Electronic Supplementary Material (ESI) for CrystEngComm.
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Supplementary Information

Lower rim dimerization of a calixarene through the encapsulation of sodium ions

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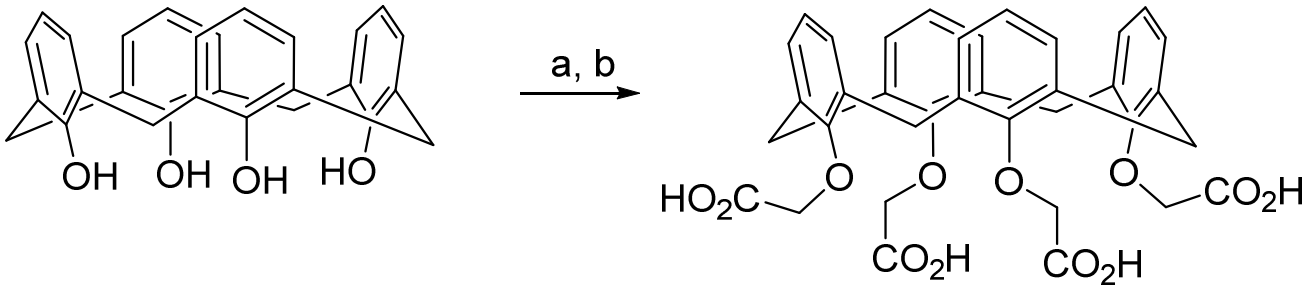
1. Material and Methods

1.1 Synthesis and sample preparation of the sodium tetra(carboxymethoxy)calix[4]arene forms

Tetra(carboxymethoxy)calix[4]arene was prepared according to known procedures[S1,S2] with some modifications (Scheme 1). Briefly, calix[4]arene (1.00 g), ethyl bromoacetate (7.88 g) and Na_2CO_3 (5.00 g) were refluxed in 50 mL of dry acetone for 24 h. The solvent was evaporated and to the crude mixture were added NaOH (4.00 g), 20 mL of ethanol and 20 mL of water. The mixture was refluxed for 24 h. The cooled solution was acidified with 2 M HCl to pH 1 and the resulting precipitate was isolated by filtration. The precipitate was then dissolved in AcOEt and the solution was washed twice with 20% aqueous HCl and three times with water to remove impurities. The organic layer in sequence was dried over $MgSO_4$ and concentrated to dryness to yield the monomeric calixarene as a residual solid. NMR spectroscopic data were in agreement with literature.[S2]



Scheme 1. Synthesis of tetra(carboxymethoxy)calix[4]arene. a) $BrCH_2COOEt/Na_2CO_3$, acetone, reflux; b) NaOH, $EtOH/H_2O$, reflux, and then HCl.

Next, a calixarene sample was weighed (10 mg) and dissolved under vigorous stirring in the following solvent systems: a mixture of methanol (5 ml) and 0.1 M HCl (0.1 ml) for 1; a mixture of methanol (2.5 ml), chloroform (2.5 ml) and 0.1 M HCl (0.1 ml) for 2; only either dimethylsulfoxide (5.0 ml) or dimethylformamide (5.0 ml) for 3 and 4, respectively. A NaCl amount of either 1.4 mg (1 and 2) or 1.8 mg (3 and 4) was dissolved together with the calixarene sample upon heating at 303 K. The newly prepared solutions were slowly cooled to 298 K and then glass crystallizers were not

sealed and then kept upon standing for 7-10 days in the dark up to complete solvent evaporation, except for 2 whose crystals were isolated out from mother solution before total solvent evaporation.

1.2 Structure determination

Suitably shaped single crystals of 1-4 were selected for the X-ray diffraction data collect on a Bruker-AXS Kappa Duo diffractometer with an APEX II CCD detector. MoKα radiation from an IμS microsource with multilayer optics was employed. Diffraction images were recorded by φ and ω scans set using APEX2 software.[S3] This software was also employed to treat the raw dataset for indexing, integrating, reducing and scaling of the reflections. Next, the crystallographic softwares were used as follows: SIR2004[S4] (structure solving), SHELXL-97[S5] (structure refinement), MERCURY,[S6] and ORTEP-3[S7] (structure analysis and representations). All structures were solved through identification of all non-hydrogen atoms in asymmetric units directly from the Fourier synthesis of the structure factors after retrieval of their phase using the direct methods. Each initial model was refined by full-matrix least squares method on F^2, adopting free anisotropic and constrained isotropic atomic displacement parameters for non-hydrogen and hydrogen atoms, respectively. In the case of hydrogens, their U_{iso} was set to either $1.2U_{eq}$ of the bonded carbon, except for methyl hydrogens in which their U_{iso} was set to $1.5U_{eq}$ of the corresponding carbon as well as of the bonded oxygen. Hydrogen coordinates were stereochemically defined and constrained in the refinements, oscillating as that of the bonded carbon or oxygen to keep idealized bond angles and lengths fixed. However, most OH hydrogens were first localized from the difference Fourier maps so that they were ideally positioned for covalent bonding to the corresponding oxygen atoms. In carboxyl groups, identification of C—O bond lengths featuring a protonated moiety instead of an ionized carboxylate one were helpful to assign the proton position. The reported hydrogen atom locations for the water molecule in 1 and 2 and methanol one in 1 give rise to satisfactory intramolecular and intermolecular hydrogen bond geometry, but their positions are not fully in

agreement with what can be seen in three-dimensional difference Fourier map plots most probably due to the weakness of high resolution intensity data. Flack parameters of 0.3(3), -0.23(19) and -0.6(5) were outputted for the noncentrosymmetric crystal structures of 1, 3 and 4. However, it is important to mention that absolute structure could be not assigned reliably due to these high uncertainties. It is striking to note that crystals of 2 were extremely small plates diffracting poorly even at medium resolution shell. In addition, they seem to loose quickly solvent when away from mother solution, undergoing decrease of long-range order (only 19% of observed reflections). These factors have precluded much the data quality outputting bad refinement statistics (Table S1). In this structure, smeared electronic density as low electron density peaks in the difference Fourier map was found, which refer probably to non-stoichiometric loosing solvent molecules. However, their assignment was not possible and the data was squeezed using Platon.[S8] Even after squeezing the data of the free calix[4]tube 2, several refinement restraints were needed to ensure proper geometry of the dimer assembled with the C and D units. DFIX and DANG restraints were employed to bond lengths and angles of two phenyl rings (C15D-C20D and C22D-C27D), as well as these restraints were applied to all carboxymethylene moieties from subunit D and to one of subunit C (C31C, C32C, O7C and O8C). FLAT restraint was also used to keep planar the atoms of three carboxylic moieties, being two of them from subunit D (C34D, O9D and O10D; and C36D, O11D and O12D) and another one from subunit C (C30C, O5C and O6C). The carboxylic atoms C32D, O7D and O8D have been refined as variable metric rigid group using AFIX 9 command. Atomic displacement parameters (ADPs) of non-hydrogen atoms were also restrained using SIMU and DELU, but even so high correlation was observed between ADPs and site occupancy factors of some carboxyl oxygen and carbon atoms from calixarene units C and D when refining them anisotropically. Therefore, they were refined isotropically as well as the water oxygen.

Concerning labeling scheme of phenyl rings, they have been labeled from one to four (C1 to C6, C8 to C13, C15 to C20, and C22 to C27, in this order). In 1 and 2, their phenyl labels have been

succeeded by the letter identifying calixarene unit [*e. g*., Ph1(A) denotes phenyl ring 1 from calixarene unit A and Cg1(A) refers to centroid calculated through its carbons].

Free calix[4]tube 1 was composed with three sodium ions, two calixarene units, wherein one of them undergoes deprotonation in one acid moiety and another one is present with two deprotonated carboxylic groups, assembling one sodium-encapsulating neutral dimer, one water and methanol molecule in its asymmetric unit. Free calix[4]tube 2 contains in its asymmetric unit two aforementioned neutral dimers and at least one water molecule, since it was not possible to find the number of solvent molecules in its structural voids using the SQUEEZE tool.[S9] The electron count in the void could be not directly attributed to number of solvent molecules due to two reasons. Firstly, the overall electron count removed from structure factors using SQUEEZE refers to highly disordered solvent molecules which can be most probably water or methanol. These two solvent molecules were doubtless found in the structure of the free calix[4]tube 1, suggesting therefore that both of them can be lodged into the large void of 2 taken into account the presence of both solvents in its crystallization system. In this way, the assignment of electron count in void to only water molecules, for instance, was not done. The second reason resides in the low amount of observed data (only 19%). The absence of strong low-order reflections precludes the reliability of the electron count using SQUEEZE.[S9] Therefore, even if anyone is sure of which solvent proportion is in the void, the electron number could be not reliably attributed to solvent content based on such poor intensity data from the solvent-loosing weakly-diffracting crystal.

Polymeric calix[4]tubes 3 and 4 are present with one calixarene unit undergoing deprotonation in two carboxylic moieties, one sodium ion in a full occupancy site (labeled as Na1, see below in section 3) and two sodium ions on a two-fold rotation axis with [001] direction (labeled as Na2 and Na3). Therefore these two sodium ions have 50% occupancy in the asymmetric unit and they are rotated on themselves to generate the full occupancy site. Such two-fold rotation gives rise to the other calixarene unit and other entire sodium to form the dimer, which is anionic in these structures.

One of the sodium ions in the crystallographic special positions counterbalances the dimer charge. Besides those, either one dimethylsulfoxide (DMSO) or one dimethylformamide (DMF) molecule is also found in the asymmetric unit of the polymeric calix[4]tubes 3 and 4, respectively.

At last, refinement convergence had been not full in the structure of the polymeric calix[4]tube 4 because of a slight disorder in the solvent DMF molecule entrapped into the calixarene upper rim. Such disorder in the solvent molecule is understood as a consequence of a competition between two sets of intermolecular interactions in the polymeric calix[4]tube 4, namely, C-$H_{(DMF)}$…$\pi_{(calix)}$ and C-$H_{(calix)}$…$O_{(DMF)}$ (see section 3 and Figure S6 in the Supplementary Information). This disorder phenomenon does not occur in the isostructural crystal form 3 because DMSO is longer than DMF. In this way, DMSO is not affected by disorder effects in 3 since it is able to establish both aforementioned interaction sets with suitable geometry. The maximum shifts precluding refinement convergence of 4 were observed in the fractional coordinates of carbonyl atoms of DMF molecule. It is important to comment that trial refinements were used with the split-atom approach. However, since the extra disordered sites were very close together, still higher correlation and more unstable refinements were observed applying the classical split-atom approach to the solvent molecule. Even splitting the carbonyl and consequently the methyl atoms of DMF, the one-site model converged easier. Isotropic refinement of its carbons and oxygen was also attempted, but full refinement convergence also was not reached. To achieve full convergence, the structure of 4 was firstly refined using conjugate gradient least-square method[S10] (CGLS instruction) and then full matrix least-square (L.S. instruction) refinement followed.

