Towards ionic channels with crown ethers and 4-*tert*-butylcalix[n]arenes (with n=6, 8)

Inauguraldissertation

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Prof. Dr. Hans-Jakob Wirz

A mes parents,

A Lucien Bloise et au Dr. Alain Manthegetti,

Ainsi qu'à Noël.

"They did not know it was impossible, so they did it" Mark Twain

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Some of the following results have been reported in these publications [1-6]:

[1] E. D. Gueneau, Synthèse et caractérisation de clusters de métaux alcalin et/ou alcalino-terreux" Thèse n° 3452.

[2] K. M. Fromm, E. D.Gueneau, H. Goesmann, C. G. Bochet, *Z. Anorg. Allg. Chem.* (**2003**), 629(4), 597-600.

[3] R. D. Bergougnant, A. Y. Robin, K. M. Fromm, *Crystal Growth & Design* (**2005**), 5(5), 1691-1694.

[4] K. M. Fromm, E. D. Gueneau, A. Y. Robin, W. Maudez, J. Sague, R. Bergougnant, *Z. Anorg. Allg. Chem.* (**2005**), 631(10), 1725-1740.

[5] K. Fromm, R. D. Bergougnant, A. Y. Robin, *Z. Anorg. Allg. Chem.* (**2006**), 632(5), 828-836.

[6] M. Dułak, R. Bergougnant, K. M Fromm, H. R. Hagemann, A. Y. Robin, and T. A. Wesołowski, *Spectrochim. Acta A*, (**2006**), 64A (2), 532-548.

Contents

I Introduction

I.1. Ion channels

Water molecules and ions play important roles in the biological cells. To transport these species through cellular membranes, life has produced a wide diversity of water and ion channels. An ion channel is an integral membrane protein or, more typically, an assembly of several proteins. Such "multi-subunit" assemblies usually involve a circular arrangement of identical or homologous proteins closely packed around a water-filled pore through the plane of the membrane or lipid bilayer (Figure I.1) [7-9]. While large-pore channels permit the passage of ions more or less indiscriminately, the archetypal channel pore is just one or two atoms wide at its narrowest point. It conducts a specific ion, such as sodium or potassium, and conveys it through the membrane single file nearly as quickly as the ions move through a free fluid. In some ion channels, access to the pore is governed by a "gate," which may be opened or closed by chemical, for example $GABA_A$ (Gamma Aminobutyric Acid type A), or electrical signals (Voltage-gated channels like these for Na^+, K^+, Cl^- ions), temperature, or mechanical force (Stretch- and heat-activated channels), depending on the channel.

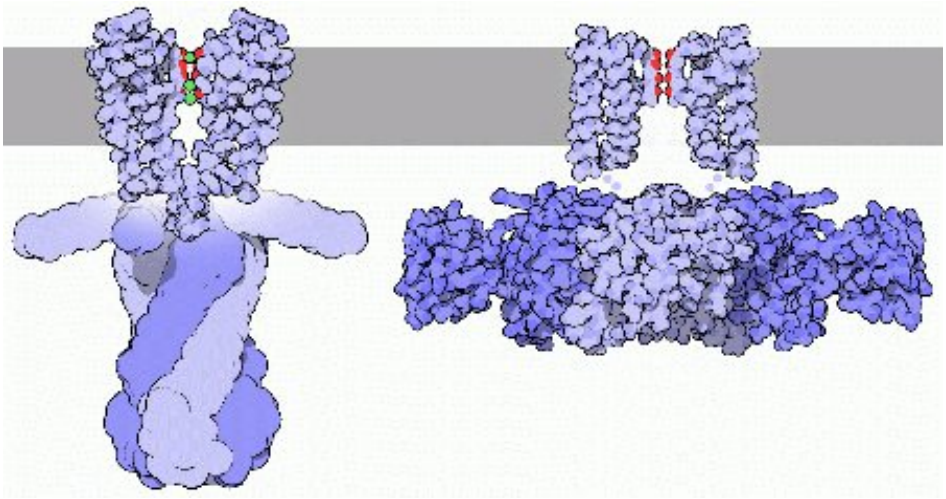
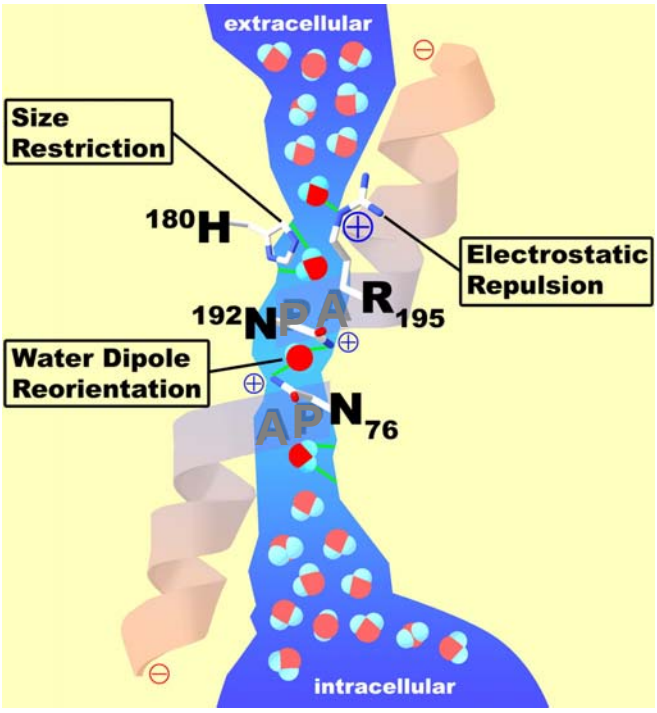


Figure I.1: Potassium channel of bacteria in an open position (right) and closed (left).

Among the most studied channels are i) aquaporin, a water channel and ii) the potassium ion channel.

Aquaporins are a class of integral membrane proteins that form pores in the membrane of biological cells and selectively conduct water molecules in and out through an unidirectionnal process, while preventing the passage of ions and other solutes thanks to simple electrostatic interactions (size restriction applied to larger molecules, dipole interaction impossible with ions). Also known as water channels, each is composed of four identical subunit proteins. Water molecules traverse the narrowest portion of the channel single file. The presence of water channels increases the permittivity of membranes to water by as much as ten fold. However, because the process is passive, aquaporins cannot reverse the direction of the osmotic gradient driving the flow of water. The 2003 Nobel Prize in Chemistry was awarded to Peter Agre (jointly with Roderick MacKinnon for his work on potassium channels) for his discovery of aquaporins and elucidation of their structure and function (Figure I.2).



H: histidine, R: arginine,A: alanine, P: Proline, N: Asparagine

Figure I.2: Functional schematic for water passage through AQP1 (AQP= aquaporin).

There are ten known types of aquaporins, and six of these are located in the kidney. The most studied aquaporins are AQP1 (Figure I.3), AQP2, AQP3, and AQP4 [10]. The number after the sign AQP (for aquaporin) are here to remember the order of discovery. There is also some difference between each protein. For example, some aquaporins are regulated by some molecule such as AQP2 which is sensitive to vasopressin. Some aquaporins can let also some other small molecules (urea, glycerol) go through the membrane such as AQP3. Finally aquaporins can be used everywhere in the body such as AQP1 (present in kidney, lung, or eyes) or be designed for a part of the body like AQP4 mainly located in the brain.

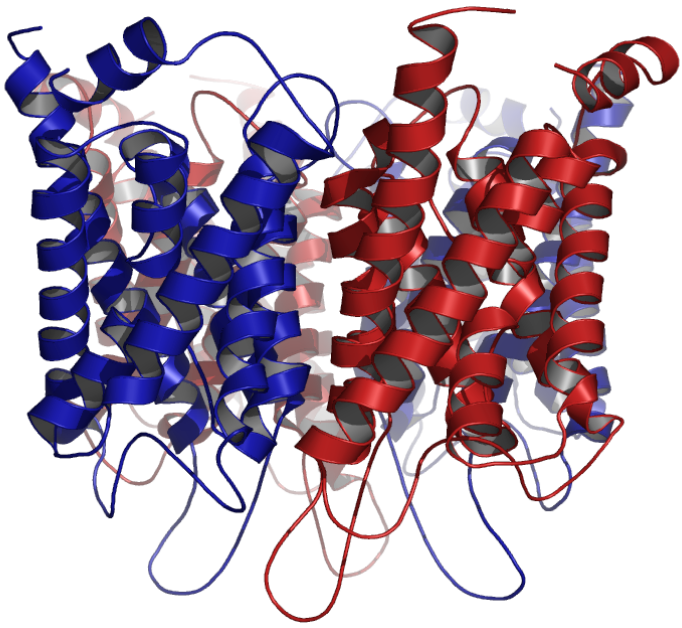
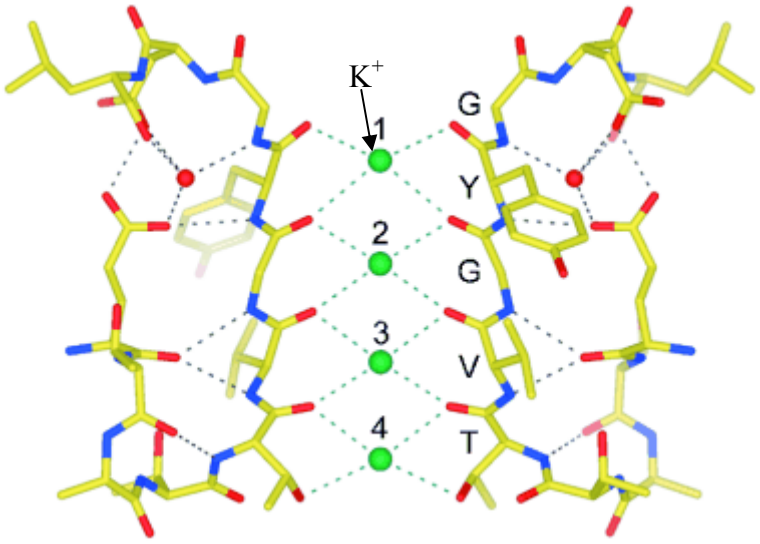


Figure I.3: Example of aquaporin representation AQP1.

In cell biology, potassium channels are among the most common type of ion channel with sodium or ATPase channel. They form potassium-selective pores that span cell membranes. Potassium channels (Figure I.4) are found in most cells, and control the electrical excitability of the cell membrane. In neurons, they shape action potentials. An action potential is a wave of electrical discharge that travels along the membrane of a cell. Ion channels support this electrical discharge, and set the resting

membrane potential. They regulate cellular processes such as the secretion of hormones, so their malfunction can lead to diseases.



T: Threonine, V: Valine, G: Glycine, Y: Tyrosine

Figure I.4: Detailed structure of the K^+-selectivity filter (two subunits).Oxygen atoms (red) coordinate K^+ ions (green spheres) at positions 1to 4 from the extracellular side. Single letter amino acid code identifies select amino acids of the signature sequence (yellow: carbon, blue: nitrogen, and red: oxygen). Green and gray dashed lines show O···K^+ and hydrogen-bonding interactions, respectively.

Some potassium channels are voltage-gated ion channels which means that such channels open or close in response to changes in the transmembrane voltage. Not all potassium channel are voltage-gated, they can also open in response to an intracellular increase of calcium ions or other signalling molecules concentration. Others are constitutively open or possess high basal activation, such as the resting potassium channels that set the negative membrane potential of neurons. When open, they allow potassium ions to cross the membrane at a rate which is nearly as fast as their diffusion through bulk water. There are over 80 mammalian genes that encode potassium channel subunits. The pore-forming subunits of potassium channels have a homo- or heterotetrameric arrangement. Four subunits are arranged around a central pore. All potassium channel subunits have a distinctive pore-loop structure that lines

the top of the pore and is responsible for ion selectivity. Each pore-loop (called H5 or P) creates interactions with potassium ions and with no other ions thanks to the organisation of their constitutive amino acid (arginine, glutamate).

Potassium channels found in bacteria are amongst the most studied of ion channels, in terms of their molecular structure. Using X-ray crystallography, profound insights have been gained into how potassium ions pass through these channels and why (smaller) sodium ions do not (A sodium ion is smaller than a potassium ion, but it does not jump into the potassium channel, because the selectivity filters have a very precise conformation which is more tuned for the larger potassium ion.). The 2003 Nobel Prize for Chemistry was awarded to Roderick MacKinnon for his pioneering work on this subject [11].

Such examples led to a lot of questions on the type of mechanism needed for ion translocation. They furnish also good examples of ion carriers which could be used in nanotechnology. Therefore great efforts have been made in chemical research to design mimicking ion channels. Among the compounds used by chemists and biologists, two families have taken an important place in the literature: crown ethers and calix[n]arenes, though porphyrins should be mentioned as well [12-34].

I.2. Crown ethers

Since the pioneering work of Pedersen [35], crown ethers have gained more and more interest in the context of anionic synthetic reagents, phase transfer catalysts, biological ion transfer, molecular switches and other applications such as sensors or biological model systems [36].

Crown ethers are compounds that, in their best known form, are cyclic oligomers of ethylene oxide. The essential repeating unit of any simple crown ether is ethyleneoxy, i.e., $-CH_2CH_2O-$, which repeats six times in 18-crown-6. Macrocycles of the $(-CH_2CH_2O-)_n$ type in which $n \geq 4$ are generally referred to as crown ethers rather than by their systematic names due to the special conformation of crown ether with complexed cations (Figure I.5). Pedersen examined molecular models of dibenzo-18-

crown-6 and felt that the interaction between receptor and guest ion was such that the host “crowned” the cation.

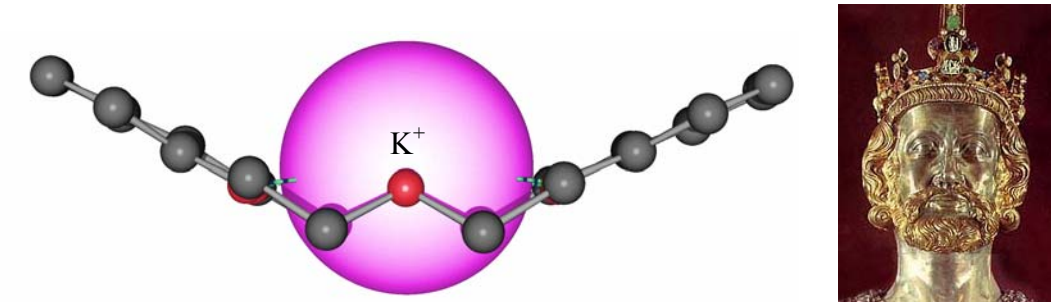


Figure I.5: Example of dibenzo18-crown-6 capping a potassium ion compared to Charles the Great with his crown.

General synthesis of crown ethers is achieved by condensation of a diol with dichloroalkanes with a SN2 mechanism in basic conditions. Alkali metal ions can act as a template during the synthesis and help the cyclisation (Figure I.6). The template effect is a well-recognized phenomenon which arises from the complexation of the crown’s precursor around the metal ion leading to what has been called a “pre-crown” complex. In large ring formation, loss of the conformational entropy due to internal rotations around the single bonds of the bifunctional chain precursor provides a major contribution to the free energy of activation. Reduction of the conformational entropy upon multiple coordination of the polyoxa chain with the metal ion should result in a greater “proximity” of the chain ends in the metal-associated chain than in the unassociated one. This is believed to be the basis for the template effect.

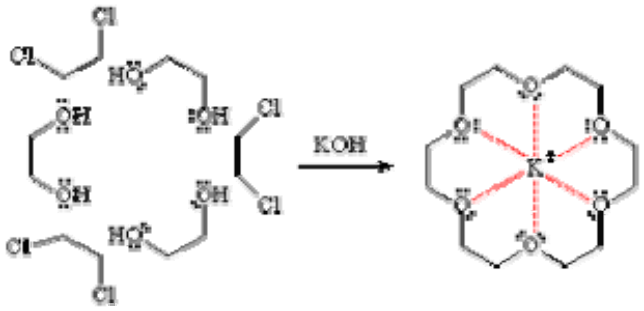


Figure I.6: Synthesis of 18-crown-6 by condensation of 1,2-ethanediol on 1,2-dichloroethane.

At the very beginning, many different methods were described for the synthesis of crown ethers. Five methods with different starting materials were reported by Pedersen [35]. Instead of diols, dicarboxylic acids [37], secondary amines [36], or sugars can be also used [39] and metal transition salts can catalyze the reaction [40]. Other molecules led to the development of related families like the aza-crowns, cryptands or macropolycyclic polyethers [41-43].

The crown ethers are known for their ability to strongly enhance cation solubility in organic solvents. The oxygen atoms are ideally situated (normally at the ionic bond distance) to coordinate with a cation inside the ring, whereas the outside of the ring is hydrophobic. The result of this hydrophilic heart-hydrophobic shell is that the complexed cation is soluble in nonpolar solvents thanks to the hydrophobic shell. The size of the inner ring of the crown ether determines the size of the cation it can solvate. Therefore, 18-crown-6 has high affinity [36] for the potassium cation, 15-crown-5 for the sodium cation and 12-crown-4 for the lithium cation (Table I.1). Besides the relative sizes of the ion and the hole in the polyether ring, some other factors drive the complexation such as the number of oxygen atoms in the polyether ring, the coplanarity of the oxygen atoms, the symmetrical placement of the oxygen atoms, the basicity of the oxygen atoms, steric hindrance in the polyether ring, the tendency of the ion to associate with the solvent and the electrical charge of the ion [36].

Cations	Ionic diameters of cations (Å)	Macrocyclic polyethers	Relative sizes of the hole in crown ether (Å)
Li^+	1.20	All 12-crown-4	1.2-1.5
Na^+	1.90	All 15-crown-5	1.7-2.2
K^+	2.66	All 18-crown-6	2.6-3.2

Table I.1: Comparison between some cation and hole diameters.

As mentioned above, crown ethers play several roles in chemistry. The most studied is of course the general complexing ability of such a macrocycle. The large diversity of crown ethers allows the complexation of a large variety of metal ions such as p-block metal ions or lanthanides [44, 45] but more than that, ions such as ammonium and primary alkylammonium ions and also cylindrical cations (benzenediazonium, benzoyl and nitrosyl cations) [46-49]. Furthermore, catenanes (molecule with interlocking rings) and rotaxanes (mechanically-interlocked molecular architectures) based on crown ethers are able to bind molecules like DNA. Some examples are found in bacteria and viruses [36]. If crown ethers are able to bind positively charged or neutral molecules, it exists the possibility of protonation of heteromacrocycles such as azacrownether in order able to bind anion like chloride or more, alkalide and electride [50, 51]. The alkalides are a class of ionic compounds where the anions are of the group I (alkali metal) elements Na, K, Rb, Cs (no known 'Lithide' exists). The cation is an alkali cation complexed by a large organic complexant. The resulting chemical form is $[A^+$ Complexant$]$ B^-, where the complexant is either a cryptand, crown ether, or aza-crown. The electrides are just like alkalides except that the anion is presumed to be simply an electron which is localized to a region of the crystal between the complexed cations.

Another important part of crown ether chemistry is the molecular switch. Molecular switching involves changes in charge state, conformation or structure that enable or prevent cation complexation in a host structure that previously could not or could, respectively, bind a guest. Photochemical switching (Figure I.7), redox and electrochemical switching are among the most common [52-60].

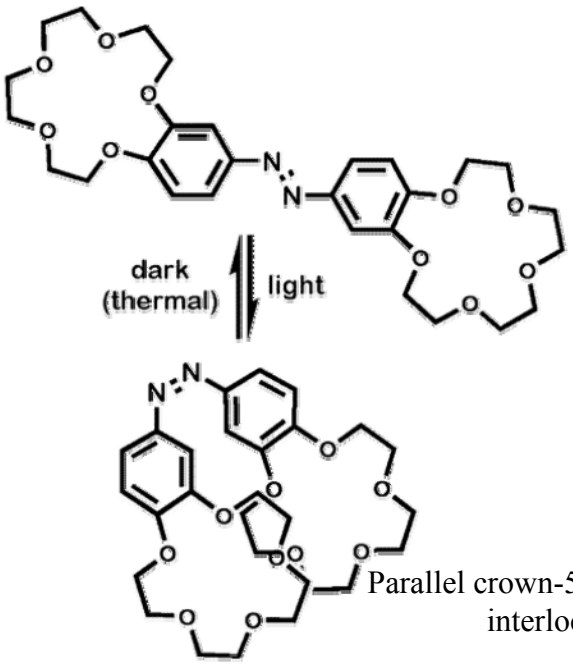


Figure I.7: Photochemical switching of crown ether systems.

The concepts of molecular switching and sensing evolved more or less simultaneously in the crown ether field. Molecular switching involves changes in charge state, conformation, or structure that enable or prevent cation complexation in a host structure that previously could not or could, respectively, bind a guest. Crowns are an integral part of many sensors as they provide the binding scaffold for the guest, the presence of which is detected by a change in some detectable property. In sensor molecules that involve crown binding sites, the complexation process takes place to a greater or lesser extent than in molecular switching. There are numerous examples in the literature of crown ethers used as sensors for a broad range of inorganic ions [61-66]. Their selective receptor properties, in conjunction with the relative ease of synthesis and structural modification, make crown ethers attractive targets as ionophores. Several recent examples of ionophores and the metals they complex can be seen in Figure I.8.

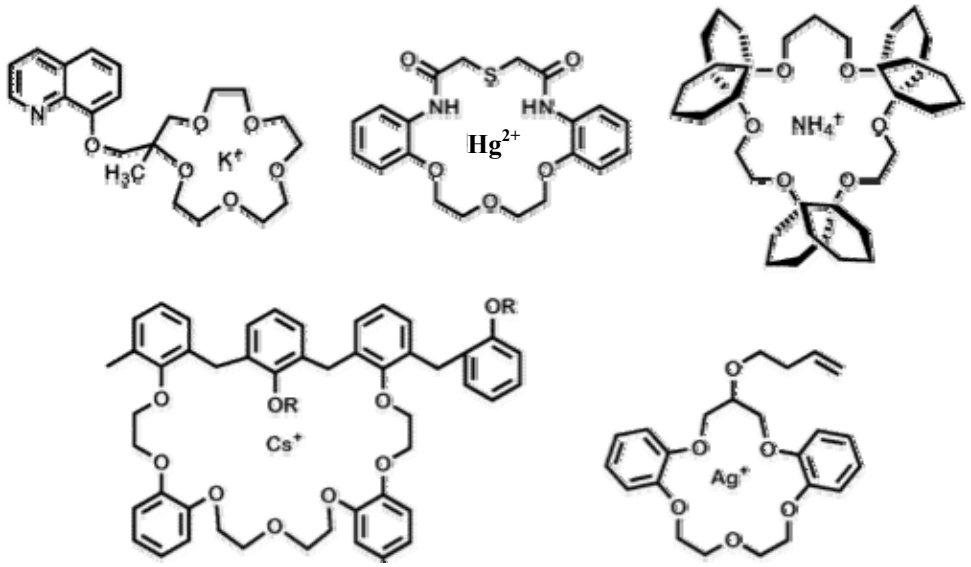


Figure I.8: Crown ether-based ionophores.

Crown ethers can also be integrated in a larger structure like in polymers or be the central core of a dendrimer unit. These structures lead to application in nanotubes [67] or light-emitting devices (LEDs) [68]. Attached to silica, crown ethers can be efficient agents in separation problems as in chromatography.

Finally, crown ethers find applications in biomimicry because relatively simple molecular structures that mimic biological function offer many advantages. Typically such models are of sufficiently low molecular weight that they can be fully characterized in the chemical sense. This includes detailed spectroscopic analysis and, in many cases, an X-ray crystal structure. Structural modifications in a functioning model can be directly assayed, and any change in the molecular arrangement can be determined precisely. Such macrocycles propose a simple chemical mechanism with one, or more identical molecules to explain a complicated biological mechanism controlled by a combination of peptides. The key issue, of course, is that the model must be functional. A compound designed to be an ion channel may, for example, have a tubular shape. Unless it can insert into a bilayer and transport ions, however, it is little more than a sculpture. Chemists are increasingly designing and synthesizing chemical structures that either function as function more complex chemical structures

or permit a detailed analysis of interactions that are thought to be important in biology. Four main applications are described in literature:

- Receptors for cation-π interactions [69-75]. Indeed, three of the twenty common amino-acids have a side chain with aromatic cycles (phenylalanine, tryptophan, tyrosine) that could serve as a π-donor for an alkali-metal cation but their interactions are not well known until now. It is of particular interest to study these phenomena.
- Amphiphiles (chemical compounds possessing both hydrophilic and hydrophobic nature) for membrane formation. The combination of hydrophobic tail and polar headgroups in alkyl-substituted crown ethers permits the formation of remarkably rigid and highly organized aggregates. Those associations are really interesting as the basis of bilayer systems or membranes.
- Ion channel model systems with the most known "chundles" reported by Lehn [76], the bola-amphiphiles of Fyles [77], Nolte's polymerized isonitriles [78], Voyer's crown substituted peptides [79], the redox-switchable systems of Hall [80], and the steroid-substituted crowns of Pechulis, Frye, et al. [81]. Figure I.9 shows the channel systems developed by Voyer (top), Fyles (middle), and Hall (bottom).
- Biological activity of crown ethers such as the ability to regulate enzyme activity, interaction with and cleavage of DNA, and action as antimicrobial agents [82-85].

