Synthesis and characterisation of asymmetrically bridged calix[4]arene and tetrathiacalix[4]arene mono amido crown derivatives

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The synthesis of asymmetrically bridged calix[4]arene and tetrathiacalix[4]arene amido crown derivatives has been achieved by the aminolysis of distal diester derivatives of calix[4]arene and tetrathiacalix[4]arene with 1,2-diaminopropane or 1,2-diaminocyclohexane (stereoisomeric mixture, mainly *trans*). The title compounds have been characterised by detailed analysis of their NMR spectra. The assignment of NMR signals and stereo differentiation in the amido crown ring formation has been studied using a chiral shift reagent.

Keywords: calixarene, thiacalixarene, calixamidocrowns, calixazacrowns, chiral shift reagents

Calixarenes are phenolic macrocyclic molecules which have been extensively used to design symmetrical and asymmetrical scaffolds for obtaining molecular receptors for recognition of cations, anions and molecules.[1–8] Realisation of chiral calixarenes (with chirality principles embedded within the calixarene scaffold or outside of it) is extremely important both from the view point of resolution of racemic mixtures.[9,10] as well as recognition of anions which unlike cations are present in various symmetrical and asymmetrical shapes. Asymmetrical calixarene derivatives are important for achieving the complementarity of shape of the anions and the molecular receptors.

Several strategies have been used to achieve asymmetrical calixarenes. These strategies include the introduction of chiral substituents in the calixarene skeleton, desymmetrisation of basic calixarene cage through different substitutents at the lower or the upper rim as well as through the less explored approach that involves the incorporation of a substituent in the *meta* position of phenol ring of calixarenes.[11–13] Since calixarenes undergo rapid conformational change, establishment of asymmetry in calixarenes is a complex issue.[14,15] Very little work seems to have been published on the characterisation of inherently asymmetric calix[4]arene derivatives. In fact, characterisation of asymmetrical calixarene derivatives becomes more difficult in the presence of enantiomeric mixtures of substrates or the recognition targets.

Despite the importance of asymmetrical (thia)calixarenes and calixarene–amidocrown compounds, (useful calixarene derivatives for recognition of anions), it is significant that there has been only one report on the subject. The reported work involves a multistep synthetic route which provides asymmetric calixarene amido crown compounds in low yields. [16] On the other hand, there seems to be no precedent in the literature for the synthesis of asymmetrically bridged tetrathiacalix (amido) crown derivatives.

We report here an easy, high yield synthesis and characterisation of inherently asymmetrically bridged tetrathiacalix[4] arene and calix[4]arene amido crown derivatives through aminolysis of diester derivatives of tetrathiacalix[4]arene and calix[4]arene.

Results and discussion

The products of the reaction (Scheme 1) were identified by the analysis of their IR, ^{1}H and ^{13}C NMR spectra, FAB MS and CHN analysis. The substitution at both the ester groups could be confirmed by disappearance of characteristic absorptions for the ester groups around 1750–1775 cm^{-1} in the IR spectra. The synthesised amides showed characteristic absorptions for –C(=O)–N– (1660–1680 cm^{-1}) and –N–H (3320–3330 cm^{-1}) groups. FAB-MS spectra of **3a–d** exhibited molecular ion peaks (M$^+$+1) at 803 (for **3a**), 579 (for **3b**), 875 (for **3c**), 651 (for **3d**), 843 (for **4a**) and 619 (for **4b**) respectively which unambiguously confirmed the formation of mono amido crown derivatives.

