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Dedicated to Full Member of the Russian Academy of Sciences V.I. Minkin on his 80th anniversary

Synthesis and Aggregation Properties of Thiacalix[4]arene Tetra-*N*-acylamides

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Abstract—First 1,3-*alternate p-tert*-butylthiacalix[4]arenes containing *N*-acylamide fragments have been synthesized by reaction of thiacalix[4]arene carboxylic acid chlorides with 2-substituted 4,5-dihydro-1,3-oxazoles, and their aggregation properties have been studied.

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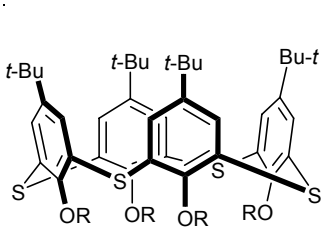
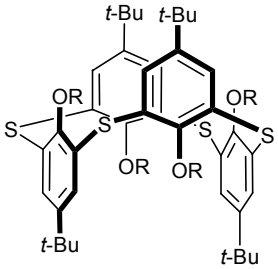
A widely used approach for the design of highly efficient and selective receptors in supramolecular chemistry implies that binding sites are fixed on a macrocyclic scaffold to ensure their required spatial arrangement. Spatially preorganized receptor structure favors enhanced binding selectivity, and the stability of the resulting complexes increases many times due to so-called macrocyclic effect [1]. In addition, the presence of several coordination sites in receptor molecules could give rise to polynuclear complexes and clusters [2].

As molecular scaffold for the synthesis of nanosized multidentate ligands we used thiacalix[4]arene which offers unique functionalization potential. The main advantages of thiacalix[4]arene scaffold for the design of multidentate ligands include the possibility for one-step preparation of initial macrocycles, the existence of thiacalix[4]arene molecules as four stereoisomers **A**–**D** capable of fixing a required spatial orientation of the binding sites, and the presence of sulfide bridges as additional coordination sites for transition metal cations [3–5].

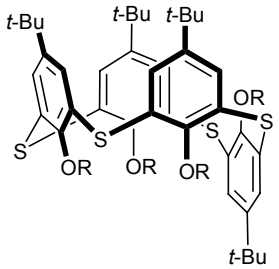
2-Substituted 4,5-dihydrooxazoles **1** are cyclic imido esters which undergo ring opening in reactions with nucleophiles or electrophiles with formation of polyoxazolines (as a result of cationic polymerization) that are nontoxic biodegradable compounds promising as pseudopeptides [6]. The dihydrooxazole ring in **1** can be opened by the action of such electrophiles as Lewis acids, strong protic acids and their esters, benzyl and alkyl halides, dihydrooxazolium salts, methyl iodide, methyl *p*-toluenesulfonate, methyl trifluoromethanesulfonate, dialkyl sulfate [7], and acyl halides [8]. Due to different electrophilicites of acyl halide and oxazolinium intermediate **2**, the reactions of acyl halides with 4,5-dihydro-1,3-oxazoles **1a**–**1c** can be



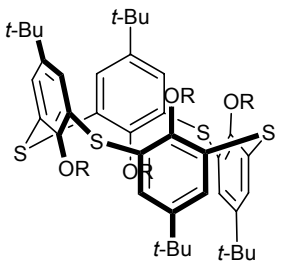
Cone (**A**)



1,3-*Alternate* (**B**)

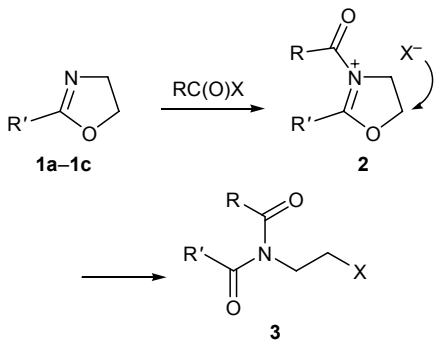


Partial cone (**C**)



1,2-*Alternate* (**D**)

Scheme 1.