Those examples give a broad overview of crown ether chemistry and its potential applications.

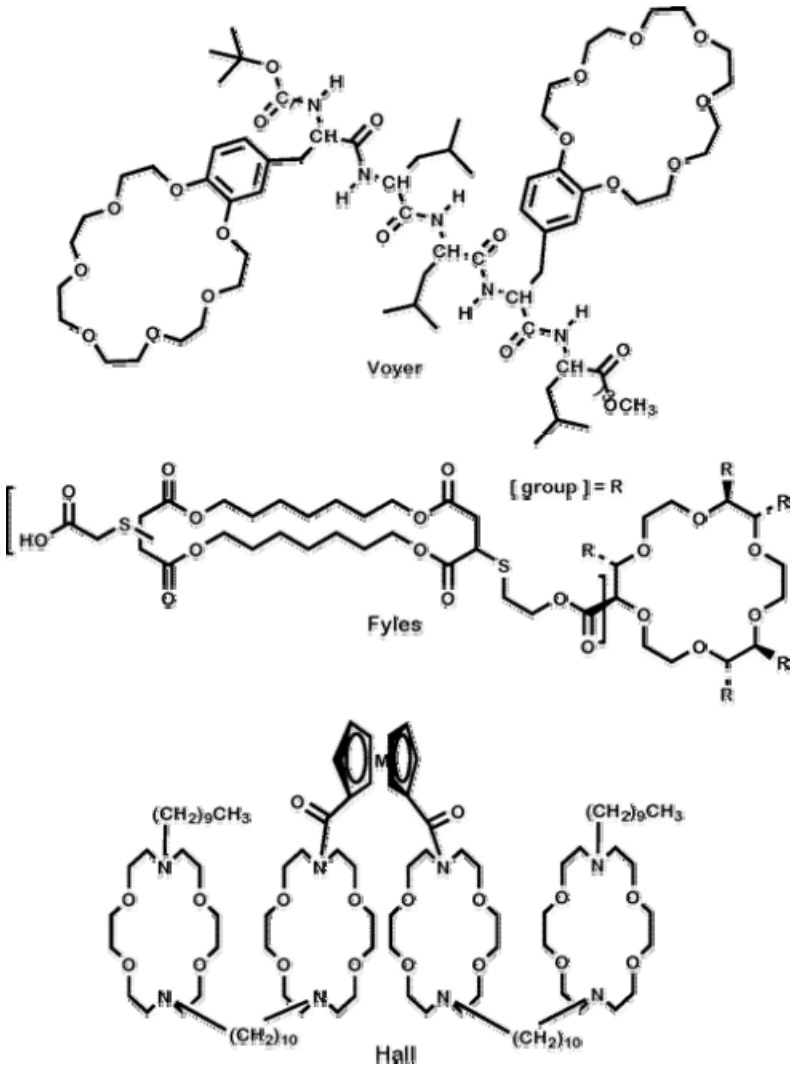


Figure I.8: Synthetic model ion channel systems.

I.3. Calix[n]arenes

Initiated by Baeyer, phenol and formaldehyde chemistry produced a lot of new polymers, among them Bakelite from Baekeland. It is only by changing phenol for p-substituted-phenol that Zinke and Ziegler isolated the first calix[n]arenes. Finally, Gutsche who was looking at molecules as enzyme mimics decided to use the Zinke

product. In this way, he initiated the chemistry of one of the most important building blocks of supramolecular chemistry [86, 87].

Calix[n]arenes are cyclic molecules composed of phenol derivative groups linked together by methylene bridges for the most general case (oxygen or sulfur bridges and others are also possible) as shown in figure I.10. The name of this compound was given by Gutsche as he noticed the analogy between an ancient vase shape (calix in Greek) and the shape of 4-tert-butylcalix[4]arene in cone conformation. Arene refers to the presence of aromatic rings in the structure.

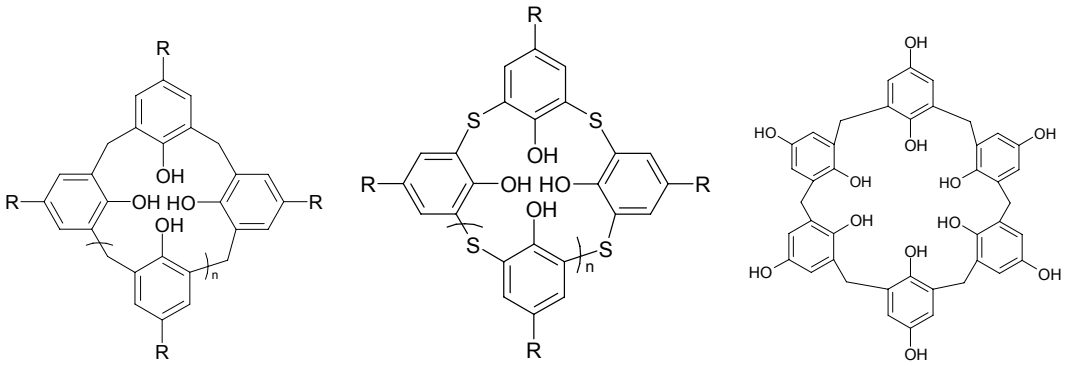


Figure I.10: Calix[n]arene, thiacalix[n]arene, p-hydroxycalix[6]arene.

Most commonly, their synthesis is achieved by condensation of formaldehyde and phenol derivatives in basic conditions. Alkali metal ions can act as a template during the synthesis and help the cyclisation (Figure I.11.a).

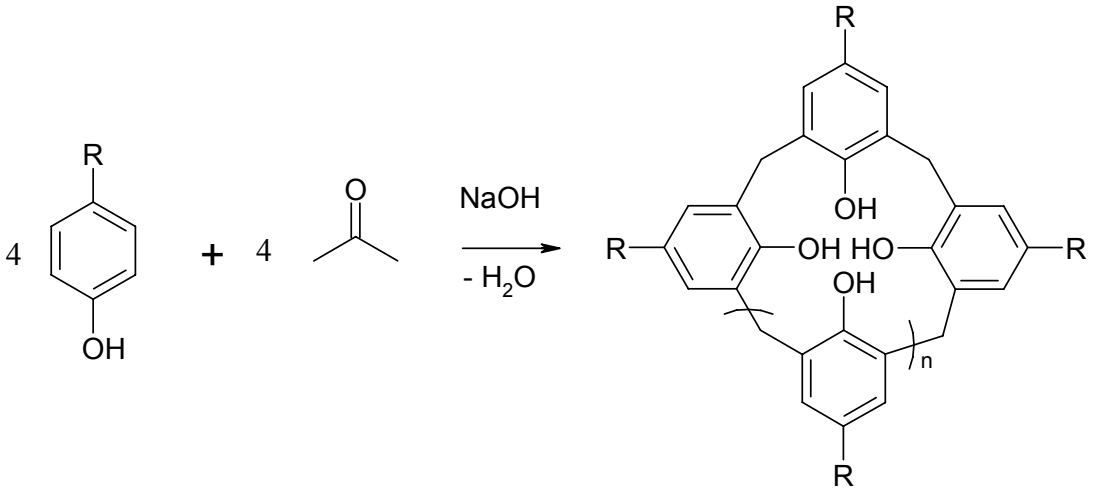


Figure I.11.a: Base-catalyzed synthesis of a calix[n]arene.

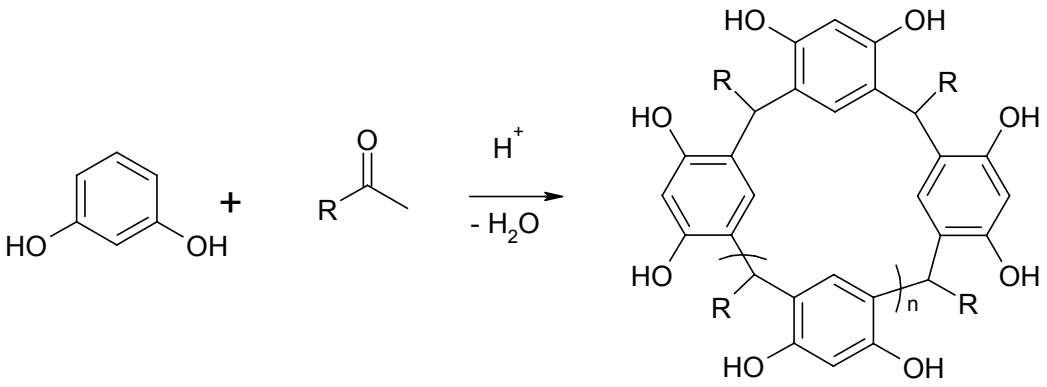


Figure I.11.a: Acid-catalyzed synthesis of calix[n]arene.

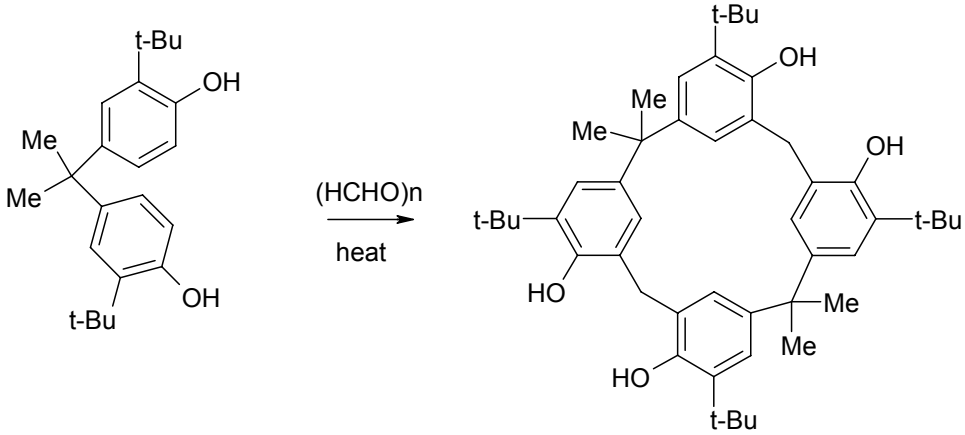
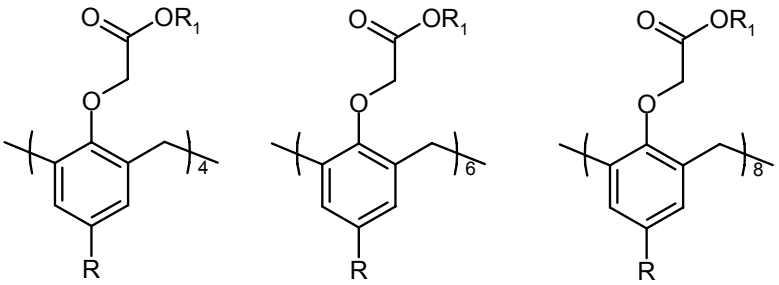


Figure I.11.c: Synthesis of calix[n]arene under neutral conditions.

Two other synthetic ways can be cited: the acid-catalyzed one and the formation under neutral conditions (Figure I.11.b and c).

A. Ester series													
	tetramers					hexamers				octamers			
	7	**10**	**13**	**16**	**19**	**8**	**11**	**14**	**17**	**9**	**12**	**15**	**18**
Li^+	15.0	1.8	6.7	1.1	27.6	11.4	4.7	1.7	2.6	1.1	0.8	0.9	0.4
Na^+	94.6	60.4	85.7	34.2	94.0	50.1	10.4	10.3	6.7	6.0	7.5	8.3	4.1
K^+	49.1	12.9	22.3	4.8	75.8	85.9	51.3	29.1	25.2	26.0	20.2	25.5	12.1
Rb^+	23.6	4.1	9.8	1.9	53.4	88.7	94.1	41.2	77.7	30.2	28.9	29.8	17.5
Cs^+	48.9	10.8	25.5	4.6	81.9	100.0	94.6	54.8	94.6	24.5	30.1	20.1	27.0



(7) R= t-Bu R_1= Et (8) R= t-Bu R_1= Et (9) R= t-Bu R_1= Et

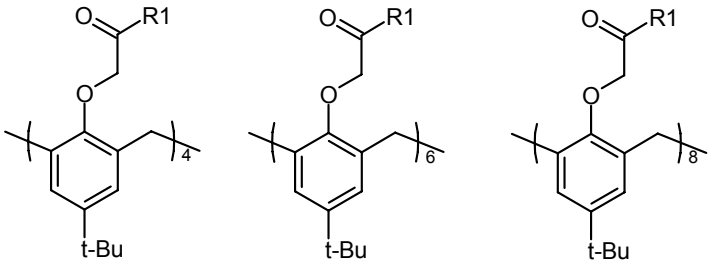
(10) R= H R_1= Et (11) R= H R_1= Et (12) R= H R_1= Et

(13) R= t-Bu R_1= Me (14) R= t Bu R_1= Me (15) R= t-Bu R_1= Me

(16) R= H R_1= Me (17) R= H R_1= Me (l8) R= H R_1= Me

(19) R= t-Bu R_1= t-Bu

B. Ketone series							
	tetramers				hexamer	octamers	
	20	**23**	**24**	**25**	**21**	**22**	**26**
Li^+	31.4	49.8	46.6	34.1	1.2	0.7	1.5
Na^+	99.2	94.0	92.8	94.3	6.2	9.9	21.6
K^+	84.1	72.6	81.4	47.7	12.8	25.1	7.7
Rb^+	53.7	23.4	43.7	27.1	11.6	20.8	1.7
Cs^+	83.8	17.2	31.6	50.7	13.6	15.3	4.5



(20) R= Me (21) R= Me (22) R= Me

(23) R= 1-Adamantyl (26) R= t-Bu

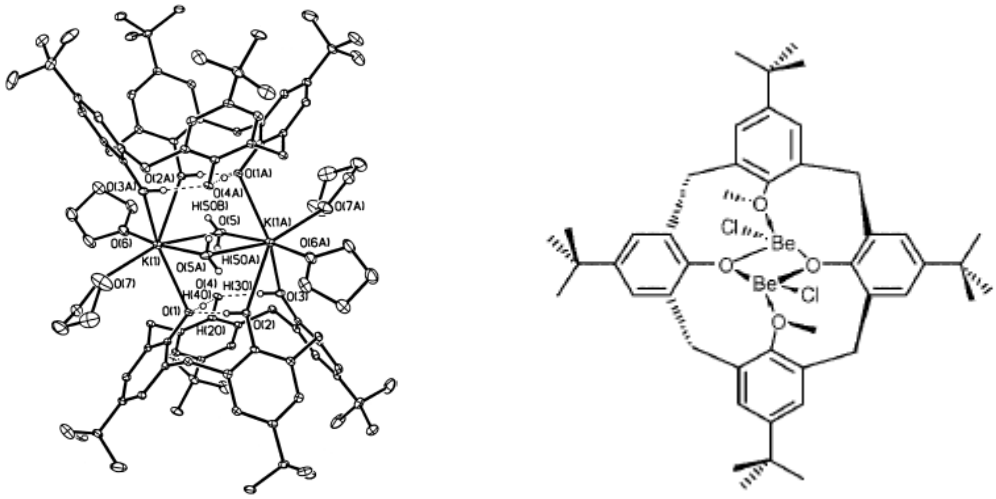
(24) R= t-Bu

(25) R= Ph

Table I.2: Examples of extraction with R-calix[n]aryl acetate and 4-tert-butylcalix[n]aryl R-ketone.

Calix[n]arenes are known for their properties in cation extraction and transport in organic solvents (table I. 2). The most detailed studies have been done for group 1 and 2 metal ions [88-97]. Calix[n]arenes allow for a lot of imagination in chemical design such as substitution of the upper rim (more accessible part of the macrocycle)

or the lower rim (narrow part of the macrocycle), or combinations with crown ethers. Still today, alkaline earth metal ion complexation remains an important domain of investigation. For example, the first complex of a Be^{2+} ion was characterized this year [98] (Figure I.12.b.).



a:[K_4(4-tertbutyl-calix[4]arene-4H)$(THF)_5]_2$THF b:[(4-tertbutylcalix[4]arene(OMe)2(OBeCl)2]

Figure I.12: Complexes with K^+ and Be^{2+} ions.

Calix[n]arenes are also used in nuclear waste treatment or to complex lanthanide ions [99-116]. Their flexibility and their ability to stabilize metal ions which have a large coordination number are useful for lanthanides and actinides. Calix[n]arenes are thought to be "cleaner agents" to remove nuclear waste [99]. Thus, calix[n]arenes show the highest selectivity for uranyl ion [99b-c] relative to other macrocyclic polydentate. Other advantages of these macromolecules are that they are easy to prepare and that the range of ligand systems for lanthanides and actinides is not broad.

In the last paragraphs, calix[n]arenes only bind one or two metal ions per molecule. Calix[n]arenes can also act as multidentate ligands and be either a cluster keeper or a cluster builder. Miyano et al. [117] supposed that they were able to create multi-metal centered structures for other applications such as synthons. A synthon is a concept in retrosynthetic analysis. It is defined as a structural unit within a molecule which is related to a possible synthetic operation. However, they are studied for their luminescence properties, catalytic activities, shape-selective organic synthesis and

clusters for MOCVD (Metal Organic Chemical Vapor Deposition) [118-127] (Figure I.13).

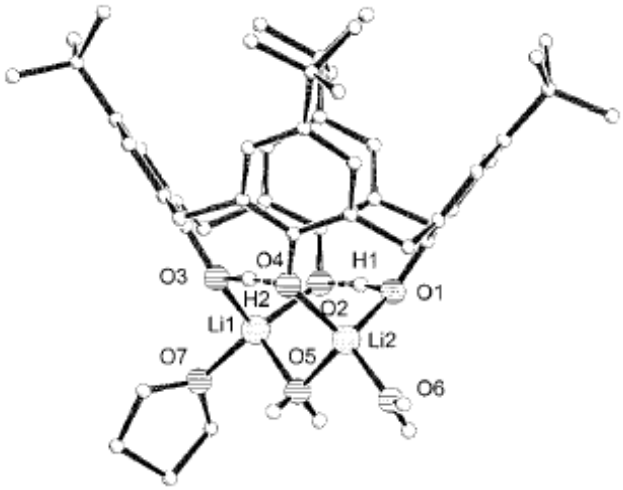


Figure I.13: Complex with Li^+ ions.

Due to their facile complexation with metal ions, calix[n]arenes are used as sensors or/and receptors. The simplest receptor, for example, can be achieved by O-acylation of phenol groups [128]. Thus, calix[n]arenes can contribute to enlarging the family of podands (molecules with pendent heteroatomic chains to bind metals) [129-133]. In some cases, Langmuir-Blodgett films or polymers of calix[n]arenes are reported to have exceptional electrochemical or optical properties [134-140]. A particular application of the latter is the fluorescent sensor for Sr^{2+} or uranium ions [141, 142]. Piezoelectric quartz crystals (PQC) are used as gas sensors, and to apply their properties in solution, they need a coating to reduce interactions with solution and enhance their selectivity. As already mentioned, calix[n]arenes can selectively bind ionic species and even organic compounds too. Xu et al. developed calix[n]arene coated PQC sensors for the detection of organic amines [143]. Calix[n]arenes are designed to complex specific molecules, and PQC transmits a quantitative signal of the complexation. If calix[n]arenes can work efficiently in interaction with PQC, they can be used alone or in association with polymers to serve as ionophores in electrodes [144-147] (Figure I.14). Due to their cation-π interactions (for example potassium

complexation thanks to the cation-π interactions of phenyl rings of 4-tertbutyl-calix[4]arene) and their specific contacts induced by designed substitutents, calix[n]arene were interesting enough to be included in soft metal ion-selective electrodes.

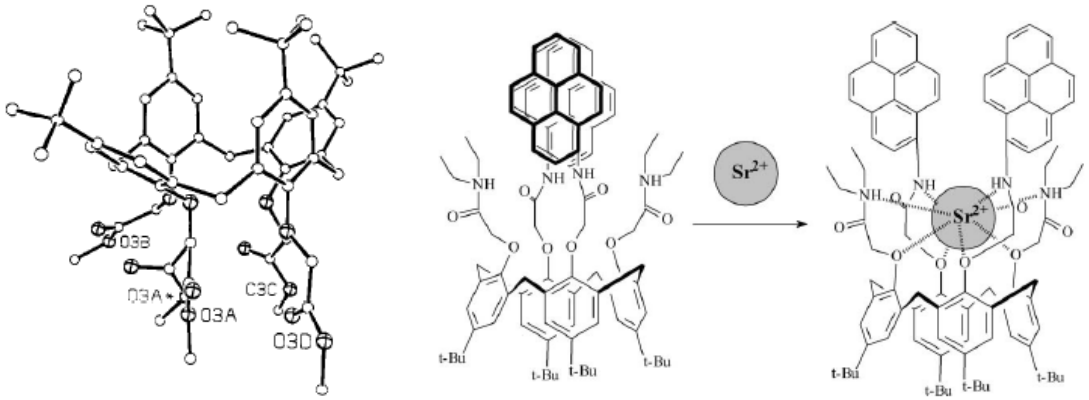
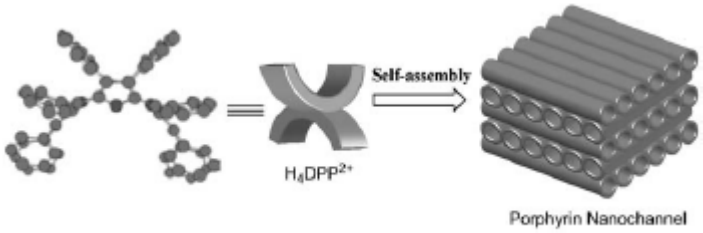


Figure I.14: Application of calix[n]arene as alkali metal receptor and fluorescent sensor.

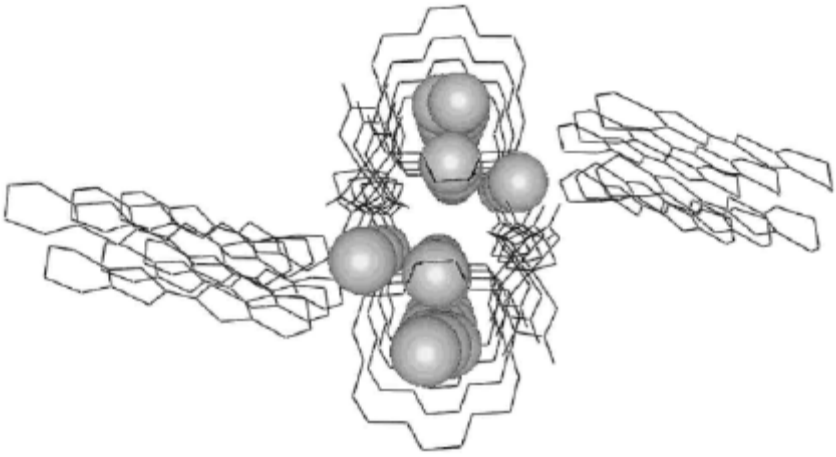
The last field in which calix[n]arenes can be applied is in biological activities. At the same time as neutral and amine complexes were reported [148-153], interactions with biological molecules were described with peptides, amino acids, DNA, and proteins [154-161]. The substitution of calix[n]arenes allows one not only to create adapted solutions for metal ion complexation but also for organic and biological molecule complexation. Coleman et al. reported the complexation of albumin by sulfonatocalix[n]arene [156]. Recently, Sgurlata et al. synthesized glycosamino acid-calix[n]arenes and studied their lectin binding ability [160]. Such systems use weak interactions such as hydrogen bonds or π-π interactions. The biocompatibility and the desire to mimick Nature, led scientists to develop new projects such as work of Reinaud et al. on transition metal enzymatic activity [162-167], and on Cu^{2+} or calcium carbonate growth studied by Volkmer [168, 169].

These examples present a general idea on the utility of calix[n]arenes in a broad domain of applications.

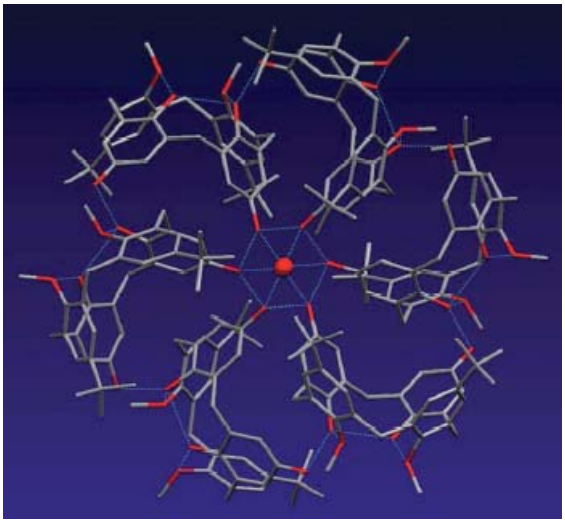
I.4. Aim of the thesis



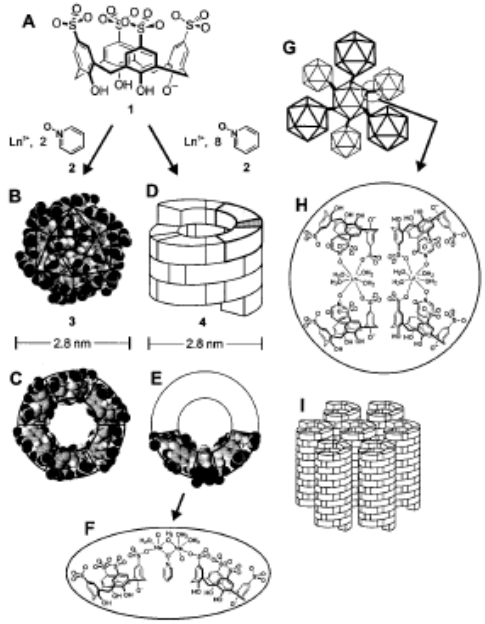
a. Channels made with dodecaphenylporphyrin H_4DPP^{2+}.



b. Channels formed by 18-crown-6-ether and H_3O^+/H_2O.



c. Channel obtained thanks to self-assembly of proximal calix[4]arene dihydroquinone around water molecule.



d. Helicoidal channels self-assembly of sulfonatocalix[4]arene Ln^{3+} ion and pyridine-N-oxide

Figure I.15: Four examples of channels with porphyrin, crown ether and calix[n]arene building blocks.

