

Borylation of Calix[4]resorcinols under Ultrasonic Irradiation

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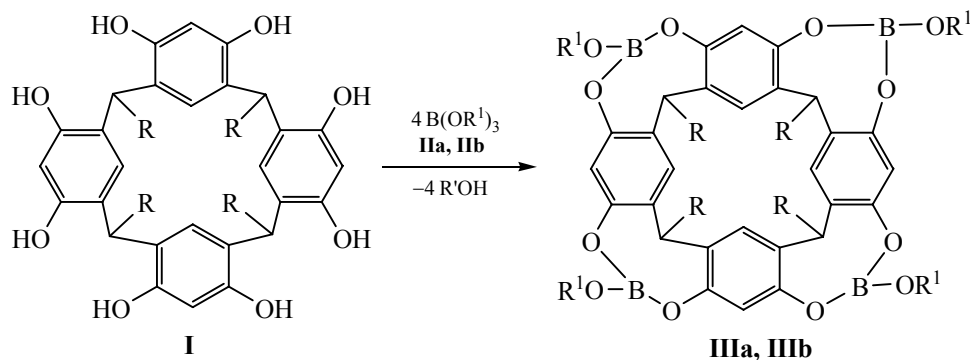
Abstract—The use of ultrasonic irradiation in the reactions of trialkyl borates with octahydroxytetraoctyl[4]metacyclophane allowed us obtaining tetraalkoxyboratocavitands with higher yields and under milder conditions. We first obtained 1,3,2-dioxaborolane and 1,3,2-dioxaborinane derivatives of tetraoctyl[4]metacyclophane using ultrasonic irradiation. Boratocavitands were converted into the corresponding amine complexes with isobutylamine.

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Since recently, an increased attention is paid to structural, chemical, physicochemical aspects and application of the class of calixarene compounds. This is due to the existence of a hydrophobic cavity in the molecules of these compounds that is capable of complexing with the molecules of neutral organic compounds and ions [1–4]. Due to the presence of hydroxy groups, the calixarenes can be subjected to functionalization, which leads to a change in the size and geometry of the cavity and its ability of effective and selective bonding. The calixarenes whose molecules include a hetero-element may have practical value as a complexing agents and metals extractants, and can serve as the models of enzymatic and other biochemical processes. Among the calixarenes with

the elements introduced through the HO groups the most attention attracted the cavitands O- and C-phosphorylated at the lower and upper rims [5–19]. At the same time, other calixarenes of the O-element type (O-sulfonylated [20], O- and C-silylated [21–26] and O-borylated cavitands [27]) have been less studied.

It is known that the reaction of calixresorcynols with triisobutyl borate in boiling toluene leads to the formation of tetraalkoxyborato-cavitands [28]. In order to increase the reactivity of borates in these reactions we exerted ourselves to search for initiators of these transformations. We have previously shown that the reaction of thiophosphorylation of trialkyl borates under ultrasonic irradiation occurs considerably faster,



