

Synthesis of Isomeric Dinitro and Diamino Derivatives of Polycyclic Crown Ethers: Dibenzo-18-crown-6 and Dibenzo-24-crown-8

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Abstract—Specific features of the synthesis of polycyclic crown ethers dibenzo-18-crown-6 and dibenzo-24-crown-8 and their dinitro and diamino derivatives have been studied. A mixture of isomers of dibenzocrown ether derivatives was obtained and separated. The spectral and thermal characteristics of the synthesized compounds and the kinetics of synthesis of dibenzo-24-crown-8 by the two-component condensation of pyrocatechol with 1-chloro-2-[2-(2-chloroethoxy)ethoxy]ethane in an alcoholic medium in the presence of a KOH template agent were studied.

Keywords: crown ether derivatives, regioisomers, dibenzo-18-crown-6, dibenzo-24-crown-8, separation of isomers

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Functional derivatives of dibenzocrown ethers are promising candidates for application as building blocks in supramolecular systems and polymer materials. The known methods of synthesis of these derivatives provide mixtures of isomers with unknown component concentrations, and, therefore, research into the separation of such mixtures is an urgent issue.

Amino and diamino derivatives of crown ethers can be further functionalized. They can be immobilized on various organic and inorganic substrates and then used to construct nitrogen-containing structures, in particular, polymers of diaminocrown ethers [1–7]. Polyamide 18-crown-6 was used in a reverse osmosis membrane, and polymers including crown ethers can be used as proton-conducting cation exchange membranes for proton-driven cation transport [6]. Macrocyclic ligands are introduced into membranes for metal separation and ion-selective electrodes [6].

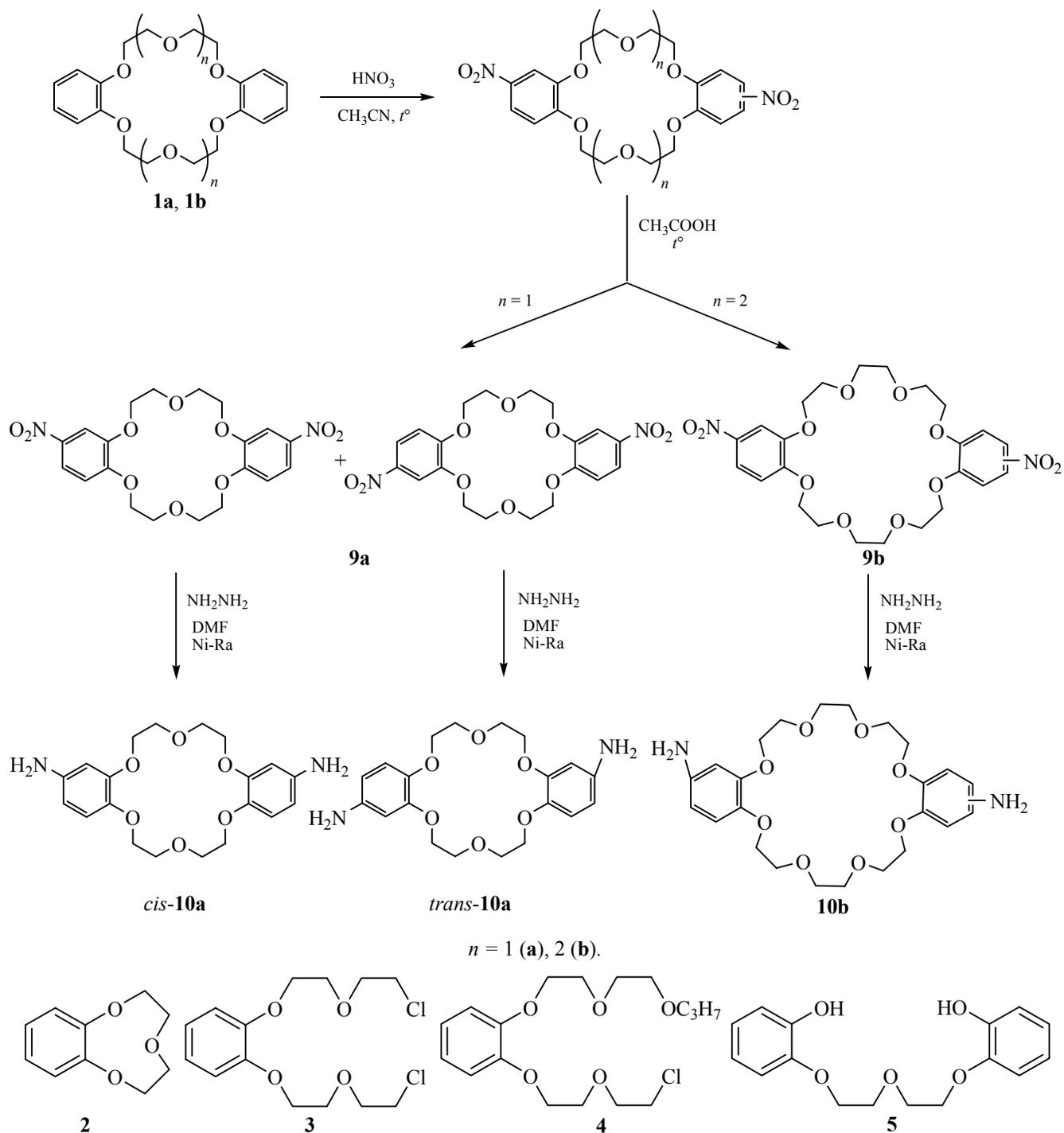
We performed research into the synthesis of regioisomers of diaminodibenzo-18-crown-6 and diamino-dibenzo-24-crown-8 from the presynthesized starting compounds: dibenzocrown ethers and dinitrocrown ethers.