Biological channels are attractive materials for ionic transport, molecular sieves or catalysts. Therefore a lot of efforts are made to explain their mechanisms and structures as well as mimicking their properties.

Aquaporins and ion channels are the most imitated channels. Porphyrines were employed to obtain aquaporin-like channels or porous materials for ions and water molecules [12-19].

Crown ether structures were also investigated with more or less rigid compounds to obtain narrow channels for water, neutral molecules or ions thanks to π-stacking, CH_2-π interaction or weak hydrogen bonds and produce gramicidin models [20-25].

For calix[n]arenes, koilands (hollow molecular units) are the precursors of calix[n]tubes [26-28] where metal ionic bridges are replaced by organic chains. Another approach was to use particular molecules like hydroquinone [29-31] or hydrogen bond interactions to create self-assembled structures in view to achieve a tubular organisation [32]. Atwood et al. were able to create capsules or tubes with calix[4]arenes with help of other molecules (helicoïdal tubes [33]) or only with hydrogen bonds [34] (Figure I.15d).

The aim of this work is to produce and study DB18C6 (dibenzo-18-crown-6) compounds with polyhalide anions with channel structures in the solid state, as well as to crystallize and study channel structures with the large and flexible calix[n]arenes (with n=6, 8).

DB18C6 is a rather rigid ligand that can possess a plane of oxygen atoms to coordinate cations when in the correct conformation. The phenyl rings are excellent to create π-stacking. As already reported [2], DB18C6 can be isolated in a close packing with an infinite channel of water molecules and hydronium ions inside the oxygen atoms crown. It is interesting to investigate whether this channel can transport hydronium species, water, and/or also can carry other ions. This study will be reported in the first part of this thesis. In a second part, new structures with DB18C6 will be described. These structures will show examples of the pH-dependency of crown ether crystallisation.

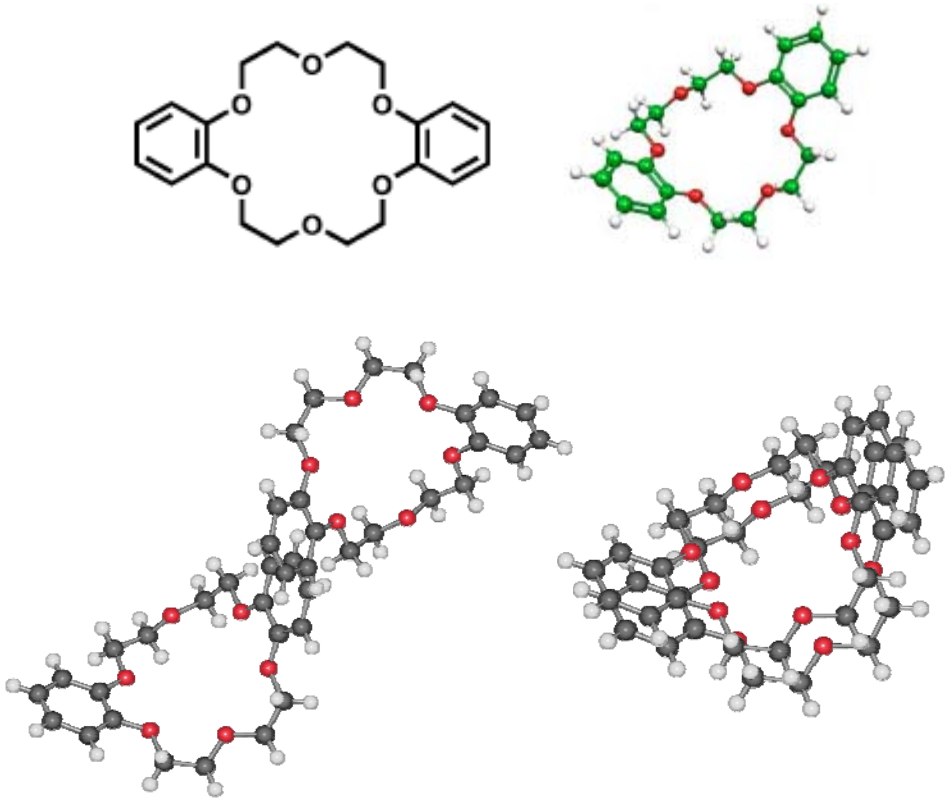
In contrast to DB18C6, calix[n]arenes, with n = 6, 8, are more flexible macrocycles. Besides, they have the possibility to form intramolecular hydrogen

bonds to stabilize certain more preferred conformations. In a biphasic apolar/polar, respectively aprotic/protic, system (THF/water), this hydrogen bonding system could be of particular interest to create preferential interactions not only intramolecularly but also intermolecular interactions with solvent molecules, and could favor some long-distance organisations. Thus, in a third part, new structures with calix[n]arenes, n = 6, 8, will be described. One structure will show the flexibility of the 4-tert-butylcalix[8]arene. The others will present rather unique systems created thanks to hydrogen bonds, and weak interactions between 4-tert-butylcalix[n]arenes and water molecules. For some structures finally, their possibilities of evolution from monomers to polymers due to the presence of alkali metal ions and solvents will be exposed.

II Results and discussion

II.1. Dibenzo-18-crown-6 (DB18C6) as a structural directing ligand

DB18C6 is a cyclic polyether ligand (scheme II.1) with two phenyl groups at opposite positions to each other. The structure is not planar and the mean planes of the phenyl groups form an angle of 150°. This has been confirmed by theoretical calculations to be a stable conformation of this ligand [6].



Scheme II.1: Scheme and representations of DB18C6.

Potentially, DB18C6 should be able to stack on top of each other to yield a 1D-chain structure due to π-π interactions. Upon stacking, the aromatic ring would be

packed in such a way as to obtain interactions of H-atoms of one with the ring current of the next neighbour molecule. Instead of having only a single interaction of DB18C6 phenyl ring with CH groups of the next DB18C6, one crown ether will eclipse the following second as illustrated in scheme II.1.

Indeed, with the aim of obtaining a 1D-structure with a metal ion, the compound $1\infty[(H_2O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}]1\infty[(H_3O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}](I_3)$ **1** was isolated [2].

This chapter deals with the study of this compound and its derivative obtained by cation substitution reactions, and the investigations of physical properties such as ionic conductivity and light absorption.

II.1.1. $1\infty[(H_2O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}]1\infty[(H_3O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}](I_3)$ 1

This compound was previously described by K. M. Fromm et al [1, 2]. It was obtained in this present work by a different synthetic method, and since it is the basis of the results described further on, its structure is discussed again in detail here. $1\infty[(H_2O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}]1\infty[(H_3O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}](I_3)$ **1** crystallises as red-brown needles in the orthorhombic space group Pccn (n° 56) with two independent half-molecules per asymmetric unit (figure II.1).

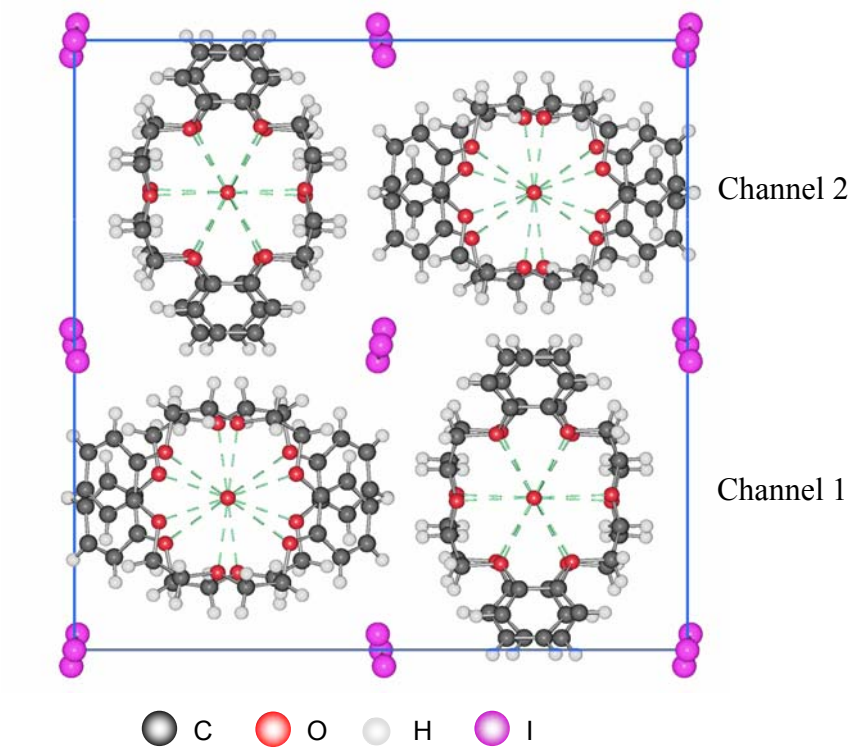


Figure II.1: View down the c-axis of the channels of **1**.

The structure of **1** is composed of two different channels in a crystallographic point of view: channel 1 around the oxygen atoms of water molecules O7 and O8 (Figure II.2) and channel 2 around the oxygen atoms of hydronium ion and water molecule O9 and O10 (Figure II.3). They result from an offset stacking of the aromatic groups of DB18C6 and are filled either with neutral water molecules H_2O or with alternating H_3O^+ cations and H_2O-molecules. Although it was impossible to attribute protons during the refinement of the structure, the presence of H_3O^+ is necessary to keep the compound neutral. At first sight, these cations seem to be statistically shared between both channels but only one channel coordinates the hydronium ion. This can be derived from the fact that packing in each channel is different. While Channel 1 stacks closer to being eclipsed, channel 2 features a twist angle from one DB18C6 to the next of circa 10°. This difference in stacking can be attributed to a different filling of the channel.

One proposal is that channel 2 carries alternately hydronium ions and water molecules. Hydronium ion O8 is coordinated by the ligand crown ether and is almost

included in the plane defined by the six oxygens of this latter (only a difference of 0.011Å). The coordination sphere is completed by the oxygens atoms O7 of the two nearby water molecules. Every hydronium ion is thus linked with the next one via a water molecule and the angle H_3O^+–H_2O–H_3O^+ is 180°. Such a conformation generates an infinite linear chain bridged by hydrogen bonds with the oxygen atoms of H_3O^+ and H_2O on a two-fold axis (1/4, 3/4, z (d)) (figure II.2). By the way, none of the oxygen positions in the centre of the channels, O7, O8, O9, nor O10, could be refined with alkali metal ions (refinement and structure factor are too bad) instead of oxygen atoms of water molecules, as will be important for the derived compound **2**.

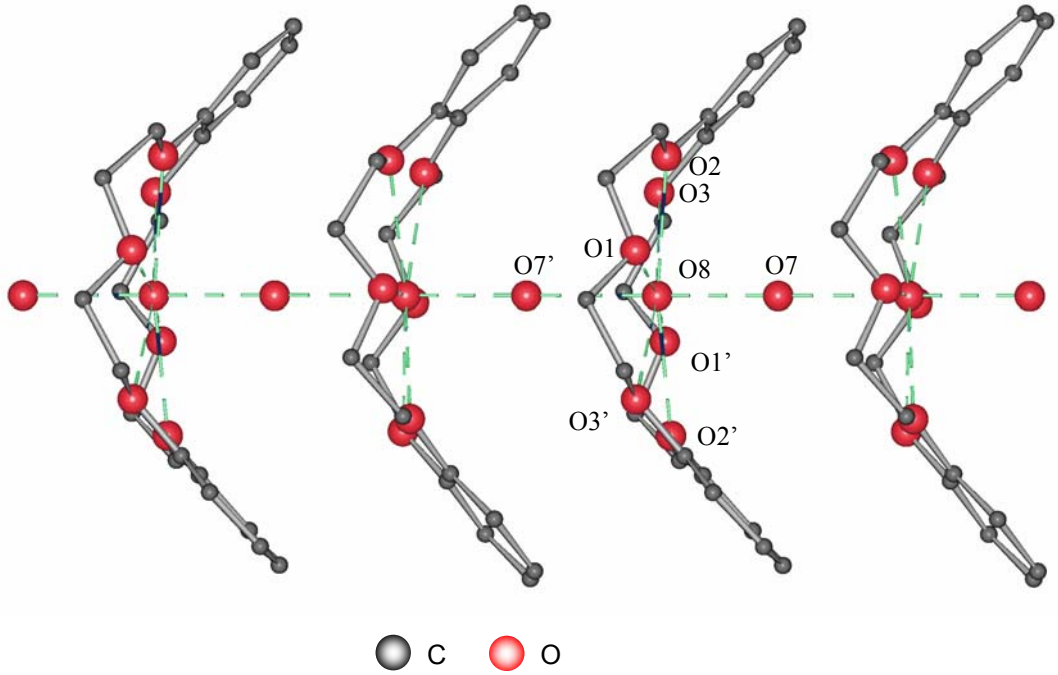


Figure II.2: Channel 2 filled with H_3O^+ and H_2O.

Channel 1 is similar to channel 2 but the hydronium ions are replaced by water molecules. In this case, the infinite column of water molecules O9, O10 is situated on the two-fold axis (1/4, 1/4, z (c)) (figure II.3).

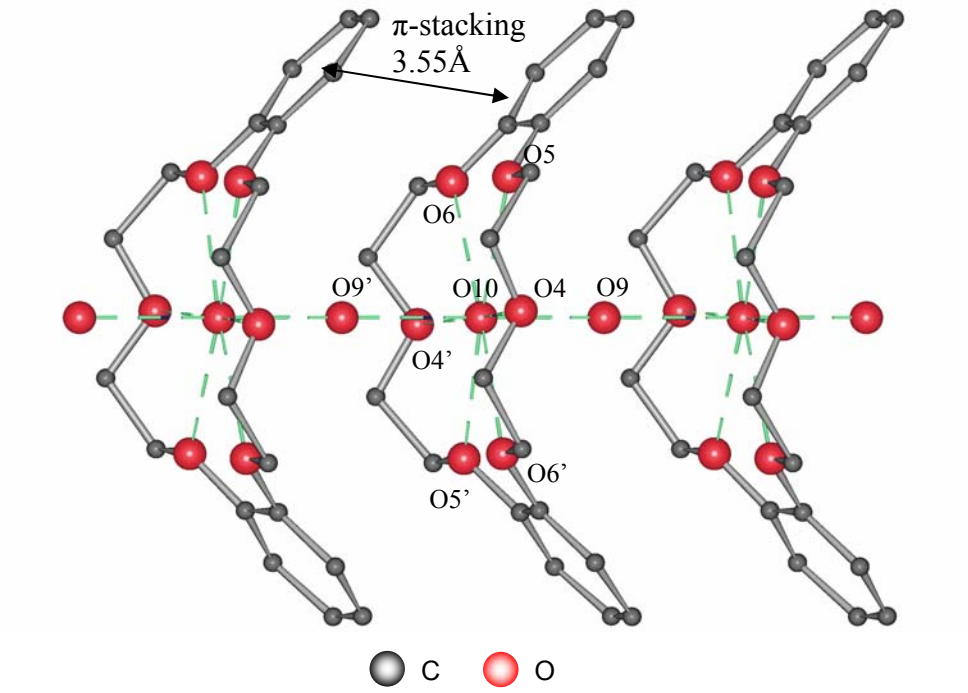


Figure II.3: Channel 1 filled only with H_2O.

The protons of the water molecules and the hydronium ions can not be assigned unambiguously. Thanks to crystallographic data, some assignments can be discussed. The O–O distances within the channels involved in hydrogen bonding are mentioned in table II-1. A better π-stacking in channel 1 and a relative freedom of coordinated oxygen O7 and O7' allow one to imagine that this channel is exclusively filled with water. On the contrary, the strong interaction of H_3O^+ and two oxygen atoms of the crown ether molecules and the weaker stacking of the crown ether allow one to suppose that channel 2 is coordinating H_3O^+ ions.

The two channels are parallel to each other and arranged in an alternating fashion way along the c-axis. The counter ions, the linear anions I_3^-, follow the same parallel orientation and fill the voids between the channels. These anions are symmetric with a I–I distance of 2.920(1) Å and the central atom I1 is located on the inversion center (1/2, 1/2, 1/2 (b)). The distance of 3.847 Å between two anions is long but testifies to weak interactions between polyiodide anions. The angle defined between terminal iodide I2 and the atoms I2 and I1 of the next I_3^- is around 150°, similar to what has been reported previously [170].

In order to estimate the cost in energy to distort the crown ether from its conformation in channel 1 to the one in channel 2, we performed computations at the DFT level (B3LYP, 6-31G**) [171a]. The conformation in channel 1 is more stable with a total energy of -1227.69284 Hartrees compared to -1227.68805 Hartrees for the crown ether ligand in channel 2. The difference in favor of the conformation of the crown ether in channel 1 represents 3.00 kcal/mol. All these facts, put together, give evidence for the H_3O^+-cation being in channel 2 rather than in channel 1.

Channel 1		Channel 2	
O10–O9	2.25(3) Å	O8–O7	2.20(4) Å
O10–O9'	2.44(3) Å	O8–O7'	2.20(4) Å
O10–O4, O4'	2.734(6) Å	O8–O1, O1'	2.793(6) Å
O10–O5, O5'	2.791(6) Å	O8–O2, O2'	2.681(6) Å
O10–O6, O6'	2.623(7) Å	O8–O3, O3'	2.679(7) Å
O10–plane (O4, O5, O6, O6', O5', O4')	0.033(9) Å	O8–plane (O1, O2, O3, O3', O2', O1')	0.011(9) Å
O9–plane (O4, O5, O6, O6', O5', O4')	2.28(3) Å	O7–plane (O1, O2, O3, O3', O2', O1')	2.18(3) Å
Angle between the planes of the phenyl groups	84.2(2)°	Angle between the planes of the phenyl groups	83.7(2)°

Table II.1: Distances O–O and O–plane (Å) angles in **1**.

With our new synthetic approach, large quantities of air-stable single crystals of cm-dimensions in length were obtained (Photo 1). Under the polarisers of a light microscope, they show a strong dichroism depending on the angles of the polarisers (Curve 1). The dichroism was measured by Dr. Jean-Pierre Rivera at the University of Geneva. [4] The red-brown color is typical for the triiodide absorption (molar absorptivities of I_3^- at 288 and 350 nm were determined to be 3.52 x 10^4 and 2.32 x 10^4 $L.mol^{-1}.cm^{-1}$, respectively.) [171b]

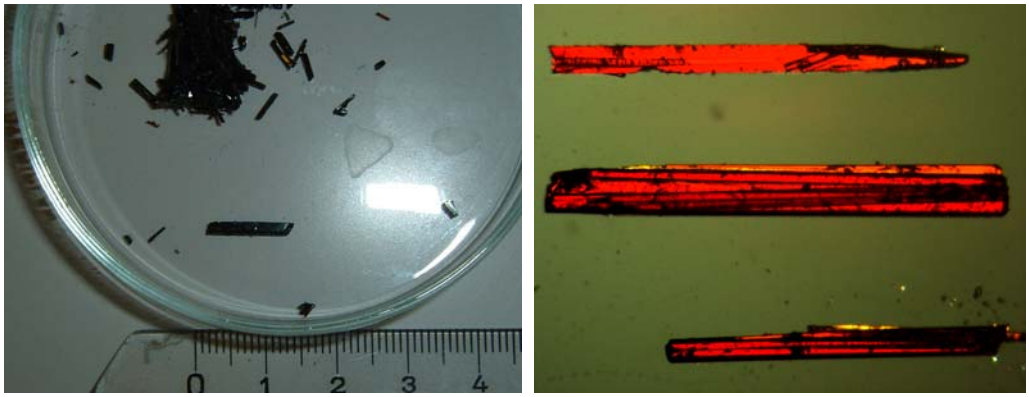
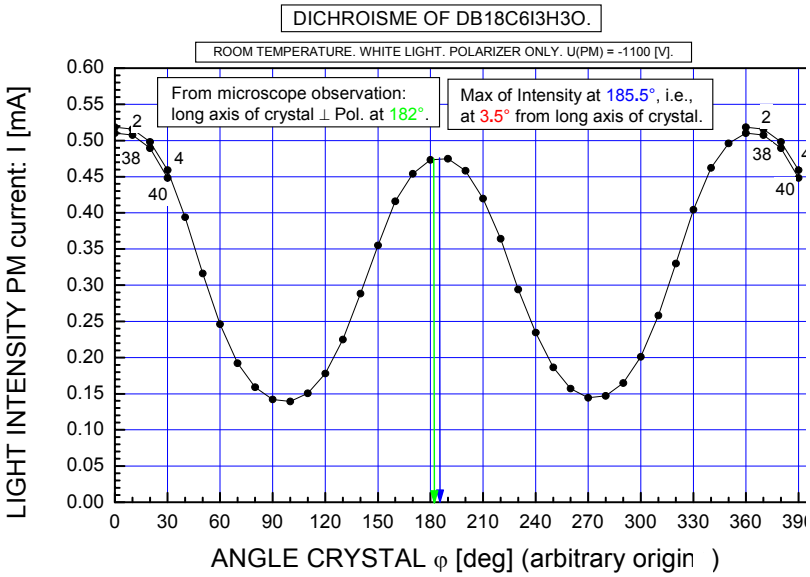


Photo I..1: Single crystals of **1** in a Petri dish and under polarized light microscope



Curve I.1:

Circular dichroism of **1**

To our knowledge, **1** is the first example of dibenzocrown ether in which the ligands are so well stacked by such a double unidirectional π-stacking. Single unidirectional π-stacking is usually observed [172]. In the case of the linear water chain, it is also the first structural example reported. There is an example with a zigzag chain and 18-crown-6 [158]. Finally, water chains inside 1D-arrangements of crown ether molecules are rare. Most of the time, monomeric complexes have been described [173, 174, 175]. In these compounds, the hydronium cation is situated at 0.2-0.3 Å out of the plane of the oxygen atoms of the crown ether. In **1**, O8 is coordinated by the crown ether.

II.1.2. 1∞[Na(DB18C6)(μ-OH)]1∞[Na(DB18C6)(μ-H_2O)] (I_3) 2

When single crystals of **1** are immersed into an aqueous solution of NaOH, ion exchange and substitution of H_2O or H_3O^+ by NaOH is observed without dissolution of the single crystals, thus compound **2** is isostructural to **1** and compound **2** "crystallizes" as red-brown needles in the orthorhombic space group Pccn (n° 56) with two independent half-molecules in the asymmetric unit. As shown in figure II.4, the organisation of the cell remains the same as in **1**: two types of channels surrounded by parallel triiodide anions. It was not possible to obtain crystals of this complex by classical synthesis due to the reactivity of diiodine (I_2) with sodium hydroxide.

In **2**, the cell parameters *a* and *b* are smaller by circa 0.1 Å than in **1** which is reflected in a by 26 $Å^3$ smaller unit cell volume.

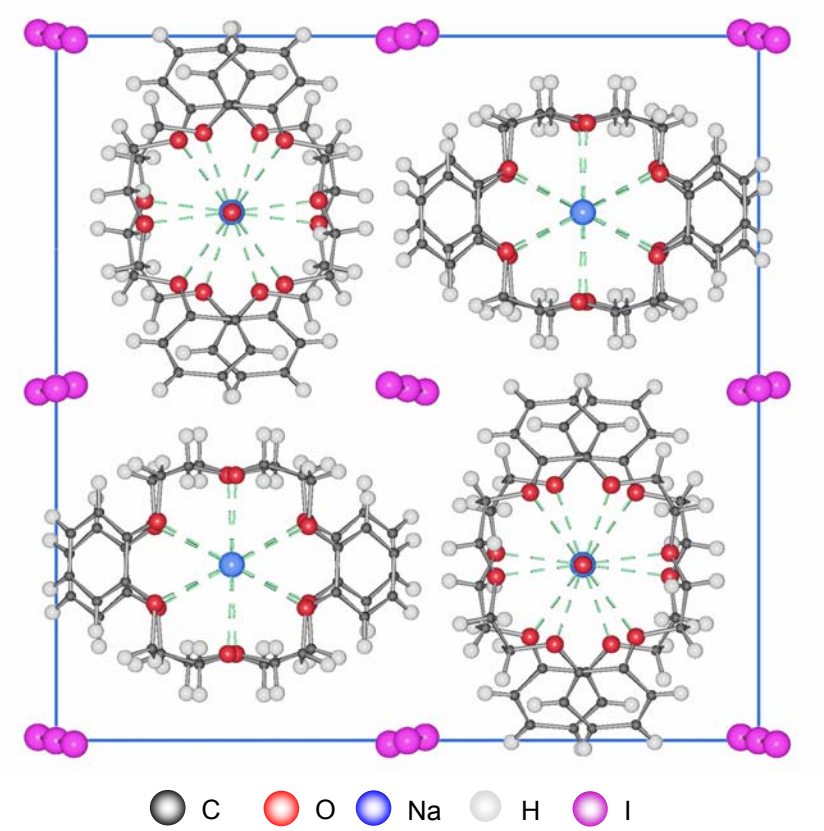


Figure II.4: Unit cell of **2**, view down the c-axis of the channels.

