Synthesis and aggregation properties of novel amino acetals with the calix[4]resorcinol platform

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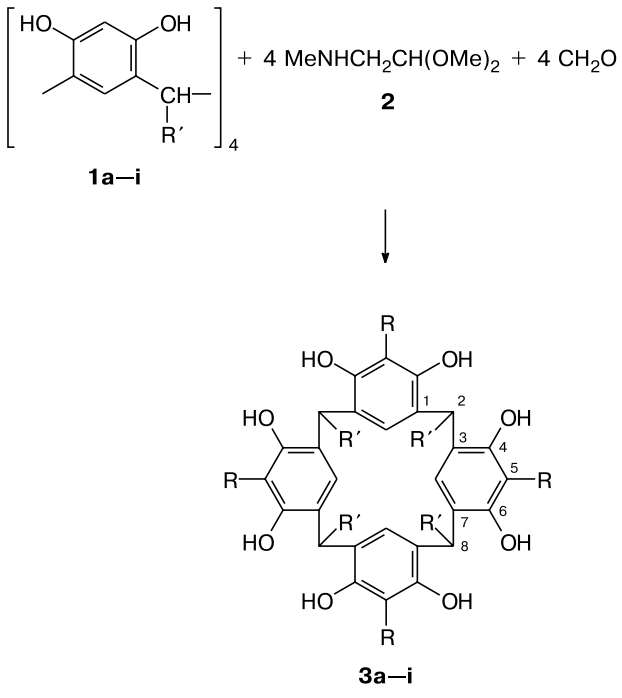
Reactions of calixarenes with methylaminoacetaldehyde dimethyl acetal and formalin gave Mannich bases with the calixarene platform. It was found by dielcometric titration that calix[4]resorcinols with acetal groups in the aminomethyl fragment form head-to-head supramolecular aggregates in chloroform at low concentrations.

Key words: calix[4]resorcinol, aminomethylation, acetals, aggregation.

Supramolecular chemistry, which is largely based on macrocyclic compounds, has been under intensive development in recent years. These compounds have an important tendency toward self-assembly producing definite supramolecular ensembles.[1—4] Calix[4]resorcinols, which are cyclic tetramers obtained by condensation of aldehydes with resorcinols, can serve as components for the creation of such supramolecular ensembles.[5—7] Structures like these are also of great interest as complexones and extracting agents. Functionalization of calix[4]resorcinols allows one to vary the size and shape of the molecular cavity and synthesize novel spatially ordered structures with practically useful properties. Compounds containing aminomethyl fragments in the aromatic rings in the *ortho*-position relative to the hydroxy groups are promising representatives of functionalized calixarenes. These compounds containing tertiary amino groups have been synthesized by the Mannich reaction.[8,9] Recently, we have synthesized the first representatives of aminomethylated calix[4]resorcinols containing NH groups or phosphonoylalkyl fragments on the upper rim of the molecule[10] and modified the calix[4]resorcinol platform with methylamino acetal fragments.[11] Compounds of the latter type can serve as a basis for design of container-type and tubular structures *via* condensation of the acetal fragments with polyphenol molecules. The goal of the present work was to obtain a series of amino acetal Mannich bases according to the previously developed method and study their conformational properties and the aggregation behavior for subsequent targeted synthesis of compounds with a desired architecture.

Reactions of calixarenes **1a**—**i** with methylaminoacetaldehyde dimethyl acetal **2** and formalin in the ratio 1 : 4 : 4 gave Mannich bases with the calix[4]resorcinol platform (**3a**—**i**) (Scheme 1). The structures and compo-

Scheme 1



3a—i

R = —$CH_2N(Me)CH_2CH(OMe)_2$; R′ = Me (**a**), Et (**b**), Pr (**c**), C_5H_{11} (**d**), C_6H_{13} (**e**), C_7H_{15} (**f**), C_8H_{17} (**g**), C_9H_{19} (**h**), $C_{11}H_{23}$ (**i**)