The methods of synthesis of functionally substituted aromatic crown ethers can be divided into two main groups: syntheses starting from dihydric phenols containing other functional groups and syntheses involving functionalization of aromatic rings. We chose the second synthetic approach, taking into account that it makes use of usual reactions to introduce substituents into the aromatic ring, and the positions of the substituents are controlled by common orientation rules (Scheme 1).

We previously studied the synthesis of dibenzo-18-crown-6 **1a** [8]. According to GLC data, the reaction was complete in 5–5.5 h. Along with compound **1a**, the postreaction mixture contained admixtures of benzo-9-crown-3 **2** and 1,2-bis[2-(2-chloroethoxy)ethoxy]benzene **3** and its reaction product with isopropanol 1-[2-(2-chloroethoxy)ethoxy]-2-[2-(2-propoxyethoxy)ethoxy]benzene **4**, as well as semicrown diol **5**.

To ensure selective cyclization to crown ethers and prevent formation of linear polymers the reactions were performed in highly diluted solutions, where intramolecular cyclization to form a macrocyclic product is more probable and occurs faster than

Scheme 1.



polymerization which requires the two reagents to collide with each other [9, 10].

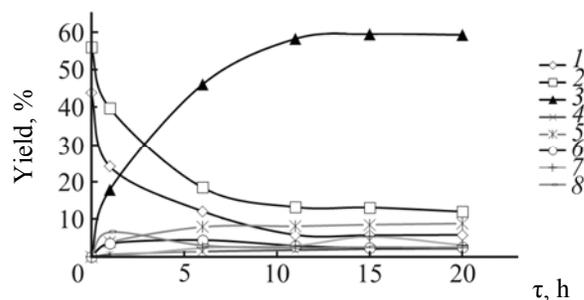
We studied the kinetics of the synthesis of dibenzo-24-crown-8 **1b** by the two-component condensation of pyrocatechol **6** with 1-chloro-2-[2-(2-chloroethoxy)-

ethoxy]ethane **7** in an alcoholic medium in the presence of KOH as a template agent. The reaction progress was followed and the molar concentrations of compound **1b** were determined by GLC. It was found that the reaction mixtures contained, along with compound **1b**, minor amounts of compounds **8a–8e**

(Scheme 2). Admixtures of benzo-18-crown-6 **8d** and dibenzo-30-crown-10 **8e** were also detected in compound **1b**. The kinetic regularities of the synthesis of compound **1b** are illustrated in the figure.

Dinitro derivatives of dibenzocrown ethers (dinitrodibenzo-18-crown-6 and dinitrodibenzo-24-crown-8) are formed as mixtures of the *trans* and *cis* isomers. Feigenbaum and Michel [11] described a long-term isomers of dinitrodibenzo-18-crown-6 by the nitration of dibenzo-18-crown-6 with a mixture of nitric and acetic acids in chloroform [11]. The synthesis gave the *cis* (mp 208–236°C) and *trans* isomers (mp 236–252°C). The broad ranges of melting points reported in the cited work cast doubt upon the purity of these products.