Geometry of all coordination and hydrogen bonds for the four structures, as well as geometric parameters of the CH…π and π…π interactions, can be found in Tables S2 to S9 together with ellipsoid plots of their asymmetric unit in Figures S7 to S10.

1.3 Mass spectrometry

The negative ion high-resolution mass spectra were obtained on Orbitrap Q-Exactive mass spectrometer (Thermo Scientific, Bremen, Germany) equipped with a heated electrospray ion source. The parameters used were: spray voltage 3 kV; capillary temperature 250 °C; Fourier transform MS resolution 140 000; S-Lens Level 50; sheath gas 10 (arbitrary units). Mass spectrum was acquired in continuous monitoring mode with a mass range of 600-1450. Sodium tetra(carboxymethoxy)calix[4]arene 3 was dissolved in dimethylsulfoxide and diluted to a concentration of 100 ng mL^{-1} using methanol, without base addition. The resulting solution was analyzed by direct infusion through the syringe pump (Hamilton 1750RN) at a flow rate of 3 µl min^{-1}. The data were evaluated by the XCALIBUR software 2.7 SP1 (Thermo Scientific, Bremen, Germany).

1.4 ^{1}H Nuclear Magnetic Resonance

The ^{1}H NMR experiments were performed at 298 K on a Bruker Avance III 500 spectrometer, operating at 500.13 MHz for ^{1}H, equipped with a 5 mm z-gradient TBI probe (^{1}H, ^{13}C and XBB), using DMSO-d6 (500 µL) to prepare a solution of sodium tetra(carboxymethoxy)calix[4]arene 3 (1 mg).

2. Description of coordination environment and supramolecular architecture in 1 and 2

Non-shared sodium ions are coordinated to eight oxygens in 1 with a square antiprismatic geometry (Figure S1c). They are bonded to the four methoxy oxygens from each calixarene unit, forming one base of the anticube. Their another base is formed by four oxygens from both carboxyl (O5A, O9A and O11A for Na1 and O7B and O11B for Na3) and carboxylate (O7A for Na1 and O5B and O9B for Na3) groups. Except for O7B, the oxygens onto these last anticube bases from the two non-shared sodium ions are simultaneously coordinated to the shared sodium Na2 in a seven-coordinated distorted capped octahedral geometry. Similar coordination environment is also found in structure 2, especially in the dimer formed with calixarene units labeled as C and D (Figure S1d). In that, the only difference resides in the fact that the shared sodium Na5 is coordinated to both oxygens O9D and O10D from a same carboxylate group rather than to one only oxygen from each moiety as in all others. This does result in a capped square antiprismatic coordination geometry for Na5 bonded to nine oxygens. The dimer assembled with calixarene units labeled as A and B conserves also a similar coordination fashion observed in 1, but without one oxygen onto each anticube basal plane of the shared sodium Na2 , which are common to Na1 (O7A, O9A and O11A) and Na3 (O5B, O7B and O9B). Consequently, this shared sodium is coordinated to six oxygens exhibiting a distorted octahedral geometry, while both non-shared ones are bonded to seven oxygens with a distorted capped octahedral environment (Figure S1d). This difference between the nanotube made up of calixarene fragments A and B in 2 and that present in 1 is due to the engagement of one carboxyl group of each unit only in hydrogen bonds while all others are coordinated to one non-shared sodium enclosed into the minor cavity and to the shared sodium ion through one of their oxygens. The non-coordinated carboxyl group from calixarene subunit A acts sealing the dimer edge through accepting hydrogen bonding from carboxyl moiety of fragment B

(O6B-H…O5A), whether the non-coordinated carboxyl group from calixarene subunit B is simultaneously hydrogen bonding donor and acceptor from water molecule found in asymmetric unit (O12B-H…O1w and O1w-H…O11B; Figure S1b). This water molecule is also a hydrogen bonding donor to carboxylate oxygen O8A (O1w-H…O8A) through the same hydrogen interacting with O11B, in a bifurcated pattern which helps to seal the nanotube at its border. The other sealing hydrogen bonding interactions between carboxyl (donor) and carboxylate (acceptor) moieties in this crystallographically independent nanotube are O10A-H…O10B and O12A-H…O8B, while a set of bifurcated and non-bifurcated hydrogen bonds take place in the other dimer in the asymmetric unit of 2 (O12C-H…O9D; O12C-H…O10D; O10C-H…O10D; O10C-H…O7D; O8D-H…O9C; O6D-H…O8C; and O6C-H…O12D). In 1, there are three sealing hydrogen bonds between carboxyl and carboxylate groups (O10A-H…O10B; O12B-H…O8A; O6A-H…O6B) and only one between carboxyl moieties (O12A-H…O7B). The carboxyl group acting as acceptor in the last hydrogen bonding is also a donor to water molecule present in the asymmetric unit, which in turn is donor to the carboxyl oxygen O11B (O1w-H…O11B) and to methanol oxygen (O1w-H…O1s) trapped in the hydrophobic cavity of subunit A (Figure S2).

3. Description of coordination environment and supramolecular architecture in 3 and 4

The non-shared sodium Na1 present in a full occupancy site exhibits square antiprismatic coordination geometry (Figure S5b), as occurs in 1 and 2. It is bonded to the four methoxy oxygens, which set one anticube basal plane, and to two carboxyl (O7 and O11) and two carboxylate (O5 and O9) oxygens, which form one another. The shared sodium ion Na2, which is located in a 50% occupancy special position coinciding with a two-fold rotation axis, as well as Na3, is coordinated to six oxygens (four carboxyl ones, O7, O11, $O7^{i}$, $O11^{i}$, and two carboxylate ones, O5 and $O5^{i}$, symmetry operator i = 1-x, 2-y, z in 3 and i = -x, 1-y, z in 4) with a distorted octahedral

environment. One carboxylate group is not bonded to Na2, it acts sealing the nanotube through hydrogen bonding acceptation from carboxyl oxygen $O8^{i}$ from a two-fold axis symmetry related calixarene unit (O8-H…$O10^{i}$; Figure S5a) besides coordinating to sodium ion Na3 through its oxygen O9. Sodium Na3 has square antiprismatic coordination geometry: one anticube basal plane is formed with carboxylate (O9 and $O9^{i}$) and carboxyl (O8 and $O8^{i}$) oxygens from two-fold axis symmetry related calixarene subunits of a same nanotube, while another one has carboxylate ($O5^{ii}$ and $O5^{iii}$, symmetry operators *ii* =-0.5+y, 0.5+x, -0.5+z and *iii* = 1.5-y, 1.5-x, -0.5+z in 3 and *ii* = -0.5+y, 0.5+x, -0.5+z and *iii* = 0.5-y, 0.5-x, -0.5+z in 4) and carboxyl ($O12^{ii}$ and $O12^{iii}$) oxygens from two-fold axis symmetry related calixarene subunits of a neighboring nanotube tied along [001] direction (Figure S4). In addition to hydrogen bond between carboxyl and carboxylate moieties already mentioned, there is another one (O12-H…$O6^{i}$) contributing to seal the nanotube centre of 3 at its edge. By symmetry, the interactions $O8^{i}$-H^{i}…O10 and $O12^{i}$-H^{i}…O6 are created, totaling four hydrogen bonds sealing the nanotube in 3 and 4 (Figure S5a).

Besides C-H…π interactions between solvent molecules and phenyl moieties into hydrophobic cavities of the dimers (C2s-H2s2…Cg3, C2s-H2s2…Cg4 and C2s-H2s3…Cg2 in 3 or C1s-H1s1…Cg4, C1s-H1s2…Cg3 and C1s-H1s3…Cg1 in 4, see Figure S6), dimethylsulfoxide and dimethylformamide do also contribute to crystal packing by accepting two non-classical hydrogen bonds from C-H phenyl moieties of a nanotube near to that trapping either dimethylsulfoxide or dimethylformamide into its aromatic cavity (C5-H…O1s and C13-H…O1s in both structures).

4. Tables

Table S1. Crystal data and refinement statistics of the sodium calix[4]tubes.

		free calix[4]tube 1	free calix[4]tube 2	polymeric calix[4]tube 3	polymeric calix[4]tube 4
structural formula		$(C_{72}H_{61}Na_3O_{24})(CH_4O)(H_2O)$	$(C_{72}H_{61}Na_3O_{24})_2(H_2O)_{0.5}$ [i]	$(C_{72}H_{61}Na_4O_{24})(C_2H_6OS)_2$	$(C_{72}H_{61}Na_4O_{24})(C_3H_7NO)_2$
fw		1429.24	2767.37[i]	1557.42	1547.35
cryst syst		orthorhombic	triclinic	tetragonal	tetragonal
space group		$P2_12_12_1$	P-1	$P4_2bc$	$P4_2bc$
Z		4	2	4	4
T (K)		296(2)	296(2)	296(2)	296(2)
unit cell dimensions	a (Å)	14.174(4)	14.840(2)	21.041(4)	21.0771(7)
	b (Å)	18.194(5)	17.911(3)	21.041(4)	21.0771(7)
	c (Å)	26.052(6)	31.010(5)	16.456(3)	16.6303(5)
	α (°)	90	79.663(11)	90	90
	β (°)	90	86.268(11)	90	90
	γ (°)	90	82.469(12)	90	90
V (Å^3)		6718(3)	8032(2)	7286(3)	7387.9(4)
calculated density (Mg/m^3)		1.413	1.144[i]	1.420	1.391
absorption coefficient (mm^{-1})		0.124	0.100[i]	0.181	0.124
Absorption correction		Multi-scan T_{min} / T_{max} = 0.901	Multi-scan T_{min} / T_{max} = 0.908	Multi-scan T_{min} / T_{max} = 0.921	Multi-scan T_{min} / T_{max} = 0.941
θ range for data collection (°)		1.56 – 26.60	1.39 – 25.39	1.94 – 25.13	2.29 – 25.40
index ranges		-17 to 17	-17 to 17	-25 to 24	-25 to 15
		-14 to 22	-11 to 21	-22 to 24	-17 to 25
		-32 to 31	-36 to 37	-19 to 18	-11 to 19
data collected		48,173	45,357	41,778	24,382
unique reflections		13,078	27,303	6,333	5,868
unique reflections with $I > 2\sigma(I)$		5,959	5,220	2,947	2,790
symmetry factor (R_{int})		0.1126	0.1181	0.2484	0.1284
completeness to θ = 25° (%)		100	93.9	99.9	99.8
F (000)		2984	2882[i]	3248	3232
parameters refined		909	1,735	490	497
goodness-of-fit on F^2		0.984	0.931	0.973	1.005
final R_1 factor for $I > 2\sigma(I)$		0.0677	0.1435	0.0689	0.0662
$wR2$ factor for all data		0.2110	0.4050	0.1666	0.2141
largest diff. peak / hole (e/Å^3)		0.377/-0.643	0.498/-0.872	0.258/-0.311	0.325/-0.357
CCDC deposit number		1,433,706	1,433,707	1,433,708	1,440,137

[i] Even though the data have been squeezed with PLATON,[S8,S9] the electron count removed from structure factors was not assigned to solvent molecules (most probably methanol and water) because of uncertainty in their proportion and missing strong reflections even at low resolution shell.