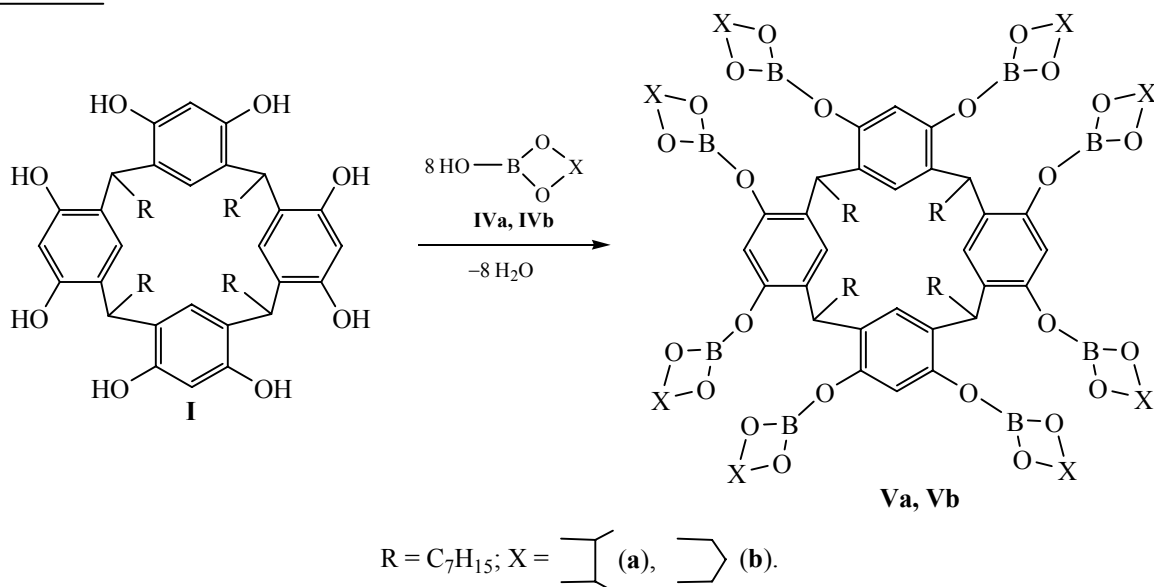
R = C₇H₁₅, R¹ = Bu-*i* (a), Bu (b).

at lower temperatures and with higher yield [29, 30]. Now we prepared tetraalkoxyborato-cavitands **IIIa** and **IIIb** by irradiation with ultrasound (22 kHz, power 400 W) a mixture of octahydroxytetraoctyl[4]metacyclophane **I** with trialkylborates **IIa** or **IIb**, respectively, in toluene (100°C, 45 min).

The IR spectrum of cavitand **III** contains characteristic absorption bands of the O–B stretching vibrations at ν 1028–1033 cm^{-1} . Compound **IIIa** is characterized by the presence in the IR spectrum of two absorption bands of moderate intensity in the region of ν 1350 and 1381 cm^{-1} of symmetric bending δ -vibration of the geminal methyl groups $(\text{CH}_3)_2\text{C}$ in the isobutoxy substituents, as in [31]. Boron atoms of the cavitands **III** appear as a narrow singlet signal in the ^{11}B NMR spectrum at δ_{B} 18 ppm, which corresponds to compounds with tricoordinated boron atoms [32–34]. In the ^1H NMR spectrum of the borate **IIIa** the methine proton of the CH_2CHAr fragment gives two triplets with chemical shift δ 4.13 ppm ($^3J_{\text{HH}}$ 6.7 Hz). The presence in the ^1H NMR spectrum of the doublet with chemical shift δ 0.94 ppm ($^3J_{\text{HH}}$ 6.7 Hz) belonging to the methyl protons of the fragment

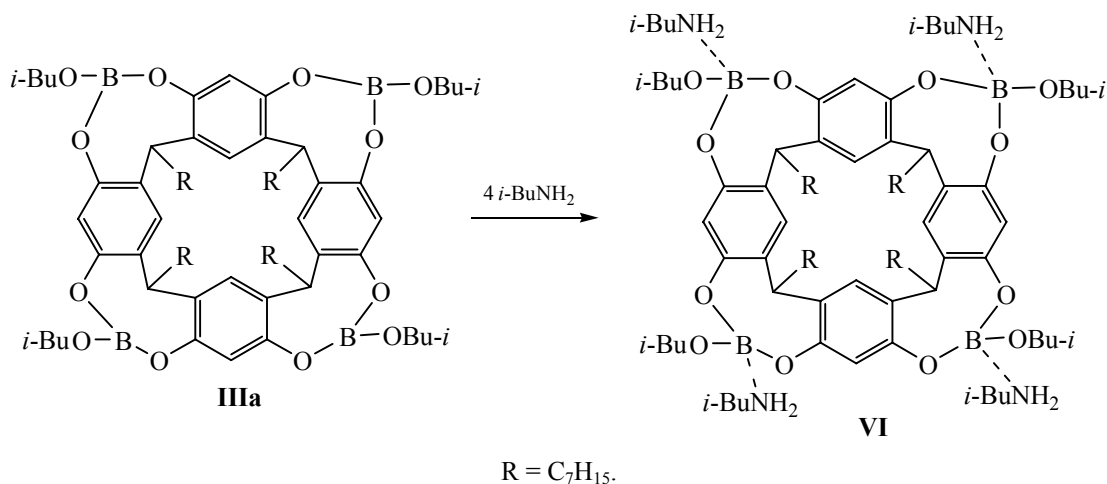
$(\text{CH}_3)_2\text{CHCH}_2\text{O}$ confirms the addition of four isobutoxy groups to the cavitand **IIIa**. The MALDI mass spectrum of the cavitand **IIIa** contains the mass peak m/z 1209 corresponding to a molecular ion of the product of substitution of all eight hydroxy groups (calculated $M = 1208.9$).