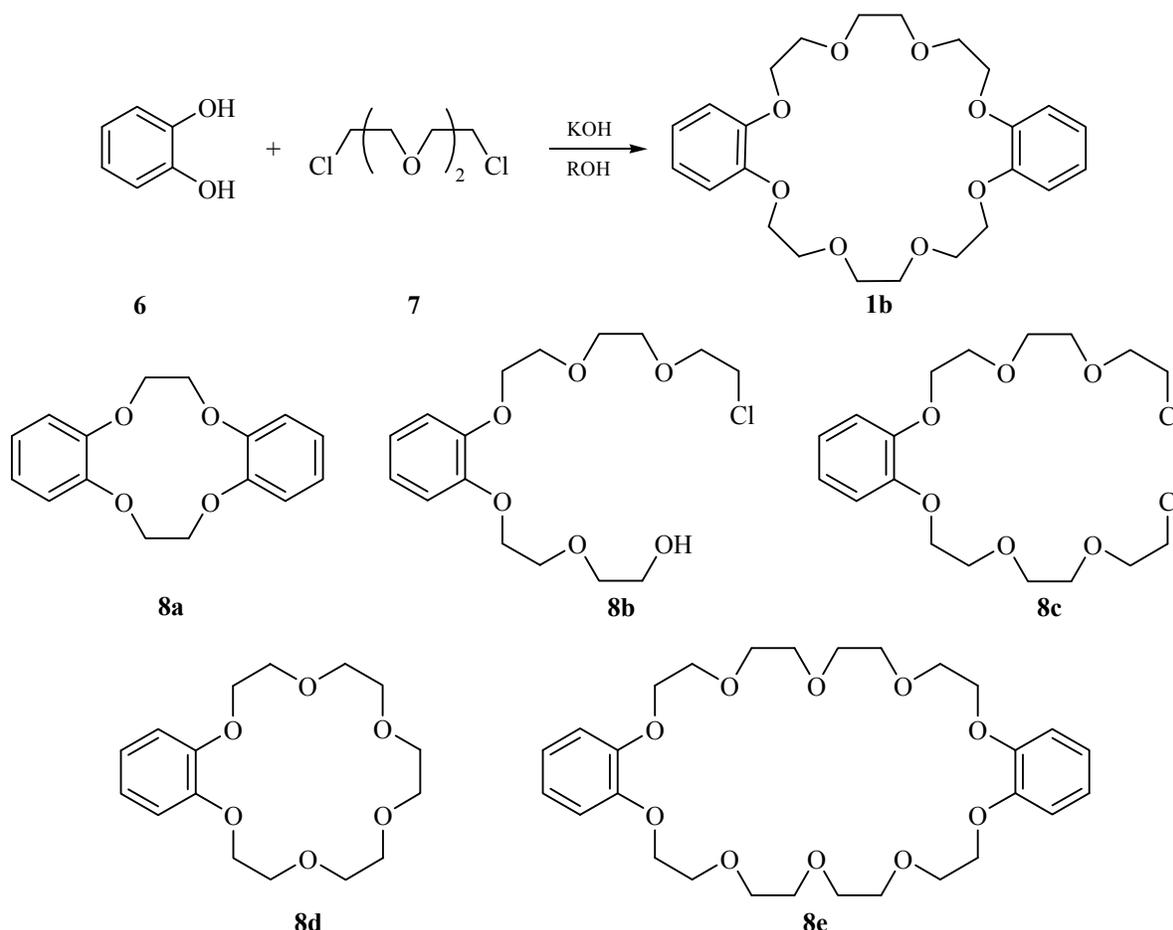
Dinitrodibenzo-18-crown-6 **9a** and dinitrodibenzo-24-crown-8 **9b** were prepared by the reaction of the corresponding dibenzocrown ethers with 58% nitric acid in acetonitrile followed by crystallization of the resulting dinitro derivatives from acetic acid. The



Kinetic curves of the synthesis of dibenzo-24-crown-8 **1b**: (1) pyrocatechol **6**, (2) triethylene glycol dichloride, (3) dibenzo-24-crown-8 **1b**, and (4–8) admixtures of compounds **8a–8e**.

isomer mixture obtained from dibenzo-18-crown-6 (mp 223–237°C) was separated into individual regioisomers by crystallization from acetic acid: *trans*-**9a** (mp 242–249°C) and *cis*-**9a** (mp 210–213°C). Because of the poor solubility of *trans*-**9a**, we could isolate the regioisomers after the obtained synthesized nitro compounds had been reduced to amino derivatives:

Scheme 2.