R′ = Me (**a**), Et (**b**), Ph (**c**).

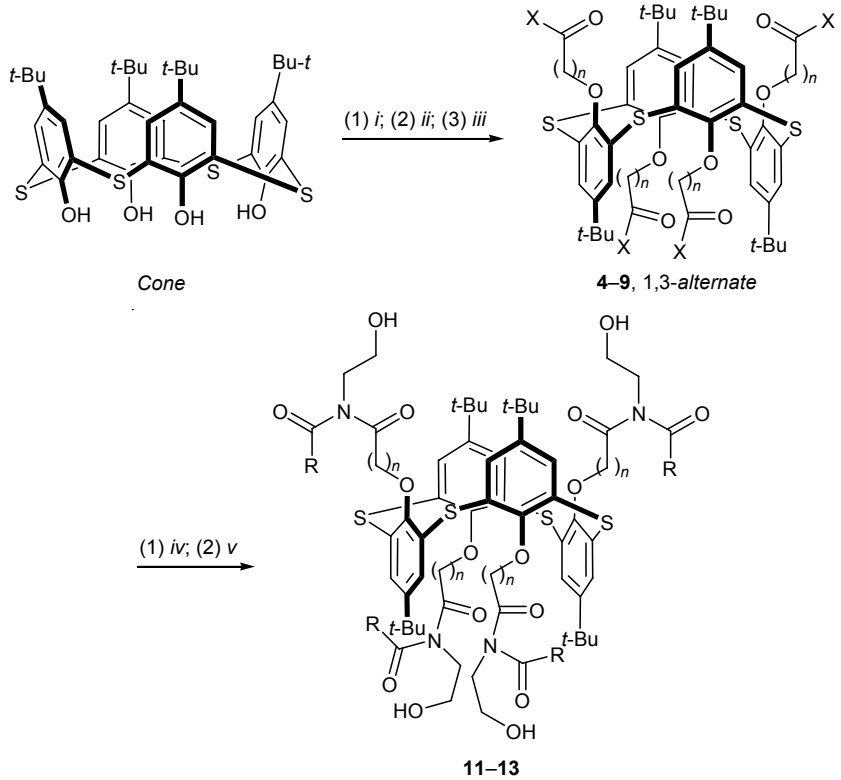
stopped after addition of one acyl halide molecule with formation of *N*-acylamides **3** (Scheme 1).

Up to now, no data have been reported on the use of calixarene derivatives as initiators of oxazoline ring opening or polymerization of oxazolines. Calixarene derivatives can be used as potential receptors and complexing agents, molecular building blocks for the synthesis of coordination polymers, and synthetic precursors for further transformations.

In the present work we selected thiacalix[4]arene tetracarbonyl chlorides in the 1,3-*alternate* conformation as electrophilic agents for opening of the oxazoline ring (Scheme 2). The synthesis of tetracarboxylic acid **6** (1,3-*alternate*) in two steps was described previously [9, 10]. In this work we performed the synthesis of **6** under microwave irradiation; the yields in both steps were comparable with those reported in [9, 10], but the reaction time was shortened from 24 to 3–3.5 h. Acid chloride **8** (1,3-*alternate*) was obtained in quantitative yield by heating acid **6** in thionyl chloride, followed by removal of excess thionyl chloride under reduced pressure. Compound **8** was used without additional purification. The reaction of **8** with 2-methyl-4,5-dihydro-1,3-oxazole (**1a**) was carried out at a reactant ratio of 1:5 under solvent-free conditions (reaction time 2 h).

The ^{1}H NMR and MALDI TOF data indicated that the major product was that resulting from $SOCl_2$-initiated polymerization of **1a** since thionyl chloride is a stronger electrophile and more efficient initiator of oxazoline ring opening than carboxylic acid chloride. The MALDI TOF mass spectrum contained only peaks

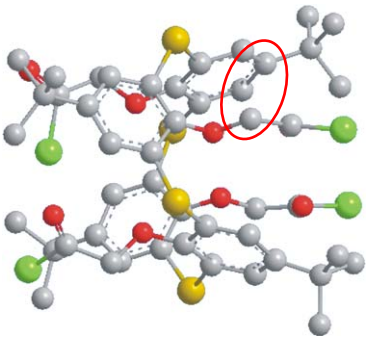
Scheme 2.