Table S2. Coordination bond lengths (Å) and angles (°) of free calix[4]tube 1.

Length		Angle	
Na1-O1A	2.415(6)	O7A-Na1-O11A	100.6(2)
Na1-O2A	2.405(6)	O9A-Na1-O11A	67.0(2)
Na1-O3A	2.439(6)	O5A-Na2-O7A	71.0(2)
Na1-O4A	2.408(6)	O5A-Na2-O9A	100.1(2)
Na1-O5A	2.568(7)	O5A-Na2-O11A	70.0(2)
Na1-O7A	2.434(7)	O5A-Na2-O5B	90.9(2)
Na1-O9A	2.558(8)	O5A-Na2-O9B	173.9(3)
Na1-O11A	2.550(7)	O5A-Na2-O11B	111.4(2)
Na2-O5A	2.403(8)	O7A-Na2-O9A	61.6(2)
Na2-O7A	2.477(8)	O7A-Na2-O11A	98.7(2)
Na2-O9A	3.045(9)	O7A-Na2-O5B	147.8(3)
Na2-O11A	2.577(7)	O7A-Na2-O9B	110.0(3)
Na2-O5B	2.500(7)	O7A-Na2-O11B	90.8(3)
Na2-O9B	2.296(7)	O9A-Na2-O11A	59.5(2)
Na2-O11B	2.514(8)	O9A-Na2-O5B	150.1(2)
Na3-O1B	2.355(6)	O9A-Na2-O9B	75.6(2)
Na3-O2B	2.500(6)	O9A-Na2-O11B	128.2(3)
Na3-O3B	2.363(6)	O11A-Na2-O5B	99.6(2)
Na3-O4B	2.449(7)	O11A-Na2-O9B	103.9(3)
Na3-O5B	2.328(7)	O11A-Na2-O11B	170.2(3)
Na3-O7B	3.013(8)	O5B-Na2-O9B	90.9(2)
Na3-O9B	2.350(6)	O5B-Na2-O11B	70.9(3)
Na3-O11B	2.788(8)	O9B-Na2-O11B	74.7(2)
		O1B-Na3-O2B	78.6(2)
Angle		O1B-Na3-O3B	134.4(2)
O1A-Na1-O2A	85.7(2)	O1B-Na3-O4B	86.3(2)
O1A-Na1-O3A	136.0(2)	O1B-Na3-O5B	70.7(2)
O1A-Na1-O4A	81.2(2)	O1B-Na3-O7B	123.8(2)
O1A-Na1-O5A	64.8(2)	O1B-Na3-O9B	150.7(3)
O1A-Na1-O7A	128.2(2)	O1B-Na3-O11B	82.2(2)
O1A-Na1-O9A	148.1(2)	O2B-Na3-O3B	81.4(2)
O1A-Na1-O11A	82.8(2)	O2B-Na3-O4B	139.9(2)
O2A-Na1-O3A	78.62(19)	O2B-Na3-O5B	82.7(2)
O2A-Na1-O4A	137.3(2)	O2B-Na3-O7B	56.97(19)
O2A-Na1-O5A	84.0(2)	O2B-Na3-O9B	125.4(3)
O2A-Na1-O7A	67.3(2)	O2B-Na3-O11B	149.4(3)
O2A-Na1-O9A	125.9(2)	O3B-Na3-O4B	83.4(2)
O2A-Na1-O11A	152.0(2)	O3B-Na3-O5B	145.7(3)
O3A-Na1-O4A	83.14(19)	O3B-Na3-O7B	74.8(2)
O3A-Na1-O7A	82.6(2)	O3B-Na3-O9B	71.4(2)
O3A-Na1-O5A	150.7(2)	O3B-Na3-O11B	128.5(2)
O3A-Na1-O9A	64.1(2)	O4B-Na3-O5B	126.8(3)
O3A-Na1-O11A	126.4(2)	O4B-Na3-O7B	150.0(2)
O4A-Na1-O5A	124.8(2)	O4B-Na3-O9B	83.3(2)
O4A-Na1-O7A	147.3(3)	O4B-Na3-O11B	61.0(2)
O4A-Na1-O9A	77.2(2)	O5B-Na3-O7B	71.1(2)
O4A-Na1-O11A	65.7(2)	O5B-Na3-O9B	94.0(2)
O5A-Na1-O7A	69.0(2)	O5B-Na3-O11B	68.6(2)

O5A-Na1-O9A	110.0(2)	O7B-Na3-O9B	70.4(2)
O5A-Na1-O11A	67.9(2)	O7B-Na3-O11B	118.8(2)
O7A-Na1-O9A	70.1(3)	O9B-Na3-O11B	68.8(2)

Table S3. Coordination bond lengths (Å) and angles (°) of free calix[4]tube 2.

Length		Angle	
Na1-O1A	2.458(8)	O4B-Na3-O7B	151.3(2)
Na1-O2A	2.390(8)	O4B-Na3-O9B	82.4(2)
Na1-O3A	2.423(7)	O5B-Na3-O7B	70.0(3)
Na1-O4A	2.348(6)	O5B-Na3-O9B	88.3(3)
Na1-O7A	2.274(7)	O7B-Na3-O9B	69.1(2)
Na1-O9A	2.455(9)	O1C-Na4-O2C	83.1(3)
Na1-O11A	2.375(7)	O1C-Na4-O3C	137.4(3)
Na2-O7A	2.342(9)	O1C-Na4-O4C	83.0(3)
Na2-O9A	2.391(8)	O1C-Na4-O5C	75.4(4)
Na2-O11A	2.416(7)	O1C-Na4-O7C	78.4(4)
Na2-O5B	2.300(8)	O1C-Na4-O9C	151.9(4)
Na2-O7B	2.372(8)	O1C-Na4-O11C	134.8(4)
Na2-O9B	2.429(8)	O2C-Na4-O3C	78.4(3)
Na3-O1B	2.438(6)	O2C-Na4-O4C	137.8(3)
Na3-O2B	2.420(7)	O2C-Na4-O5C	138.0(5)
Na3-O3B	2.317(7)	O2C-Na4-O7C	66.2(4)
Na3-O4B	2.407(7)	O2C-Na4-O9C	84.7(4)
Na3-O5B	2.336(9)	O2C-Na4-O11C	141.1(5)
Na3-O7B	2.584(7)	O3C-Na4-O4C	85.6(3)
Na3-O9B	2.396(6)	O3C-Na4-O5C	139.8(5)
Na4-O1C	2.435(9)	O3C-Na4-O7C	126.0(5)
Na4-O2C	2.453(9)	O3C-Na4-O9C	63.4(4)
Na4-O3C	2.372(8)	O3C-Na4-O11C	76.1(4)
Na4-O4C	2.371(9)	O4C-Na4-O5C	75.0(4)
Na4-O5C	2.387(15)	O4C-Na4-O7C	147.2(5)
Na4-O7C	2.499(12)	O4C-Na4-O9C	122.1(5)
Na4-O9C	2.520(12)	O4C-Na4-O11C	68.4(4)
Na4-O11C	2.691(12)	O5C-Na4-O7C	74.2(5)
Na5-O5C	2.463(17)	O5C-Na4-O9C	97.7(5)
Na5-O7C	2.627(18)	O5C-Na4-O11C	64.1(5)
Na5-O9C	2.527(15)	O7C-Na4-O9C	73.6(5)
Na5-O11C	2.753(16)	O7C-Na4-O11C	107.4(4)
Na5-O5D	2.585(17)	O9C-Na4-O11C	57.7(5)
Na5-O7D	2.504(18)	O5C-Na5-O7C	70.8(5)
Na5-O9D	2.540(18)	O5C-Na5-O9C	95.6(4)
Na5-O10D	2.69(5)	O5C-Na5-O11C	62.2(5)
Na5-O11D	2.635(16)	O7C-Na5-O9C	71.3(5)
Na6-O1D	2.250(12)	O7C-Na5-O11C	102.1(3)
Na6-O2D	2.417(13)	O9C-Na5-O11C	56.8(5)
Na6-O3D	2.386(14)	O5C-Na5-O5D	138.2(7)
Na6-O4D	2.296(13)	O5C-Na5-O7D	146.0(8)
Na6-O5D	2.317(16)	O5C-Na5-O9D	98.2(6)
Na6-O7D	2.458(13)	O5C-Na5-O10D	94.1(10)
Na6-O9D	2.586(15)	O5C-Na5-O11D	93.9(6)