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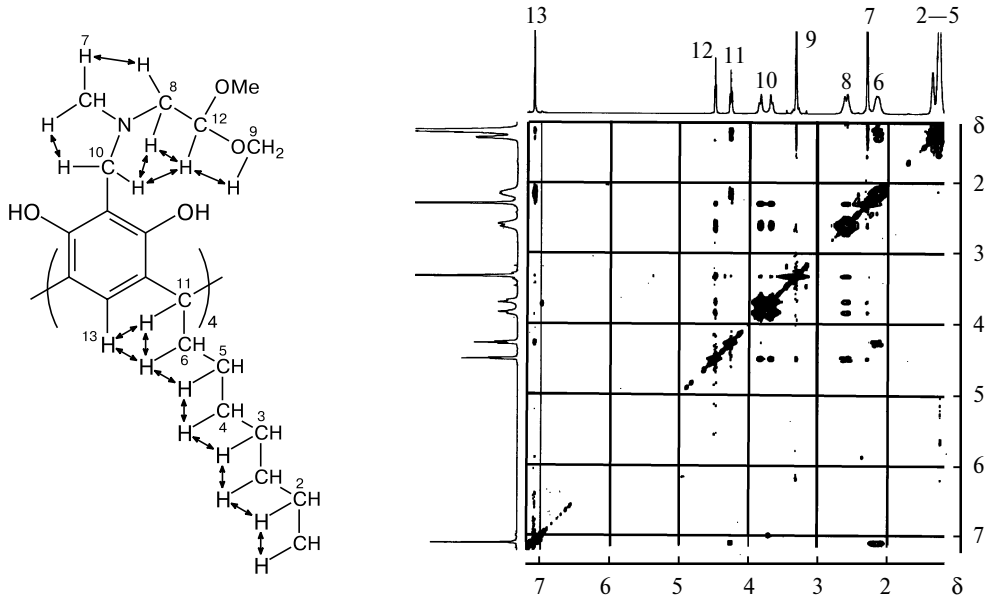

Fig. 1. 2D ROESY spectrum of compound **3f**.

sitions of compounds **3a—i** were proved by ^{1}H and ^{13}C NMR spectroscopy and elemental analysis.

Analysis of the ^{1}H NMR spectra of compounds **3a—i** showed that the position of the multiplet for the protons of the CH groups linking the aromatic fragments is virtually independent of the carbon chain length of substituent R′, which provides evidence for the same conformation.

A 2D ROESY study of compounds **3a,b,d,f** in chloroform revealed that they exist in the "cone" conformation.

The 2D ROESY spectrum of compound **3f** in chloroform (Fig. 1) contains only the expected cross peaks: C(6)H_2 and C(5)H_2, C(10)H_2 and NMe, C(11)H and C(6)H_2, C(11)H and C(5)H_2, C(11)H and C(4)H_2, C(12)H and NMe, and C(8)H_2 and C(10)H_2, which confirms the cone conformation.

Dielcometric titration is a technique used to study the aggregation of amphiphilic compounds in nonpolar solvents by measuring a change in the polarization of the medium in an applied electric field.[12] Aggregation of amphiphilic compounds change the dielectric characteristics of the medium. A plot of the dielectric constant ε *vs.* the concentration of such compounds becomes nonlinear (its slope changes) because of aggregation occurring at certain points called critical micelle concentrations (cmc).[13—16] The parameter ε can be regarded not only as a characteristic of the medium polarity but also as a measure of its ordering.[13]

We used this technique for investigation of the self-assembly of calix[4]resorcinols **3**.

It is known that the motive forces of head-to-head aggregation of amphiphilic compounds in nonpolar media that gives rise to reverse micelles are electrostatic interactions or hydrogen bonding.[13—16]

The plots of ε *vs.* the concentration of compounds **3a,d,e,h,i** are shown in Fig. 2.