Alhough the symmetrically bridged amido crown analogues (**5a**, **b**, Fig. 1) show much simpler NMR spectral pattern *i.e.*, a pair of doublet for methylene bridge protons as shown in our previous publication.[17] The NMR spectra of apparently asymmetrically bridged amido crown analogues (**3a–d**) were more difficult to interpret. The similarity of splitting pattern of their ^{1}H NMR spectra with those of other asymmetrical calix[4]arenes provided clues to the presence of chiral unit in these molecules.[18,19] Asymmetrically bridged calix[4]arene amido crown derivatives (**3a, 3b, 4a, 4b**) gave two signals for methylene carbons in the range of δ 29–32 ppm in ^{13}C NMR spectra which suggest that they exist in their cone conformation[20–22] thereby indicating the introduction of asymmetry in the amido crown ring. For example, methylene bridge protons in **3a** appeared as six doublets (2:2:1:1:1:1) at δ 4.28, 4.03, 3.57, 3.53, 3.42 and 3.38 respectively. The –OCH$_2$ protons merged with signals for one proton of –NHC**H**(CH$_3$)– and appeared as a triplet and a pair of doublets (1:1) at δ 4.68, 4.49 and 4.42. Similarly, both the distereotopic protons of –NHC**H**$_2$– appeared at different places as broad doublets at δ 4.17 and 3.24. (Fig. S1, see Electronic Supplementary Information, ESI)

A similar ^{1}H NMR pattern was observed for the debutylated analogue (**3b**) (Fig. S2, see ESI)**,** while in the DEPT-135 spectrum, it exhibited six signals for the aromatic –CH and two signals for methylene carbon to suggest its asymmetric structure. The NMR signals were assigned with the help of HSQC (Fig. S3, see ESI) and DQF-COSY spectrum (Fig. S4, see ESI).

It was determined that the two-dimensional ^{1}H–^{1}H correlations are extremely helpful in determining the molecular structure of **3b** as shown in Fig. S4 (ESI). The NMR spectrum of tetrathiacalix[4]arene mono amido crown derivatives **3c** and **3d** were found to be similar to their calix[4]arene analogues. The tetrathiacalix[4]arene mono-amido crown derivatives therefore were inferred to be present in their cone conformation with a chiral centre in the amide ring. For instance, compound **3c** exhibited prominent NMR signals at δ 8.83 and 8.71 for hydroxyl protons and at δ 8.34 and 8.00 for amide protons while signals for aromatic protons appeared at δ 7.66, 7.61 and 7.50 (1:1:2). The ArOCH$_2$– protons appeared at δ 4.82 (t, 2H), 4.51 (d, 1H) and 4.35(d, 1H) while NHCH$_2$ protons appeared at δ 4.18 and δ 3.26 respectively. The (CH$_3$)$_3$C- protons appeared at δ 1.18 and δ 1.11 while –CH$_3$ and –NHC**H**(CH$_3$)– appeared at δ 1.26 and δ 4.57 respectively. The complicated NMR spectral pattern observed could be interpreted with the help of NOESY spectral analysis (Fig. S5a, see ESI). The molecular structure assigned on the basis of NOESY correlations of tetrathiacalix[4]arene mono amido crown constitution

