18-hexaen-34-one (IIId). Yield 1.6 g (15%), mp 228–229°C, R_f 0.49 (ethyl acetate). IR spectrum, ν, cm^{-1}: 3325 (NH), 1703 (C=O). ^{1}H NMR spectrum, δ, ppm: 1.06 m (4H, H_{aliph}), 1.15 m (10H, H_{aliph}), 1.57 m (4H, H_{aliph}), 3.36 m (2H, HCC=O), 4.13 m (2H, HC–N), 3.58 m (2H, OCH_2CH_2O), 3.92 m (2H, OCH_2CH_2O), 4.13 m (4H, OCH_2CH_2O), 3.30 br.s (1H, NH), 6.96 d (2H, H$^{\alpha}$, *J* 8.35 Hz), 7.23 t (2H, H^{b}, *J* 8.4 and 7.7 Hz), 6.83 t (2H, H^{c}, *J* 7.7 and 7.4 Hz), 7.07 d (2H, H^{d}, *J* 7.4 Hz). Mass spectrum, *m/z* (I_{rel}, %): 477 [*M*]$^+$ (10), 449 (3), 310 (19), 297 (100), 148 (12), 134 (21), 131 (23), 121 (20), 119 (18), 107 (17), 91 (28), 77 (19), 55 (58). Found, %: C 75.31; H 8.31; N 2.90. $C_{30}H_{39}NO_4$. Calculated, %: C 75.44; H 8.23; N 2.93.

25-Acetyl-8,11,14-trioxa-33-azapentacyclo-[19.11.1.1^{22,32}.0^{2,7}0^{15,20}]tetratriaconta-2,4,6,15(20),16,18-hexaen-34-one (IV). A solution of 0.5 g (1.05 mmol) of azacrownophane **IIId** in 3 ml of acetic anhydride was boiled for 3 h. The excess acetic anhydride was evaporated in a vacuum. To the residue 10 ml of saturated sodium carbonate solution was added, and the reaction product was extracted into chloroform (3 × 30 ml). The combined extracts were dried with anhydrous $MgSO_4$ and evaporated in a vacuum. The residue was recrystallized from ethanol. Yield 0.4 g (74%), colorless crystals, mp 245–246°C, R_f 0.21 (ethyl acetate). IR spectrum, ν, cm^{-1}: 1692 (C=O), 1649 (NC=O). ^{1}H NMR spectrum, δ, ppm: 1.24–2.13 m (18H, H_{aliph}), 2.68 s (3H, CH_3), 3.08 m (1H, HCC=O), 3.24 m (1H, HCC=O), 5.06 br.s (1H, HC–N), 6.63 br.s (1H, HC–N), 3.55 m (2H, OCH_2CH_2O), 3.79 m (2H, OCH_2CH_2O), 4.02 m (2H, OCH_2CH_2O), 4.19 m (2H, OCH_2CH_2O), 6.26 d (1H, H$^{\alpha}$, *J* 7.94 Hz), 6.42 d (1H, H$^{\alpha}$, *J* 7.93 Hz), 6.80 m (2H, H^{b}), 6.76 t (1H, H^{c}, *J* 7.4 and 7.3 Hz), 6.51 t (1H, H^{c}, *J* 7.4 Hz), 6.82 d (1H, H^{d},

J 7.3 Hz), 6.98 d (1H, H^d, *J* 7.3 Hz). Mass spectrum, *m/z* (I_{rel}, %): 519 $[M]^+$ (9), 491 (12), 476 (5), 449 (9), 448 (13), 151 (20), 149 (5), 134 (20), 133 (21),131 (27), 121 (12), 119 (11), 107 (21), 91 (26), 55 (39), 44 (52), 43 (100). Found, %: C 74.02; H 8.09; N 2.65. $C_{32}H_{41}NO_5$. Calculated, %: C 73.96; H 7.95; N 2.70.

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