Na6-O11D	2.625(16)	O7C-Na5-O5D	83.2(5)
Angle		O7C-Na5-O7D	136.5(7)
O1A-Na1-O2A	81.9(3)	O7C-Na5-O9D	161.8(7)
O1A-Na1-O3A	137.8(3)	O7C-Na5-O10D	146.2(8)
O1A-Na1-O4A	82.5(2)	O7C-Na5-O11D	95.6(6)
O1A-Na1-O7A	104.7(3)	O9C-Na5-O5D	106.7(6)
O1A-Na1-O9A	153.7(3)	O9C-Na5-O7D	80.3(5)
O1A-Na1-O11A	83.3(3)	O9C-Na5-O9D	125.3(7)
O2A-Na1-O3A	81.1(3)	O9C-Na5-O10D	80.6(10)
O2A-Na1-O4A	136.9(3)	O9C-Na5-O11D	160.2(7)
O2A-Na1-O7A	66.8(3)	O11C-Na5-O5D	158.3(6)
O2A-Na1-O9A	120.6(3)	O11C-Na5-O7D	88.4(5)
O2A-Na1-O11A	147.3(3)	O11C-Na5-O9D	84.5(5)
O3A-Na1-O4A	84.1(2)	O11C-Na5-O10D	45.2(7)
O3A-Na1-O7A	103.5(3)	O11C-Na5-O11D	142.6(6)
O3A-Na1-O9A	64.6(3)	O5D-Na5-O7D	74.0(6)
O3A-Na1-O11A	127.9(3)	O5D-Na5-O9D	97.0(4)
O4A-Na1-O7A	156.3(3)	O5D-Na5-O10D	123.7(9)
O4A-Na1-O9A	87.9(3)	O5D-Na5-O11D	55.8(5)
O4A-Na1-O11A	69.0(2)	O7D-Na5-O9D	59.8(6)
O7A-Na1-O9A	75.7(3)	O7D-Na5-O10D	51.8(10)
O7A-Na1-O11A	89.2(3)	O7D-Na5-O11D	101.2(4)
O9A-Na1-O11A	70.4(3)	O9D-Na5-O10D	45.9(8)
O7A-Na2-O9A	75.8(3)	O9D-Na5-O11D	70.2(5)
O7A-Na2-O11A	86.6(3)	O10D-Na5-O11D	116.0(9)
O9A-Na2-O11A	70.8(2)	O1D-Na6-O2D	79.7(4)
O7A-Na2-O5B	89.9(3)	O1D-Na6-O3D	135.1(4)
O7A-Na2-O7B	162.1(3)	O1D-Na6-O4D	85.3(5)
O7A-Na2-O9B	116.9(3)	O1D-Na6-O5D	68.6(5)
O9A-Na2-O5B	164.2(4)	O1D-Na6-O7D	144.7(7)
O9A-Na2-O7B	120.6(3)	O1D-Na6-O9D	141.3(6)
O9A-Na2-O9B	92.2(3)	O1D-Na6-O11D	74.3(4)
O11A-Na2-O5B	115.7(3)	O2D-Na6-O3D	80.3(5)
O11A-Na2-O7B	92.1(3)	O2D-Na6-O4D	135.9(4)
O11A-Na2-O9B	147.2(3)	O2D-Na6-O5D	81.7(6)
O5B-Na2-O7B	74.5(3)	O2D-Na6-O7D	80.8(5)
O5B-Na2-O9B	88.4(3)	O2D-Na6-O9D	138.1(5)
O7B-Na2-O9B	72.2(3)	O2D-Na6-O11D	138.5(6)
O1B-Na3-O2B	82.1(2)	O3D-Na6-O4D	81.9(5)
O1B-Na3-O3B	137.9(3)	O3D-Na6-O5D	145.8(6)
O1B-Na3-O4B	83.1(2)	O3D-Na6-O7D	68.8(5)
O1B-Na3-O5B	68.7(3)	O3D-Na6-O9D	73.0(5)
O1B-Na3-O7B	122.7(3)	O3D-Na6-O11D	140.1(6)
O1B-Na3-O9B	144.5(3)	O4D-Na6-O5D	130.1(6)
O2B-Na3-O3B	84.6(2)	O4D-Na6-O7D	128.4(7)
O2B-Na3-O4B	138.8(3)	O4D-Na6-O9D	71.7(5)
O2B-Na3-O5B	95.4(3)	O4D-Na6-O11D	73.7(5)
O2B-Na3-O7B	64.2(2)	O5D-Na6-O7D	79.7(6)
O2B-Na3-O9B	128.3(2)	O5D-Na6-O9D	102.9(5)
O3B-Na3-O4B	81.2(2)	O5D-Na6-O11D	58.9(6)

O3B-Na3-O5B	152.6(3)	O7D-Na6-O9D	59.7(6)
O3B-Na3-O7B	85.6(2)	O7D-Na6-O11D	102.7(5)
O3B-Na3-O9B	70.8(2)	O9D-Na6-O11D	69.6(5)
O4B-Na3-O5B	114.4(3)		

Table S4. Coordination bond lengths (Å) and angles (°) of polymeric calix[4]tube 3.

Length		Angle	
Na1-O1	2.494(8)	O4-Na1-O7	152.6(3)
Na1-O2	2.350(7)	O4-Na1-O9	125.5(3)
Na1-O3	2.449(7)	O4-Na1-O11	70.5(2)
Na1-O4	2.396(7)	O5-Na1-O7	69.9(3)
Na1-O5	2.809(9)	O5-Na1-O9	114.5(3)
Na1-O7	2.379(8)	O5-Na1-O11	67.3(2)
Na1-O9	2.851(9)	O7-Na1-O9	66.5(3)
Na1-O11	2.366(8)	O7-Na1-O11	95.1(3)
Na2-O5	2.875(10)	O9-Na1-O11	70.6(2)
Na2-O7	2.508(8)	O5-Na2-O5^{i}	92.4(4)
Na2-O11	2.465(7)	O5-Na2-O7	67.2(2)
Na3-O5	2.994(11)	O5-Na2-O7^{i}	157.6(3)
Na3-O8	2.951(10)	O5-Na2-O11	65.0(2)
Na3-O9	2.847(10)	O5-Na2-O11^{i}	89.9(3)
Na3-O12	3.025(10)	O7-Na2-O7^{i}	134.4(5)
Angle		O7-Na2-O11	89.5(2)
O1-Na1-O2	82.1(2)	O7-Na2-O11^{i}	104.2(2)
O1-Na1-O3	135.8(3)	O11-Na2-O11^{i}	144.4(4)
O1-Na1-O4	79.4(2)	O5^{ii}-Na3-O5^{iii}	87.7(5)
O1-Na1-O5	61.5(2)	O5^{ii}-Na3-O8^{i}	150.3(4)
O1-Na1-O7	90.2(3)	O5^{ii}-Na3-O8^{iii}	72.9(2)
O1-Na1-O9	155.0(3)	O5^{ii}-Na3-O9	135.6(2)
O1-Na1-O11	122.8(3)	O5^{ii}-Na3-O9^{i}	102.3(2)
O2-Na1-O3	85.2(2)	O5^{ii}-Na3-O12^{ii}	73.5(2)
O2-Na1-O4	132.3(3)	O5^{ii}-Na3-O12^{iii}	65.6(2)
O2-Na1-O5	124.5(3)	O8-Na3-O8^{i}	133.0(2)
O2-Na1-O7	69.9(3)	O8-Na3-O9	78.3(3)
O2-Na1-O9	81.5(3)	O8-Na3-O9^{i}	71.9(3)
O2-Na1-O11	151.9(3)	O8-Na3-O12^{ii}	126.4(2)
O3-Na1-O4	78.6(2)	O8-Na3-O12^{iii}	78.0(2)
O3-Na1-O5	150.0(3)	O9-Na3-O9^{i}	100.1(5)
O3-Na1-O7	124.4(3)	O9-Na3-O12^{ii}	149. 8(2)
O3-Na1-O9	61.0(2)	O9-Na3-O12^{iii}	75.9(2)
O3-Na1-O11	84.3(2)	O12^{ii}-Na3-O12^{iii}	122.2(3)
O4-Na1-O5	82.8(3)		

Symmetry operators i = 1-x, 2-y, z; ii = -0.5+y, 0.5+x, -0.5+z and iii = 1.5-y, 1.5-x, -0.5+z.

Table S5. Coordination bond lengths (Å) and angles (°) of polymeric calix[4]tube 4.

Length		Angle	
Na1-O1	2.476(7)	O4-Na1-O7	153.3(3)
Na1-O2	2.366(6)	O4-Na1-O9	125.6(3)
Na1-O3	2.461(7)	O4-Na1-O11	70.0(2)
Na1-O4	2.397(7)	O5-Na1-O7	68.4(3)
Na1-O5	2.710(9)	O5-Na1-O9	115.1(3)
Na1-O7	2.394(8)	O5-Na1-O11	68.9(2)
Na1-O9	2.841(10)	O7-Na1-O9	66.4(3)
Na1-O11	2.371(7)	O7-Na1-O11	95.7(3)
Na2-O5	2.608(9)	O9-Na1-O11	72.0(2)
Na2-O7	2.562(8)	O5-Na2-O5^{i}	103.6(4)
Na2-O11	2.409(6)	O5-Na2-O7	67.7(2)
Na3-O5	3.435(13)	O5-Na2-O7^{i}	169.7(3)
Na3-O8	3.058(9)	O5-Na2-O11	70.2(2)
Na3-O9	2.739(10)	O5-Na2-O11^{i}	91.6(3)
Na3-O12	3.373(11)	O7-Na2-O7^{i}	121.5(4)
Angle		O7-Na2-O11	90.5(2)
O1-Na1-O2	81.1(2)	O7-Na2-O11^{i}	103.6(2)
O1-Na1-O3	134.3(2)	O11-Na2-O11^{i}	151.0(4)
O1-Na1-O4	79.7(2)	O5^{ii}-Na3-O5^{iii}	73.24(19)
O1-Na1-O5	63.3(2)	O5^{ii}-Na3-O8	139.56(18)
O1-Na1-O7	91.4(3)	O5^{ii}-Na3-O8^{i}	71.59(18)
O1-Na1-O9	154.6(3)	O5^{ii}-Na3-O9	102.9(2)
O1-Na1-O11	124.7(2)	O5^{ii}-Na3-O9^{i}	130.9(2)
O2-Na1-O3	85.7(2)	O5^{ii}-Na3-O12^{ii}	69.18(19)
O2-Na1-O4	132.4(3)	O5^{ii}-Na3-O12^{iii}	57.56(19)
O2-Na1-O5	122.9(3)	O8-Na3-O8^{i}	147.8(2)
O2-Na1-O7	69.6(2)	O8-Na3-O9	85.8(2)
O2-Na1-O9	79.7(3)	O8-Na3-O9^{i}	76.8(2)
O2-Na1-O11	151.6(3)	O8-Na3-O12^{ii}	75.4(2)
O3-Na1-O4	77.6(2)	O8-Na3-O12^{iii}	124.1(2)
O3-Na1-O5	150.8(3)	O9-Na3-O9^{i}	113.7(6)
O3-Na1-O7	124.3(3)	O9-Na3-O12^{ii}	75.0(2)
O3-Na1-O9	60.3(2)	O9-Na3-O12^{iii}	150.1(2)
O3-Na1-O11	82.9(2)	O12^{ii}-Na3-O12^{iii}	112.5(2)
O4-Na1-O5	85.3(3)		

Symmetry operators i = -x, 1-y, z; ii = -0.5+y, 0.5+x, -0.5+z and iii = 0.5-y, 0.5-x, -0.5+z.