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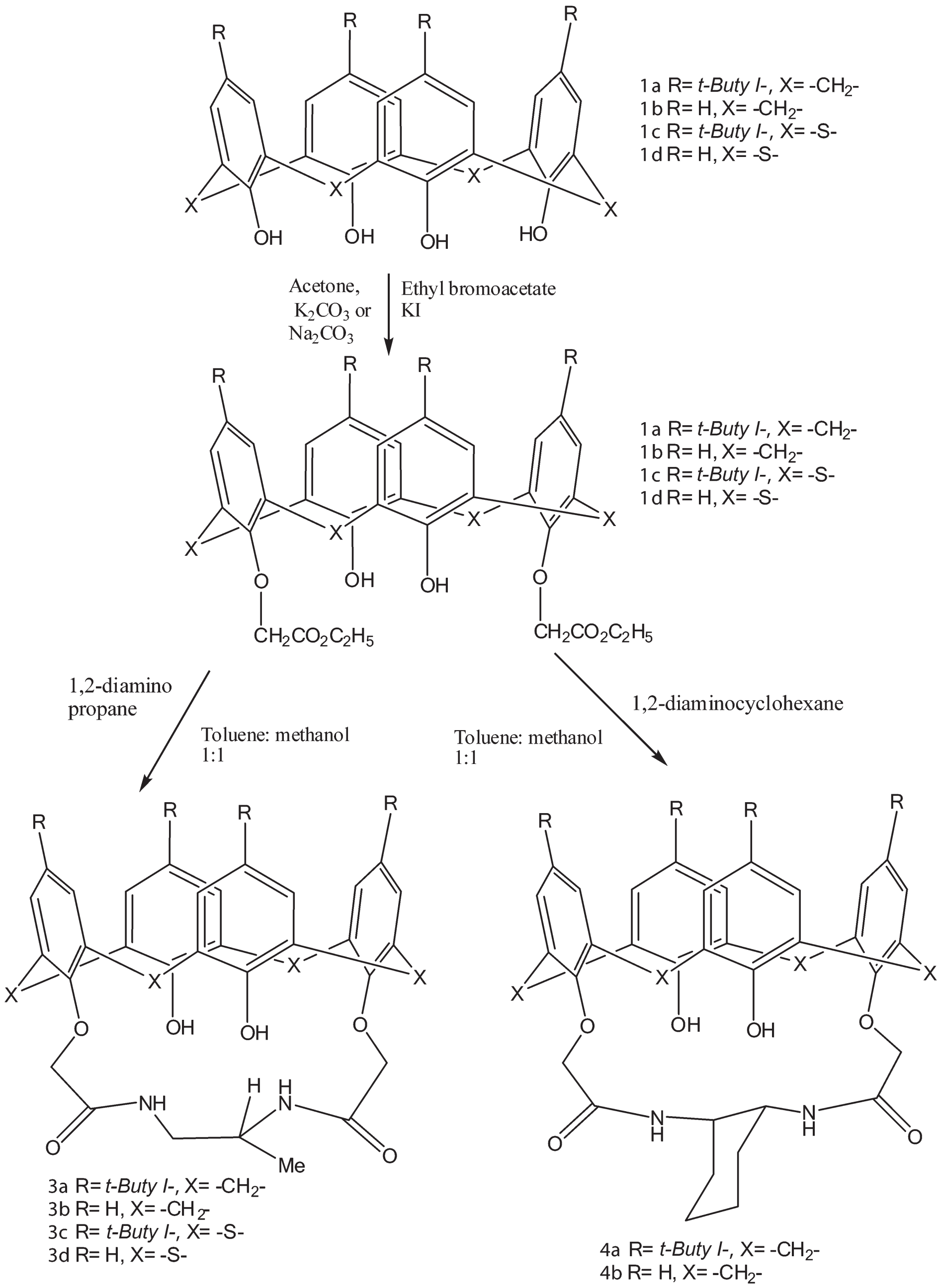

Scheme 1 Synthesis of asymmetrically bridged calix[4]arene mono amido crowns analogues (**3a–d**, **4a–b**).

as represented in Fig. S5b (ESI) was further confirmed by ^{13}C NMR spectrum (DEPT-135) of **3c** (Fig S5b, see ESI).

The NMR spectra of **4a** and **4b** gave a series of multiplets as given in the experimental section. The compounds clearly gave signals for methylene carbons in the range of δ 29–32 ppm in the ^{13}C NMR spectra which suggested that they are present in their cone conformation. The FAB MS and elemental analysis established them to be calix[4]arene mono amido crown compounds.

The aminolysis of calix[4]arene or tetrathiacalix[4]arene diethyl acetate, in principle, can lead to the formation of biscalixarenes due to the reaction of two terminal amino group. However, no trace of bis-calix[4]arenes or bis-tetrathiacalix[4] arenes could be detected.