Thermal characteristics of dibenzocrown ethers and their dinitro and diamino derivatives

Compound	Endothermic peak, °C	Exothermic peak, °C	Weight loss at 400°C, %
Diaminodibenzo-18-crown-6 (10a)	175	350	65
Diaminodibenzo-24-crown-8 (10b)	130	361	33
Dibenzo-18-crown-6 (1a)	160	271	58
Dibenzo-24-crown-8 (1b)	100	327	83
Dinitrodibenzo-18-crown-6 (9a)	176	350	62
Dinitrodibenzo-24-crown-8 (9b)	146	343	65

cis-diaminodibenzo-18-crown-6 *cis*-**10a** (mp 170–172°C) and *trans*-diaminodibenzo-18-crown-6 *trans*-**10a** (mp 197–198°C).

Attempted synthesis of isomeric dinitrodibenzo-24-crown-8 **9b** both by the procedure in [12] and by the nitration of compound **1b** with nitric acid in acetonitrile followed by crystallization of the resulting nitro derivative from acetic acid afforded nitro derivatives respectively of mp 148.5–149.5°C and mp 150–151.5°C; unlike dinitro derivative **9a** they were readily soluble in most organic solvents.

A few methods of synthesis of diamino derivatives of dibenzocrown ethers are known. Diaminodibenzo-24-crown-8 **10b** were synthesized in a yield of 56% by the reduction of dinitro derivative **9b** with hydrogen on Pd/C in ethanol [12] in a pressure reactor at 3.5 at or in DMF, acetic acid, and ethylene glycol [13–15].

The mixture of regioisomers of compound **10b** was obtained by the procedure in [4, 16–18] by the reduction of dinitrodibenzo-24-crown-8 **9b** (a mixture of 4,4' and 4,5' isomers) with hydrazine hydrate in the presence of Raney nickel. Diamino derivatives **10a** and **10b** were also prepared by the reduction of the corresponding dinitrodibenzocrown ethers with hydrazine hydrate in the presence of Pd or Ni catalysts in DMF at 75–80°C. It was found that the diamino-crown ether obtained in the presence of 10% Pd/C is quite difficult to separate from the catalyst, because the product forms a fairly strong complex with palladium. The reduction catalyzed by the highly active Raney nickel is much more facile.

In view of the fact that diaminodibenzocrown ethers can be used to obtain high-temperature polymers, we assessed the thermal stability of dibenzocrown ethers **1a** and **1b** and derivatives **9a**, **9b** and **10a**, **10b**. The thermal degradation of compounds **1**, **9**, and **10** involves two steps: melting and decomposition.

The thermoanalytical curves of these compounds show endothermic peaks from 100 (**1b**) to 176°C (**9a**). These temperature points correspond to melting of compounds **1b** and **9a**. The exothermic peaks from 271 to ~400°C correspond to weight losses of 33 (**1a**) and ~65% (**1b**, **9**, **10**).

Comparison of the thermal characteristics of dibenzocrown ethers and their derivatives is presented in the table. The endothermic and exothermic peaks makes it possible to determine the melting and decomposition points, respectively.

Thus, we synthesized and characterized the regioisomers of dinitrodibenzo-18-crown-6 and diaminodibenzo-18-crown-6 and determined the optimal conditions of the preparation of the polycyclic ether dibenzo-24-crown-8 and its dinitro and diamino derivatives.

EXPERIMENTAL

The Fourier transform IR spectra were measured on a Bruker Vertex 70 instrument with an ATR unit (Miracle ATR, PIKE, ZnS crystal). The ¹H and ¹³C NMR spectra were registered on a Bruker Avance III NanoBay spectrometer [300.28 (¹H) and 75.51 MHz (¹³C)] in the thermostabilization mode at 25°C.