Again two different channels can be identified: Channel 1 with an almost perfect stacking of DB18C6 ligands and channel 2 with offset packing of DB18C6 molecules.

Channel 1 can be described as a linear chain of alternating hydroxide anions and sodium cations in which the cations are coordinated by DB18C6 in a motif of a distorted hexagonal bipyramid. Each bipyramid is linked to the other via a hydroxide anion (figure II.4). The distance Na^+–OH^- is 1.92(1) Å and shorter than the distance O8–O7 in **1**. On the contrary, the distance Na^+–$OH_{(bridging)}$ is longer than the distance O8-O7' in **1** with a length of 2.771 (1) Å. The π-stacking is less strong with a distance of 3.877 Å on average between aromatic rings. The presence of OH^- is confirmed by a ^{1}H-NMR with a signal at 2.6 ppm which is in agreement with the literature but shifted by 0.4 to 0.6 ppm [176, 177] and by IR. The broad characteristic band of water in **1** is replaced by two signals around 3616 cm^{-1} in **2**. One can be attributed to ν_{OH^-} stretching deformation of hydroxide anions and the second attributed to the ν_{OH} stretching of independent water molecules.

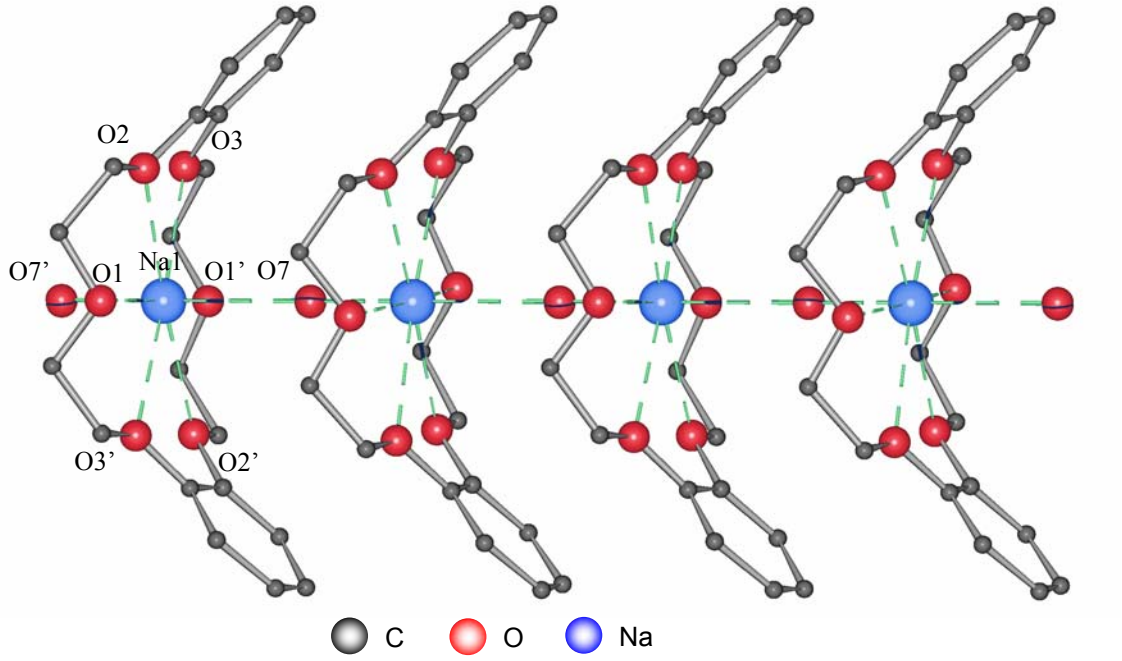


Figure II.5: Channel 1 filled with Na^+ and OH^-.

In channel 2, there is an alternation of sodium ions and water molecules (figure II.6). Here, the distance Na^+–O is 2.35(1)Å and longer by circa 0.15 Å than in the H_2O-H_2O channel 2 of **1**. This effect results probably from the repulsive dipole-dipole interactions between sodium cation and hydrogen atoms of water molecules. The Na–$O_{(DB18C6)}$ distances are between 2.645(7) and 2.800(7) Å long. This corresponds well to average values observed in the literature [178, 179]. The Na–O(water) distance with 2.35(1) Å fits also with literature and with the cationic complex of $[Na(DB18C6)(H_2O)_2]^+$ already reported [180]. As for **1**, those distances are given in table II-2.

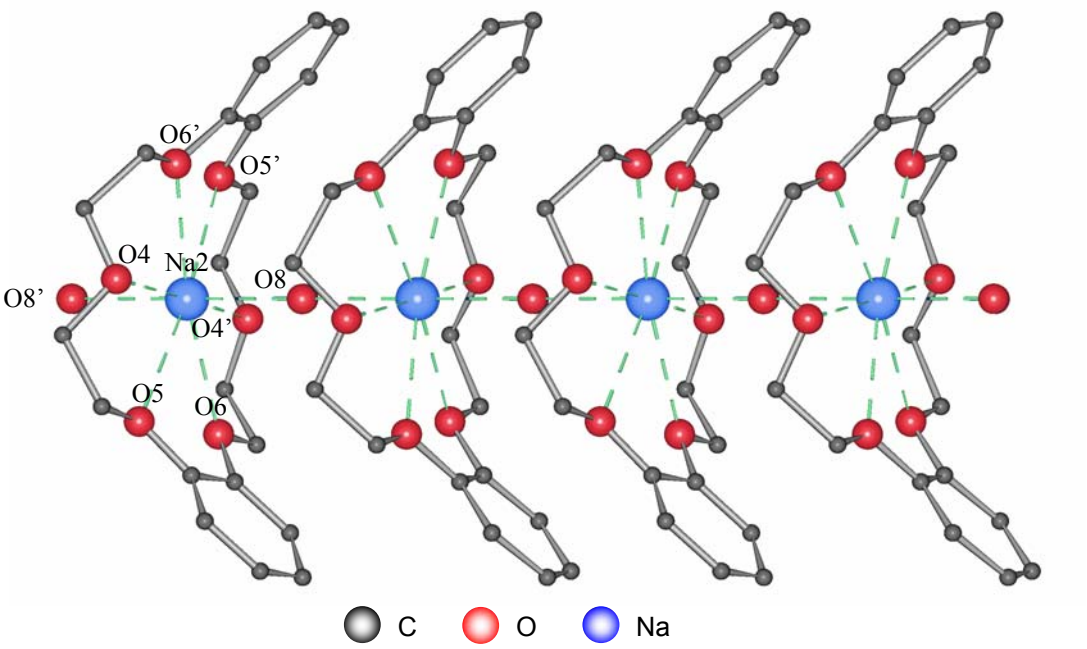


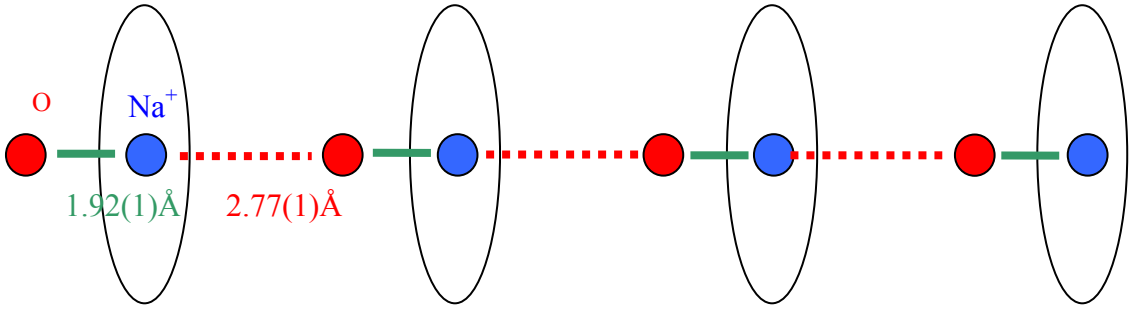
Figure II.6: Channel 2 filled with Na^+ and H_2O.

The charge of channel 2 is neutralised by I_3^-. The triiodide anions are still parallel to the channels and there is the same angle between two adjacent triiodides. Instead of 150° in **1**, in this case the angle is 151° in **2**.

Channel 1		Channel 2	
Na^+1–O7	1.92(1) Å	Na^+2–O8	2.35(1) Å
Na^+1–O7’	2.77(1) Å	Na^+2–O8’	2.35(1) Å
Na^+1–O1, O1’	2.794(6) Å	Na^+2–O4, O4’	2.800(7) Å
Na^+1–O2, O2’	2.689(7) Å	Na^+2–O5, O5’	2.764(6) Å
Na^+1–O3, O3’	2.684(7) Å	Na^+2–O6, O6’	2.645(7) Å
Na^+1–-plane (O1, O2, O3, O3’, O2’, O1’)	0.003(8) Å	Na^+2–plane (O4, O5, O6, O6’, O5’, O4’)	0.014(7) Å
O7–plane (O1, O2, O3, O3’, O2’, O1’)	2.77(1) Å	O8–plane (O4, O5, O6, O6’, O5’, O4’)	2.37(1) Å
Angle between the planes of the phenyl groups	85.5(2)°	Angle between the planes of the phenyl groups	85.2(2)°

Table II.2: Distances Na^+-O and Na^+-plane (Å) and angles in **2**.

In channel 1, two types of bonds can be identified: a short one 1.92(1) Å and a longer one of 2.77(1) Å on the other side of the crown ether. Such phenomenon is due to the natural tendency of ions to make ion pairs when they are not solvated (Scheme II.2). It seems not possible to have other electrostatic interactions between oxygen atoms of crown ether or water molecule to compensate this interaction. Furthermore, the hydrogen atom of the OH^- is on the Na–O axis for reason of symmetry and can create some dipole-dipole ineraction which explain the distance of 2.77(1)Å.



Scheme II.2: Coordination of sodium ion in channel 1.

In channel 2, the O–Na distances vary as well. Water molecules screened by DB18C6 are by 0.1 Å further away from the sodium cation and non-screened water molecules are 0.1 Å closer to the sodium cation than in the ideal position of water

molecules coordinating to water molecules in **1**. To compensate for these effects, the crown ether molecules open their angle between the phenyl groups. The angle between the phenyl groups in channel 1 gains 0.5°, and 1° in channel 2 as compared to the same in **1**. Such values confirm the coordination of sodium ions by crown ether [180, 181].The differences between **1** and **2** are summed up in table II-3.

As for **1**, the positions of the central channel atoms were tested for different elements. In **2**, none of the positions described here for Na^+ gave a stable refinement for oxygen of water molecule occupation. Split refinements resulted also in unstable refinements.

	1	**2**
a (Å)	22.047(2)	21.988(4)
b (Å)	22.084(2)	21.988(4)
c (Å)	9.3800(6)	9.382(2)
distance $I–I_{intra}$ (Å)	2.920(1)	2.921(1)
distance $I–I_{inter}$ (Å)	3.846(1)	3.839(1)
Angle between the plane of the phenyl groups in channel 1(°)	84.2(2)	85.5(2)
Angle between the plane of the phenyl groups in channel 2 (°)	83.7(2)	85.2(2)
$A^+1–O$ in channel 1 (Å)	2.25(3)	1.92(1)
$A^+1–O'$ in channel 1 (Å)	2.44(3)	2.77(1)
$A^+2–O$ in channel 2 (Å)	2.20(4)	2.35(1)
$A^+2–O'$ in channel 2 (Å)	2.20(4)	2.35(1)

$A^+ = H_3O^+, Na^+$

Table II.3: Difference between structures **1** and **2**.

II.1.3. Ionic conduction experiments

The transformation of the structure **1** into **2** is the proof that ions can be exchanged in the solid state even in the heart of crown ether channels and therefore a

study of ion exchange was conducted. In the first part, proton exchange was studied and in the second part sodium exchange was analyzed in more detail.

II.1.3.1 SEM (scanning electron microscope) images of 1

1 forms needle-like single crystals, some of which have a rectangular hole inside as shown in figure II.7.a, and some of which are smaller and without hole (figure II.7.b). To avoid the conduction via capilliary like in a drinking straw, crystals without hole were used for the following measurements.

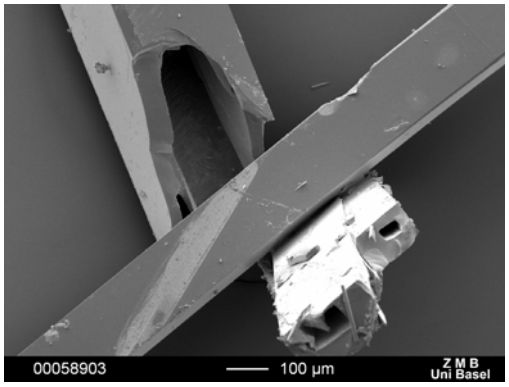


Figure II.7.a: Hole in crystal of **1** (Size 100 µm).

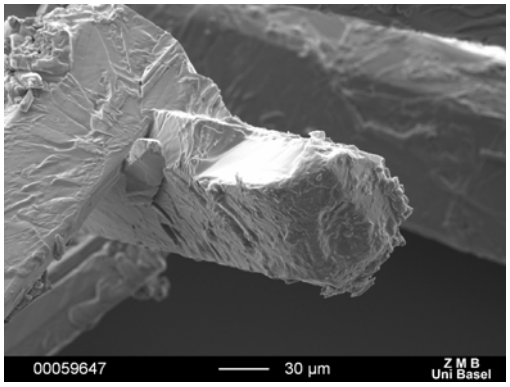


Figure II.7.b: Crystal of **1** without hole (Size 30 µm).

Crystals with rectangular openings were cut several times to find out whether the channels cross the total length of the single crystal, which was indeed the case (Figure 7.c, d).

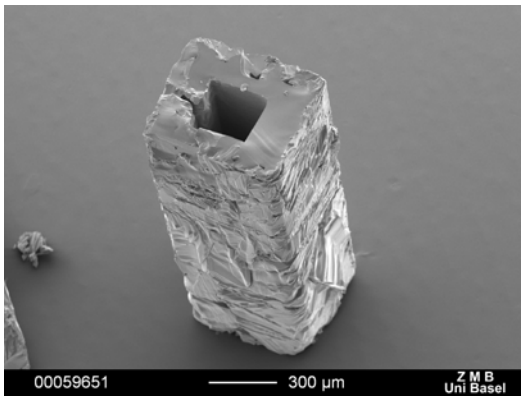


Figure II.7c: Hole in a once cut crystal of **1** (Size 300 µm).

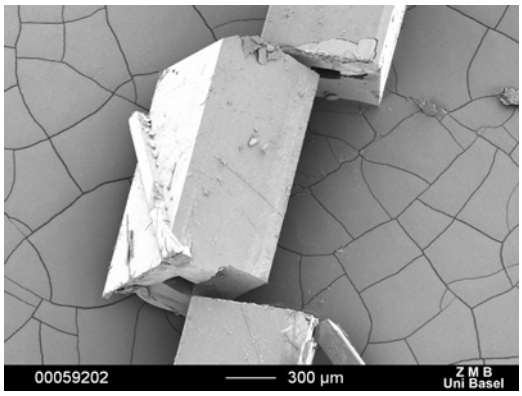


Figure II.7d: Several times cut crystal of **1** (Size 300 µm).

II.1.3.2 Description of the devices A and B.

In order to measure the ion conductivity across single crystals of **1** which are not hollow, a small device A was constructed for the fixation of such a single crystal (Figure II. 8a).

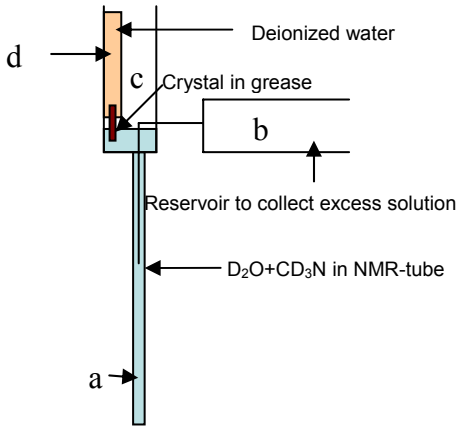


Figure II.8a: Device A for proton exchange measurement.

The device A is composed of four parts (figure II.8a):

-One NMR-tube a), easy to remove to follow the experiment by ^{1}H-NMR,

-One reservoir b), to collect the excess of deuterated solution above the NMR tube composed of a syringe with a needle descending into an NMR tube,

-One compartment c), composed of a syringe where a single crystal of **1** is in touch with deuterated solution, no grease was in contact with the deuterated solution,

-One reservoir d), containing slightly acidic water due to dissolved carbon dioxide and on which the crystal of **1** is fixed with non-proton-conducting grease (the grease was tested previously as a blank and was proved to be non-conducting).

A single crystal of **1** without a hole bridges the two latter reservoirs. In the upper one, a solution of a weak aqueous acid provides the protons and water. In the lower compartment, a solution of D_2O and acetonitrile collects the protons and/or water exchanged via the compound **1**. The measurement of proton concentration was followed with a 250 MHz Bruker Advance spectrometer, at 298 K. In order to make sure that the grease is not conducting and that nothing can pass via the interface grease-single crystal, a long crystal of **1** was chosen and put in contact with the deuterated solution only with the tip of the crystal. The deuteration of the crystal seems not to occur during the experiment.

For the measurement of ion (i.e. sodium ion), transport through single crystals of **1**, respectively **2**, device B was constructed (Fig. II.8b).

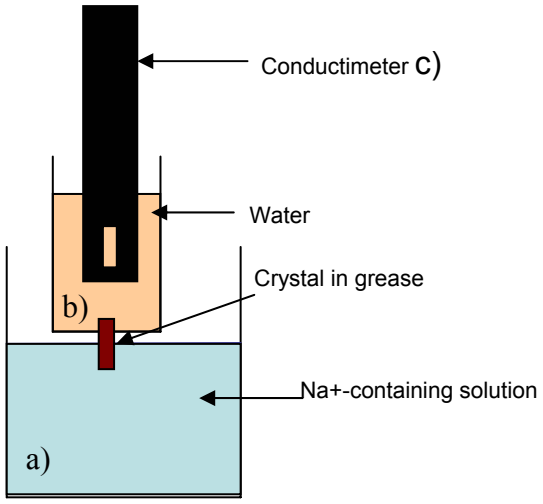


Figure II.8b: Device B for a sodium cation exchange measurement.

The device B is composed of three parts:

-One reservoir a) containing a 1 M in Na^+ solution (NaOH),

-One compartment b) filled with deionised water on which **1** is fixed,

-And one conductimeter c) in this latter.

Again, a single crystal of **1** bridges the two compartments. In the upper one, the dionized water will be enriched with Na^+ due to osmotic pressure. The measurement of conductivity was recorded by a WTW Tetracon 325 conductimeter and its WTW 720 unit. Upon ion or NaOH transport of **1**, transformation into compound **2** should occur. In order to make sure that the grease is not conducting and that nothing can pass via the interface grease-single crystal, a long crystal of **1** was chosen and put in contact with the sodium hydroxide solution only with the tip of the crystal.

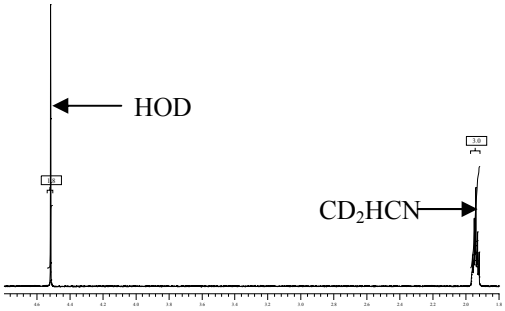
II.1.3.3 Results

II.1.3.3.1. Measurement of proton transport

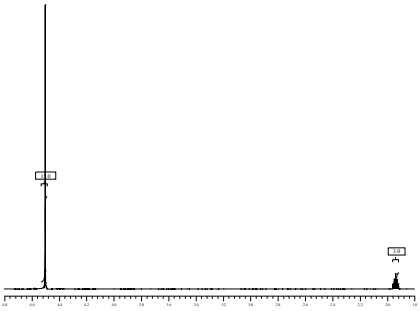
The measurement of proton transport is made with a solution of water at pH=6 (acidified by dissolved carbon dioxide). The D_2O/CD_3CN solution is in ratio (3:1). The measurements were done at room temperature (26°C).

A measurement was carried out every thirty minutes. Curve II.1 reports the results as derived from the integration of the ^{1}H-proton signals at 4.515 ppm, the signal for water increases with time (Spectrum I. 1. a-d).

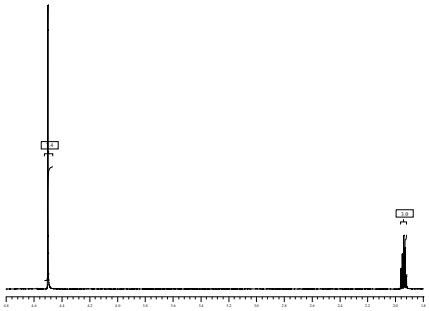
As we observe no shift of the water signal during the time of the measurement, nor appearance of a second signal, the concentration of H_3O^+ is either very much smaller than the one of H_2O, and it is not possible to differentiate two signals on the ^{1}H-NMR spectrum, or there is simply H_2O transport through single crystals of **1**. Therefore the number of proton was integrated on the H_2O signal.



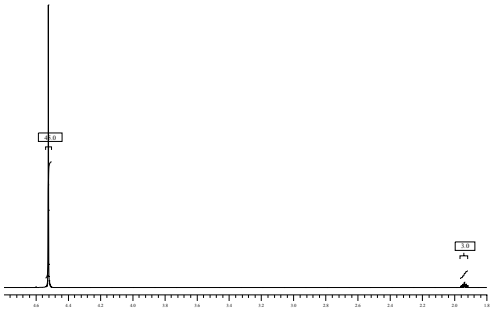
Spectrum I. 1. a: t=0 min



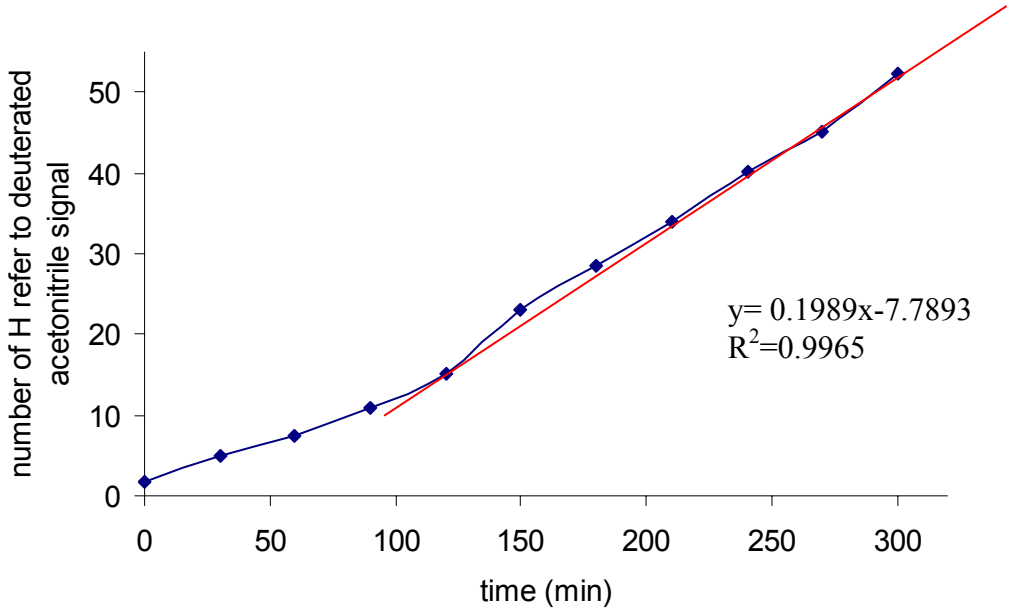
Spectrum I.1. c: t=120 min



Spectrum I. 1. b: t=60 min



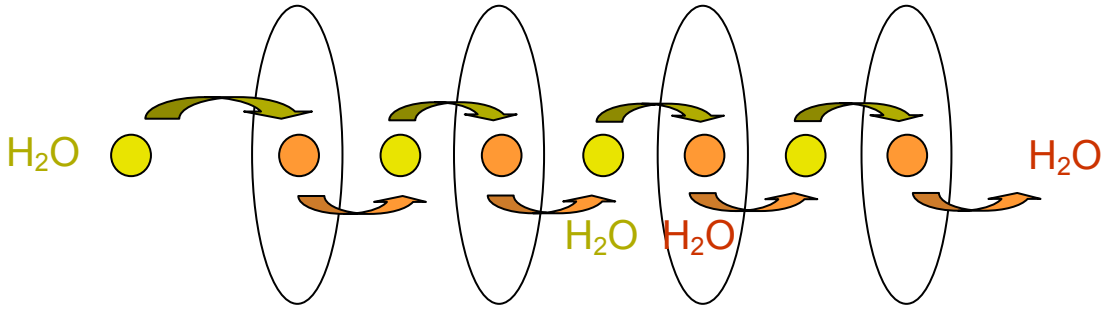
Spectrum I. 1.d d: t=180 min



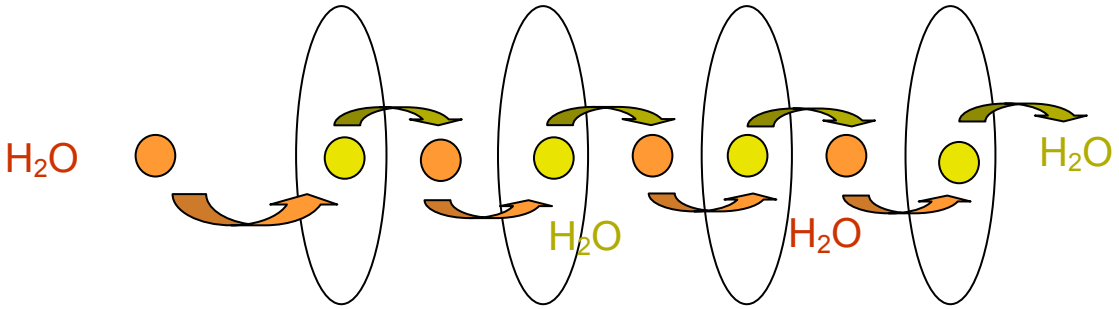
Curve II.1: H_2O/H_3O^+ increase in D_2O/CD_2HCN

As shown in curve II.1, the transport is linear and around 9 to 10 H atoms exchange per hour with respect to the signal for CD_2HCN, which stem from the 99% deuterated acetonitrile molecules. Absolute quantification is however not possible here as the quantity of protonated H_2O in D_2O is not indicated, but was present to some extent in the initial D_2O in spite of its indication of 100% D_2O.