The ^{1}H NMR spectral analysis with the help of a chiral shift reagent ytterbium(III) tris [3-(trifluoromethyl-hydroxylmethylene)-(e)-camphorato] revealed that *tert*-butyl protons of **3a** which initially appeared as two singlets (1:1 ratio) got split into six singlets (1:1:1:1:2:2 ratio) along with significant downfield shifts on addition of chiral shift reagent (Fig. 2). The appearance of six singlets for *tert*-butyl protons in 1:1:1:1:2:2 ratio could be attributed to the existence of two enantiomers, each having three different kinds of *tert*-butyl groups in 1:1:2 ratio (Fig. S3, ESI). The integration ratios of the protons also substantiated the results obtained from the optical rotation studies which confirmed the presence of two enantiomers in an almost equimolar ratio. The other protons in **3a** also showed significant downfield shifts in the NMR experiment on addition of the shift reagent. Similar observations could be discerned with **3c**. However, in the case of their debutylated analogues (**3b** and **3d**), complicated splitting pattern with significant downfield shifts could be observed in similar experiments. Due to the absence of *tert*-butyl groups in this case, we were unable to find characteristic signals for analysis of the splitting pattern but on the basis of their structural similarity with the *tert*-butyl analogue, we expect them to be a

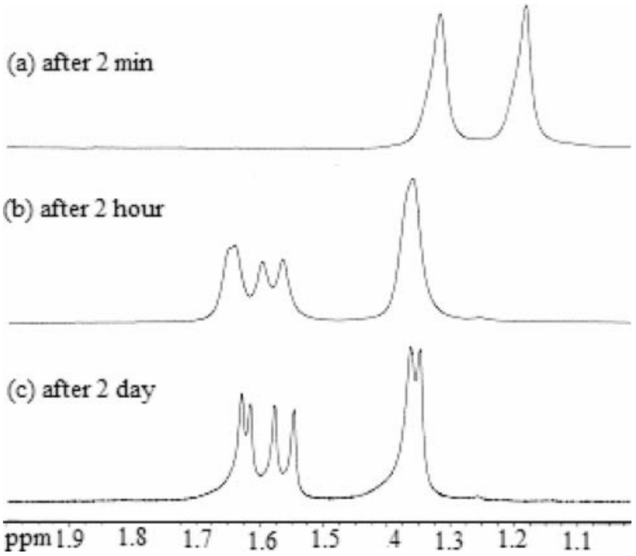

Fig. 1 Molecular structure of a symmetrically bridged calix[4] arenemono amido crown analogues.[17]

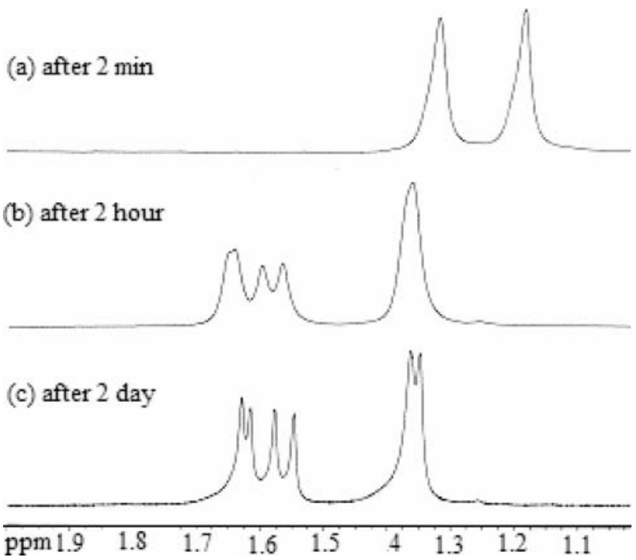

Fig. 2 Partial ^{1}H NMR spectrum (298 K, 300 MHz) of **3a** showing splitting in *tert*-butyl protons on the addition of chiral shift reagent ytterbium(III) tris [3-(trifluoromethyl-hydroxylmethylene)-(e)-camphorato] derivative (a) after 2 minutes; (b) after 2 hours; and (c) after 2 days.

racemic mixture of two enantiomers. The addition of H_2O or D_2O to the complex formed between the chiral shift reagent and calix[4]arene amido crown derivative in $CDCl_3$ resulted in a spectrum similar to the free calix[4]arene amido crown, *i.e.*, two signals for the *tert*-butyl groups of **3a**. This observation indicated that the formation of a complex between the chiral shift reagent and calixarene amido crown is a reversible process and the complexation requires anhydrous conditions to choose appropriate stereoisomer for cyclisation and to limit numerous conformational possibilities to yield the most stable amido crown compounds.