It can be seen in Fig. 2 that the cmc values of calix[4]resorcinols **3** with different hydrocarbon chain lengths change only slightly (in the range 5—7.5 · 10^{-5} mol L^{-1}). An analogous aggregation behavior has been

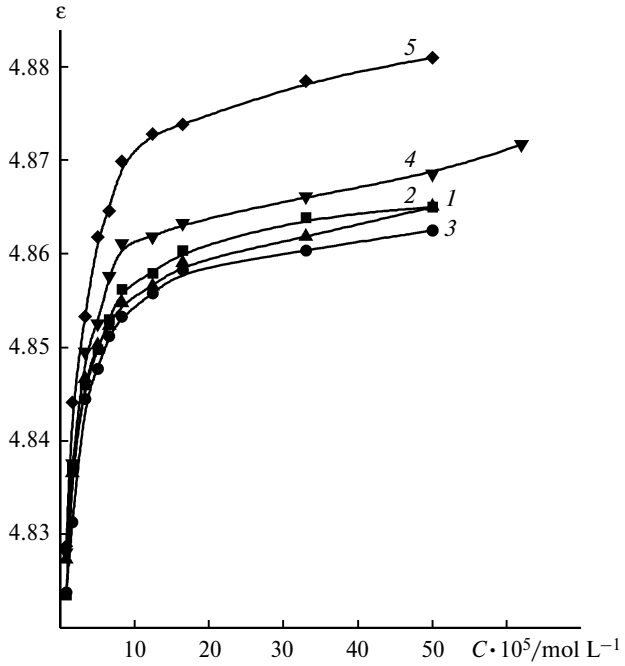

Fig. 2. Plots of the dielectric constant ε *vs.* the concentration for solutions of compounds **3a** (*1*), **3d** (*2*), **3e** (*3*), **3h** (*4*), and **3i** (*5*) in chloroform.

reported earlier[15,16] for structurally simpler aminomethylated calix[4]resorcinols with cmc = (7—10)·10^{-5} mol L^{-1}. A comparison of the aforementioned values shows that the presence in calix[4]resorcinols **3** of acetal groups tending to form additional hydrogen bonds facilitates the aggregation of these compounds in chloroform by reducing their cmc approximately by half. For the series of compounds **3**, the ε value changes only slightly after the cmc (see Fig. 2). Such a pattern is characteristic of the formation of low-polarity associates.[12,15] The data obtained suggest that calix[4]resorcinols with acetal groups in the aminomethyl fragment, like their simpler aminomethylated analogs,[15] form head-to-head supramolecular aggregates in chloroform at low concentrations.

Experimental

^{1}H and ^{13}C NMR spectra were recorded on a Bruker MSL-400 spectrometer (400.13 and 100.62 MHz, respectively) in $CDCl_3$ at 20 °C with reference to residual protons of the solvent ($CDCl_3$). IR spectra were recorded on a UR-20 spectrometer (Nujol) in the 400—3600 cm^{-1} range. Calix[4]resorcinols **1a**—**i** were prepared as described earlier.[7]