Developing these studies, we increased a number of reagents capable of borylate calixarenes at the hydroxyl groups. Previously, we used cyclic hydroxylboranes as borylating agents at the S–H group of phosphorus thioacids [34]. Now we explored the phosphorylating ability of hydroxyborinanes in the reactions with calixarenes. However, the reaction did not occur even at prolonged boiling in xylene medium using a Dean–Stark trap to remove the liberated water. At the same time, under the influence of ultrasonic irradiation (100°C, 50 min) the reaction of calixarene **I** with 2-hydroxy-4,5-dimethyl-1,3,2-dioxaborolane **IVa** (a mixture of isomers prepared from 1,3-butanediol) and 2-hydroxy-1,3,2-dioxaborinane **IVb** in toluene resulted in the formation of cyclic borylated cavitands **Va** and **Vb**.



In the ^{11}B NMR spectrum of 1,3,2-dioxaborolane derivative **Va** there are two signals of equal intensity at δ_{B} 21.7 and 22.3 ppm, caused by the formation of a mixture of isomers due to the presence of chiral centers in the 1,3,2-dioxaborolane fragments. At the same time, 1,3,2-dioxaborinane cavitand **Vb** has a singlet signal at δ_{B} 8.17 ppm. This result agrees with the tricoordinated nature of the boron atoms in

compounds **V**. In the ^1H NMR spectrum of the 1,3,2-dioxaborolane cavitand **Va** the methyl protons of the borolane $\text{CH}_3\text{CHCHCH}_3$ fragments appear as doublets at δ 1.25 ppm ($^3J_{\text{HH}}$ 6.0 Hz). In the ^1H NMR spectrum of the mixture of the isomers of 1,3,2-dioxaborinane derivative **Vb** there are two triplets related to the methylene protons at the oxygen atoms of the propylene fragment ($\text{OCH}_2\text{CH}_2\text{CH}_2\text{O}$) at δ_1 4.00 ppm



and δ_2 4.7 ppm ($^3J_{\text{HH}}$ 5.7 Hz). In the MALDI mass spectrum of compound **Va** there is a mass peak m/z 1661 of the molecular ion $[M]^+$ (calculated M 1663.8). Accordingly, the product **Vb** in the MALDI mass spectrum gives the mass peak of its molecular ion $[M + 8H]^+$, m/z 1564 (calculated M 1552.3).

Reaction of the borocavitands with amines exemplifies their complexing properties. Reaction of the cavitand **IIIa** with isobutylamine in benzene at 50°C (1 h) affords a solid amine complex **VI**.