In conclusion, we have observed that special disposition of amino groups in calix[4]arene and tetrathiacalix[4]arene can induce cyclisation to give chiral calix[4]arene- and tetrathiacalix[4]arene amido crown derivatives that can be conveniently characterised by NMR spectroscopic methods. The study also suggests that cyclisation process can choose an appropriate isomer from the stereoisomeric mixture to give the most stable amido crown compounds (mainly *trans*). Further investigation to use them for resolution of target organic racemates and anion recognition for development of sensor materials is being undertaken in our laboratories.

Experimental

All the reagents used in the study were purchased from Sigma-Aldrich, Alpha Aesar, or Merck and were considered chemically pure. The solvents used were distilled. Column chromatography was performed on silica gel (60–120 mesh) obtained from Merck. ^{1}H NMR, DQF-COSY, NOESY, ^{13}C NMR, DEPT-135 and HSQC spectra were recorded on a 300 MHz Bruker DPX 300 instrument at room temperature using tetramethylsilane (TMS) at 0.00 as an internal standard. IR spectra were recorded on a Nicolet Protégé 460 spectrometer in KBr disks while the FAB mass spectra were recorded on a JEOL SX 102/DA-6000 Mass spectrometer/Data System using Argon/Xenon (6 kV, 10mA) as the FAB gas. Melting points were determined on an electrothermal melting point apparatus obtained from M/S Toshniwal and were uncorrected. Elemental analyses were carried out on a Perkin Elmer's 240C-CHN analyser.

Preparation of starting materials

p-tert-Butylcalix[4]arene **1a** and *p-tert*butyltetrathiacalix[4]arene **1c** were obtained by base catalysed condensation of *p-tert*-butylphenol with formaldehyde or sulfur as reported previously.[26,27] Their debutylated analogues (**1b, 1d**) were obtained by the $AlCl_3$ catalysed dealkylation reaction.[28,29] The synthesis of diester derivatives (**2a–d**) was achieved by the reaction of bromoethylacetate in the presence of potassium carbonate (for **2a** and **2b**)[30,31] or sodium carbonate (for **2c** and **2d**).[32]

Synthesis of calix[4]arene (amido)mono-crown derivatives; general procedure

Diesters (**2a, 2b, 2c** and **2d**) and 1,2-diaminopropane or *trans*-1,2-diaminocyclohexane (20–30 equiv.) were refluxed in toluene: ethanol (1:1 ratio) for 48–72 h. The solvent was removed under reduced pressure to yield a yellowish semisolid (or solid) which was recrystallised twice from $CHCl_3$:CH_3OH to yield calix[4]arene amido mono crown derivatives as white solids that were further purified by column chromatography if necessary (for **3c** and **3d**).