Table S6. Geometry of intramolecular hydrogen bonds (HB) and selected intermolecular contacts of free calix[4]tube 1 made up of subunits A and B.

Contact type	D—H· · ·A	D—H (Å)	H· · ·A (Å)	D· · ·A (Å)	D—H· · ·A (°)
intra HB	O6A-H60...O6B	0.82	1.64	2.452(9)	169
	O10A-H100...O10B	0.82	1.63	2.442(10)	173
	O12A-H12A...O7B	0.82	1.84	2.662(8)	175
	O12B-H120...O8A	0.82	1.72	2.530(13)	169
inter HB	O8B-H8B...O1W	0.82	1.92	2.628(15)	144
	O1S-H10S...O6B	0.99	1.85	2.77(2)	152
	O1W-H2W...O11B	0.85	2.16	2.95(2)	155
	O1W-H1W...O1S	0.85	2.18	2.84(2)	134
inter CH...π	C1S-H1SA...Cg3(A)	1.09	2.50	3.43(2)	142
	C1S-H1SC...Cg1(A)	1.09	2.99	3.70(2)	123
	C1S-H1SC...Cg4(A)	1.09	2.70	3.49(2)	129
	C18A-H18A...Cg3(B)	0.93	3.59	4.480(9)	162
	C27A-H27A...Cg3(B)	0.93	3.30	4.216(11)	166

Table S7. Geometry of intramolecular hydrogen bonds (HB) and selected intermolecular contacts of free calix[4]tube 2 made up of subunits A, B, C and D.

Contact type	D—H· · ·A	D—H (Å)	H· · ·A (Å)	D· · ·A (Å)	D—H· · ·A (°)
intra HB (A-B)	O10A-H10A...O10B	0.82	1.68	2.492(11)	171
	O12A-H12A...O8B	0.82	1.67	2.447(9)	157
	O6B-H6B...O5A	0.82	1.88	2.65(2)	156
intra HB (C-D)	O6C-H6C...O12D	0.82	2.17	2.76(7)	129
	O10C-H10C...O7D	0.82	2.29	3.08(2)	162
	O10C-H10C...O10D	0.82	2.46	3.04(5)	129
	O12C-H12E...O9D	0.82	2.69	3.44(2)	154
	O12C-H12E...O10D	0.82	2.39	2.93(4)	124
	O6D-H6D...O8C	0.82	2.20	2.98(4)	158
	O8D-H8D...O9C	0.82	1.82	2.47(4)	135
inter HB	O12B-H12D...O1W	0.82	2.12	2.83(2)	145
	O1W-H1W...O8A	0.99	2.20	2.83(2)	120
	O1W-H1W...O11B	0.99	2.71	3.43(2)	129
inter CH...π	C25A-H25A...Cg1(D)	0.93	3.13	3.879(13)	140
	C26A-H26A...Cg3(D)	0.93	2.81	3.617(12)	145
	C25C-H25C...Cg1(B)	0.93	3.21	3.850(11)	128
	C26C-H26C...Cg2(B)	0.93	3.36	4.028(13)	130
	C26C-H26C...Cg3(B)	0.93	3.13	3.813(14)	132
		Cg...Cg distance			
inter π...π	Cg4(A)...Cg2(D)	4.014(6) Å			
	Cg4(B)...Cg4(C)	4.064(5) Å			

Table S8. Geometry of intramolecular hydrogen bonds (HB) and selected intermolecular contacts of polymeric calix[4]tube 3.

Contact type	D—H· · ·A	D—H (Å)	H· · ·A (Å)	D· · ·A (Å)	D—H· · ·A (°)
intra HB	O8-H8...O10	0.82	1.67	2.442(11)	155
	O12-H120...O6	0.82	1.69	2.450(11)	154
inter CH...O	C5-H5...O1S	0.93	2.49	3.397(15)	166
	C13-H13...O1S	0.93	2.40	3.307(16)	165
inter CH...π	C2S-H2S2...Cg2	0.96	3.19	4.055(17)	151
	C2S-H2S3...Cg3	0.96	3.35	3.991(16)	126
	C2S-H2S3...Cg4	0.96	3.33	4.129(16)	143

Table S9. Geometry of intramolecular hydrogen bonds (HB) and selected intermolecular contacts of polymeric calix[4]tube 4.

Contact type	D—H· · ·A	D—H (Å)	H· · ·A (Å)	D· · ·A (Å)	D—H· · ·A (°)
intra HB	O8-H8...O10	0.82	1.70	2.498(11)	163
	O12-H120...O6	0.82	1.65	2.441(11)	160
inter CH...O	C5-H5...O1S	0.93	2.83	3.75(2)	169
	C13-H13...O1S	0.93	2.46	3.39(2)	174
inter CH...π	C1S-H2S1...Cg4	0.96	2.89	3.71(3)	144
	C1S-H2S2...Cg3	0.96	2.96	3.81(3)	148
	C1S-H2S3...Cg1	0.96	3.06	3.74(3)	130

5. Figures

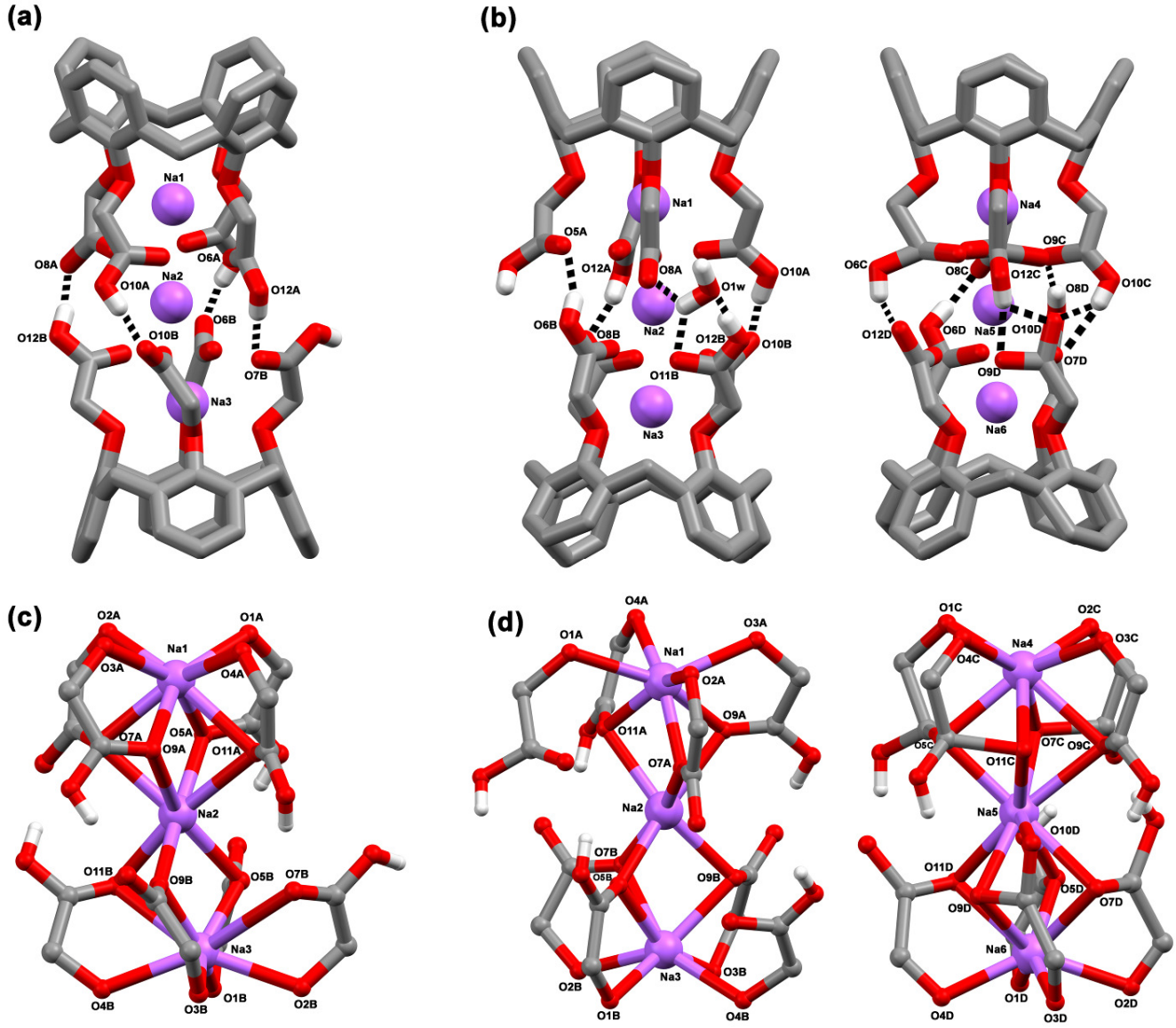


Figure S1. The free dimeric nanotubes present in the asymmetric units of (a) 1 and (b) 2. In these panels, coordination bonds were hidden for clarity and hydrogen bonds sealing the central region of the dimers are shown as dashed black lines. The coordination environment of sodium ions found in (c) 1 and (d) 2. In these panels, hydrogens and carbons from cones were omitted to highlight only coordination bonds hidden in panels (a) and (b). In all panels, only carboxyl hydrogens are displayed.