The channel 1 is probably the only one to conduct protons in form of neutral water because if hydronium ions were transported, moving counter-ions would be also needed. In absence of moving counter-ions in the crystal structure, the conclusion is that only half of the structure is conducting. This relatively slow process could be due to the hopping conduction through the channel as seen in scheme II.3a and b. The slight difference in the beginning is maybe due to the use of a dried crystal.



Scheme II.3.a: Illustration of the hopping process of water through channel 2 of **1**.

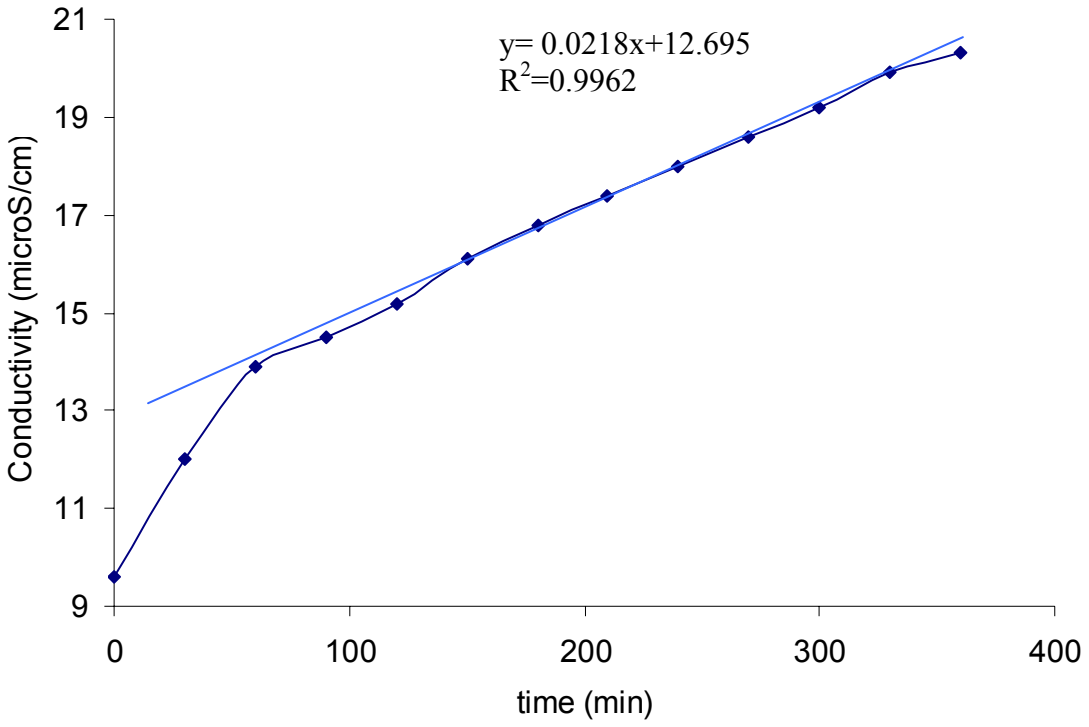


Scheme II.3.b: Illustration of the hop process of water for channel 2 of **1**.

II.1.3.3.2. Measurement of the sodium ion conductivity

The measurement of ion conductivity was made with a similar device B using a solution of NaOH 1 M. It was carried out at room temperature (26°C).

The conductivity was measured every thirty minutes. Curve II.2 shows the results.



Curve II.2: Conductivity of a water solution doped with sodium cation.

Here again, a linear behavior of the conductivity as a function of the time is observed. The same singularity at the beginning noticed previously for proton exchanges is also present. Besides of the fact of crystal hydration, the ionic nature of the guest plays an important role in the shape of the curve II.2. The conductivity augments fast, which is probably due to cation and anion migration, in that case Na^+ and OH^-. After the measurement, iodide test with silver nitrate was made. No iodide trace was found in the final solution.

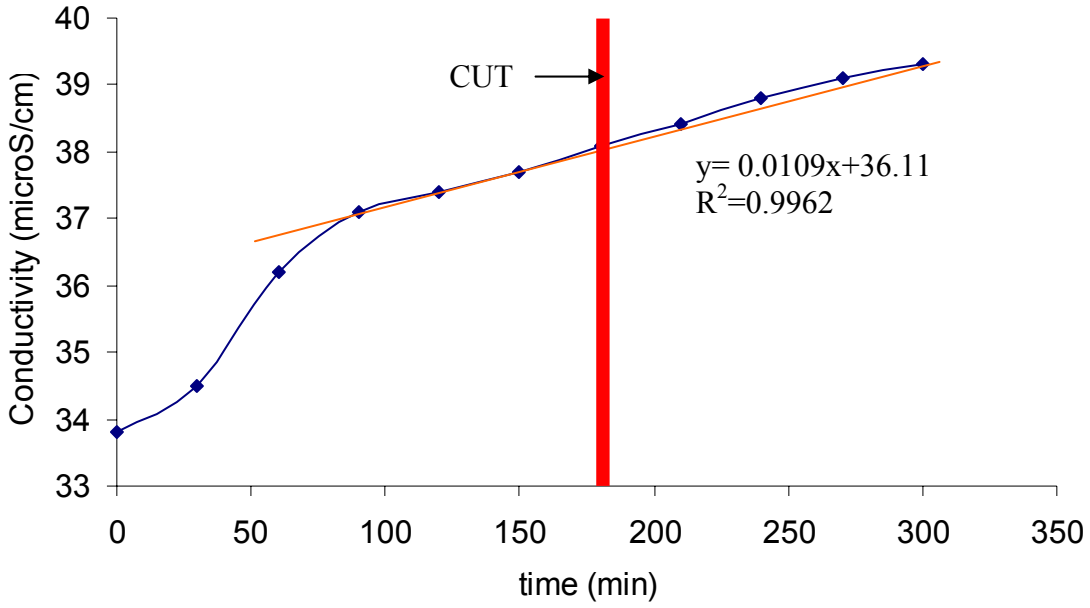
Na^+ ions seem to move faster in the channel because the slope change occurs after 60 minutes instead of 120 minutes as in case of H_2O. This can be due to the fact that DB18C6 is fitted to sodium ions with a diameter of 1.90 Å while water molecules are bigger with a diameter of 2.8 Å. Another effect can be the hydration energy set free upon migration of Na^+ from the channel into the water solution.

Analysis of the water enriched with sodium ions via this osmotic process reveals a concentration of $6.50 \cdot 10^{-4}$ mol/L. In other words, for an initial volume of 4 mL, $2.60 \cdot 10^{-6}$ moles of sodium ions are present in the solution after six hours. If these moles are reported to the crystal surface, it is $3 \cdot 10^{-5}$ mol/mm^2/h of sodium ions which are passing through the crystal. If this number is calculated in reference to the surface of a monocrystal of **1** and the number of conducting channels, there is $7.31.10^{-17}$ mol/channel/h of sodium ions going through a single channel of a monocrystal of **1**. In term of flux, crystals of **2** transport $4.3.10^{-7}$ mol/h. This value is comparable with extraction abilities of crown ethers in solution. For example, NaCl can be carried with a flux of $0.31.10^{-7}$ mol/h or NaI with a flux of 15.10^{-7} mol/h, which means that our value is on the average between these two latter.

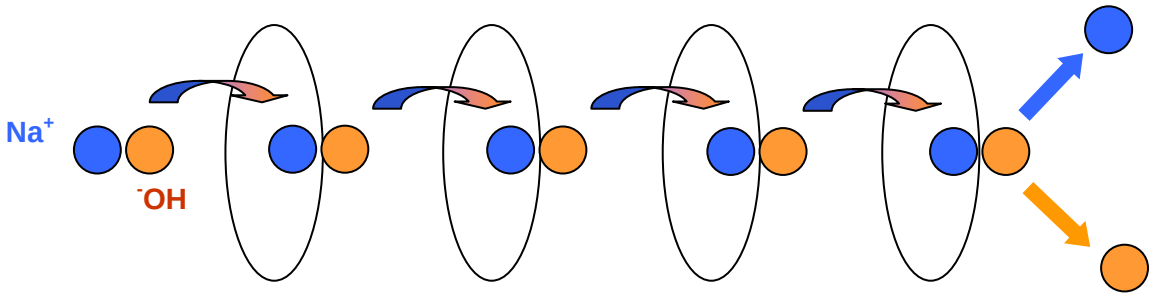
Crystals of **1** and **2** can transport ionic species or water molecules through crown ether tunnels. They show a preference for transporting Na^+ and OH^- ions. It seems to be a specific transport depending on ion size. Furthermore, only one channel, i.e. the channel where there is neutral species (water molecules) or a pair of ions (cation and anion), is conducting.

In a third experiment, similar to the second, the crystal was cut to half of its length in order to find out a length dependency of the conduction process. As the slope remains the same, length dependency can be excluded. Such an experiment favors the

hopping process theory for NaOH. The data in curve II.3 support a hopping process as revealed Scheme (II.4a, b).



Curve II.3: Conductivity of a water solution doped with sodium cations.



Scheme II.4: Illustration of the hopping process of sodium hydroxide for channel 1 of **1**.

II.1.4. [Na(DB18C6)I(THF)][Na(DB18C6)(H_2O)$_2$]I(THF)$_2$(CHI_3) 3

In order to synthesize 2 in larger amount, the synthesis was carried out under basic conditions. For solubility reasons, sodium *tert*butoxide is used instead of sodium hydroxide. During the preparation of the solution, the base reacts with iodine to give sodium iodide. NaI reacts with DB18C6 in a mixture of THF/H_2O/acetone (due to the amount of base, acetone was added to avoid biphasic system creation during the reaction) to yield the complex ionic compound [Na(DB18C6)I(THF)][Na(DB18C6)(H_2O)$_2$]I(THF)$_2$(CHI_3) **3** in 80 % yield.

3 crystallises in space group $P2_1/m$ (N°. 11) with four molecules per unit cell. The cationic complex of **3,** [Na(DB18C6)(H_2O)$_2$]$^+$, consists of a sodium cation to which a DB18C6-ligand in equatorial, and two water molecules in axial positions of a hexagonal bipyramidal coordination sphere are coordinated. The neutral complex in **3** is a [Na(DB18C6)I(THF)] unit (Figures II.9 and II.10).

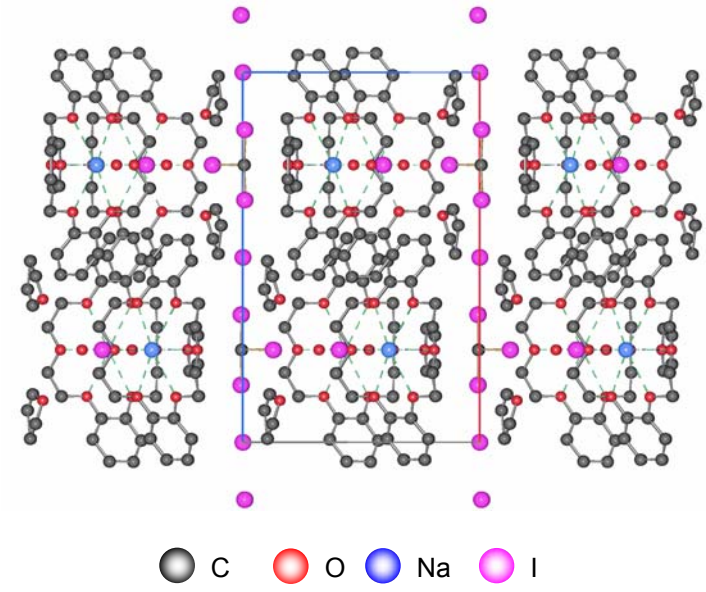


Figure II.9: View of the packing onto the *ab*-plane in **3**, including the solvent molecules, but excluding H-atoms for clarity.

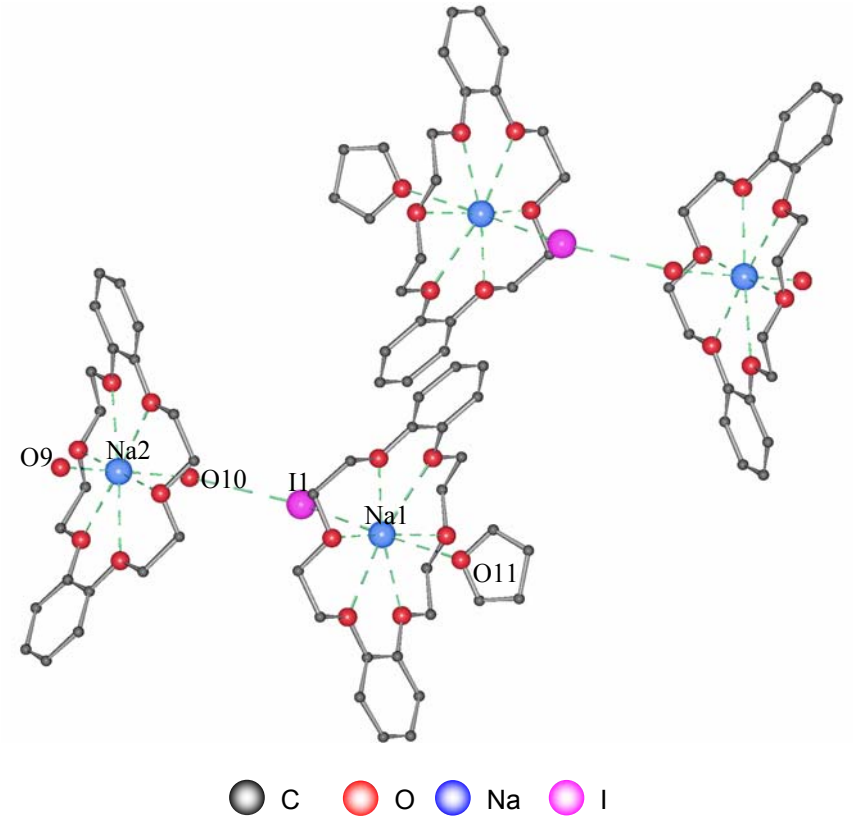


Figure II.10: Cationic dimers of **3** linked via H-bonding, (H-atoms omitted for clarity).

The counter ion to the cationic complex is a free iodide ion, I2 which is not directly coordinated to the cation. Furthermore, there are free solvent molecules, two THF and one CHI_3 molecules, filling voids in the structure, but not coordinated to the cationic or neutral complexes of **3**. The Na–O(DB18C6) distances are between 2.74(1) and 2.778(9)Å long. This corresponds well to average values observed in the literature [178, 179], but is longer than in similar complexes with the smaller crown ether 15C5 [182 ,183]. On average, the Na2–O (DB18C6) distances in the complex cation of **3** are by 0.03 Å shorter than in the neutral moiety of **3**. The sodium cation Na2 is 0.051 (8) Å out of the mean plane formed by the six surrounding oxygen

atoms of the crown ether, slightly exo of the folding of the latter oxygen atoms. The aromatic rings of the DB18C6 in the cationic unit form a dihedral angle of 133.6(3) ° towards O10. The two neutral water ligands, O9 and O10, act as axial ligands to the sodium cation, with Na2–O(H_2O) distances of 2.48(2) (Na2–O10) and 2.14(5) Å (Na2–O9). The difference in these distances is due to the fact that one water molecule, O9, is a terminal ligand, whereas the other, O10, is linked to the iodide of the neutral unit of **3** via H-bonding. The hydrogen bond to I1 has a distance of O10–I1 3.51(3) Å, the Na2–O10–I1 angle being 130(2) °. The H-bonded system between the neutral unit and the cationic complex of **1** is shown in Figure II.10. I1 is directly coordinating to Na1 with 3.523(4) Å. This is by ca. 0.5 Å longer than in the non-linked, isolated neutral complex [Na(DB18C6)I(THF)] [178], showing the dramatic elongation effect on the Na–I bond when I atom is implicated in hydrogen bond with water molecule O10.

$$Na^+ + I^- + DB18C6 + THF\ (dried) \xrightarrow{\text{Inert atmosphere}} [Na(DB18C6)I(THF)]\ [178]$$

Reaction II.1: Synthesis of neutral complex [Na(DB18C6)I(THF)] [178]

The coordination sphere of Na1 is completed by one DB18C6 ligand and a terminally bonded THF ligand (Na1–O11 3.00(4) Å) opposite I1 to yield an overall neutral complex. The folding of the DB18C6 ligand in the neutral complex of **3**, in the direction of the THF ligand, results in an angle of 125.7(3) ° between the mean planes through the phenyl rings. Even though the folding is directed in the same way as in the neutral literature compound [178], the folding is not as important (ca. 90° in

[Na(DB18C6)I(THF)] [178]). In [Na(DB18C6)I(THF)], electrostatic interactions involviing oxygen atoms compensate for the ionic interaction between sodium and iodide ions. Therefore the folding is important. In the case of **3**, the iodide anion is involved in two interactions. One is an hydrogen bond, and the second is an interaction with sodium ion. This latter is less strong than in [Na(DB18C6)I(THF)] as mentioned above. As a result, the compensation of the DB18C6 is less important and the folding too.

The arrangement of the crown ether complexes extends in two dimensions, with the sodium ions Na1 and Na2, and I1 on the crystallographic sites (x, 1/4, z (e)) which correspond to a mirror plane. In addition to the free anion I2, which is placed on a crystallographic inversion centre (0, 1/2, 1/2 (c)), two disordered THF molecules and a CHI_3 molecule co-crystallize in the solid state parallel to the *bc* plane (Figure II.11).

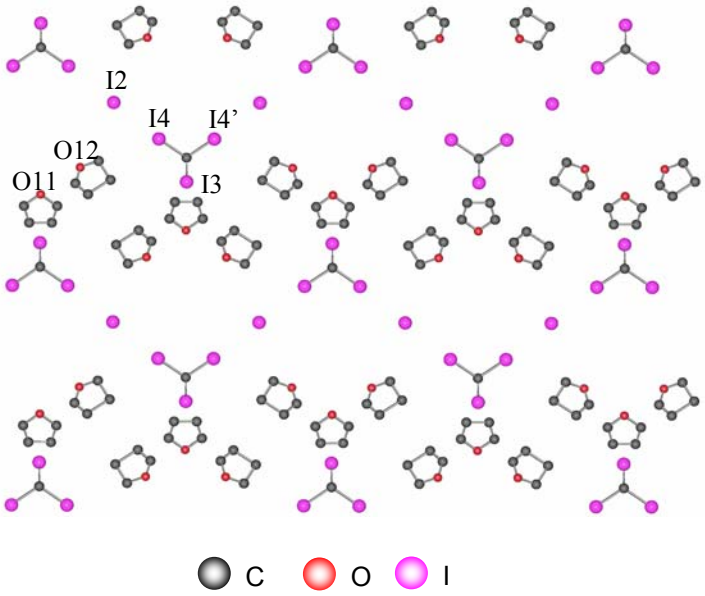


Figure II.11: A layer of solvent and counter ions contained in **3** showing the arrangement o CHI_3 and I2, (H-atoms omitted).

Atom I3 of the iodoform is also placed on a crystallographic mirror plane (x, 1/4, z (e)). I4 of the iodoform points toward the free anion I2 at a distance of 3.787(2) Å, leading to a linear arrangement of I4, I2 and I4 of a second iodoform molecule within the sheet. The *bc*-plane is thus divided into strands containing iodide ions and iodine atoms of iodoform, and chains containing the THF molecules. The sheets containing solvent molecules and counter ions alternate with the planes containing the crown ether complexes (Figure II.9).

The presence of iodoform, which was not added as a solvent, can be explained when looking at the reaction conditions. In a first step, NaO*t*Bu is hydrolysed in the THF/H_2O mixture to yield HO*t*Bu and NaOH. To avoid demixion of the solution due to the amount of base, acetone was deliberately added and it reacted typically with excess iodine under basic conditions to yield CHI_3 and by-products (iodoform reaction) [184].

In compound **3**, a fragment of a chain structure is achieved by hydrogen bonding, which does not completely propagate in one dimension, but is stopped by terminal THF ligands to sodium. If THF was not present in compound **3**, a structure similar to the known [Na(DB18C6)Br(H_2O)][Na(DB18C6)(H_2O)$_2$]Br(H_2O) [185], with an infinite one-dimensional hydrogen bonding system would possibly have been observed. A comparison of the fragments [Na(DB18C6)I(THF)] and [Na(DB18C6)(H_2O)$_2$]$^+$ with analogues in the literature shows that the distances fit in general with previous results [178, 185, 186].

The distance Na1–I1 with 3.523(4) Å of **3**, is longer than in the structure [Na(DB18C6)I(THF)] [178], where Na-I is 3.094(3) Å. The average Na–O(crown

ether) distances in **3** are with 2.738(7) and 2.766(7) Å longer than in $[Na(DB18C6)(H_2O)_2]^+$ in which a dimer of a cationic complex is observed with average Na-O(crown ether) distances of 2.705 and 2.727 Å [185]. In the latter compound, the Na-O distance to the terminal water molecule is with 2.313(9) Å by 0.7 Å shorter than the Na–O (THF) distance in **3**, but by ca. 0.2 Å longer than for the similar Na–O (H_2O) bond of **3**.

II.1.5. $[(H_5O_2)(Br4\text{-}DB18C6)_2][FeBr_4]$ 4

In order to synthesize the tribromide analogue to **1**, several attempts were made and the following compounds show the results of these reaction.

Attempting to generate tribromide in situ, bromine was added to a solution of DB18C6 in THF in the presence of water and iron (iron is here to generate Br^- anions). The compound $[(H_5O_2)(Br4\text{-}DB18C6)_2][FeBr_4]$ **4**, is observed in 60 % yield.

The structures 4 to 7 are the results of some attempts to get isostructural compounds of **1** but with tribromide ion as counter ion. Even if the structures are not the one expected, these examples illustrate a whole range of interaction which pack molecules together.

4 crystallises in the monoclinic space group *C*2/c (N°.15) with four molecules per unit cell (Figure II.12.a and b).

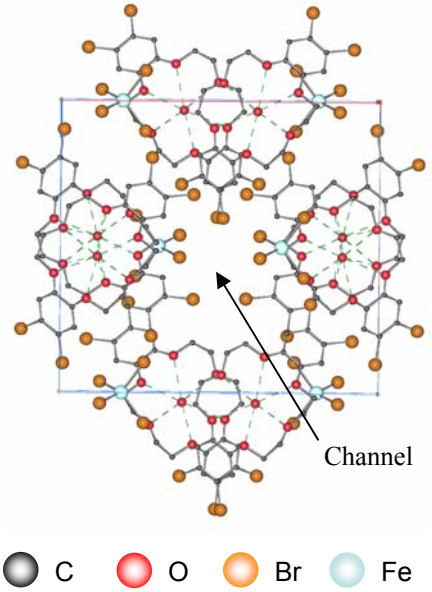


Figure II.12.a: Unit cell of **4** containing bromo-containing channels (*ab*-plane) (H-atoms omitted for clarity).