1,3-Distal-5,11,17,23-tetra-tert-butyl-25,27-(5-methyl-3,8-dioxo-1,10-dioxa-4,7-diazadecano)calix[4]arene (**3a**): White solid, yield: 74%, m.p. > 200 °C (decomposed). IR (KBr, υ_{max}/ cm^{-1}): 3373, 1695. ^{1}H NMR (300 MHz, $CDCl_3$, δ in ppm): 8.31 (d, 1H, J = 7.5 Hz, –CONH), 8.27 (s, 1H, –OH), 8.23 (s, 1H, –OH), 8.22 (d, 1H, J = 7.5 Hz, –CONH), 7.14 (s, 1H, ArH), 7.12 (s, 2H, ArH), 7.04 (s, 2H, ArH), 7.01 (s, 1H, ArH), 6.98 (s, 2H, ArH), 4.68 (t, 3H, $ArOCH_2$ and $CONHCH(CH_3)CH_2NHCO$), 4.49 (dd, 2H, J = 14.1 Hz, 6.6 Hz, $ArOCH_2$), 4.28 (d, 2H, J = 11.7 Hz, $ArCH_2Ar$), 4.22 (d, 1H, J = 13.2 Hz, $CONHCH(CH_3)CH_2NHCO$), 4.03 (d, 2H, J = 11.1 Hz, $ArCH_2Ar$), 3.57 (d, 1H, J = 7.5 Hz, $ArCH_2Ar$), 3.53 (d, 1H, J = 7.5 Hz, $ArCH_2Ar$), 3.42 (d, 1H, J = 5.7 Hz, $ArCH_2Ar$), 3.38 (d, 1H, J = 5.7 Hz, $ArCH_2Ar$), 3.24 (broad d, 1H, J = 13.5 Hz, $CONHCH(CH_3)CH_2NHCO$), 1.31 (t, 3H, J = 7.2 Hz, $–CH_3$), 1.24 (s, 18H, $–C(CH_3)_3$), 1.13 (s, 18H, $–C(CH_3)_3$). DEPT-135 NMR (75 MHz, $CDCl_3$, δ in ppm): 127.3, 126.5, 126.3, 126.2, 126.1 (aromatic CH), 74.8 ($ArOCH_2$), 45.0 (NHCH), 43.6 ($NHCH_2$), 32.9 ($ArCH_2Ar$), 31.97, 31.50 ($–C(CH_3)_3$), 17.68 ($–CH_3$). FAB MS *m/z*: 803 (M++1, 100%). Anal. Calcd for $C_{51}H_{66}N_2O_6$: C, 76.27; H, 8.28; N, 3.49. Found: C, 76.55; H, 8.35; N, 3.36%. UV (λ_{max}, MeOH): 280 nm.

1,3-Distal-5,11,17,23-tetrahydro-25,27-(5-methyl-3,8-dioxo-1,10-dioxa-4,7-diazadecano)calix[4]arene Calix[4]arene(isopropylene amido) crown (**3b**): White solid, yield: 72%, m.p. > 340 °C (decomposed). IR (KBr, υ_{max}/ cm^{-1}): 3577, 3351, 1688. ^{1}H NMR (300 MHz, $CDCl_3$, δ in ppm): 8.31–8.15 (m, 4H, NH and OH), 7.21–7.15 (m, 6H, ArH), 7.00 (d, 2H, J = 7.2 Hz, ArH), 6.92 (d, 2H, J = 7.5 Hz, ArH), 6.76–6.70 (m, 2H, ArH), 4.71 (t, 2H, J = 14.1 Hz), 4.54 (d, 1H, J = 14.1 Hz), 4.47 (d, 1H, J = 14.1 Hz), 4.29 (t, 2H, J = 13.5 Hz), 4.05 (d, 2H, J = 14.1 Hz), 3.64 (d, 1H, J = 5.7 Hz), 3.59 (d, 1H, J = 5.7 Hz), 3.48 (s, 2H), 3.45 (d, 2H, J = 14.1 Hz), 3.27 (d, 1H, J = 13.5 Hz), 1.34 (d, 3H, J = 6.9 Hz, CH_3). DEPT-135 NMR (75 MHz, $CDCl_3$, δ in ppm): 130.5, 129.7, 129.5, 129.4, 127.4, 121.0, 120.9 (ArCH), 74.8 (OCH_2), 45.0 (NHCH), 43.6 ($NHCH_2$), 32.3 ($ArCH_2Ar$), 17.8 (CH_3). FAB MS *m/z*: 579 (M^++1, 100%). Anal. Calcd for $C_{35}H_{34}N_2O_6$: C, 72.65; H, 5.92; N, 4.84. Found: C, 72.98; H, 5.76; N, 4.89%.