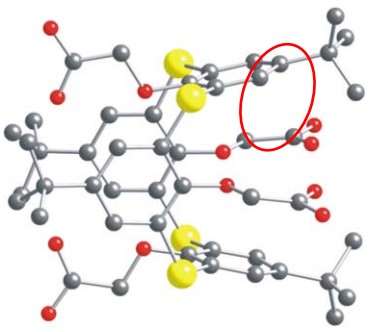
4, **6**, **8**, *n* = 1; **5**, **7**, **9**, **11**–**13**, *n* = 3; **4**, **5**, X = OEt; **6**, **7**, X = OH; **8**, **9**, X = Cl; **11**, R = Me; **12**, R = Et; **13**, R = Ph; *i*: $Br(CH_2)_nCOOEt$, Cs_2CO_3, acetone; X = OEt; *ii*: THF–H_2O, LiOH (**4** → **6**) or EtOH–H_2O, KOH (**5** → **7**); *iii*: $SOCl_2$; *iv*: **1a**–**1c**, KI, MeCN; *v*: $Na_2S_2O_3$, H_2O.

corresponding to oxazoline polymerization products (*m*/*z* 359–785, 2–7 units) and initial macrocycle (*m*/*z* 1028). Presumably, macrocycle **8** with thionyl chloride forms stable host–guest complexes which do not decompose on distillation under reduced pressure. In order to remove traces of thionyl chloride and decompose inclusion complexes, the mixture was heated in boiling benzene with subsequent azeotropic distillation. After removal of thionyl chloride, no polymerization products were detected.

The reactions of thiacalixarene tetracarbonyl chloride **8** with 2-methyl-4,5-dihydro-1,3-oxazole (**1a**) were carried out under different conditions. Methylene chloride and its mixture with hexane, acetonitrile, chloroform, DMF, and THF–acetonitrile were used as solvent, the temperature was varied from 40 to 153°C, and the reaction time was 0.5–3 h (until initial compound **1a** disappeared according to the TLC data). However, in no case individual compounds were isolated. The ^{1}H NMR spectra of the reaction mixtures contained numerous signals from *tert*-butyl groups in the region δ 1.22–1.26 ppm and aromatic protons in the calixarene macrocycle. These findings, as well as the MALDI TOF mass spectra, showed that mixtures of partially and completely substituted compounds were formed in the reactions of **8** with dihydrooxazole **1a**. A probable reason is steric shielding of the reaction centers in molecule **8** by the neighboring *tert*-butyl groups.



8



6

Structures of acid chloride **8** according to semiempirical calculations (molecular mechanics, MMPlus) and of acid **6** according to the X-ray diffraction data [12].

The *tert*-butyl groups in thiacalix[4]arenes with a short spacer (one methylene group) in the 1,3-*alternate* conformation create hindrances to reaction with the C(O)Cl groups and formation of compound **10**. This follows from the results of semiempirical calculations of the structure of **8** (see figure) [11] and X-ray diffraction data for related compounds (acid **6** [12] and ester **4** [13]). It is seen that the *tert*-butyl and C(O)Cl groups appear close to each other, which may reduce the reactivity of electrophilic centers.