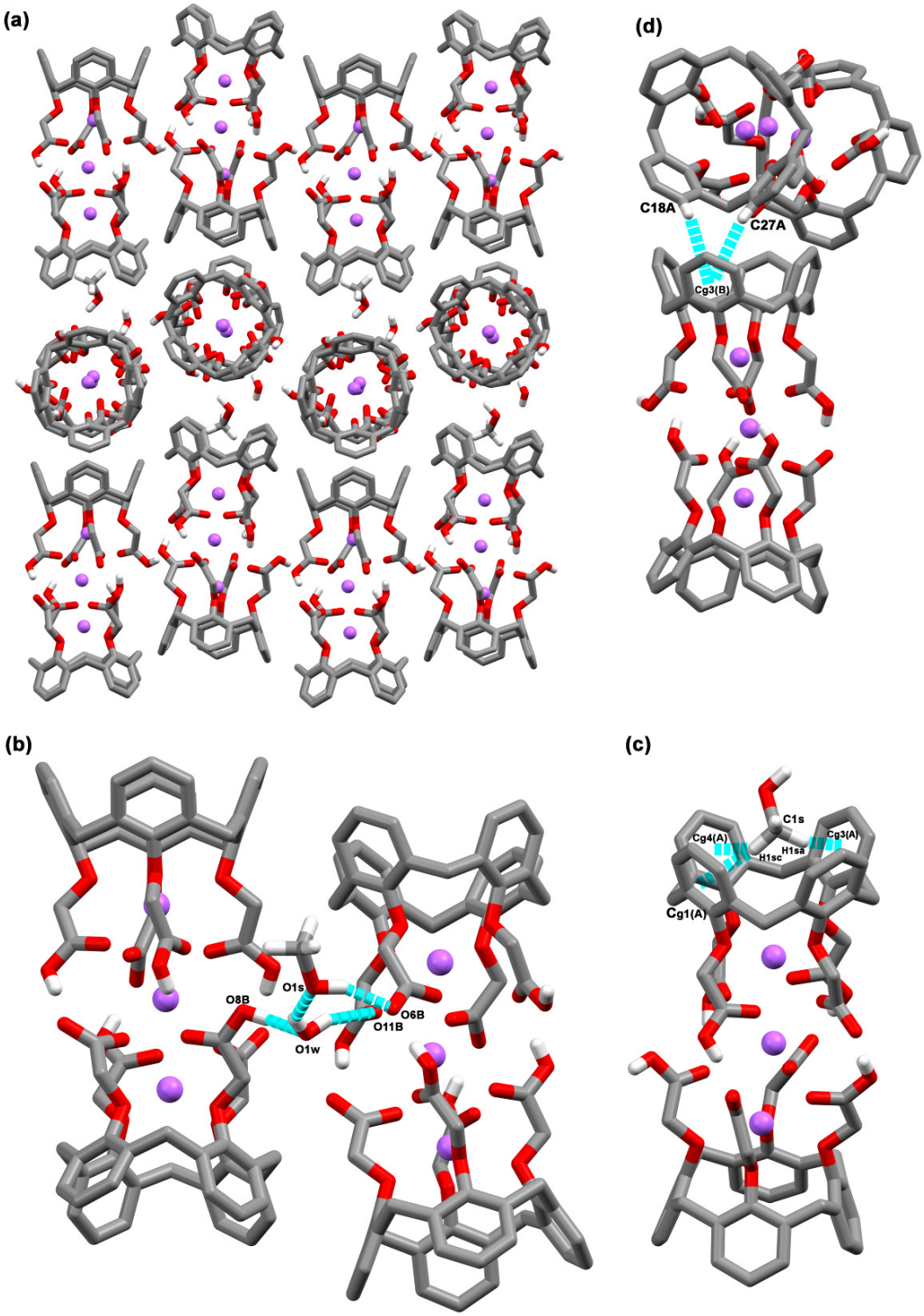


Figure S2. (a) Crystal packing of the dimeric nanotubes in 1 and the main intermolecular interactions (dashed cyan lines) responsible for lattice stabilization: (b) hydrogen bonds involving carboxyl and carboxylate moieties from calixarene units, methanol and water molecules; C-H…π contacts (c) trapping each methanol molecule into the cone from unit A [Cg1(A), Cg3(A) and Cg4(A) centroids were calculated through C1A to C6A, C15A to C20A and through C22A to C27A, respectively] and (d) including partially phenyl moieties from unit A into the cone from unit B [Cg3(B) centroid was calculated through C15B to C20B].

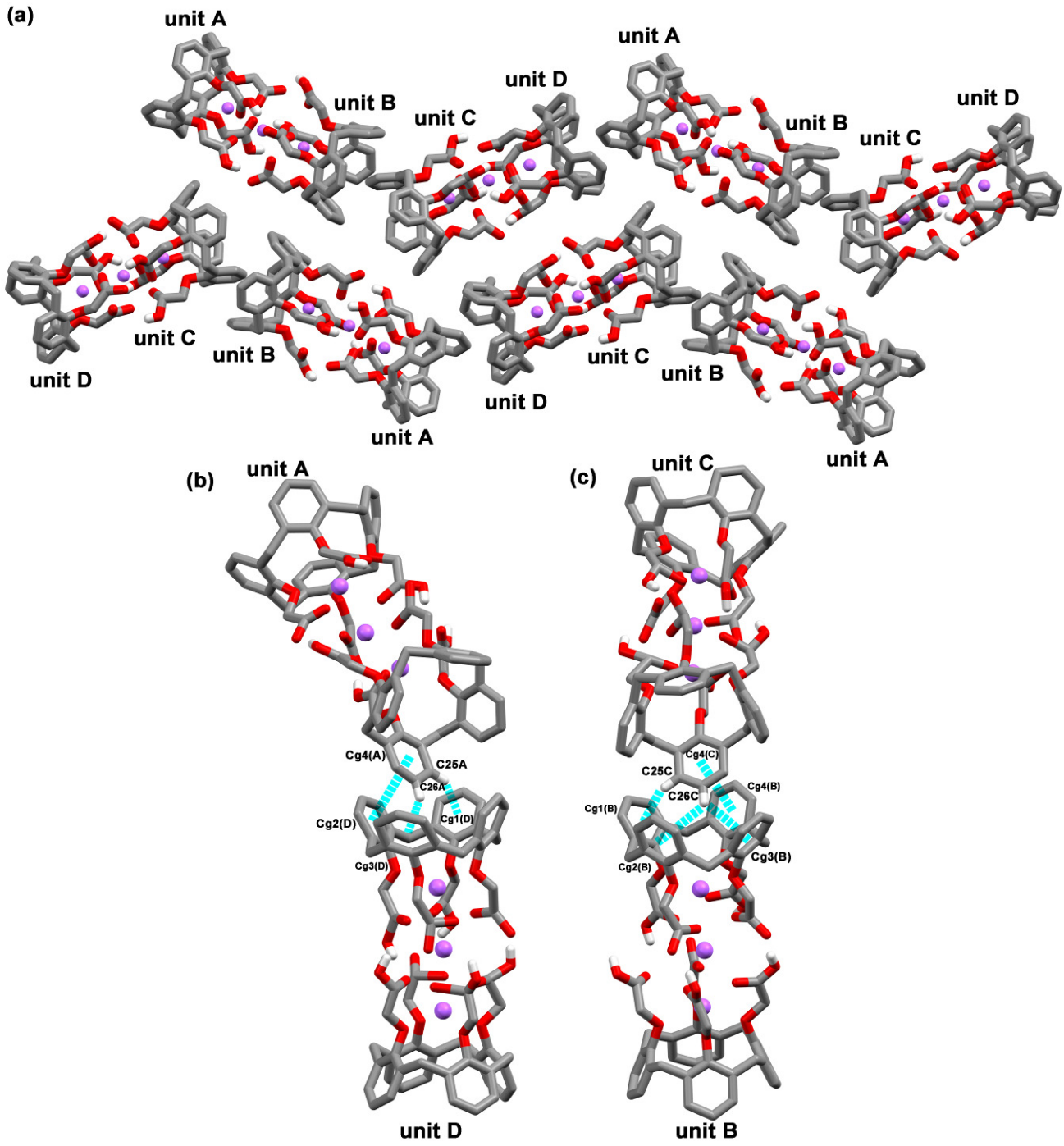


Figure S3. (a) Crystal packing of the dimeric nanotubes in 2 and the C-H…π and π…π contacts (dashed cyan lines) including (b) one phenyl moiety from calixarene unit A [its Cg4(A) centroid was calculated through C22A to C27A] into the cone from unit D of another nanotube [Cg1(D), Cg2(D) and Cg3(D) centroids were calculated through C1D to C6D, C8D to C13D, and C15D to C20D] and (c) one phenyl moiety from calixarene unit C [its Cg4(C) centroid was calculated through C22C to C27C] into the cone from unit B of a neighboring nanotube [Cg1(B), Cg2(B), Cg3(B) and Cg4(B) centroids were calculated through C1B to C6B, C8B to C13B, C15B to C20B, and C22B to C27B].