1,3-Distal-5,11,17,23-tetra-tert-butyl-25,27-(5-methyl-3,8-dioxo-1,10-dioxa-4,7-diazadecano)-2,8,14,20-tetrathiacalix[4]arene (**3c**): White solid, separated by column chromatography using hexane:ethyl acetate (5:5) as the eluent, yield: 44%, m.p. > 230 °C (decomposed). IR (KBr, υ_{max}/ cm^{-1}): 3373, 1695. ^{1}H NMR(300 MHz, $CDCl_3$, δ in ppm): 8.83 (s, 1H, –OH), 8.71 (s, 1H, –OH), 8.34 (d, 1H, *J* = 8.4 Hz, –CONHCH), 8.00 (d, 1H, *J* = 8.4 Hz, –$CONHCH_2$), 7.66 (s, 2H, ArH), 7.61 (s, 2H, ArH), 7.50 (s, 4H, ArH), 4.82 (t, 2H, *J* = 13.2 Hz, $ArOCH_2$), 4.57 (broad s, 1H, –$CONHCH(CH_3)CH_2NHCO$), 4.51 (d, 1H, *J* = 13.2 Hz, $ArOCH_2$), 4.35 (d, 1H, *J* = 13.2 Hz, $ArOCH_2$), 4.20 (d, 1H, *J* = 6.9 Hz, $CONHCH(CH_3)CH_2NHCO$), 3.26 (broad d, 1H, *J* = 12.9 Hz, $CONHCH(CH_3)CH_2NHCO$), 1.26 (broad t, 3H, *J* = 7.2 Hz, –CH_3), 1.18 (s, 18H, –$C(CH_3)_3$), 1.11 (s, 18H, –$C(CH_3)_3$). DEPT-135 (75 MHz, $CDCl_3$, δ in ppm): 138.0, 137.7, 137.6, 136.2, 135.8 (aromatic CH), 76.1 ($ArOCH_2$), 45.2 (NHCH), 43.6 ($NHCH_2$), 31.76, 31.42 (–$C(CH_3)_3$), 17.25 (–CH_3).FAB MS *m/z*: 875 (M^++1, 100%). Anal. Calcd for $C_{47}H_{58}N_2O_6S_4$: C, 64.50; H, 6.68; N, 3.20. Found: C, 64.81; H, 6.73; N, 3.25%.

1,3-Distal-5,11,17,23-tetrahydro-25,27-(5-methyl-3,8-dioxo-1,10-dioxa-4,7-diazadecano)-2,8,14,20-tetrathiacalix[4]arene (**3d**): White solid, separated by column chromatography using hexane:ethyl acetate (5:5) as the eluent, yield: 38%, m.p. > 200 °C (decomposed). IR (KBr, υ_{max}/ cm^{-1}): 3378, 1685. ^{1}H NMR (300 MHz, $CDCl_3$, δ in ppm): 8.71 (s, 1H, -OH), 8.49 (s, 1H, -OH), 8.18 (d, 1H, *J* = 8.4 Hz, –CONHCH), 7.80 (d, 1H, *J* = 8.4 Hz, –$CONHCH_2$), 7.65 (d, 2H, ArH_{meta}), 7.56 (d, 2H, ArH_{meta}), 7.53 (d, 2H, ArH_{meta}), 7.46 (d, 2H, ArH_{meta}), 6.92 (t, 2H, *J* = ArH_{para}), 7.53 (t, 2H, *J* = ArH_{para}), 4.83 (t, 2H, *J* = 13.2 Hz, $ArOCH_2$), 4.60 (broad d, 2H, –$CONHCH(CH_3)CH_2NHCO$ and $ArOCH_2$), 4.42 (d, 1H, *J* = 13.2 Hz, $ArOCH_2$), 4.17 (d, 1H, *J* = 6.9 Hz, $CONHCH(CH_3)CH_2NHCO$), 3.23 (broad d, 1H, *J* = 12.9 Hz, $CONHCH(CH_3)CH_2NHCO$), 1.26 (broad t, 3H, *J* = 7.2 Hz, –CH_3), 138.0, 137.7, 137.6, 136.2, 135.8 (aromatic CH), 76.1 ($ArOCH_2$), 45.2 (NHCH), 43.6 ($NHCH_2$), 31.76, 31.42 (–$C(CH_3)_3$), 17.25 (–CH_3). FAB MS *m/z*: 651 (M^++1, 100%). Anal. Calcd for $C_{31}H_{26}N_2O_6$: C, 57.21; H, 4.03; N, 4.30. Found: C, 57.53; H, 3.94; N, 4.18%.