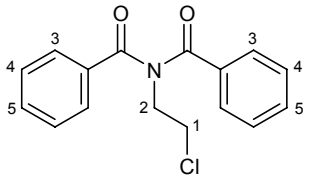
In order to reduce steric hindrances, we have synthesized acid chloride **9** (1,3-*alternate*) with a longer spacer (three methylene groups). Tetraester **5** was prepared in 58% yield from *p*-*tert*-butylthiacalix[4]arene and ethyl 4-bromobutanoate in acetone in the presence of cesium carbonate (60 h on heating). Under microwave irradiation (350 W), the reaction time was shortened to 3.5 h. Tetraester **5** was hydrolyzed to acid **7** (yield 92%) by treatment with potassium hydroxide in aqueous ethanol, followed by acidification, and acid **7** was converted into acid chloride **9** by reaction with thionyl chloride as in the synthesis of **8**.

By heating a suspension of acid chloride **9**, dihydrooxazole **1a**, and KI in acetonitrile at a molar ratio of 1:5:8 for 3 h we obtained pure compound **11** in 43% yield. In the ^{1}H NMR spectrum of **11**, protons of the *tert*-butyl groups resonated as a singlet at δ 1.23 ppm, aromatic protons gave a singlet at 7.33 ppm, methylene proton signals appeared as two triplets at δ 1.44 and 1.99 ppm, and the singlet at δ 2.06 ppm was assigned to protons in the acetyl group. This spectrum corresponds to the symmetric 1,3-*alternate* structure.

The reactions of **9** with 2-ethyl- and 2-phenyl-4,5-dihydro-1,3-oxazoles **1b** and **1c** afforded compounds **12** and **13** which were isolated by column chromatography in 79 and 20% yield, respectively. The ^{1}H NMR spectrum of **12** was analogous to the spectrum of **11** with the difference that a triplet at δ 1.44 ppm and a quartet at δ 2.33 ppm (CH_3CH_2) were present instead of methyl proton singlet at δ 2.06 ppm. The ^{1}H NMR spectrum of **13** was more complex: the *tert*-butyl groups gave two closely located singlets, aromatic protons appeared as a multiplet at δ 7.28–7.55 ppm and two triplets at δ 7.77 and 8.01 ppm (*o*-H), and methylene protons resonated as two pairs of triplets at δ 1.36 and 1.46 ppm. The MALDI TOF mass spectra of **11**–**13** were consistent with the assumed structures.

Molecules **11**–**13** contain 2-hydroxyethyl fragment instead of 2-chloroethyl. This unambiguously follows from the IR data (νOH 3307–3380 cm^{-1}) and high-resolution MALDI TOF mass spectra (*m*/*z* 1406, 1460, and 1652, respectively). Substitution of the chlorine atom by hydroxy group is likely to occur during the isolation procedure when the reaction mixture was treated with an aqueous solution of sodium thiosulfate to remove liberated iodine.

Imide **14** isolated from the reaction mixture according to the same procedure retained the terminal chlorine atom (MALDI TOF, *M* 286).



14

It is known that thiacalixarenes with water molecules form stable inclusion compounds [14]. As shown by X-ray analysis, even unsubstituted thiacalix[4]arene gives a complex with water in a mixture of methylene chloride with methanol and a few drops of water, which is very rarely observed for water-insoluble host compounds (probability 1:20000) [15]. Presumably, in the reaction with thiacalixarene derivative the chlorine atom is replaced by water molecule residing in the calixarene cavity (supramolecular catalysis).

A characteristic feature of polyfunctional calixarene derivatives is their ability to form aggregates in various media [16]. It is very important to consider aggregation while estimating the complexing power of ligands since their self-association could reduce the efficiency of their interactions with guest molecules [17, 18]. Compounds **11**–**13** possess *N*-acylamide fragments and hydroxy groups which favor self-association via intermolecular hydrogen bonding.

The aggregation of macrocycles **11**–**13** was studied in methylene chloride in the concentration range from 10^{-4} to 10^{-2} M by the dynamic light scattering technique. The sizes of particles formed in solution are given in table. No aggregation was observed for compound **11** even in a 10^{-2} M solution, whereas *N*-propionyl and *N*-benzoyl derivatives **12** and **13** turned out to be more prone to self-association. Replacement of the methyl group by more lipophilic ethyl or phenyl changes the hydrophilic–lipophilic balance and enhances amphiphilic properties of macrocycles **12** and **13**. Therefore, variation of the substituent in the 2-position of initial dihydrooxazole derivatives may be used to control the complexing ability and amphipilic properties of the resulting compounds, which is important for purposeful design of supramolecular systems based thereon.