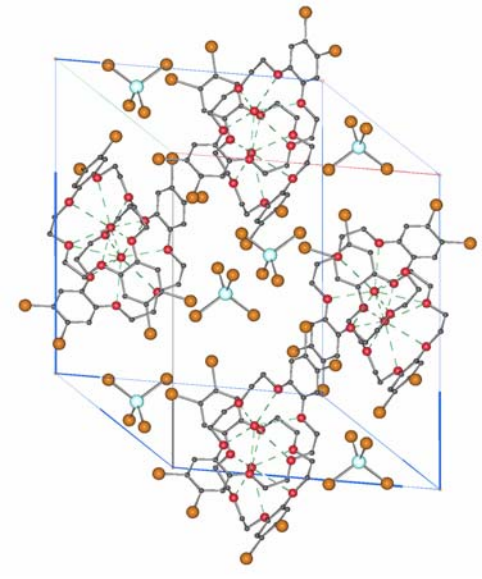


Figure II.12.b: Unit cell of **4** showing the zigzag line of $FeBr_4^-$, (H-atoms omitted for clarity).

Its formation can be explained by a series of reactions. In a first step, the crown ether molecule is twice brominated at each of the two phenyl groups to yield 4,5,4',5'-tetrabromodibenzo-18-crown-6 (further on called "Br4-DB18C6") [187, 188], and HBr. The latter generates acidic conditions necessary for the presence of H_3O^+. Iron powder, which is present, is attacked and oxidized by bromine to give the large $[FeBr_4]^+$ anion present in the structure of **4**. The iron atom of the anion is found on a crystallographic two-fold axis (0, y, 0.25(*e*)). The large cation present in **4**, $[(H_5O_2)(Br4\text{-}DB18C6)_2]^+$, is formed by two Br4-DB18C6 ligands that form a tennis ball-like cavity with a corresponding seam (Figure II.13.a and b).

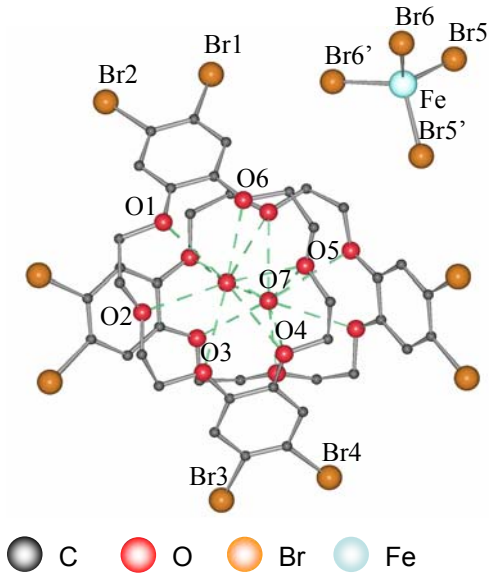


Figure II.13a: Molecular entity of **4**, (H-atoms omitted for clarity).

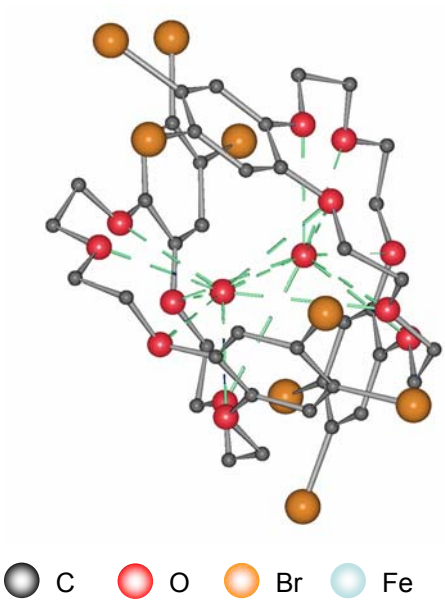


Figure II.13.b: Side-view of the capsule formed by two Br4-DB18C6 ligandscontaining $H_5O_2^+$, (H-atoms omitted for clarity).

In the centre of this cavity, two oxygen atoms, O7 and its symmetry equivalent O7' are found. O7 is located 1.36(1)Å over the mean plane formed by the six oxygen atoms of the crown ether, and the O7–O(crown) distances are rather long and range from 2.84(1) Å (with O5) to 3.26(1) Å (with O1). The shorter ones probably correspond to strongest hydrogen bonding interactions wheras the longer ones to electrostatic repulsion of O7 towards O1. Hydrogen atoms could not all be located on O7 and only two could be attributed, pointing in the directions of O5/O4 and O2/O3, indicating bifurcating H-bonding. The O7–O7' distance of 2.42(2) Å indicates that the present species is a $H_5O_2^+$ cation stabilized in this cavity with the third proton being split over two positions somewhere along the bisector between O7 and O7'. Crystallographically, this means that a water molecule and a hydronium cation are delocalized over both positions, and that the third hydrogen atom could be delocalized over both oxygen atoms. In the IR spectrum of **4**, typical broad bands for $H_5O_2^+$ are found at 3359cm^{-1} and around 1700cm^{-1}. Similar distances and IR bands are observed in other structures in which $H_5O_2^+$ has been characterized, and compared

to literature compounds, the O7–O7' distance of **4** is in the range of normal O–O contacts (2.288 Å to 2.473 Å) for $H_5O_2^+$ [189, 190]. The dibromo-substituted phenyl rings of one crown ether molecule form an angle of 127.4(7)° to each other, which is on average wider than in the nonbrominated DB18C6, although, the latter can also adopt flatter angles in some cases, as seen for instance in **3**. Interestingly, one of the aromatic rings only deviates by 15.6(3)° from the mean plane formed by the six oxygen atoms of the crown ether whereas the other is 48.8(2)° out of plane. This is probably due to the fact that the two macrocycles do not have the same interactions with $H_5O_2^+$ ion. Indeed, one phenyl ring (C11 to C16) interacts more or less with H atoms of $H_5O_2^+$ ion which create attraction of the electronic clouds of benzene (angle increase until 48.8(2)°) whereas the other phenyl ring (C1 to C6) with the same $H_5O_2^+$ ion is in interaction with oxygen atom. This latter ineraction is a repulsive interaction (angle decrease until 15.6(3)°).

In the packing of **4**, the "tennis balls" arrange along the *c*-axis, and bromine atoms of the ligand as well as from the $[FeBr_4]^-$ point into channels along the same direction (Figure II.12a and b). Hydrogen bonds between the four bromine atoms of $[FeBr_4]^-$ and H-atoms of crown ether phenyl rings are observed around 3 Å and involve Br5–H5 and Br6–H2. One bromine atom of each phenyl ring, Br1 and Br4, is also involved in hydrogen bonding to H9A and H20A, respectively (3.02 and 3.25Å) of neighbouring crown ether molecules. Shorter contacts of bromine with hydrogen atoms are present in the structure, but cannot be counted as efficient H-bonding due to small C–Br–H angles inferior 90°.

The NMR signals of **4** in $CDCl_3$ are complicated to interpret due to the similarity of H_3O^+ and $H_5O_2^+$. In **4**, a signal at 7.704 ppm seems to testify to the presence of $H_5O_2^+$. Two other signals at 3.747 ppm and 1.444 ppm could reveal the dissociation of $H_5O_2^+$ into H_3O^+ and H_2O when crystals are dissolved in $CDCl_3$. Further analysis, with COSY (Correlation spectroscopy) and HSQC (Heteronuclear single quantum correlation), reveal the interaction of H7a with H17a, H10b with H10b and H20b with H20b (CH_2) from one crown ether with their homologues on the partner crown ether in the bowl. This indicates that the tennis-ball shaped capsule exists in solution.

Nevertheless, this conformation is not totally stable as it can be observed signal for H_3O^+ and H_2O.

II.1.6. [(H_3O)(Br4-DB18C6)$(Br_3)Br_2$] 5

The same reaction carried out as for 4 with bromine in excess, but without iron present, yields the compound [(H_3O)(Br4-DB18C6)$(Br_3)Br_2$] **5** in 61% yield.

5 crystallises in the triclinic space group *P*-1 with two molecules per unit cell. Again the DB18C6 has been brominated to yield the Br4-DB18C6 (Figure II.14.a and b).

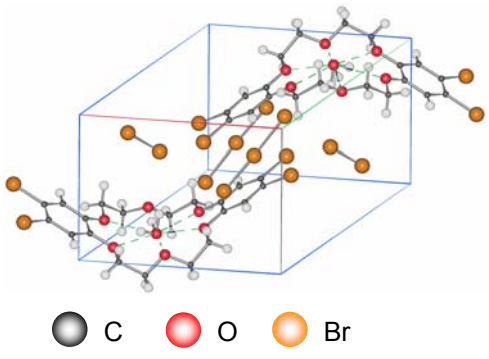


Figure II.14.a: Crystal cell of **5,** (H-atoms omitted for clarity).

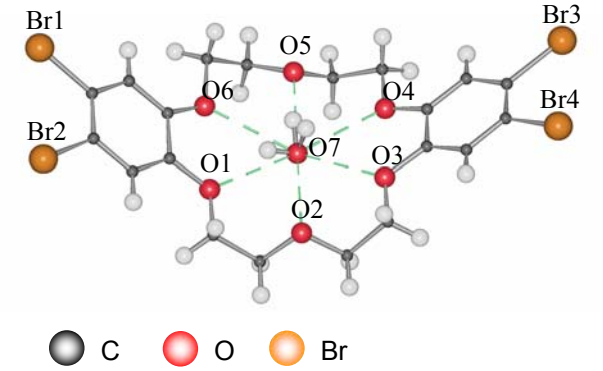


Figure II.14.b: Cationic complex of **5,** (H-atoms omitted for clarity).

The ionic compound **5** consists of a complex cation in which H_3O^+ (O7) is coordinated by the six oxygen atoms of a Br4-DB18C6. The O7 atom is by 0.32(1)Å out of the mean plane formed by the six oxygen atoms of the macrocycle on the exo position to the cavity, indicating pyramidal H_3O^+ as discussed previously [2, 175, 186], with O7–O(crown) distances between 2.54(1) Å to O5 and 2.80(1) Å to O2. This large difference in distances shows the different coordination of H_3O^+ by the crown ether. Hydrogen atoms of the cation could be located in the Fourier map. They are arranged in a pyramidal fashion around O7, two hydrogen atoms pointing towards O5 and O3 with a strong H-bond of 1.6(1) Å for H71–O5 (O7–H71–O5 163(8) °),

and a weaker H-bond of 2.0(1) Å for H72–O3 (O7–H72–O3 of 143(9) °). The distances between O (crown ether) and O7 varying from 2.557(2) to 2.81(1) Å are on average similar to the values in structures with 18crown6 [173, 191] varying from 2.55(5) to 2.84(5) Å or with DB18C6 [174], varying from 2.570(7) to 2.797(8) Å. The disorder observed in the structure by Reich et al. [174] seems to be absent in **5**. The third hydrogen atom of H_3O^+ does not point to an oxygen atom of the crown ether, but to Br3 of an adjacent crown ether molecule, with a distance of 3.103 Å. Indeed, it seems that Br3 points more in the direction of the crown below than the other three close bromine atoms attached to adjacent crown ether molecules. The two phenyl rings form an angle of 120.9(2) ° to each other, by ca. 7° smaller than in **4**, and exclude the hydronium cation. This time, and in contrast to **4**, both phenyl rings are almost equally out of the mean plane formed by the six oxygen atoms of the crown ether, i.e. by ca 30°.

The cations stack on top of each other, all folded in the same direction within one stack, and 8.138 Å apart from each other. The neighbouring stack of cations runs in the opposite direction and intercalates with the dibromo-arene groups between the dibromo arene groups of the crown ether molecules of the first stack (Figure II.15).

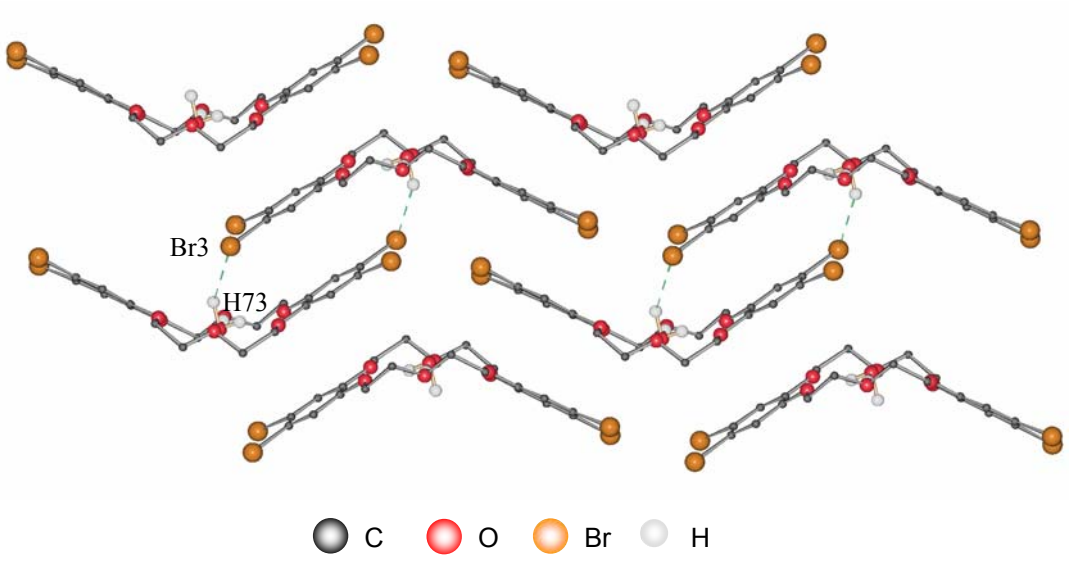


Figure II.15: Packing of cationic complexes in **5** (H-atoms omitted for clarity).

The aromatic rings are offset with respect to each other by ca. 4.7 Å and the closest distance between rings is ca. 3.7 Å, indicating only weak π-π-interactions. The

arrangement is such that sheets of cations are formed in the (x, 1/4, z)-plane. The two-dimensional cationic arrays are separated by the anions, consisting of Br_3^-, and by bromine, Br_2. Bromine and tri- bromide are arranged in a plane (Figure II.16).

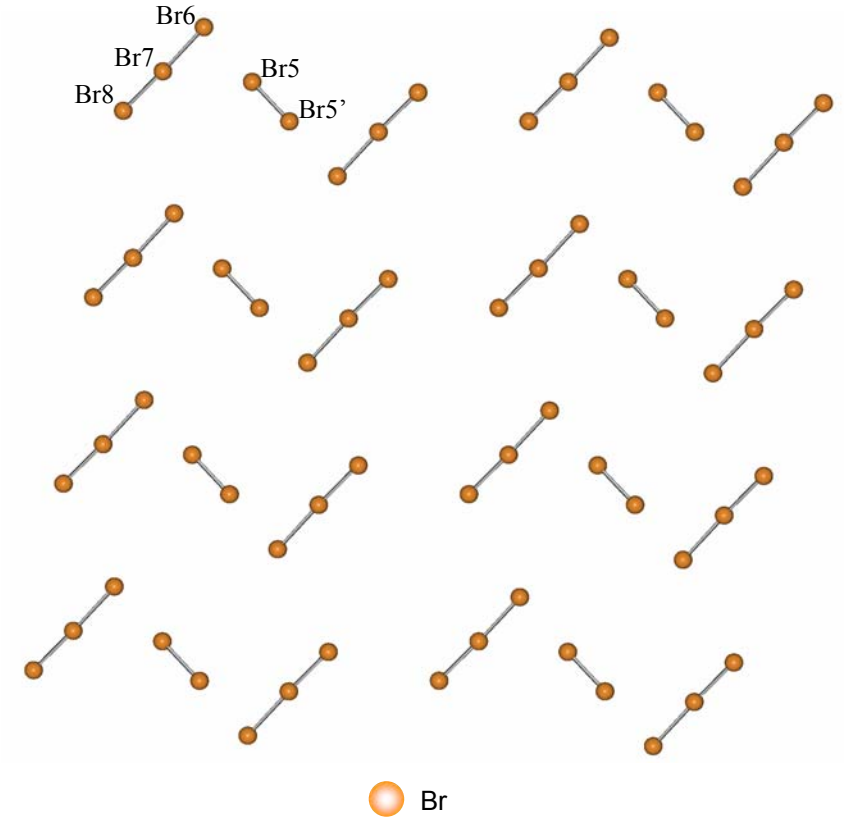


Figure II.16: Layer of **5** containing counter ions Br_3^- and Br_2 molecules, (H-atoms omitted for clarity).

The Br–Br distance in Br_2 is 2.331(3) Å long, whereas in the tribromide, the distances are 2.401(3) and 2.627(2) Å for Br7–Br8, respectively Br6–Br7. The Br6–Br7–Br8 angle is almost linear at 177.6(5)°. Weak interactions between the tribromide and bromine can be considered at a distance Br6–Br5 of 3.187(2) Å. A crystallographic inversion centre is found in the middle of the bromine molecule. The short Br–Br distances of **5** are by 0.02 Å longer than the shortest one in solid bromine [192]. The longer interbromine contacts are in the range of normal Br-Br found for polybromide ions [193, 194].

The signal at 3.74 ppm found in the ^{1}H-NMR spectrum of **4** is also present in the one of **5**. This value is more or less in accordance with the signal of 4.33 ppm given

by You et al. [195] for H_3O^+ in $CDCl_3$.The difference could result from a change in the environment. Indeed in **4** and **5**, hydronium ions are isolated from the environment by coordination with crown ether.

II.1.7. [K(Br2-DB18C6)(Br_3)] 6

The reaction of DB18C6 with a solution of Br_2 titrated by KOH until neutralisation of Br_2 acidity in THF/water solvent yields after fifteen days, single crystals of [K (Br2-DB18C6)(Br_3)] **6** in 73% yield.

6 crystallises in the triclinic space group *P*-1 (N° 2) with two molecules per unit cell. DB18C6 is only twice brominated. This may be due to pH conditions fixed around 7 whereas before, the pH was 1 for **4** and **5**. The structure is comparable to [K(DB18C6)](I_3) [196]. The main motif consists of a potassium ion coordinated by the six oxygens atoms of the crown ether, a Br_3^- ion (K–Br 3.15(1) Å) and a water molecule (K–O 2.1(2) Å). The potassium ion is also out of the plane defined by the six oxygen of Br2-DB18C6 by 0.30(1) Å. It is furthermore strongly coordinated by O6 of the Br2-DB18C6 with 2.71(2) Å whereas the other K–O(Br2-DB18C6) distances are longer with 2.73(1) to 2.82(2) Å

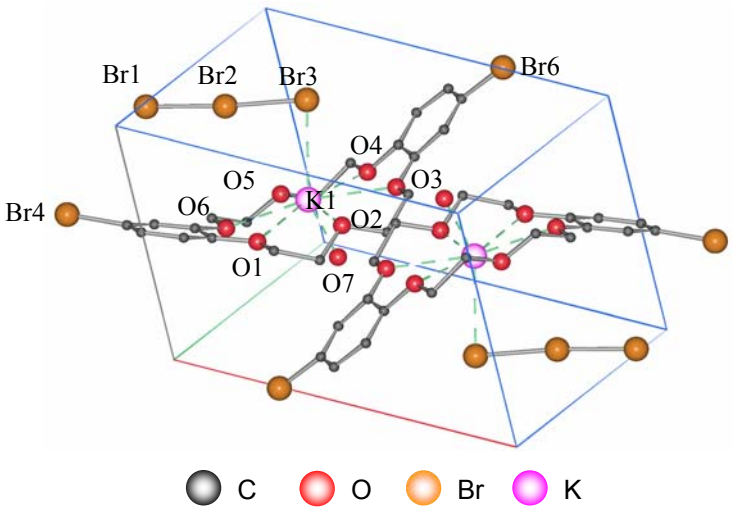


Figure II.17: Unit cell of **6** (H-atoms omitted for clarity).

The structure of **6** is held together by electrostatic interactions: hydrogen bonds (O7–Br3 3.370 Å, O7–Br6 3.287 Å), Br3–Br6 contacts (2.900 Å) or ionic bond K–Br3 (3.15(1) Å) depicted in figure II.18.These contacts create a chain Br3–K–O7. In addition, Br3, Br6 and O7 define an equilateral triangle of interactions. Finally, crown-ether chains stack themselves in a wave-like fashion thanks to weak π-CH_2 (C19 and C20 with aromatic ring C1-C6) interactions.

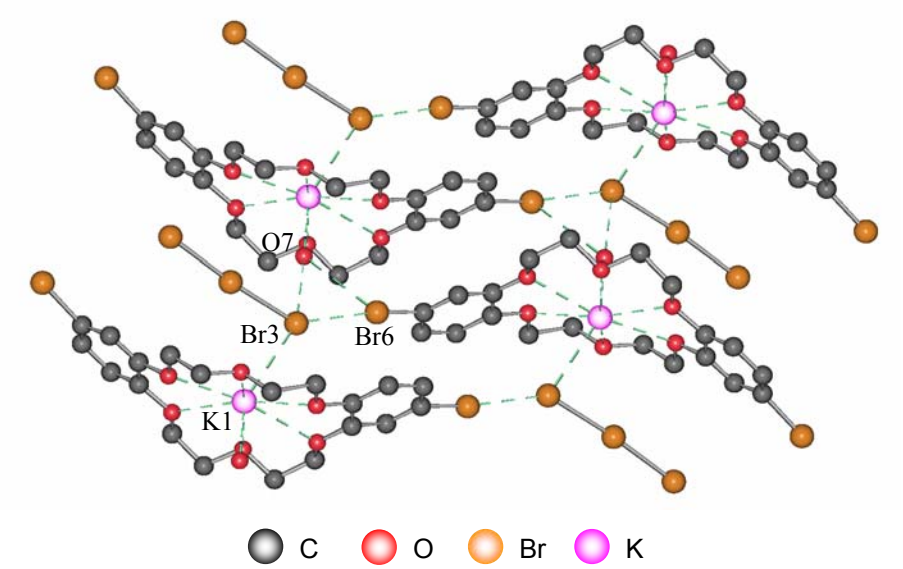


Figure II.18: Contacts of different Br in **6** (H-atoms omitted for clarity).

In comparison to the structure of Schröder and al. [196], the introduction of a water molecule between Br2-DB18C6 and Br_3^- ion opens the structure. The interactions with potassium ions and DB18C6 are destroyed in **6** and a slipping of crown ethers occurs. The empty space is filled with a Br_3^- ion which coordinates directly to the potassium cation.

II.1.8. [(Me_3NPh)(DB18C6)(Br_3)] 7 (Me_3NPh = trimethylphenylammonium ion)

In order to avoid alkali metal ions to be competing with H_3O^+ to be coordinated by DB18C6, phenyltrimethyammonium tribromide ([Me_3NPh]Br_3) was used to do the anion exchange I_3^- by Br_3^-. This however gave not the desired Br_3^- analogue to **1**, but [(Me_3NPh)(DB18C6)(Br_3)] **7** in 82% yield without bromination. There is no bromination in this case because of the low concentration of Br_3^- ions, the active species.

7 crystallises in the monoclinic space group $P2_1/m$ (N°.11) with two molecules per unit cell. The cation of **7**, $[Me_3NPh]^+$ is coordinated by the crown ether via H18A–O1 and H12–O1 contacts and by π-stacking between C14/C15 and phenyl ring of the following DB18C6 with distances of 3.89(1) and 3.63(1) Å respectively. The DB18C6 molecules are stacked to form channels in the direction of the *a*-axis (Figure II.19).

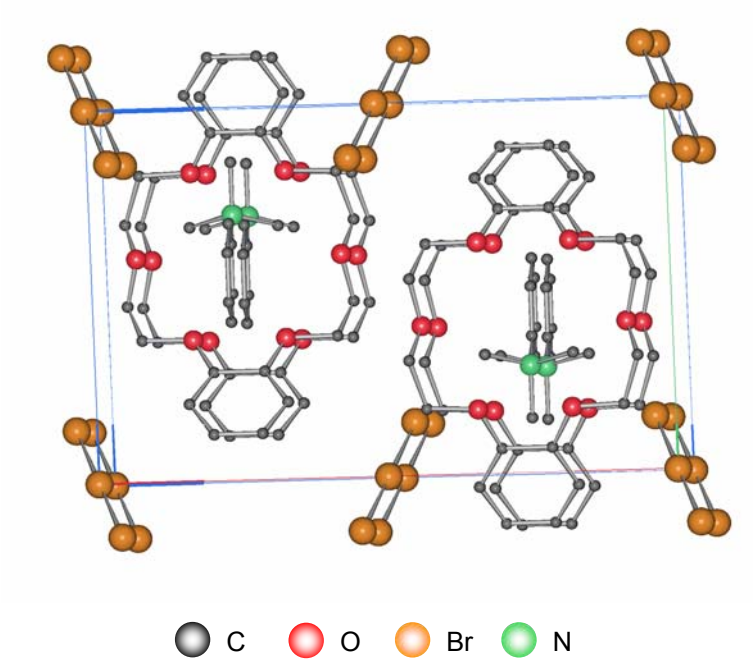


Figure II.19: View along the *bc*-plane of **7**, showing parallel arrangement of crown ether molecules, (H-atoms omitted for clarity).

The stacking is however not a close one as in **1**, as the large $[Me_3NPh]^+$ cations are intercalated between the DB18C6 ligands with the phenyl group pointing in the direction of the middle of one DB18C6 molecule, and the methyl groups pointing opposite into the cavity formed by the next DB18C6 ring (Figure II.20).