The reaction products were identified on a Khromatek-Kristall 500.2 chromatograph coupled to a Thermo ISQ mass spectrometer, using a TR-5MS quartz capillary column (*l* 15 m, *d* 0.25 mm, film thickness 0.25 μm, carrier gas helium, split ratio 1 : 25, oven temperature 150–280°C, ramp rate 20 deg/min). The mass spectra were obtained at the ionization energy 70 eV and ion source temperature 280°C. The products were identified by library search on NISTUSA database (2008), as well as by the molecular ions based on mass spectrum–structure correlations.

Isomeric dinitrodibenzo-18-crown-6 ethers 9a. Dibenzo-18-crown-6 **1a**, 36 g (0.1 mol), in 160 mL of

acetonitrile was heated to 70°C, after which 40 mL (1.5 mol) of 58% nitric acid was added with stirring over the course of 0.5 h. The reaction mixture was stirred for 40 min at 70°C and then poured into 1000 g of ice or ice water. In 0.5 h the precipitate was filtered off and washed with 500 mL of water. The reaction product was dried in air to constant weight to obtain 42.5 g (94.8%) of the isomer mixture, mp 220–224°C. The mixture was recrystallized from acetic acid to obtain 2.87 g of *trans*-dinitrodibenzo-18-crown-6, mp 240–249°C, and 2.49 g of *cis*-dinitrodibenzo-18-crown-6, mp 208.1–213.8°C.

***cis*-Dinitrodibenzo-18-crown-6 (*cis*-9a).** Yield 2.49 g, mp 208.1–213.8°C. IR spectrum, ν , cm^{-1} : 624 w, 653 m, 722 w, 742 s (NO_2), 784 w, 806 m, 863 w, 867 m, 898 w, 929 w, 956 m, 972 m, 998 w, 1043 m, 1055 m, 1082 m, 1100 s (C–N), 1135 s (C–O–C), 1221 s, 1237 s, 1261 s, 1274 s, 1343 s (NO_2), 1405 w, 1426 w, 1451 m, 1505 s (CH_2), 1512 sh (NO_2), 1588 m (C–C_{Ar}), 2883 w (C–H), 2926 w (C–H). ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 3.79–3.93 m (8H, CH₂), 4.16–4.30 m (8H, CH₂), 7.16 d (2H_{Ar}, $J = 8.9$ Hz), 7.72 d (2H_{Ar}, $J = 1.7$ Hz), 7.88 dd (2H_{Ar}, $J = 8.9$, $J = 1.7$ Hz). ¹³C NMR spectrum (DMSO-*d*₆), δ_{C} , ppm: 68.05, 68.30, 68.39, 106.77, 111.31, 117.44, 140.51, 147.63, 153.74. Found, %: C 52.10; H 5.33; N 6.19. C₂₀H₂₂N₂O₁₀. Calculated, %: C 53.33; H 4.92; N 6.22.

***trans*-Dinitrodibenzo-18-crown-6 (*trans*-9a).** Yield 2.87 g, mp 240–249°C. IR spectrum, ν , cm^{-1} : 616 w, 649 w, 722 w, 744 s (NO_2), 798 m, 849 w, 883 w, 898 m, 923 w, 967 m, 995 m, 1035 w, 1048 w, 1096 m, 1118 w, 1140 s (C–O–C), 1235 s, 1247 m, 1275 s (NO_2), 1346 s (NO_2), 1399 w, 1417 w, 1428 w, 1454 w, 1474 w, 1492 w, 1512 s (CH_2), 1591 (C–C_{Ar}), 2887 w (C–H), 2955 w (C–H), 3079 w (C–H), 3093 w (C–H). ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 3.79–3.91 m (8H, CH₂), 4.11–4.20 m (8H, CH₂), 7.16 d (2H_{Ar}, $J = 9.0$ Hz), 7.72 d (2H_{Ar}, $J = 2.6$ Hz). ¹³C NMR spectrum (DMSO-*d*₆), δ_{C} , ppm: 68.02, 68.35, 68.41, 106.62, 111.26, 117.53, 140.53, 147.65, 153.74. Found, %: C 53.36; H 5.44; N 6.20. C₂₀H₂₂N₂O₁₀. Calculated, %: C 53.33; H 4.92; N 6.22.

Isomeric diaminodibenzo-18-crown-6 ethers 10a.