Hydrodynamic radii of compounds **11**–**13** in methylene chloride

Compound no.	Concentration, M	Hydrodynamic radius, nm/polydispersity index
11	10^{-2}	No aggregation
	10^{-3}	No aggregation
12	10^{-2}	111±2/0.25±0.07
	10^{-3}	83±4/0.16±0.02
	10^{-4}	No aggregation
13	10^{-2}	58±2/0.24±0.03
	10^{-3}	No aggregation

In summary, we were the first to synthesize tetra-*N*-acyl amide derivatives of *p*-*tert*-butylthiacalix[4]arene in the 1,3-*alternate* conformation as promising polyfunctional ligands for both host–guest complexation with organic substrates (including biologically important ones) and formation of nanoparticles possessing numerous surface binding sites.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Bruker Avance 600 instrument (600.13 MHz); the chemical shifts were measured relative to the residual proton signals of deuterated solvents. The mass spectra were obtained on a Bruker Ultraflex MALDI-TOF mass spectrometer using *p*-nitroaniline or 2,5-dihydroxybenzoic acid as matrix. The IR spectra were recorded in the region 400–4000 cm^{-1} from samples placed between KBr plates on a Bruker Vector 22 spectrometer with Fourier transform (resolution 1 cm^{-1}; scan number 64).

The solvents and reactants were purified according to known procedures [19] prior to use. Commercial 2-methyl-, 2-ethyl-, and 2-phenyl-4,5-dihydro-1,3-oxazoles **1a**–**1c** (99%, Aldrich) were used without additional purification. The purity of the isolated compounds was checked by TLC on Silufol UV-254 and Fluka plates (0.060–0.2 mm); spots were visualized by irradiation with a VL-6.LC lamp (6W, λ 254 nm) or (in some cases) by treatment with iodine vapor. The melting points were determined on a Boetius PHMK 05 microscope.

Microwave-assisted syntheses were carried out in a CEMMARS Xtraction microwave reactor.

For dynamic light scattering experiments, solutions of compounds **11**–**13** in methylene chloride (HPLC grade, LAB-SCAN) with required concentrations were prepared and filtered through a Millipore filter to remove dust particles. The dynamic light scattering data were obtained on a Malvern Zetasizer Nano instrument and analyzed using Malvern Dispersion Technology Software 5.10.

Tetraethyl 2,2′,2″,2‴-[5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis(oxy)]tetraacetate (1,3-*alternate*) (4). A thick-walled glass reactor was charged with 0.40 g (0.56 mmol) of 5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetraol, 0.74 g (2.27 mmol) of freshly calcined cesium carbonate, 0.2 mL (2.2 mmol) of ethyl bromoacetate, and 0.018 g (0.056 mmol) of tetrabutylammonium bromide in 5 mL of anhydrous acetone. The mixture was stirred for 30 min at room temperature and kept for 3 h in a microwave reactor (300 W). Yield 81%. The physical constants of the product were consistent with those reported in [9, 10].

2,2′,2″,2‴-[5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis-(oxy)]tetraacetatic acid (1,3-*alternate*) (6). A thick-walled glass reactor was charged with 3.82 g (3.6 mmol) of compound **4** and 1.24 g (52 mmol) of lithium hydroxide in a mixture of 22 mL of THF and 15 mL of water, and the mixture was kept for 3.5 h in a microwave reactor (350 W). Yield 96%. The physical constants of the product were consistent with those reported in [10].