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetramethylpentacyclo[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 (3a). Methylaminoacetaldehyde dimethyl acetal **2** (1.43 g, 12 mmol) and 30% formalin (1.20 g, 12 mmol) were added to a solution of calixarene **1a** (1.31 g, 2.4 mmol) in benzene—EtOH (1 : 1, v/v; 60 mL). The reaction mixture was kept at 20 °C for 24 h. The solvent was removed in water aspirator vacuum and the residue was reprecipitated from chloroform with hexane and dried at 40 °C (0.06 Torr, oil pump) to a constant weight. The yield of compound **3a** was 2.19 g (85%), m.p. 106—108 °C. Found (%): C, 62.67; H, 8.15; N, 4.91. $C_{56}H_{84}N_4O_{16}$. Calculated (%): C, 62.92; H, 7.87; N, 5.24. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 1.74 (d, 12 H, CH<u>Me</u>, *J* = 6.9 Hz); 2.31 (s, 12 H, NMe); 2.64 (br.m, 8 H, NC<u>H</u>$_2$CH); 3.34 (s, 24 H, OMe); 3.77 (m, 8 H, $C_{arom}C\underline{H}_2N$); 4.51 (br.m, 8 H, C<u>H</u>Me, C<u>H</u>$(OMe)_2$); 7.05 (s, 4 H, *m*-CH_{arom}); 8.33 (br.s, 8 H, HO). ^{13}C NMR, δ: 151.68 ($C_{arom}OH$); 125.65 ($C_{arom}CH$); 123.09 (CH_{arom}); 108.00 ($C_{arom}CH_2$); 102.69 (CH(OMe)); 59.05 (NCH_2); 55.65 ($ArCH_2$); 54.06 (OMe); 42.32 (NMe); 28.63 (CH); 20.03 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetraethylpentacyclo[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 (3b) was obtained analogously from calixarene **1b** (1.44 g, 2.4 mmol), acetal **2** (1.43 g, 12 mmol), and 30% formalin (1.20 g, 12 mmol). The yield was 2.43 g (90%), m.p. 116—118 °C. Found (%): C, 63.67; H, 7.85; N, 4.68. $C_{60}H_{92}N_4O_{16}$. Calculated (%): C, 64.06; H, 8.19; N, 4.98. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.89 (br.m, 12 H, $CH_2\underline{Me}$); 2.11 (m, 8 H, MeC<u>H</u>$_2$CH); 2.35 (s, 12 H, NMe); 2.64 (br.m, 8 H, NC<u>H</u>$_2$CH); 3.33 (s, 24 H, OMe); 3.85 (br.s, 8 H, $C_{arom}C\underline{H}_2N$); 4.18 (br.m, 4 H, $CH_2C\underline{H}(OMe)_2$); 4.53 (br.m, 4 H, $C_{arom}C\underline{H}CH_2$); 7.12 (s, 4 H, *m*-$CH_{arom}$). ^{13}C NMR, δ: 152.21 ($C_{arom}OH$); 124.06 ($C_{arom}CH$); 123.43 ($C_{arom}H$); 108.16 ($C_{arom}CH_2$); 102.04 ($\underline{C}H(OMe)_2$); 58.22 (NCH_2); 55.36 ($ArCH_2$); 53.75 (OMe); 42.49 (NMe); 35.34 (CH); 22.51 (Me$\underline{C}H_2$); 14.10 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetrapropylpentacyclo[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 (3c) was obtained analogously from calixarene **1c** (1.57 g, 2.4 mmol), acetal **2** (1.43 g, 12 mmol), and 30% formalin (1.20 g, 12 mmol). The yield was 2.06 g (73%), m.p. 102—104 °C. Found (%): C, 64.60; H, 8.67; N, 4.40. $C_{64}H_{100}N_4O_{16}$. Calculated (%): C, 65.08; H, 8.47; N, 4.75. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.95 (t, 12 H, $CH_2\underline{Me}$, *J* = 7.0 Hz); 1.26 (m, 8 H, C<u>H</u>$_2$CH$_2$CH); 2.17 (m, 8 H, MeCH$_2$C<u>H</u>$_2$); 2.31 (s, 12 H, NMe); 2.60 (br.m, 8 H, NC<u>H</u>$_2$CH); 3.33 (s, 24 H, OMe); 3.85 (br.s, 8 H, $C_{arom}CH_2N$); 4.31 (br.m, 4 H, $CH_2C\underline{H}(OMe)_2$); 4.51 (br.m, 4 H, $C_{arom}C\underline{H}Me$); 7.13 (s, 4 H, *m*-CH_{arom}); 8.25 (br.s, 8 H, HO). ^{13}C NMR, δ: 152.13 ($C_{arom}OH$); 124.18 ($C_{arom}CH$); 123.55 ($C_{arom}H$); 108.16 ($C_{arom}CH_2$); 102.12 ($\underline{C}H(OMe)_2$); 58.30 (NCH_2); 55.41 ($ArCH_2$); 53.79 (OMe); 42.51 (NMe); 35.64 (CH); 32.93 ($\underline{C}H_2CH_2CH$); 22.19 ($\underline{C}H_2CH$); 14.12 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetrapentylpentacyclo[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 (3d) was obtained analogously from calixarene **1d** (4.95 g, 6.44 mmol), acetal **2** (3.83 g, 32.22 mmol), and 30% formalin (3.22 g, 32.22 mmol). The yield was 6.76 g (81.2%), m.p. 108—110 °C. Found (%): C, 66.51; H, 9.17; N, 4.15. $C_{72}H_{116}N_4O_{16}$. Calculated (%): C, 66.87; H, 8.98; N, 4.33. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.86 (t, 12 H, $CH_2\underline{Me}$, *J* = 7.1 Hz); 1.22 (m, 24 H, $(C\underline{H}_2)_3CH_2$); 2.19 (m, 8 H, CHC<u>H</u>$_2$); 2.30 (s, 12 H, NMe); 2.61 (d, 8 H, NC<u>H</u>$_2$CH, *J* = 5.36 Hz); 3.32 (s, 24 H, CH(O<u>Me</u>)$_2$); 3.77 (br.s, 8 H, $C_{arom}C\underline{H}_2N$); 4.27 (t, 4 H, $C_{arom}C\underline{H}CH_2$, *J* = 7.88 Hz); 4.49 (t, 4 H, C<u>H</u>$(OMe)_2$, *J* = 5.36 Hz); 7.10 (s, 4 H, *m*-CH_{arom}). ^{13}C NMR, δ: 152.11 ($C_{arom}OH$); 124.29 ($C_{arom}CH$); 123.65 ($C_{arom}H$); 108.16 ($C_{arom}CH_2$); 102.14 ($\underline{C}H(OMe)_2$); 58.30 (NCH_2); 55.41 ($ArCH_2$); 53.79 (OMe); 42.53 (NMe); 35.59 ($\underline{C}H_2CH$); 32.38 ($CH_2\underline{C}H$); 32.05 ($CH(CH_2)_2\underline{C}H_2$); 27.91 ($CHCH_2\underline{C}H_2$); 22.73 ($CH_2Me$); 14.15 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetrahexylpentacyclo[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 (3e) was obtained analogously from calixarene **1e** (5 g, 6.07 mmol), acetal **2** (3.61 g, 30.34 mmol), and 30% formalin (3.03 g, 30.34 mmol). The yield was 7.60 g (93%), m.p. 136—138 °C. Found (%): C, 67.79; H, 9.14; N, 3.86. $C_{76}H_{124}N_4O_{16}$. Calculated (%): C, 67.66; H, 9.20; N, 4.15. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.87 (t, 12 H, $CH_2\underline{Me}$, *J* = 5.87 Hz); 1.27 (m, 32 H, $(C\underline{H}_2)_4Me$); 2.14 (m, 8 H, CHC<u>H</u>$_2$CH$_2$); 2.26 (s, 12 H, N<u>Me</u>); 2.59 (br.s, 8 H, NC<u>H</u>$_2$CH); 3.28 (s, 24 H, CH(O<u>Me</u>)$_2$); 3.72 (br.s, 8 H, $C_{arom}C\underline{H}_2N$); 4.26 (m, 8 H, $C_{arom}C\underline{H}CH_2$, C<u>H</u>$(OMe)_2$); 7.07 (s, 4 H, *m*-$CH_{arom}$). ^{13}C NMR, δ: 152.18 ($C_{arom}OH$); 123.69 ($C_{arom}CH$); 122.89 ($C_{arom}H$); 108.03 ($C_{arom}CH_2$); 101.91 ($\underline{C}H(OMe)_2$); 58.07 (NCH_2); 55.21 ($ArCH_2$); 53.82 (OMe); 42.35 (NMe); 34.41 ($\underline{C}H_2CH$); 33.62