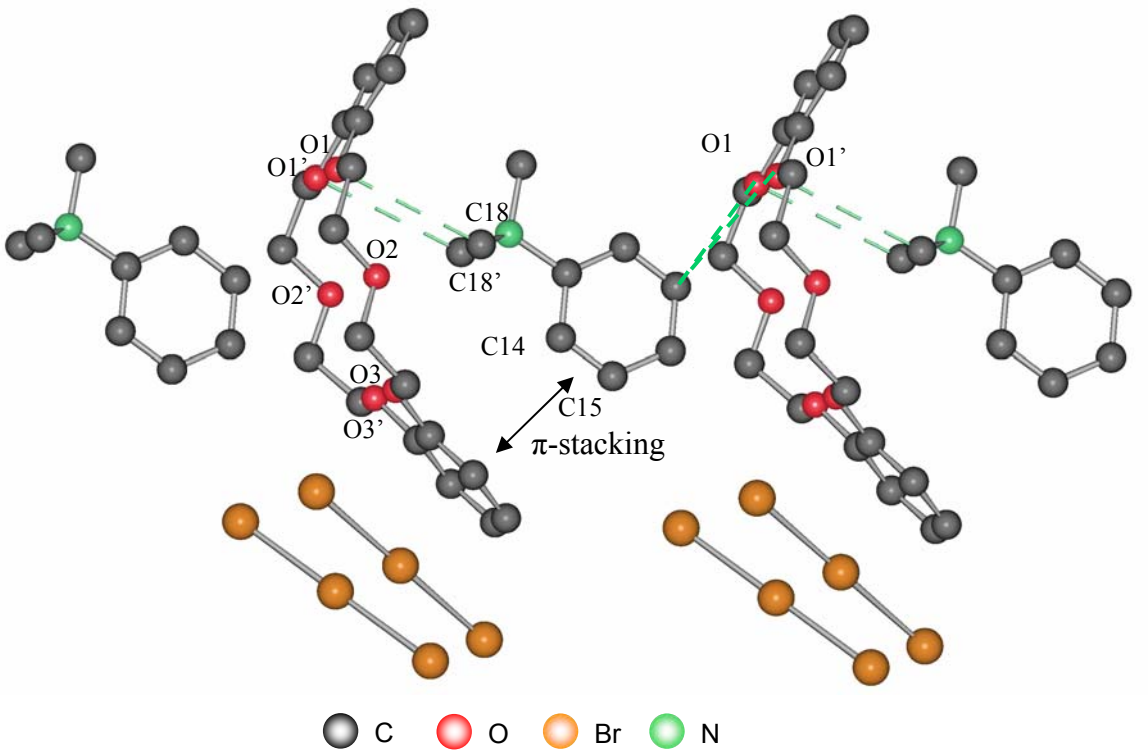


Figure II.20: Intercalation of large cations between parallel DB18C6 ligands in **7** (H-atoms omitted for clarity).

A crystallographic mirror plane runs through the carbon atoms of the phenyl ring of the cation, its nitrogen atom and a methyl group, all placed on (x, 1/4, z, (*e*)). Hydrogen bonds are observed between cation and crown ether, the shortest one being formed from hydrogen on a methyl group, H18A, to O1 of the crown ether with 2.534(1) Å. Another short contact is found between H12 of the phenyl ring of $[Me_3NPh]^+$ and O1 of the next macrocycle with 2.572(1) Å. The crown ether molecules are in a folded "butterfly" configuration with angles between the two phenyl rings of 106.8(2) °. This is ca. 10° wider than the angles observed in **1** [2], but by ca. 20°smaller than in **1**. The direction of folding of the crown ethers alters from one channel to the next, adopting the opposite direction. The distance between two DB18C6 molecules within one channel corresponds to 9.520 Å (the length of the *a*-axis). The tribromide anions are linear due to an inversion centre on which Br1 is placed (0, 0, 0 (*a*)), and possess Br1–Br2 and Br1–Br2-distances of 2.5454(6) Å.

They do not run parallel to the channel propagation direction, but are inclined by 40.48° with respect to the *a*-axis.

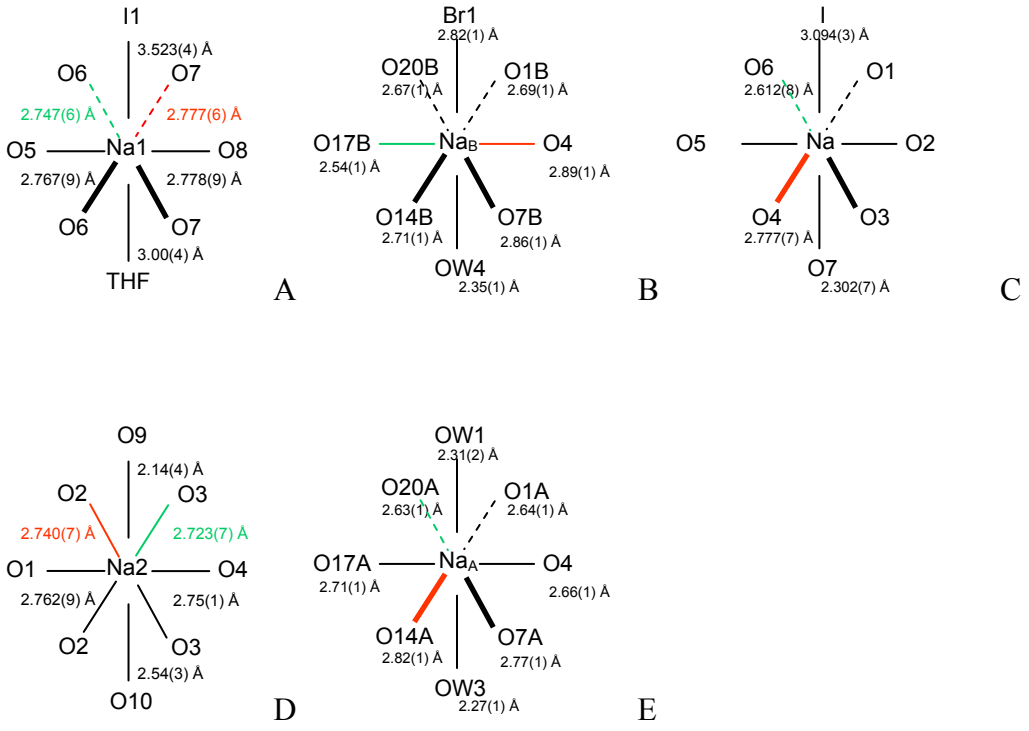
II.1.9. Comparison of structures 1 to 7: a large range of crown ether crystals

The compounds **1-7** present the different types of organization of crown ether molecules. **3** is a combination of two defined complexes found in the literature: [Na(DB18C6)I(THF)] and [Na(DB18C6)(H_2O)$_2$]X. In fact, it is interesting to notice the difference between a bridging and a non-bridging iodide ion upon the coordination of the crown ether ligand toward Na^+. In **3**, coordination is assumed principally by O6 of the crown ether ligand whereas some weaker interactions exist with other oxygen atoms of the crown ether. Table II.4 and scheme II.5 compare the coordination of Na^+ by DB18C6 in our and other literature compounds.

Interatomic distances	**A** in **3**	**B** in infinite chain [186]	**C** in [197]		**D** in **3**	**E** in infinite chain [186]
Na–O(CE) shorter (Å)	2.747(6)	2.54(1)	2.612(8)	Na–O(CE) shorter (Å)	2.723(7)	2.63(1)
Na–O(CE) larger (Å)	2.777(6)	2.89(1)	2.777(7)	Na–O (CE) larger (Å)	2.740(7)	2.82(1)
Na–O(ligand) (Å)	3.00(4)	2.35(1)	2.302(7)	Na–O(water) (Å)	2.14(4)	2.31(2)
Na–X (X=I, Br, H_2O) (Å)	3.523(4)	2.82(1)	3.094(3)	Na–O(water) (Å)	2.54(3)	2.27(1)

A: [Na(DB18C6)I(THF)]; B: [Na(DB18C6)Br(H_2O)]; C: [Na(DB18C6)I(THF)]; D: [Na(DB18C6)(H_2O)$_2$]$^+$; E: [Na(DB18C6)(H_2O)$_2$]$^+$.

Table II.4: Comparison of average distances in **1** and similar literature compounds.



Scheme II.5: Representation of the coordination of Na^+ by DB18C6.

In most cases, the Na–O distances are distributed asymmetrically around the crown ether ligand, with the shortest and the longest bonds being separated by one medium long Na–O contact.

The crown ether molecules in **3** seem to be arranged by serendipity in a linear chain via the coordinated sodium ion interactions. The introduction of a cation as bridging agent favors the appearence of well-organized chains of crown ether ligands like in **7**. These chains are maintained by hydrogen bonds. Finally, if the cation is small (and capable of H-bonding), infinite π-stacking of the crown ether molecules as in **1** can be observed. In this last case, small cations have to be linked by a small molecule in order to allow close packing of the macrocyclic ligands (H_2O in **1**, acetonitrile in reference [195]). In the absence of an efficient linker, only one single π-stacking is observed by the crown ether ligands as in **5** and **7**, as in the structure published by Kloo et al. [172]. $[(H_3O)(Br4\text{-}DB18C6)](Br_3)Br_2$ (**5**), shows the same organisation of the crown ether ligands as in compound $[(H_3O)(DB18C6)(I_3)]$ [172]. The tribromide and triiodide point in the same direction along the intertwined crown

ether stacks whereas the main difference lies in the presence of bromine molecules, which limits the symmetry operations in the crystal. In table II.5, the environment of H_3O^+ for **5** and the literature compound are given. In comparison with the structure described by Kloo, the distances are the same for the longer coordination bonds of H_3O^+. For the shorter distance, there is a difference of 0.13 Å which can be attributed to a better stabilization of H_3O^+ in **5** due to the existence of H_3O^+ ion in a unique pyramidal shape whereas in the structure described by Kloo H_3O^+ ion exists in pyramidal and in planar shape.

Interatomic distances	$[(H_3O)(Br_4\text{-DB18C6})](Br_3)Br_2$ **5**	$[H_3O(DB18C6)(I_3)]$ [172]
H_3O^+–O(crown ether)shortest (Å)	2.56(1)	2.69
H_3O^+–O (crown ether) long (Å)	2.81(1)	2.81
H_3O^+-plane (Å)	0.35(1)	0.061-0.78

Table II.5: Environment of H3O$^+$ for **5** and the literature compound.

For **6**, the tribromide ion acts as a π-stacking inhibitor. Indeed, the strong coordination Br–K^+ stops the creation of an electrostatic interaction to allow the superposition of Br2-DB18C6 like in $[DB18C6]KI_3$ [196]. Once more, the ionic interaction wins over weak interactions. In the case of **5**, Br3–H73(H_3O^+) contacts for result in the eviction of the tribromide from the coordination sphere. This anion stays as a spectator ion along the stacked Br4-DB18C6.

If **1** and $[(H_3O)(DB18C6)(I_3)]$ are compared, a pH-dependency can be presumed. The same starting materials produce two different products **1** (under neutral pH condition) and $[(H_3O)(DB18C6)(I_3)]$ (in very acidic condition). As the pH is the only difference between their synthese, it can be supposed that it is an important driving force in crystallization. For **5** and **6,** it is much more difficult to emit a clear opinion because of the presence of potassium ions in the structure of **6**. Nevertheless, the bromination is pH dependent. Br4-DB18C6 and Br2-DB18C6 are to different molecules also. They crystallize thanks to different interactions. It can be this

information that one can retain to consider an influence of the pH in the crystallization of 5 and 6.

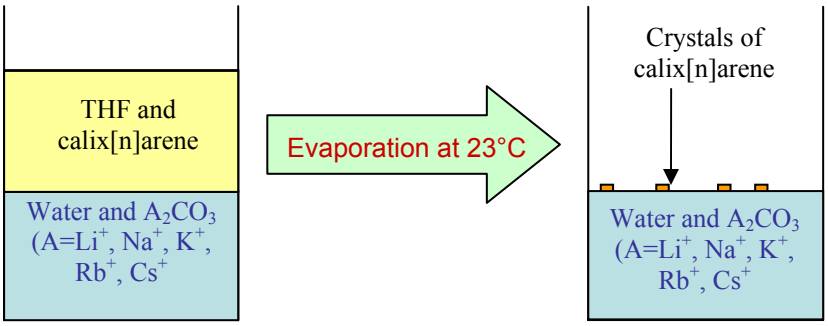
Finally, crown ethers are able to create capsules around dihydronium like in **4** or around purinium [198]. For small guests, crown ethers are able to build a cage similar to the form of a tennis ball with a corresponding seam. The two crown ether molecules are arranged approximately perpendicular to each other. Cohesion is assured by hydrogen bonds between ethyl hydrogen atoms and ether oxygen atoms (H20a–O5, H17a–O6) as well as phenyl hydrogen atoms and ether oxygen atoms of opposite macrocycles (H15–O1). For larger guests, crown ethers molecules may stack parallel to each other either via weak interactions (π-stacking and hydrogen bonds [198]) or due to symmetry as in **7**.

To resume on crown ether containing compounds, for compounds **1** and **2**, cation exchange was shown to take place by immersion of a crystal of **1** into a solution of NaOH. Conductiviy measurements indicate a hopping process of conduction within a channel system of **1** or **2**. Further studies will be carried out to further strengthen this theory such as conduction versus gradient of concentration measurement.

Other attempts of obtaining similar structures by anion exchange have failed so far and gave other interesting packing situations of crown ether molecules such as the capsule in **4**.

II.2. Towards ionic channels with 4-tert-butylcalix[n]arenes (with n=6,8): crystal structures

Calix[n]arenes are relatively flexible molecules. Calix[4]arene can be observed in four conformations (cone, partial cone, 1,2-alternate or 1,3-alternate) in solution [86], calix[6]arenes are well-known for their inversion mechanism [86] whereas calix[8]arene, which can take even more conformations, remains mainly in pleated loop conformation even in solution because of intramolecular hydrogen bonds stabilization [86]. Intramolecular bonds seem to have a stabilising effect such as double bonds in the Kekulé model for benzene, i.e. intramolecular hydrogen bonds are here to maintain a kind of flat conformation like the conjugated double bonds in benzene assure the planarity of the molecule. In addition, it seems that the more deprotonated, the more flexible calix[n]arenes are [206]. In order to avoid the flexibility problem of these large molecules, a two-phase system containing a basic aqueous phase and an organic solvent was chosen to favour only one conformation among the others. The amount of base in the aqueous phase produces the demixion suitable for the two-phase system. Indeed the saturation of the aqueous phase by a salt decreases the miscibility of THF in water. This allows to a certain extent to orient the calix[n]arene molecules (with n=6, 8) such that the OH-groups are pointed more or less into the aqueous phase, whereas the rest of the ligand molecule is dissolved in the organic solvent phase. This technique consists in layering a calix[n]arene dissolved in THF onto a basic solution of water. After a relatively slow removal of THF by evaporation through Parafilm TM at 23°C, single crystals can grow at the interface air/water. Scheme II.6 describes this process.



Scheme II.6: Description of the crystallisation technique.
THF are less miscible due to the presence of basic salts in water

II.2.1. [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$] 8

In a first experiment, the aqueous solution is a 1 M of K_2CO_3. Upon layering of this aqueous phase with a 1.5 mM solution of calix[6]arene in THF, single crystals of [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$] **8** appear after 1-2 days at the H_2O/air interface after slow evaporation of THF.

8 crystallises in the hexagonal space group $P6_3/m$ (n° 176) with two molecules per unit cell (Figure II.21). Each asymmetric unit is made of half a 4-tert-butylcalix[6]arene molecule, one and a half non-coordinating THF molecules, six water molecules, one sixth of a hydrogen carbonate anion (this anion is due to the equilibrium between carbonate and hydrogen carbonate in aqueous solution and to a titration of H_3O^+ present in non dried THF) and one sixth of a potassium cation.

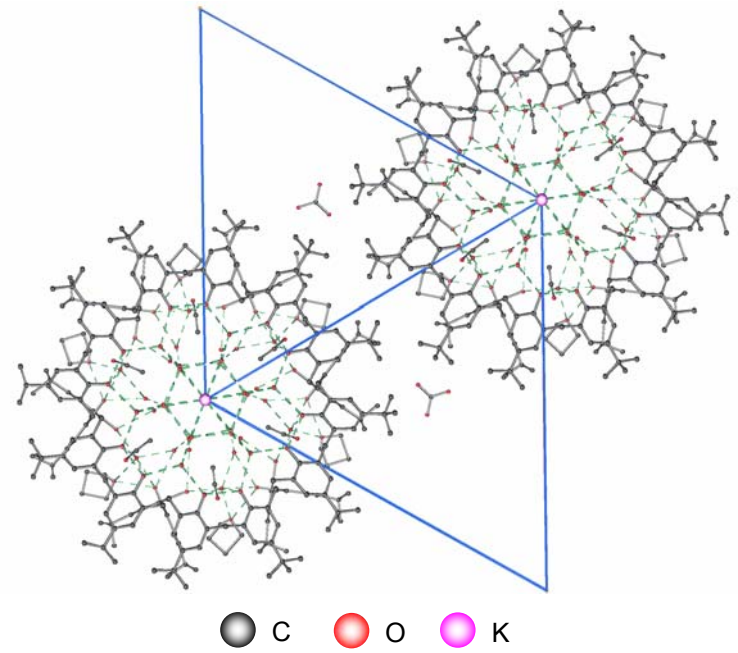


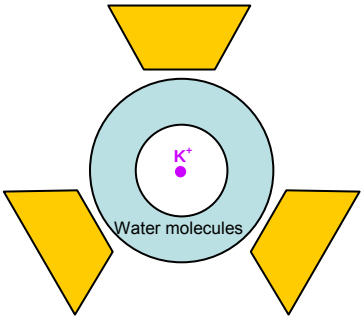
Figure II.21: Unit cell of **8** (H-atoms omitted for clarity).

The potassium ion K1 is located at (0, 0, 0, (a)) and (0, 0, 0.5, (b)) on the axis of symmetry C_3. It is hydrated by O10, O11 and their ten symmetry equivalents with average bond length of 3.39(1) Å. The distance between K and O(water) is relatively long but if coordination is excluded to the benefits of hydration interactions, this value is less uncommon [199, 200, 201].In this case the potassium ion is not coordinated by water but much more solvated which could explain this number of twelve water molecules around the cation. Each sphere of solvation is linked to the next by hydrogen bonds (O11–O12 2.906 Å) and to 4-tert-butylcalix[6]arene via O10–O7–O3 and O11–O7–O3 with hydrogen bonds (O7–O10 2.686(8) Å, O7–O11 2.761(9) Å, O7–O3 2.703(5) Å.

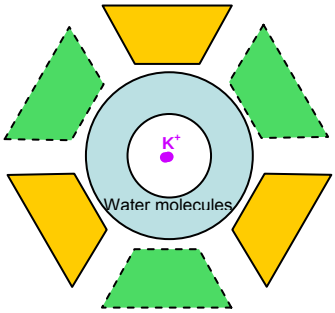
Around each solvated cation, there is a 4-tert-butylcalix[6]arene, two THF molecules and some water molecules. One THF molecule is non-coordinating to water molecules. In contrast, the second one forms with O5 the apex of a trigonal bipyramid of oxygen atoms. The base of the bipyramid (O1–O8–O8') is located on the plane made by the six oxygen atoms of the 4-tert-butylcalix[6]arene. The water molecule of O4 is far from O8 and O8' with a distance of 2.812(4) Å and can easily create H-bonding contacts which stabilise the edifice.

The 4-tert-butylcalix[6]arene adopts a flattened cone conformation. The Ph-CH_2-Ph angles are obtuse with values of 113.6(3)°, 114.5(3)° and 120.9(4)°.

The overall arrangement of **8** can thus be described as a wheel with a potassium ion at the centre surrounded by water molecules which are themselves surrounded by three calix[6]arene molecules (Scheme II.7.a and b). This arrangement is stacked on top of each other, rotated each time by 60° to yield a channel structure.



Scheme II.7.a: Schematic packing of one 4-tert-butylcalix[6]arene wheels around a center symbolized by potassium ion.



Scheme II.7.b Schematic packing of two 4-tert-butylcalix[6]arene wheels around a center symbolized by potassium ion.

From a side view, the packing of 4-tert-butylcalix[6]arene channel is compact (Figure II.22). Each indentation of the 4-tert-butylcalix[6]arene wheel fills perfectly the incurvation of both adjacent wheels.

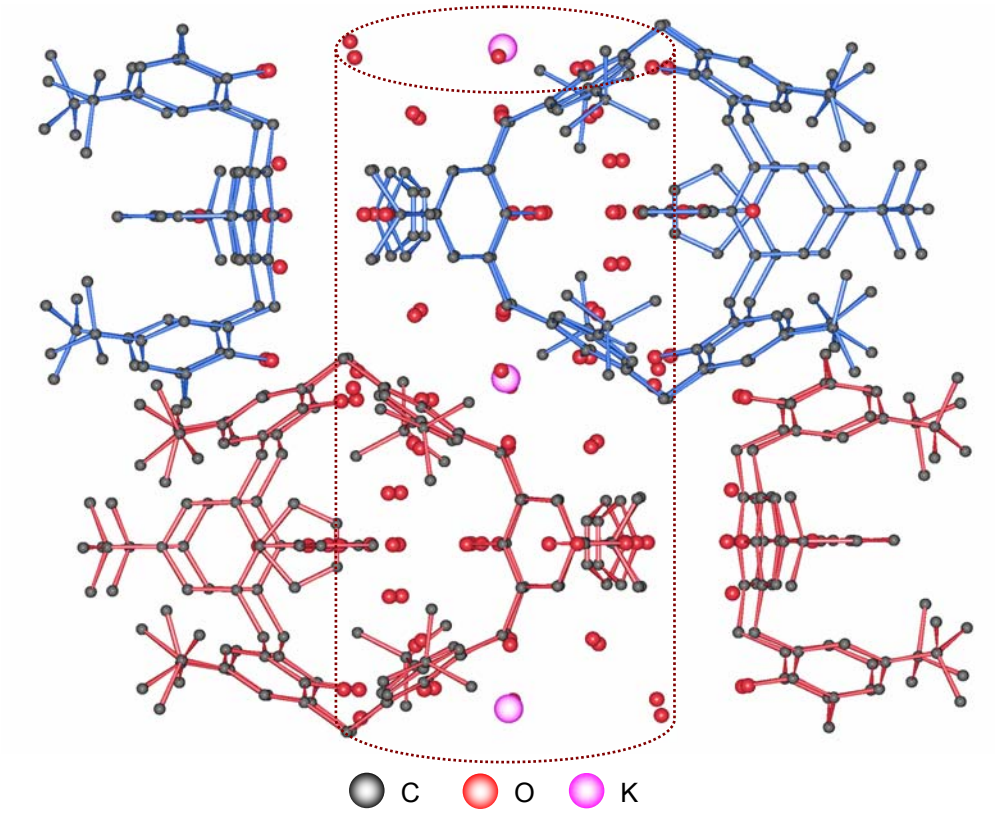


Figure II.22: Close packing of 4-tert-butylcalix[6]arene wheels (dashed line indicates channel direction, around which the calix[6]arene molecules arrange).

Finally, the hydrogenocarbonate anion is at the periphery of the 4-tert-butylcalix[6]arene moieties. It can assume the role of a linker between each wheel channel of 4-tert-butylcalix[6]arene ligands thanks to hydrogen-bonding interactions between O13 and H37c with a length of 2.797 Å (Figure II.23). Although H-atoms were not located due to the high symmetry of the compound (H-atom of HCO_3^- ion is located on a high symmetry position and is hard to find in this case), the characteristic C=O stretching vibration of hydrogen carbonate can be found around 1751 cm^{-1} which confirms the presence of hydrogen carbonate in the structure. No other clue of the hydrogen carbonate ion in the IR spectrum can be found due to the low signal of this one referred to the signal of 4-tert-butylcalix[6]arene.

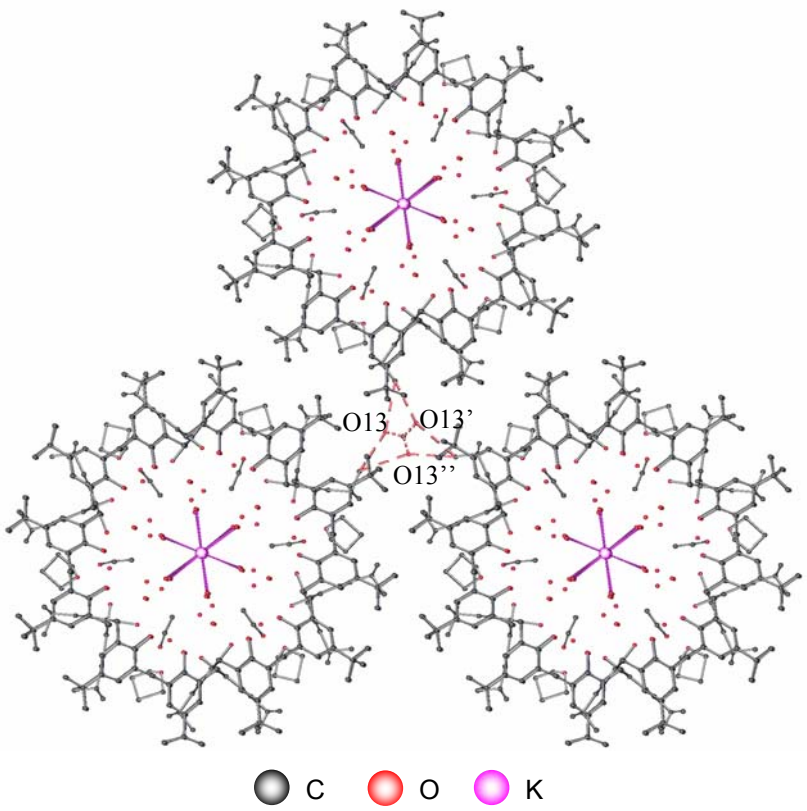


Figure II.23: Contact between 4-tert-butylcalix[6]arene wheel with O13.