Tetraethyl 4,4′,4″,4‴-[5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis(oxy)]tetrabutanoate (1,3-*alternate*) (5). *a*. A mixture of 3.00 g (4.17 mmol) 5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetraol, 6.55 g (33.3 mmol) of ethyl 4-bromobutanoate, and 8.16 g (25 mmol) of cesium carbonate in 100 mL of anhydrous acetone was heated for 72 h under reflux while stirring in an argon atmosphere. The mixture was filtered, and the filtrate and precipitate were treated separately. The filtrate was concentrated under reduced pressure, and 50 mL of methanol was added to the residue to isolate 2.25 g (46%) of **5**. The precipitate was treated with water (80 mL) and chloroform (2×50 mL). The organic layer was separated and dried over $MgSO_4$, the solvent was removed on a rotary evaporator, and the residue, 0.27 g (5%) was recrystallized from methanol. Overall yield 51%, mp 160°C. IR spectrum, ν, cm^{-1}: 2963 (C–H_{arom}), 1733 (C=O), 1268 (COC). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 t (12H, OCH_2CH_3, J = 8.0 Hz), 1.26 s (36H, *t*-Bu), 1.39 m (8H, $OCH_2CH_2CH_2$), 2.11 t (8H, $OCH_2CH_2CH_2$, J = 8.0 Hz), 3.91 t (8H, $OCH_2CH_2CH_2$, J = 8.0 Hz), 4.10 q (8H, OCH_2CH_3, J = 8.0 Hz), 7.36 s (8H, H_{arom}).

b. A thick-walled glass reactor was charged with a mixture of 0.2 g (0.28 mmol) of 5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetraol, 0.57 g (1.12 mmol) of cesium carbonate, 0.2 mL (1.4 mmol) of ethyl 4-bromobutanoate, and 0.01 g (0.031 mmol) of tetrabutylammonium bromide in 3 mL of acetone, and the mixture was subjected to microwave irradiation (350 W) for 3.5 h. When the reaction was complete, the product was isolated as described above in *a*. Yield 54%.

4,4′,4″,4‴-[5,11,17,23-tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis-(oxy)]tetrabutanoic acid (1,3-*alternate*) (7). A mixture of 2.2 g (1.9 mmol) of compound **5** and 2.1 g (37.5 mmol) of potassium hydroxide in ethanol–water (4:1) was stirred for 7.5 h on heating under reflux. The mixture was cooled to room temperature, 40 mL of aqueous HCl (1:3) was added to pH 1, and the white precipitate was filtered off. Yield 1.83 g (92%), white powder, mp 305°C. IR spectrum, ν, cm^{-1}: 3237 (OH), 2961 (C–H_{arom}), 1708 (C=O), 1264 (COC). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 1.18 m (8H, $OCH_2CH_2CH_2$), 1.21 s (36H, *t*-Bu), 1.99 t (8H, $OCH_2CH_2CH_2$, J = 8.0 Hz), 3.79 t (8H, $OCH_2CH_2CH_2$, J = 8.0 Hz), 7.33 s (8H, H_{arom}).

Acid chlorides 8 and 9 (*general procedure*). A 0.07 M solution of acid **6** or **7** in thionyl chloride was heated for 2 h under reflux. Excess thionyl chloride was removed, 20 mL of anhydrous benzene was added, and residual thionyl chloride was removed by azeotropic distillation. The residue was dried under reduced pressure. Compounds **8** and **9** were isolated as white powders in quantitative yield and were used in further syntheses without additional purification.

Reaction of acid chlorides 8 and 9 with 2-substituted 4,5-dihydro-1,3-oxazoles 1a–1c (*general procedure*). Potassium iodide was added to a solution of acid chloride **8** or **9** in acetonitrile (0.8 M, ratio **8** (**9**)–KI 1:8), and the resulting solution was added dropwise under stirring to a solution of 5 equiv of **1a**–**1c** in acetonitrile (0.8 M). The mixture was heated for 2–5 h

under reflux, the precipitate was filtered off, and the filtrate was evaporated. The residue was treated with an aqueous solution of $Na_2S_2O_3$, the mixture was stirred for 2 h at room temperature, and the precipitate was filtered off, washed with water, and dried under reduced pressure (water-jet pump).