($CH_2\underline{C}H$); 31.94 ($CH(CH_2)_2\underline{C}H_2$); 29.45 ($CH(CH_2)_3\underline{C}H_2$); 28.15 ($CHCH_2\underline{C}H_2$); 22.68 ($\underline{C}H_2Me$); 14.06 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-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 (3f) was obtained analogously from calixarene **1f** (5 g, 5.68 mmol), acetal **2** (3.38 g, 28.41 mmol), and 30% formalin (2.84 g, 28.41 mmol). The yield was 7 g (87.7%), m.p. 116—118 °C. Found (%): C, 68.32; H, 9.60; N, 4.18. $C_{80}H_{132}N_4O_{16}$. Calculated (%): C, 68.38; H, 9.40; N, 3.99. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.92 (t, 12 H, $CH_2\underline{Me}$, J = 7.00 Hz); 1.34 (m, 40 H, $(C\underline{H}_2)_5CH_3$); 2.19 (br.m, 8 H, $CHC\underline{H}_2$); 2.34 (s, 12 H, NMe); 2.63 (d, 8 H, $NC\underline{H}_2CH$, J = 5.38 Hz); 3.37 (s, 24 H, OMe); 3.72 (m, 8 H, $C_{arom}C\underline{H}_2N$); 4.32 (t, 4 H, $C_{arom}C\underline{H}CH_2$, J = 7.77 Hz); 4.54 (t, 4 H, $CH_2C\underline{H}(OMe)_2$, J = 5.38 Hz); 7.15 (s, 4 H, m-CH_{arom}). ^{13}C NMR, δ: 150.30 ($C_{arom}OH$); 124.28 ($C_{arom}CH$); 123.57 ($C_{arom}H$); 108.13 ($C_{arom}CH_2$); 58.26 (NCH_2); 55.39 ($ArCH_2$); 54.02 (OMe); 53.79 (OMe); 42.51 (NMe); 33.32 (CH); 33.61 ($CH\underline{C}H_2$); 31.92 ($CH(CH_2)_4\underline{C}H_2$); 29.75 ($CH(CH_2)_2\underline{C}H_2$); 29.42 ($CH(CH_2)_3\underline{C}H_2$); 28.22($CHCH_2\underline{C}H_2$); 22.64 ($Me\underline{C}H_2$); 14.10 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetraoctylpentacyclo[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 (3g) was obtained analogously from calixarene **1g** (3.0 g, 3.2 mmol), acetal **2** (1.9 g, 15.97 mmol), and 30% formalin (1.60 g, 15.97 mmol). The yield was 4.45 g (95%), an oily substance. Found (%): C, 68.82; H, 9.49; N, 3.77. $C_{84}H_{140}N_4O_{16}$. Calculated (%): C, 69.04; H, 9.59; N, 3.84. ^{1}H NMR, δ: 0.88 (t, 12 H, $CH_2\underline{Me}$, J = 6.57 Hz); 1.26 (m, 48 H, $CH_2(C\underline{H}_2)_6Me$); 2.16 (m, 8 H, $CHC\underline{H}_2CH_2$); 2.30 (s, 12 H, NMe); 2.60 (br.m, 8 H, $NC\underline{H}_2CH$); 3.33 (s, 24 H, OMe); 3.76 (br.m, 8 H, $C_{arom}C\underline{H}_2N$); 4.28 (t, 4 H, $C_{arom}C\underline{H}CH_2$, J = 7.80 Hz); 4.50 (m, 4 H, $CH_2C\underline{H}(OMe)_2$); 7.12 (s, 4 H, m-CH_{arom}). ^{13}C NMR, δ: 152.11 ($C_{arom}OH$); 124.27 ($C_{arom}CH$); 123.61 ($C_{arom}H$); 108.14 ($C_{arom}CH_2$); 102.15 ($CH(OMe)_2$); 58.30 (NCH_2); 55.39 ($ArCH_2$); 53.79 (OMe); 42.51 (NMe); 33.33 (CH); 33.63 ($CH\underline{C}H_2$); 31.91 ($CH(CH_2)_4\underline{C}H_2$); 29.78 ($CH(CH_2)_2\underline{C}H_2$); 29.39 ($CH(CH_2)_3\underline{C}H_2$); 28.25 ($CHCH_2\underline{C}H_2$); 22.70 ($Me\underline{C}H_2$); 14.12 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetrananylpentacyclo[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 (3h) was obtained analogously from calixarene **1h** (3 g, 3.02 mmol), acetal **2** (1.80 g, 15.12 mmol), and 30% formalin (1.51 g, 15.12 mmol). The yield was 3.68 g (79%), m.p. 72—74 °C. Found (%): C, 69.96; H, 9.83; N, 3.58. $C_{90}H_{148}N_4O_{16}$. Calculated (%): C, 70.13; H, 9.61; N, 3.64. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.85 (t, 12 H, $CH_2\underline{Me}$, J = 6.57 Hz); 1.24 (m, 56 H, $CH_2(C\underline{H}_2)_7Me$); 2.19 (m, 8 H, $CHC\underline{H}_2CH_2$); 2.31 (s, 12 H, $N\underline{Me}$); 2.64 (br.m, 8 H, $NC\underline{H}_2CH$); 3.34 (s, 24 H, $CH(O\underline{Me})_2$); 3.78 (br.m, 8 H, $C_{arom}C\underline{H}_2N$); 4.29 (t, 4 H, $C_{arom}C\underline{H}CH_2$, J = 7.80 Hz); 4.55 (m, 4 H, $CH_2C\underline{H}(OMe)_2$); 7.11 (s, 4 H, m-CH_{arom}). ^{13}C NMR, δ: 152.37 ($C_{arom}OH$); 124.14 ($C_{arom}CH$); 122.39 ($C_{arom}H$); 108.53 ($C_{arom}CH_2$); 102.57 ($\underline{C}H(OMe)_2$); 58.71 (NCH_2); 55.77 ($ArCH_2$); 54.13 (OMe); 42.88 (NMe); 34.03 ($CH\underline{C}H_2$); 33.70 (CH); 32.31 ($CH(CH_2)_4\underline{C}H_2$); 30.16, 30.14, 30.05, 29.71, 28.61 ($CH(\underline{C}H_2)_nCH_2$); 23.04 ($Me\underline{C}H_2$); 14.45 (Me).