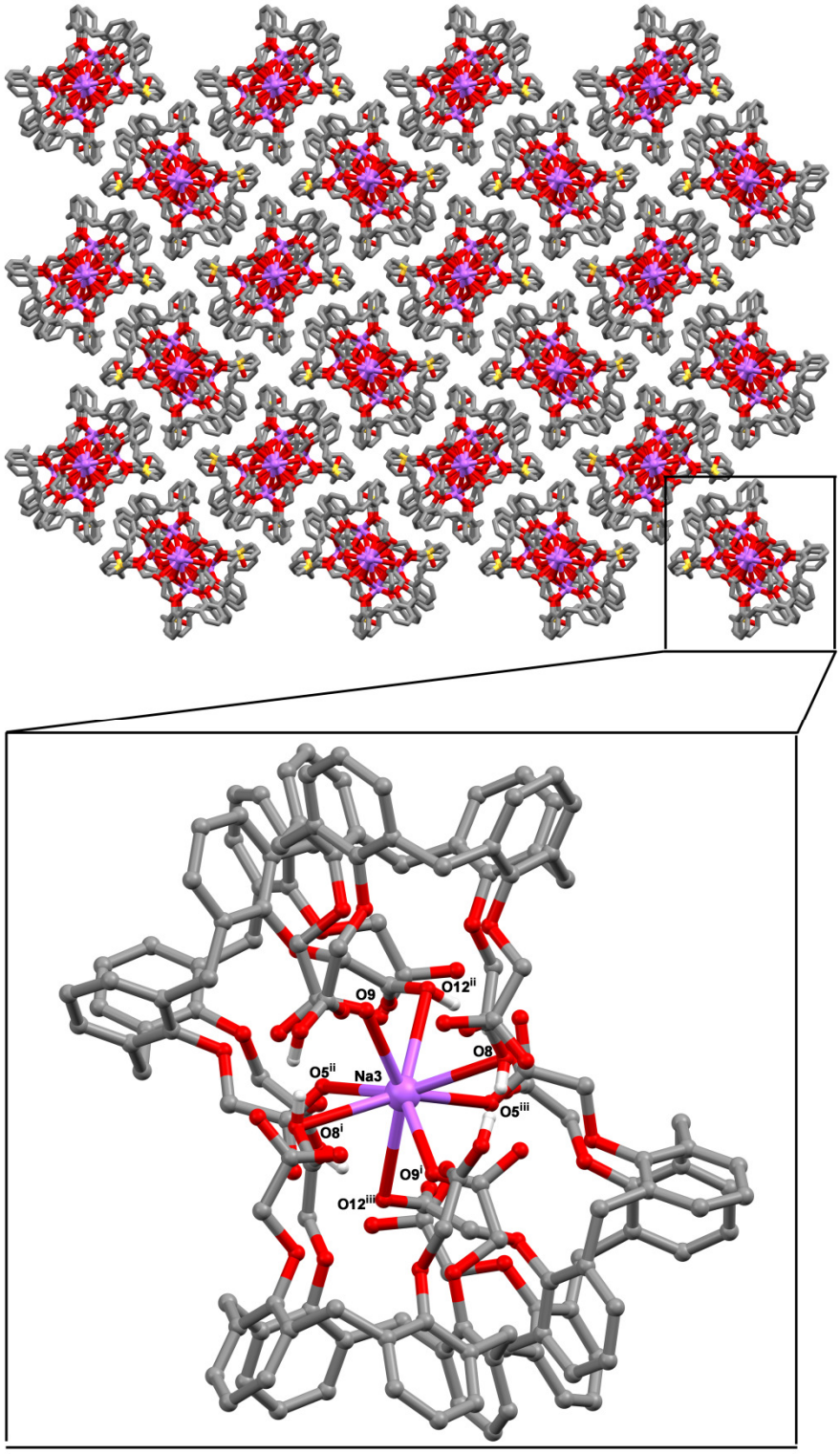


Figure S4. Crystal packing of isostructural polymeric calix[4]tubes 3 and 4 showing their 1D polymeric chains running as drawing projection. One polymer is framed onto the box to show the coordination surrounding of Na3 sodium ion holding together the nanotubes. Polymeric calix[4]tube 3 was chosen for drawing. Symmetry operators for calix[4]tube 3: *i* = 1-x, 2-y, z; *ii* = -0.5+y, 0.5+x, -0.5+z and *iii* = 1.5-y, 1.5-x, -0.5+z; and for calix[4]tube 4: *i* = 2-x, 1-y, z; *ii* = 0.5+y, -0.5+x, 0.5+z and *iii* = 1.5-y, 1.5-x, 0.5+z.

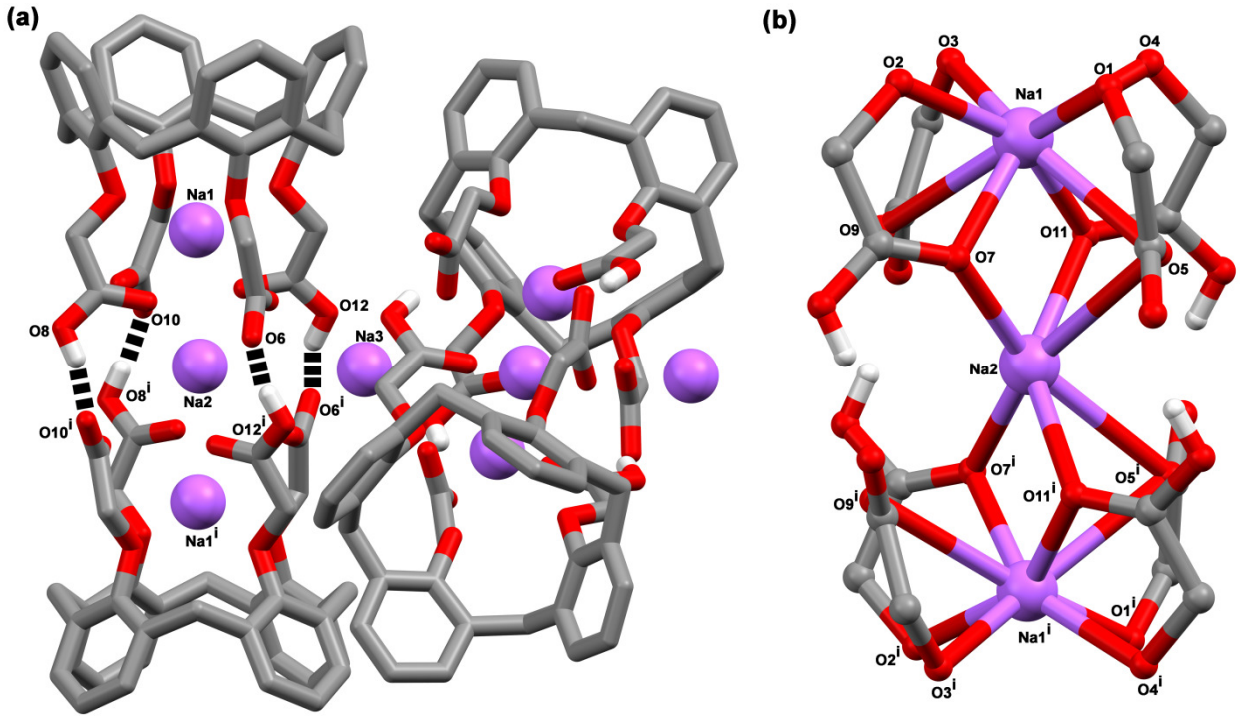


Figure S5. (a) Two dimeric nanotubes coordinated to Na3 into a 1D polymeric chain found in the isostructures of 3 and 4. In this panel, coordination bonds were hidden for clarity and hydrogen bonds sealing the central region of the nanotubes are shown as dashed black lines. (b) The coordination environment of sodium ions trapped into nanotube structure of 3. In this panel, hydrogens and carbons from cones were omitted to highlight only coordination bonds hidden in the previous panel. In all panels, polymeric calix[4]tube 3 was chosen for drawing and only carboxyl hydrogens are displayed. Symmetry operator *i* = 1-x, 2-y, z.

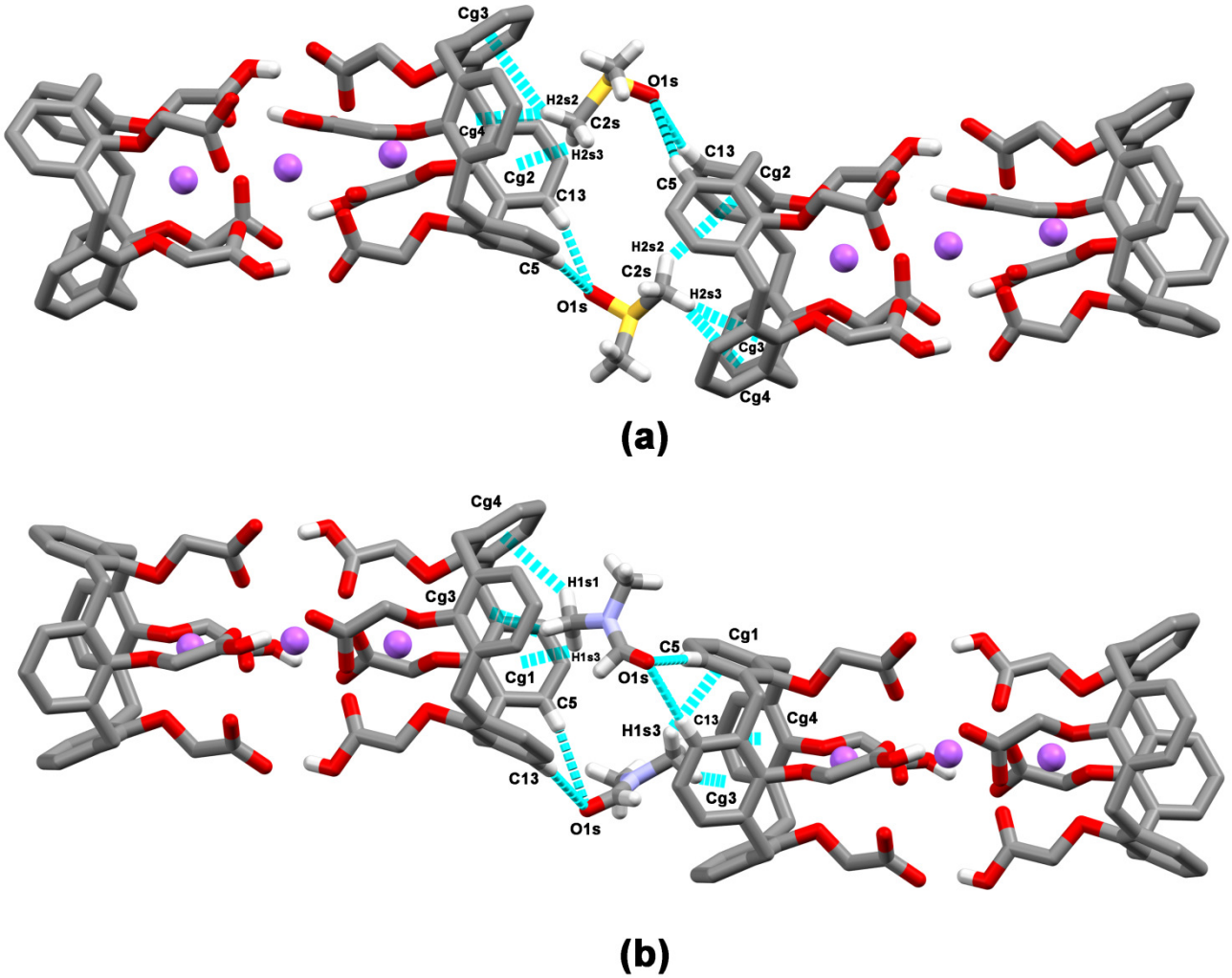


Fig S6. One (a) dimethylsulfoxide or (b) one dimethylformamide molecule is trapped into each cone of isostructural nanotubes assembled in 3 and 4. Solvent molecules do also interact with a neighboring polymeric chain through non-classical hydrogen bonding acceptation. Cg1, Cg2, Cg3, and Cg4 centroids were calculated through C1 to C6, C8 to C13, C15 to C20, and C22 to C27.