The ^{11}B NMR spectrum of complex **VI** includes a broad signal at δ_{B} -0.7 ppm, which indicates an increase in the coordination number of boron atoms to four like in the $\text{Et}_2\text{O} \cdot \text{BF}_3$ complex (δ_{B} 0.0 ppm) in which the boron atom is also tetracoordinated [34]. The IR spectrum of compound **VI** contains a broad strong absorption band at ν 3423 cm^{-1} belonging to the stretching vibrations of H_2N^+ fragment. The cavitands **Va** and **Vb** are also capable of forming amine complexes with isobutylamine. The ^{11}B NMR spectra of these complexes contain broad signals at -6.8 ppm of the complex of compound **Va** and -6.2 ppm of the complex of cavitand **Vb**.

Thus, we have developed a method of ultrasonic initiation of the reactions of trialkyl borates with the calixarenes and obtained new calixarene cyclic boron derivatives.

EXPERIMENTAL

The IR spectra were recorded on a Bruker Vector 22 Fourier Spectrometer (400–4000 cm^{-1}) from liquid films between KBr plates or from tablets with KBr. The ^1H NMR spectra were registered on a Bruker

Avance-400 spectrometer with an operating frequency 400 MHz and a Bruker Avance-600 spectrometer (600 MHz) from solutions of compounds in CDCl_3 . The ^{11}B NMR spectra were recorded on a Bruker Avance-600 instrument (192.5 MHz, external reference $\text{BF}_3 \cdot \text{Et}_2\text{O}$) from solutions in CDCl_3 . The MALDI mass spectra were obtained on a Bruker Ultraflex mass spectrometer (UV laser, 337 nm, the matrix was 1,8,9-trihydroxyanthracene). The reaction under the ultrasonic irradiation was performed using an ultrasonic dispersing unit of low frequency UZDN-1U42 with a conical nozzle of the concentrator emitter. The temperature of the reaction mixture was measured using a custom-constructed thermocouple temperature measuring unit RT-04.

2,8,14,20-Tetraheptylpentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]-octacos-1(25)3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaen-6,10,12,16,18,22,24,4-tetraisobutylborate (IIIa). A mixture of 1.2 g of calixresorcinol **I** and 1.25 g of borate **IIa** in 25 ml of water-free toluene in a dry argon atmosphere was subjected to the ultrasound irradiation for 45 min at 100°C. After cooling, the reaction mixture was evaporated in a vacuum (0.5 mm Hg, 1 h, 40°C and 0.02 mm Hg, 1 h, 40°C). 1.4 g (85%) of compound **IIIa** was isolated, mp 246–247°C (published: [28]: mp 244–245°C). IR spectrum (from a tablet), ν , cm^{-1} : 3085 w, 3061 w [$\nu(\text{C-H, Ar})$]; 2956 m, 2923 s, 2871 s [$\nu_{\text{as,s}}(\text{CH}_3)$, $\nu_{\text{as,s}}(\text{CH}_2)$, $\nu(\text{CH})$]; 1604 m, 1495 s [$\nu(\text{C=C, Ar})$]; 1462 s [$\delta_{\text{as}}(\text{CH}_3)$]; 1350 m, 1381 m [$\delta_{\text{s}}(\text{CH}_3)_2\text{C gem}$]; 1081 m [$\nu(\text{O-C})$]; 1033 m [$\nu(\text{O-B})$]. ^1H NMR spectrum, δ , ppm, (J , Hz): 0.90 t (12H, $\text{CH}_3\text{CH}_2\text{CH}_2$, $^3J_{\text{HH}}$ 6.7); 0.94 d [4H, $(\text{CH}_3)_2\text{CHCH}_2\text{O}$, $^3J_{\text{HH}}$ 6.7]; 1.26 m [40H, $\text{CH}_3(\text{CH}_2)_5$]; 1.79 m [4H, $\text{OCH}_2\text{CH}(\text{CH}_3)_2$]; 2.07 m (8H, CH_2CH_3); 3.44 d (8H, OCH_2CH , $^3J_{\text{HH}}$ 6.8); 4.13 two t (4H, $\text{CH}_2\text{CH-Ar}$, $^3J_{\text{HH}}$

6.7); 6.30 m (4H, C₆H-*ortho*); 7.20 m (4H, C₆H-*meta*). Found, %: C 71.18; H 9.07; B 3.93. C₇₂H₁₀₈B₄O₁₂. Calculated, %: C 71.54; H 9.00; B 3.58.