4,6,10,12,16,18,22,24-Octahydroxy-5,11,17,23-tetrakis[(2,2-dimethoxyethyl)methylaminomethyl]-2,8,14,20-tetraundecylpentacyclo[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 (3i) was obtained analogously from calixarene **1i** (5 g, 4.5 mmol), acetal **2** (2.69 g, 22.6 mmol), and 30% formalin (2.26 g, 22.6 mmol). The yield was 7.33 g (98%), m.p. 66—68 °C. Found (%): C, 71.46; H, 10.35; N, 3.55. $C_{98}H_{164}N_4O_{16}$. Calculated (%): C, 71.17; H, 9.92; N, 3.38. IR, ν/cm^{-1}: 1610 (CH_{arom}); 3300 (OH). ^{1}H NMR, δ: 0.87 (t, 12 H, $CH_2\underline{Me}$, J = 6.57 Hz); 1.29 (m, 72 H, $CH_2(C\underline{H}_2)_9Me$); 2.14 (m, 8 H, $CHC\underline{H}_2CH_2$); 2.29 (s, 12 H, NMe); 2.58 (br.m, 8 H, $NC\underline{H}_2CH$); 3.31 (s, 24 H, $CH(O\underline{Me})_2$); 3.76 (br.m, 8 H, $C_{arom}CH_2N$); 4.29 (t, 4 H, $C_{arom}C\underline{H}CH_2$, J = 7.80 Hz); 4.44 (m, 4 H, $CH_2C\underline{H}(OMe)_2$); 7.12 (s, 4 H, C_{arom}). ^{13}C NMR, δ: 152.12 ($C_{arom}OH$); 124.29 ($C_{arom}CH$); 123.57 ($C_{arom}H$); 108.14 ($C_{arom}CH_2$); 102.14 ($\underline{C}H(OMe)_2$); 58.30 (NCH_2); 55.41 ($ArCH_2$); 53.78 (OMe); 42.53 (NMe); 33.66 ($CH\underline{C}H_2$); 33.35 (CH); 31.96 ($CH(CH_2)_4\underline{C}H_2$); 29.86, 29.80, 29.77, 29.74, 29.41, 28.29, 28.22 ($CH(CH_2)_7\underline{C}H_2$); 22.70 ($Me\underline{C}H_2$); 14.10 (Me).

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