1,3-Distal-5,11,17,23-tetra-tert-butyl-25,27-(cyclohexan-1,2-diyl {diamino[bis(2-oxoethoxy)]})calix[4]arene (**4a**): White solid, yield: 57%, m.p. > 200 °C (decomposed). IR (KBr, υ_{max}/ cm^{-1}): 3412, 3351, 1682. ^{1}H NMR (300 MHz, $CDCl_3$, δ in ppm): 8.70 (broad t, 1H, CONH), 8.51 (s, 1H, OH), 8.10 (broad t, 1H, CONH), 7.80 (s, 1H, OH), 7.13 (s, 2H, ArH), 7.04 (s, 2H, ArH), 7.00 (s, 2H, ArH), 6.98 (s, 2H, ArH), 4.67–3.23 (m, 14H, $ArCH_2Ar$, OCH_2 and CH), 2.07–1.49 (broad m, 8H, CH_2-cyclohexyl), 1.24 (s, 18H, –$C(CH_3)_3$), 1.15 (s, 18H, –$C(CH_3)_3$). FAB MS *m/z*: 843(M^++1, 100%). Anal. Calcd for $C_{54}H_{70}N_2O_6$: C, 76.92; H, 8.37; N, 3.32. Found: C, 77.14; H, 8.40; N, 3.34%.

1,3-Distal-5,11,17,23-tetrahydro-25,27-(cyclohexan-1,2-diyl {diamino[bis(2-oxoethoxy)]})calix[4]arene (**4b**)*:* White solid, yield: 64%, m.p. > 200 °C (decomposed). IR (KBr, υ_{max}/ cm^{-1}): 3423, 3344, 1680. ^{1}H NMR (300 MHz, $CDCl_3$, δ in ppm): 8.68 (broad t, 1H, CONH), 8.50 (s, 1H, OH), 8.07 (broad t, 1H, CONH), 7.80 (s, 1H, OH), 7.07–6.97 (m, 7H, ArH), 6.86 (d, 2H, *J* = 7.2 Hz, ArH), 6.67 (t,1H, *J* = 6.9 Hz, ArH), 6.58 (t, 2H, *J* = 6.9 Hz, ArH), 4.67–3.23 (m, 14H, $ArCH_2Ar$, OCH_2 and CH), 2.07–1.49 (broad m, 8H, CH_2-cyclohexyl). DEPT-135 (300 MHz, $CDCl_3$, δ in ppm): 129.7, 129.4, 128.8, 128.6, 126.6, 121.45, 119.6 (ArCH), 74.8 (OCH_2), 50.0 (NHCH), 31.6, 31.3 ($ArCH_2Ar$), 28.2, 22.1 (CH_2). FAB MS *m/z*: 619 (M^++1, 100%). Anal. Calcd for $C_{38}H_{38}N_2O_6$: C, 73.77; H, 6.19; N, 4.53. Found: C, 73.56; H, 6.26; N, 4.58%.

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Figures S1–S5 are available in the Electronic Supplementary Information and may be downloaded through www.ingentaconnect.com/content/stl/jcr

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