2,8,14,20-Tetraheptylpentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]-octacosa-1(25)3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-6,10,12,16,18,22,24,4-tetrabutylborate (IIIb) was prepared similarly from 1.2 g of calixresorcinol **I** and 1.25 g of borate **IIb**, yield 1.3 g (81%). IR spectrum (from a film), ν , cm⁻¹: 3087 w, 3061 w [ν (C–H, Ar)]; 2959 m, 2931 s, 2871s [$\nu_{as,s}$ (CH₃), $\nu_{as,s}$ (CH₂), ν (CH)]; 1611 m, 1487 s [ν (C=C, Ar)]; 1338 s [δ_s (CH₃)]; 1073 m [ν (O–C)]; 1028 m, 970 m [ν (O–B)]. ¹H NMR spectrum, δ , ppm, (*J*, Hz): 0.94 t [12H, CH₃(CH₂)₅CH₂CH, ³*J*_{HH} 7.3]; 0.96 t [12H, CH₃(CH₂)₄O, ³*J*_{HH} 7.0]; 1.30 m (CH₂); 1.40 m (CH₂); 1.57 m (CH₂); 1.75 d.t. (8H, CH₂CH₂CH–Ar, ³*J*_{HH} 6.7); 2.33 m (8H, CH₃CH₂(CH₂)₄CH–Ar); 3.79 t (8H, OCH₂CH₂CH₂CH₃, ³*J*_{HH} 6.7); 4.35 t (4H, CH₂CH–Ar, ³*J*_{HH} 6.7); 6.14 m (4H, C₆H-*ortho*); 7.20 m (4H, C₆H-*meta*). Found, %: C 71.23; H 8.88; B 3.77. C₇₂H₁₀₈B₄O₁₂. Calculated, %: C 71.54; H 9.00; B 3.58.

3,5,10,12,17,19,24,26-Octakis(4,5-dimethyl-1,3,2-dioxaborolano)-1,8,15,22-tetrakis(hexyl)[4]metacyclophane (Va). A mixture of 0.5 g of calixresorcinol **I** and 0.52 g of borinane **Va** in 15 ml of water-free toluene in a dry argon atmosphere was subjected to the ultrasound irradiation for 50 min at 100°C. After cooling to 20°C the reaction mixture was evaporated in a vacuum (0.5 mm Hg, 1 h, 40°C and 0.02 mm Hg, 1 h, 40°C). 0.75 g (80%) of compound **Va** was isolated. IR spectrum, from a film, ν , cm⁻¹: 3028 w [ν (C–H, Ar)]; 2980 s, 2929 s, 2858 s [$\nu_{as,s}$ (CH₃), $\nu_{as,s}$ (CH₂), ν (CH)]; 1619 m, 1501 s [ν (C=C, Ar)]; 1466 s [δ_{as} (CH₃)]; 1382, 1318 m [δ_s (CH₃)]; 1079 s, 1023 m [ν (O–C)]; 897 m [ν (O–B)]. ¹H NMR spectrum, δ , ppm, (*J*, Hz, isomeric mixture): 0.92 t [12H, CH₃(CH₂)₅CH₂CH, ³*J*_{HH} 7.3]; 1.25 d (48H, CH₃CHCHCH₃-ring, ³*J*_{HH} 6.0); 1.32 m (CH₂); 2.32 m (CH₂); 4.04 and 4.23 two m (4H, CH₂CHAr); 4.56 and 4.57 two q [16H, CH₃CHCHCH₃-ring, ³*J*_{HH} 6.0]; 6.16 m (4H, C₆H-*ortho*); 7.20 m (4H, C₆H-*meta*). Found, %: C 63.19; H 8.81; B 5.49. C₈₈H₁₃₆B₈O₂₄. Calculated, %: C 63.47; H 8.26; B 5.20.