A suspension of 2.0 g (4.4 mmol) of dinitrodibenzo-18-crown-6 in 30 mL of DMF was heated with stirring to 110°C and then 1.6 g of Raney Ni was added, followed by dropwise addition of 3.3 g (66 mmol) of hydrazine hydrate at 75–80°C. The catalyst was filtered off, the mother liquor was evaporated, and the residue was washed with 50 mL of ethanol and dried to a constant weight.

***cis*-Diaminodibenzo-18-crown-6 (*cis*-10a).** Yield 93.2%, mp 167.0–170.5°C. IR spectrum, ν , cm^{-1} : 602 w, 655 w, 722 w, 743 w, 783 m (NH_2), 795 m (NH_2), 828 m, 849 w, 905 w, 928 w, 956 w, 984 m, 1040 w, 1060 w, 1080 w, 1129 s (C–O–C), 1187 m, 1232 s, 1345 m, 1449 m, 1489 w, 1512 s (CH_2), 1592 w (C–C_{Ar}), 1614 w (NH_2), 2874 w (C–H), 2920 w (C–H), 3228 w (NH_2), 3326 w (NH_2). ¹H NMR spectrum (CD₃CN), δ , ppm: 3.76–3.84 m (8H, CH₂), 3.94–4.05 m (8H, CH₂), 6.14 d. d (2H_{Ar}, $J = 8.5$, $J = 2.5$ Hz), 6.31 d (2H_{Ar}, $J = 2.5$ Hz), 6.66 d (2H_{Ar}, $J = 8.5$ Hz). ¹³C NMR spectrum (CD₃CN), δ_{C} , ppm: 68.44, 69.13, 70.27, 70.40, 101.70, 106.57, 114.42, 141.24, 143.19, 149.82. Found, %: C 61.71; H 7.26; N 7.27. C₂₀H₂₆N₂O₆. Calculated, %: C 61.53; H 6.71; N 7.17.

***trans*-Diaminodibenzo-18-crown-6 (*trans*-10a).** Yield 89.3%, mp 197.0–198.5°C. IR spectrum, ν , cm^{-1} : 622 w, 663 w, 698 w, 720 w, 752 w, 767 w, 784 w, 792 w, 802 w, 836 m (NH_2), 905 w, 929 w, 947 w, 956 w, 987 w, 1062 m, 1087 w, 1130 s (C–O–C), 1184 m, 1228 s, 1281 m, 1346 w, 1356 w, 1384 w, 1444 w, 1455 w, 1511 s (CH_2), 1592 w (C–C_{Ar}), 1615 w (NH_2), 1673 w, 2833 w (C–H), 2884 w (C–H), 2928 w (C–H), 2968 w (C–H), 3357 w (NH_2), 3382 w (NH_2), 3422 w (NH_2), 3433 w (NH_2). ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 3.72 m (8H, CH₂), 3.88–4.02 m (8H, CH₂), 4.62 br.s (4H, NH₂), 6.06 d. d (2H_{Ar}, $J = 8.4$, $J = 2.0$ Hz), 6.24 d (2H_{Ar}, $J = 2.0$ Hz), 6.63 d (2H_{Ar}, $J = 8.4$ Hz). ¹³C NMR spectrum (DMSO-*d*₆), δ_{C} , ppm: 67.77, 69.01, 69.29, 69.39, 100.80, 105.37, 115.99, 139.11, 143.52, 149.28. Found, %: C 60.46; H 7.52; N 7.17. C₂₀H₂₆N₂O₆. Calculated, %: C 61.53; H 6.71; N 7.17.

Dinitrodibenzo-24-crown-8 ethers 9b. Acetonitrile, 80 mL, was added to 26.88 g (0.06 mol) of a mixture of 4,4'- and 4,5'-isomers of dibenzo-24-crown-8. The mixture was heated to 70°C, after which 19 mL (26 g, 0.24 mol) of 58% nitric acid was added over the course of 0.5 h. The reaction mixture was stirred for 20 min at 70°C and then poured into 400 g of ice water. The precipitate was filtered off, washed with 100–200 mL of water, dried, and recrystallized from 40 mL of acetic acid. Yield 31.7 g (98.5%, mixture of regioisomers), mp 152.5–155.0°C. IR spectrum, ν , cm^{-1} : 655 m, 722 w, 743 s (NO_2), 797 w, 813 m, 873 m, 906 w, 941 w, 967 w, 992 m, 1017 m, 1043 m, 1080 m, 1095 m, 1130 s (C–O–C), 1140 s, 1236 s, 1265 s, 1280 s, 1338 s (NO_2), 1375 w, 1426 w, 1450 w, 1470 w, 1482 w, 1508 s (CH_2), 1510 sh (NO_2), 1590 (C–C_{Ar}), 2879 (C–H), 2904 (C–H), 2942 (C–H). ¹H NMR spectrum (CDCl₃), δ , ppm: 3.80–3.85 m (8H, CH₂), 3.91–