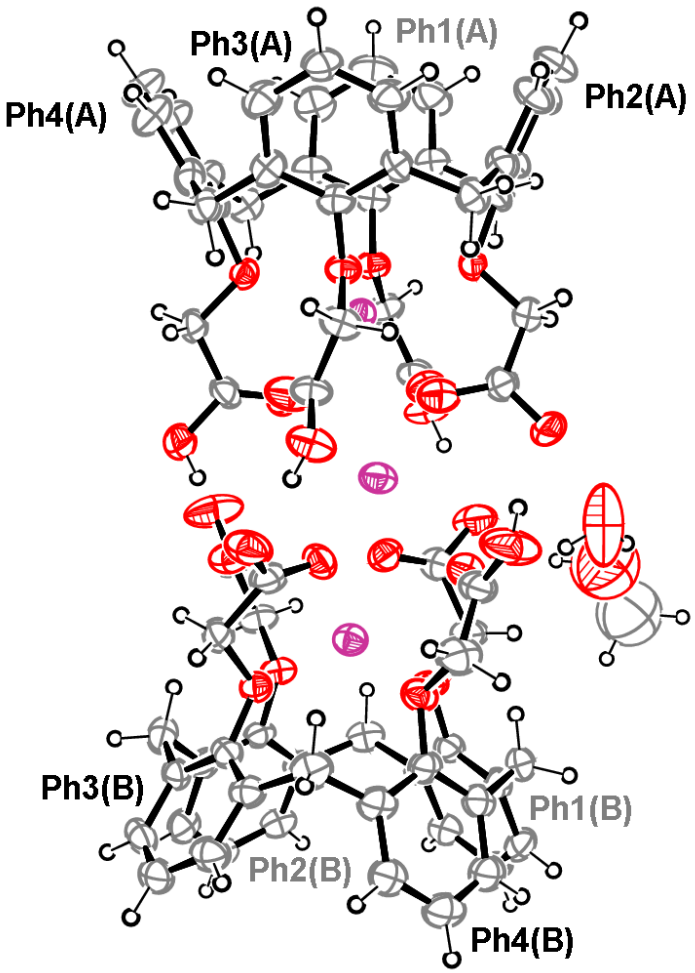


Figure S7. Asymmetric unit of free calix[4]tube 1 showing the labeling scheme of phenyl rings. Hydrogen and non-hydrogen atoms are drawn as arbitrary radius spheres and 30% probability ellipsoids, respectively.

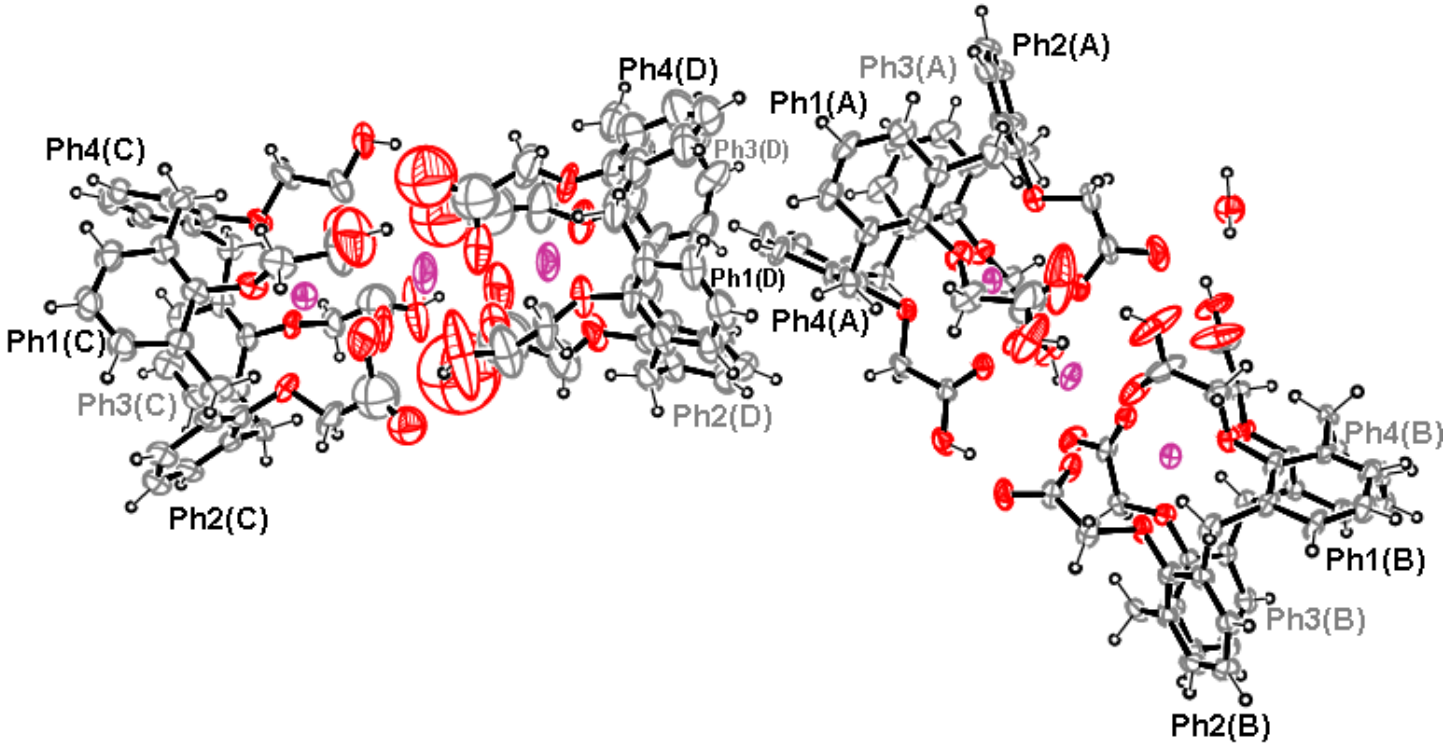


Figure S8. Asymmetric unit of free calix[4]tube 2 showing the labeling scheme of phenyl rings. Hydrogen and non-hydrogen atoms are drawn as arbitrary radius spheres and 30% probability ellipsoids, respectively.

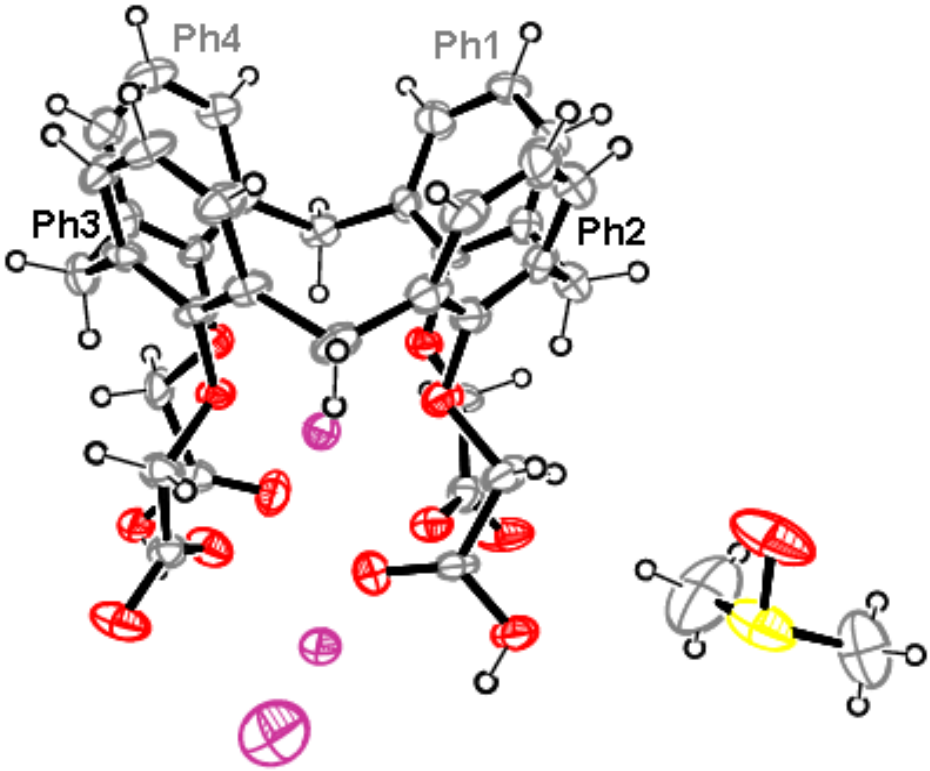


Figure S9. Asymmetric unit of polymeric calix[4]tube 3 showing the labeling scheme of phenyl rings. Hydrogen and non-hydrogen atoms are drawn as arbitrary radius spheres and 30% probability ellipsoids, respectively.

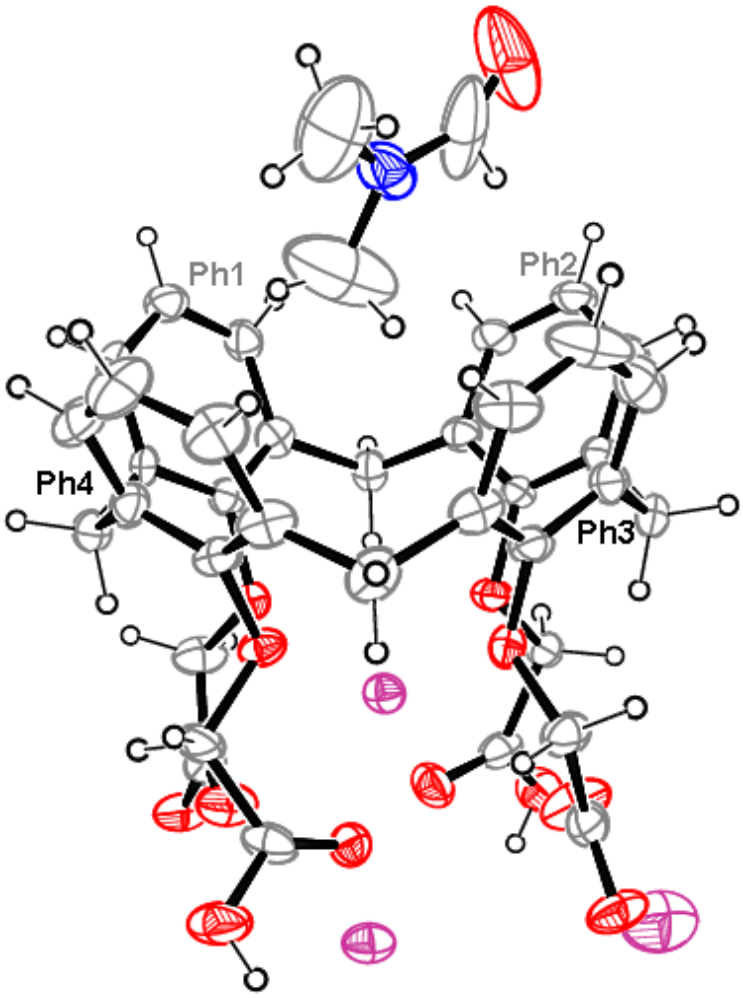


Figure S10. Asymmetric unit of polymeric calix[4]tube 4 showing the labeling scheme of phenyl rings. Hydrogen and non-hydrogen atoms are drawn as arbitrary radius spheres and 30% probability ellipsoids, respectively.

6. References

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