3,5,10,12,17,19,24,26-Octakis(1,3,2-dioxaborinano)-1,8,15,22-tetrakis(hexyl)[4]metacyclophane (Vb) was prepared similarly from 0.5 g of calixresorcinol **I** and 0.46 g of borinane **Vb**, yield 0.7 g (80%). IR spectrum (from a film), ν , cm⁻¹: 3087 w, 3026 w [ν (C–H, Ar)]; 2954 m, 2928 s, 2856 s [$\nu_{as,s}$ (CH₃), $\nu_{as,s}$ (CH₂), ν (CH)]; 1618 m, 1605 m, 1489 m [ν (C=C, Ar)]; 1350 s [δ_s (CH₃)]; 1161 s, 1070 m, 1056 m [ν (O–C)]; 934 m

[ν (O–B)]. ¹H NMR spectrum, δ , ppm, (*J*, Hz): 0.91 t [12H, CH₃(CH₂)₅CH₂CH, ³*J*_{HH} 6.7]; 1.31 br.s (CCH₂C); 1.94 m (16H, OCH₂CH₂CH₂O); 3.87 t (4H, CH₂CH–Ar, ³*J*_{HH} 6.7); 4.00 t (32H, OCH₂CH₂CH₂O, ³*J*_{HH} 5.7); 6.19 br.s (4H, C₆H-*ortho*); 7.16 m (4H, C₆H-*meta*). Found, %: C 61.93; H 7.45; B 5.56. C₈₀H₁₂₀B₈O₂₄. Calculated, %: C 61.90; H 7.79; B 5.57.

Tetraisobutylamine complex of 2,8,14,20-tetraheptylpentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25)3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaen-6,10,12,16,18,22,24,4-tetraisobutylborate (VIa). To a solution of 0.5 g of the boratocavitand **IIIa** in 20 ml of water-free benzene in a dry argon flow at 20°C at stirring was added 0.12 g of isobutylamine. The solution obtained was stirred for 1 h at 50°C, cooled to 20°C, and evaporated in a vacuum (0.5 mm Hg, 1 h, 40°C and 0.08 mm Hg, 1 h, 40°C). 0.5 g (80%) of the solid complex **VIa** was isolated. IR spectrum, (from a tablet), ν , cm⁻¹: 3423 v.s. br [ν (⁺NH₂)]; 2957 m, 2927 s, 2855 s [$\nu_{as,s}$ (CH₃), $\nu_{as,s}$ (CH₂), ν (CH)]; 1618 m, 1495 m [ν (O–C)]; 1465 s [δ_{as} (CH₃)]; 1370 m, 1341 m [δ_s (CH₃)₂C gem]; 1168 m [ν (O–C)]; 1087 m [ν (O–B)]. ¹H NMR spectrum, δ , ppm, (*J*, Hz): 0.90 d [24H, (CH₃)₂CHCH₂N, ³*J*_{HH} 4.5]; 0.94 d [24H, (CH₃)₂CH·CH₂O, ³*J*_{HH} 6.5]; 0.99 t (12H, CH₃CH₂CH₂, ³*J*_{HH} 6.5); 1.31 br.s [40H, CH₃(CH₂)₅]; 1.83 m [16H, NCH₂CH·(CH₃)₂]; 2.19 m (8H, CH₂CH₃); 2.83 m [4H, NCH₂CH·(CH₃)₂]; 3.44 d (8H, OCH₂CH(CH₃)₂, ³*J*_{HH} 6.5]; 4.34 m (4H, CH₂CH–Ar); 6.30 m (4H, C₆H-*ortho*); 7.24 m (4H, C₆H-*meta*); 8.20 m (8H, NH₂). Found, %: C 70.33; H 9.77; B 2.59; N 3.91. C₈₈H₁₅₂B₄NO₁₂. Calculated, %: C 70.40; H 10.20; B 2.88; N 3.73.

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