3.98 m (8H, CH₂), 4.18–4.25 m (8H, CH₂), 6.86 d (2H_{Ar}, *J* = 8.9 Hz), 7.71 t (2H_{Ar}, *J* = 2.4 Hz), 7.86 d.t (2H_{Ar}, *J* = 8.9, *J* = 2.4 Hz). ¹³C NMR spectrum (CDCl₃), δ_C, ppm: 69.67, 69.79, 69.86, 69.90, 71.12, 71.79, 108.42, 108.44, 111.41, 111.44, 118.19, 118.21, 141.66, 141.70, 148.56, 148.60, 154.47, 154.48. Found, %: C 53.58; N 5.18. C₂₄H₃₀N₂O₁₂. Calculated, %: C 53.50; N 5.20.

Diaminodibenzo-24-crown-8 ethers 10b. Hydrazine hydrate, 78 mL (1.6 mol), was added dropwise at 60°C to 31.7 g (0.075 mol) of dinitrodibenzo-24-crown-8 in 250 mL of ethanol, after 9.5 g of Raney Ni was added. The resulting mixture was left for 40 min and then refluxed until decolorized, filtered hot, evaporated to leave a brown oily residue which was purified by flash chromatography and dried in a desiccator to a constant weight. Yield 23.9 g (85.6%, mixture of regioisomers), mp 132.0–133.7°C. IR spectrum, ν, cm⁻¹: 627 m, 667 w, 763 w, 775 w, 828 m (NH₂), 844 m (NH₂), 891 m, 945 m, 979 m, 1051 s, 1080 s, 1118 s (C–O–C), 1182 m, 1225 s, 1274 m, 1326 w, 1349 w, 1443 m, 1509 s (CH₂), 1586 s (C–C_{Ar}), 1617 m (NH₂), 2868 m (C–H), 2923 m (C–H), 3366 m (NH₂), 3448 m (NH₂). ¹H NMR spectrum (CD₃CN), δ, ppm: 3.66 t (8H, CH₂, *J* = 2.9 Hz), 3.70–3.80 m (8H, CH₂), 3.85 br.s (4H, NH₂), 3.94–4.05 m (8H, CH₂), 6.14 d. d. d (2H_{Ar}, *J* = 8.4, *J* = 2.5, *J* = 0.7 Hz), 6.29 d (2H_{Ar}, *J* = 2.5 Hz), 6.68 d (2H_{Ar}, *J* = 8.4 Hz). ¹³C NMR spectrum (CD₃CN), δ_C, ppm: 69.37, 69.41, 70.52, 70.57, 70.86, 70.89, 71.07, 71.11, 71.46, 71.60, 102.77, 102.87, 107.28, 107.31, 141.58, 141.62, 144.17, 144.22, 151.07, 151.11. Found, %: C 60.18; N 5.85. C₂₄H₃₄N₂O₈. Calculated, %: C 60.25; N 5.86.

Kinetic study of the formation of dibenzo-24-crown-8 ether 1b. Sampling from the reaction mixture was performed in regular time intervals using alcoholic HCl as a stop reagent (pH 3–4). Quantitative analysis of the samples was performed by GLC on a Khromatek-Kristall 5000.2 chromatograph equipped with a flame ionization detector and a BP-5 quartz capillary column (*l* 30 m, *d* 0.32 mm, film thickness 0.5 μm, carrier gas helium, flow rate 2.8 mL/min, split ratio 1 : 25, oven temperature 150–300°C, ramp rate 10 deg/min). The component concentrations in the analyzed sample were calculated by percent area normalization.

The thermal characteristics of the synthesized compounds were studied by TGA and DSC on a TA Instruments STD Q600 simultaneous thermal analysis instrument in air at a heating rate of 10 deg/min in alundum crucibles in the temperature range 20–400°C.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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