4,4′,4″,4‴-[5,11,17,23-Tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis-(oxy)]tetrakis[*N*-acetyl-*N*-(2-hydroxyethyl)butanamide] (1,3-*alternate*) (11). Yield 58%, mp 179°C. IR spectrum, ν, cm^{-1}: 3307 (OH), 2957 (C–H_{arom}, *t*-Bu), 1741 (C=O), 1650 (C=O), 1263 (COC). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.23 s (36H, *t*-Bu), 1.44 t (8H, OCH_2CH_2OH, J = 8.0 Hz), 1.68 br.s (4H, OH), 1.99 t (8H, OCH_2CH_2OH, J = 8.0 Hz), 2.06 s (12H, Me), 3.45 m (8H, $OCH_2CH_2CH_2$), 3.88 t (8H, $OCH_2CH_2CH_2$, J = 8.0 Hz), 4.12 t (8H, $OCH_2CH_2CH_2$, J = 4.0 Hz), 7.33 s (8H, H_{arom}). Mass spectrum, *m*/*z*: 1427 $[M + Na]^+$, 1443 $[M + K]^+$. $C_{72}H_{102}N_4O_{16}S_4$. Calculated: M 1406.

4,4′,4″,4‴-[5,11,17,23-Tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis-(oxy)]tetrakis[*N*-(2-hydroxyethyl)-*N*-(1-oxopropyl)-butanamide] (1,3-*alternate*) (12). Yield 79%, mp 158°C. IR spectrum, ν, cm^{-1}: 3369 (OH), 2962 (C–H_{arom}, *t*-Bu), 1739 (C=O), 1649 (C=O), 1267 (COC). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.14 t (12H, CH_2CH_3, J = 8.0 Hz), 1.23 s (36H, *t*-Bu), 1.44 t (8H, OCH_2CH_2OH, J = 8.0 Hz), 1.77 br.s (4H, OH), 2.06 t (8H, OCH_2CH_2OH, J = 8.0 Hz), 2.33 q (8H, CH_2CH_3), 3.46 m (8H, $OCH_2CH_2CH_2$), 3.89 t (8H, $OCH_2CH_2CH_2$, J = 8.0 Hz), 4.14 t (8H, $OCH_2CH_2CH_2$, J = 4.0 Hz), 7.35 s (8H, H_{arom}). Mass spectrum, *m*/*z*: 1483 $[M + Na]^+$. $C_{76}H_{108}N_4O_{16}S_4$. Calculated: M 1460.

4,4′,4″,4‴-[5,11,17,23-Tetra-*tert*-butyl-2,8,14,20-tetrathiacalix[4]arene-25,26,27,28-tetrayltetrakis-(oxy)]tetrakis[*N*-benzoyl-*N*-(2-hydroxyethyl)butanamide] (1,3-*alternate*) (13). The precipitate was subjected to column chromatography on alumina using acetonitrile as eluent. Yield 20%, mp 90°C. IR spectrum, ν, cm^{-1}: 3380 (OH), 2961 (C–H_{arom}, *t*-Bu), 1724 (C=O), 1646 (C=O), 1270 (COC). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.18 br.s (36H, *t*-Bu), 1.35 m (8H, OCH_2CH_2OH), 2.05–2.14 m (8H, OCH_2CH_2OH), 3.60–3.68 m (8H, $OCH_2CH_2CH_2$), 3.87 m (8H, $OCH_2CH_2CH_2$), 4.25–4.38 m (8H, $OCH_2CH_2CH_2$), 7.32 s (8H, H_{arom}), 7.28–7.55 m (12H, *m*-H, *p*-H), 7.77 t (4H, *o*-H, J = 8.0 Hz), 8.01 t (4H, *o*-H, J = 8.0 Hz). Mass spectrum, *m*/*z*: 1692 $[M + K]^+$. $C_{92}H_{108}N_4O_{16}S_4$. Calculated: M 1652.

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