Crystals containing calix[8]arene crystals are less known than others because of their lower ability to crystallise. Nevertheless, crystals were obtained with lanthanide and transition metals ions, mainly in pleated loop conformation. This can be explained by the influence of the intramolecular hydrogen bonds. It seems that this molecule has some problem to create long-distance interactions to keep or promote a crystal structure. In addition, in the solid state, each coordination sphere defined by calix[8]arene will act as a chemical Siege Perilous. The complexation forces have to be stronger than the intramolecular hydrogen bonds otherwise these latter will destroy organisation. This makes crystallization of such compounds extremely challenging, and therefore this series of new crystal structures presented in the following chapters can be proudly added to those reported in literature.

II.2.2. [Li_{15}(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4$] 9

When 4-tert-butylcalix[8]arene was treated with the superbase butyllithium/sodium ethoxide in dried THF (dried on sodium/benzophenone and freshly distilled), single crystals of [Li_{15}(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4$] **9** were obtained in good yield. Nevertheless, they turn into a hydroxide gel when exposed to air or can self-ignite.

9 crystallizes in the orthorhombic space group Pmmn (N° 59) with two molecules per unit cell (Figure II.24 a and b). Each asymmetric unit is composed of a quarter of a 4-tert-butylcalix[8]arene molecule which traps 3 and 3/4 of lithium ions (2, 3x1/2 and 1/4 lithium atoms in the representation) with help from three halves of THF molecules and two ethoxide anions, three non-coordinating halves of THF molecules one quarter of O^{2-} ion (a few amount of peroxide is present in THF) and one half of OH^- ion (due to moisture) complete this unit.

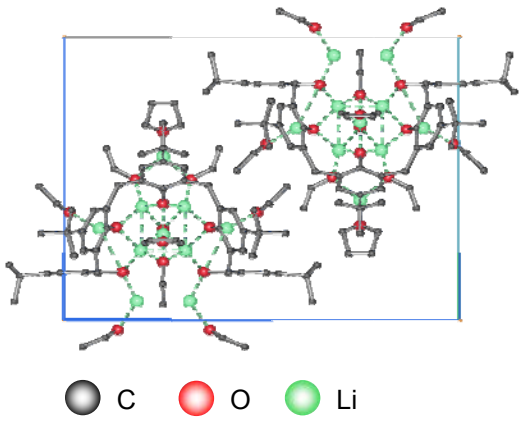


Figure II.24.a: view onto *ac*-plane of unit cell of **9** (H atoms omitted for clarity)

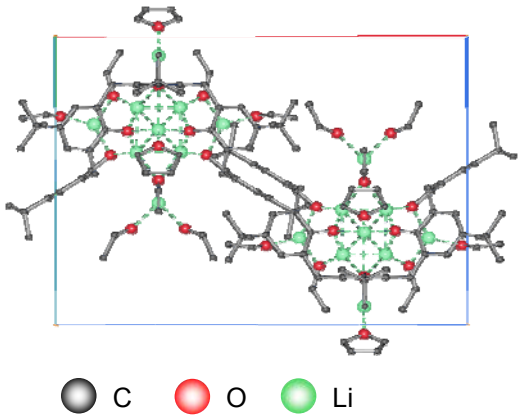


Figure II.24.a: view onto *bc*-plane of unit cell of **9** (H atoms omitted for clarity)

The 4-tert-butylcalix[8]arene molecule stabilizes an aggregate of fifteen lithium ions. The lithium cluster can be divided in different parts. A cube of eight lithium ions trapping an O^{2-} constitutes the heart of the structure. Five faces are capped by lithium ion which are linked to the cube by two edge-bridging oxygen atoms. Eight of

these bridging oxygen atoms come from the 4-tert-butylcalix[8]arene and two of the ethoxide anions). Finally, each pair of oxygen atoms on the same face is coordinated by terminal lithium ions. On the sixth face of the cube, the cluster is open and instead of one lithium ion, two are present. These two lithium ions are coordinated by two THF ligands (Figure II. 25).

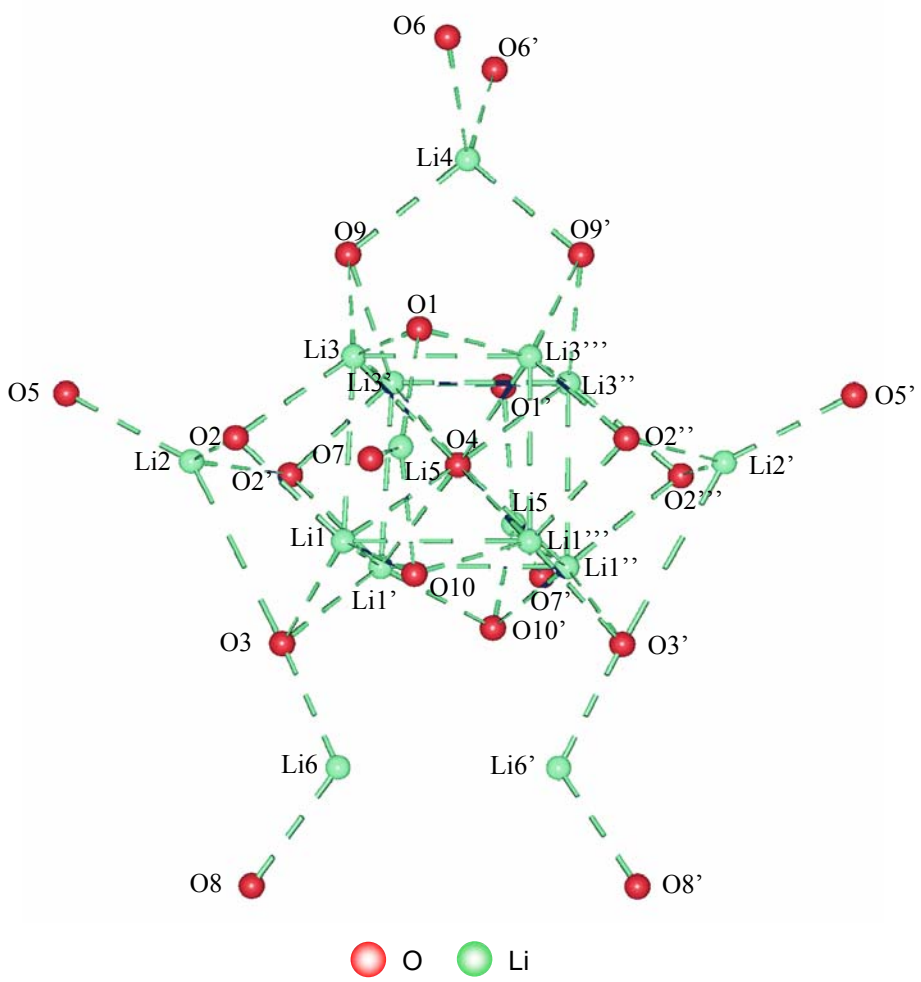


Figure II.25: Lithium-cluster of **9**

The heart of this cluster is rather unusual [202-205]. The O^{2-} anion is surrounded by a cube of eight Li^{+} cations. The distances O4–Li1 and O4–Li3 with 2.085(1) and 2.12(1) Å respectively are longer than in the reported structures with similar lithium ion cubes [202-205]. The distances Li1–Li3 are also by 0.1 Å longer with 2.44(1) Å in comparison with 2.312(6) and 2.373(5) Å [204]. This could be the result of the exo oxygen atoms which complete the lithium ion coordination of Li1, Li3 and their

symmetry equivalents. Indeed, each Li1 and Li3 is coordinated by four oxygen atoms which interact with Li^+ ion. Each exo oxygen atom tends to create some stronger bonds than others. It results in an increase of the average length O4 (inner cube)–Li.

The conformation of the 4-tert-butylcalix[8]arene is interesting. All the tert-butyl groups are pointing to the outside of the structure like a hedgehog. All the hydroxyl groups define a hydrophilic pocket inside the calix[8]arene (figure II.26). In literature, 4-tert-butylcalix[8]arene seems to organize generally itself in a pleated loop conformation in the solid state because of intramolecular hydrogen bonds. It is rather uncommon to obtain a saddle conformation in the solid state [206]. The angles between the planes defined by phenyl groups (1) are rather the same between adjacent phenyl rings i.e. 60.7(1) ° and 70.9(2) ° as in the pleated loop conformation. An inversion in the organization of the Ph-CH_2-Ph orientation from in-out sequence to in-in-out-out sequence can explain this conformation.

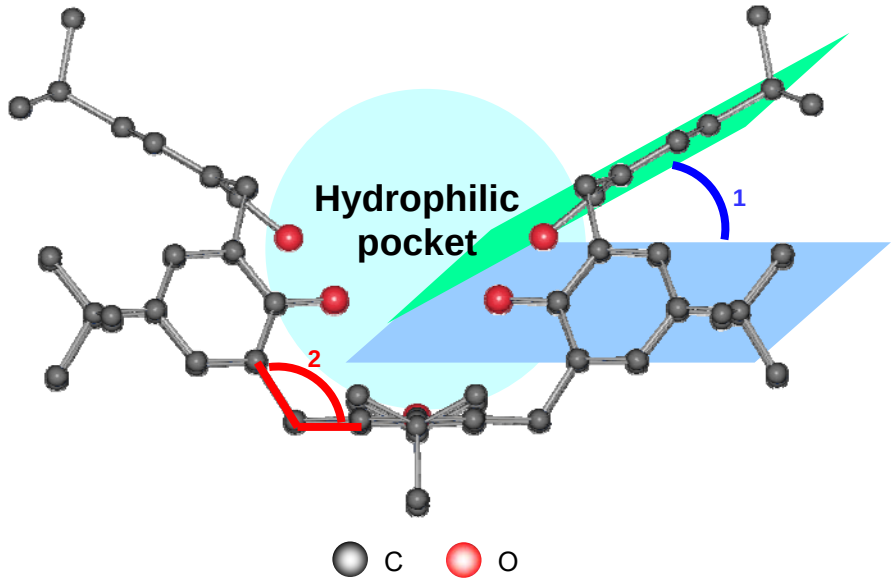


Figure II.26: 4-tert-butylcalix[8]arene ligand in a saddle conformation.

If the angles between the planes defined by phenyl groups are not really different from the angle of the pleated loop conformation, the Ph-CH_2-Ph angles (2), which are taking in account the CH_2 bridges, are opened up with values from 115.3(5)° to 120.3(5)°. This is larger than in the pleated loop conformation (109° to 116°) or in

chair conformation (108° to 118°). Here the system is relatively constrained. In the pleated loop conformation every phenyl group can be included in a mean plane and the sp^3-hybridization of the CH_2 group gives, as expected, a regular tetrahedron shape. In the chair conformation, 4-tert-butylcalix[8]arene is in 1,5-alternate conformation; two phenyl groups constrain the structure which involves a kind of deformation of the tetrahedral shape of the CH_2 groups. In a saddle conformation, the system is mainly directed by phenyl groups and they induce a bigger deformation of the tetrahedral shape of the CH_2 groups.

The following structures containing 4-tert-butylcalix[8]arene are described in the order of compounds with the smallest alkali to the largest alkali metal ions.

II.2.3. $[Li_2$(4-tert-butylcalix[8]arene-2H)$(THF)_7(H_2O)_{12}]$ 10

Reaction of 4-tert-butylcalix[8]arene with lithium carbonate affords single crystals of $[Li_2$(4-tert-butylcalix[8]arene-2H)$(THF)_7(H_2O)_{12}]$ **10** in a quantitative yield.

10 crystallizes in the monoclinic space group $P2_1/c$ (N° 14) with four molecules per unit cell. Each asymmetric unit is constituted by one 4-tert-butylcalix[8]arene which traps two Li^+ ions, the coordination of lithium ions is completed by three water molecules, there are also nine free water molecules and seven THF molecules. Among these latter, two are involved in the coordination of water molecules (Figure II.27).

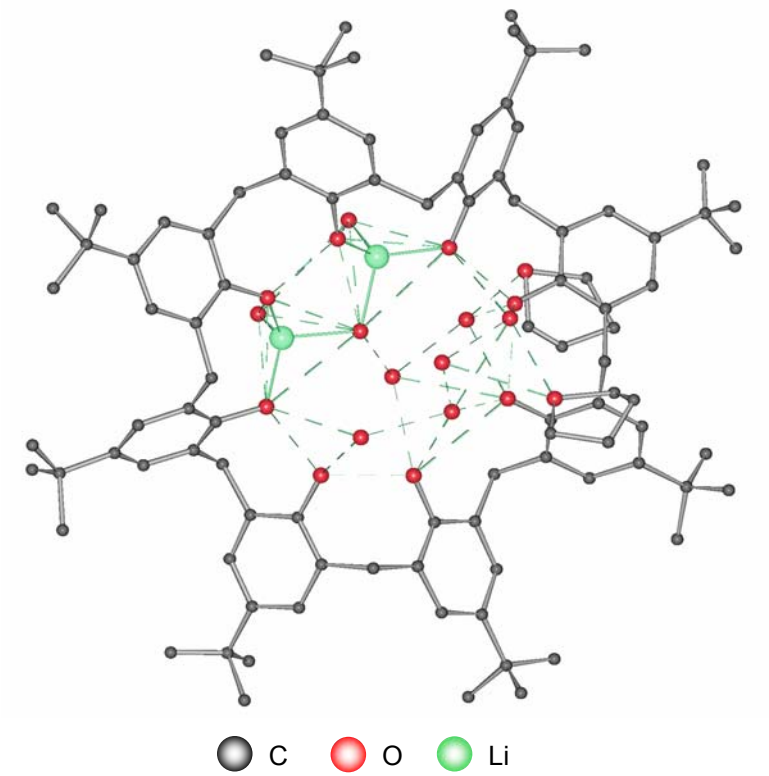


Figure II.27: Asymmetric unit of **10** ((H-atoms and non-coordinating solvent omitted for clarity).

The 4-tert-butylcalix[8]arene adopts a pleated loop conformation which is the most common one in solid state structures [86, 207, 209]. This conformation is stabilized via hydrogen bonds between protonated oxygen atoms of the 4-tert-butylcalix[8]arene. The Li^+ ions are each coordinated by two oxygen atoms of 4-tert-butylcalix[8]arene (O2, O3 for Li1 and O4, O5 for Li2) with an average distance of 1.96(2) Å which is in agreement with the value found in the literature for Li-O bond [208]. It is not possible to discriminate which oxygen atoms of 4-tert-butylcalix[8]arene are more coordinating Li-ions in both cases. Li-ions are separated from each other by a distance of 3.31(1) Å. This coordination is completed by a bridging water molecule (O19) between the lithium cations, and two water molecules (O18 for Li1 and O16 for Li2) which give rise to a heteroatomic ladder fragment between two adjacent 4-tert-butylcalix[8]arene ligands via H-bonds between O16, O18 and their symmetry equivalents (Figure II.28, scheme II.8). The hydrogen bond distances O16–O16' and O18–O18' are 2.833(2) Å. Furthermore O16 and O16' are

linked via hydrogen bonds to THF molecules with an average distance of 2.715(2) Å. This arrangement is defined as a dimer of 4-tert-butylcalix[8]arene around Li-ions.

The dimers are separated from each other by a layer of distorted THF and water molecules which do not interact with them. This lack of organization was due to weakest electrostatic forces that cannot create any type of interaction or self-assembly between solvent molecules as observed inside the dimer.

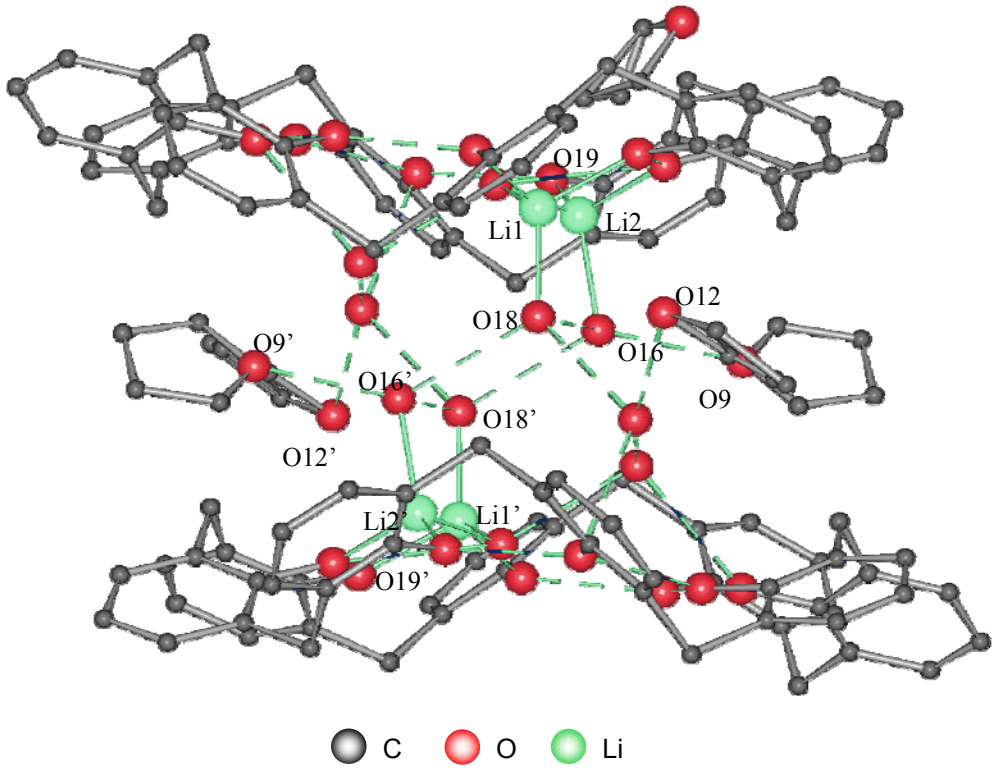
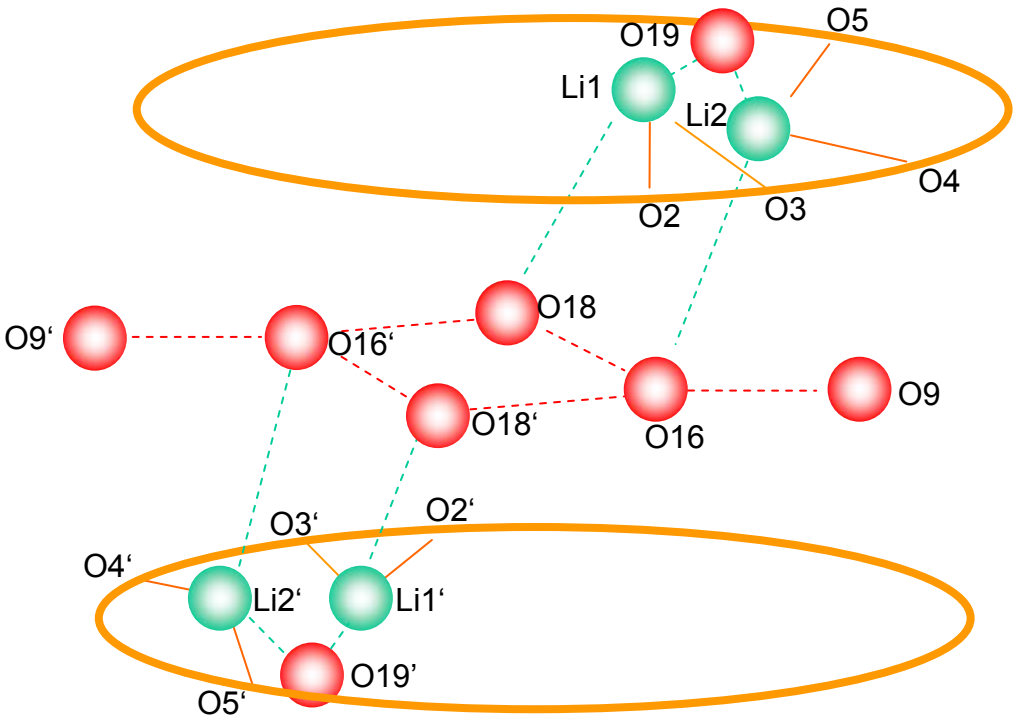


Figure II.28: Dimer of 4-tert-butylcalix[8]arene around lithium-oxygen ladder (H-atoms, non-coordinating solvent and ^{t}butyl groups omitted for clarity).



Scheme II.8: Main coordinating oxygen atoms of lithium ions.

II.2.4. $[Na_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_4(H_2O)_5]$ 11

In a Schlenk flask under an inert atmosphere, excess Na metal and 4-tert-butylcalix[8]arene in THF gave a pale yellow solution. After concentration, single crystals of $[Na_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_4(H_2O)_5]$ **11** were obtained in quantitative yield (water molecules came from moisture present in the THF).

11 crystallizes in the monoclinic space group $P2_1/c$ (N° 14) with two molecules per unit cell. Each asymmetric unit consists of a 4-tert-butylcalix[8]arene ligand which coordinates two Na^+ ions. The coordination sphere of each cation is completed by three THF and two water molecules (Figure II.29.a). In this case also, there are some free THF and water molecules included in the structure.

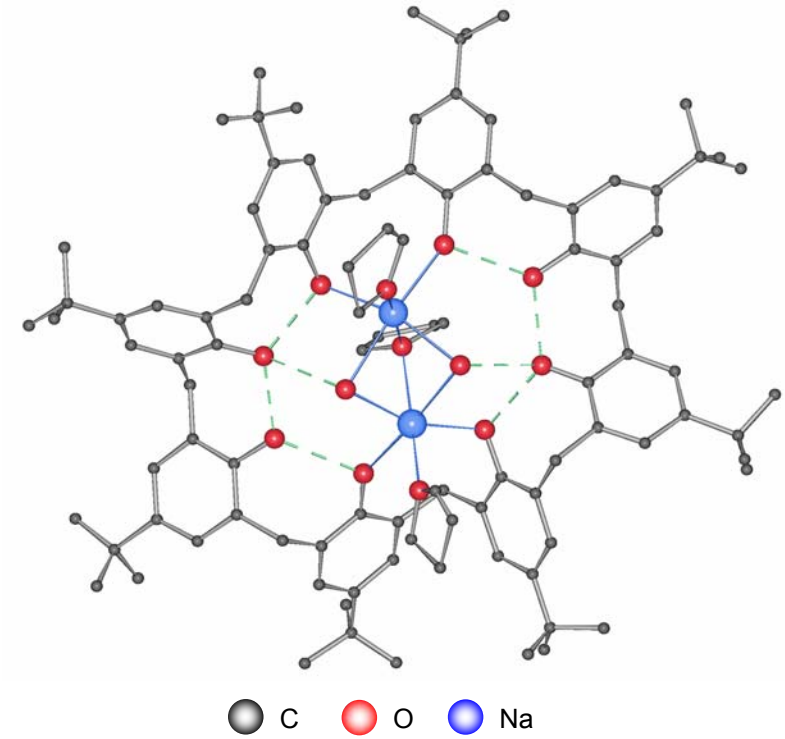


Figure II.29a: Asymmetric unit of **11** (H-atoms and non-coordinating solvent omitted for clarity).

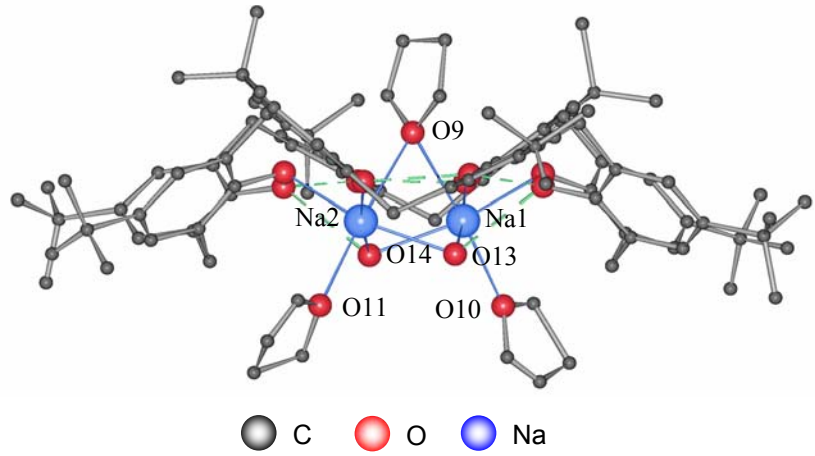


Figure II.29b: Asymmetric unit of **11** in "UFO" position (H-atoms and non-coordinating solvent omitted for clarity).

11 contains 4-tert-butylcalix[8]arene in a pleated loop conformation (figure II.29.b) [86, 207, 209]. The Na^+ ions are coordinated by the 4-tert-butylcalix[8]arene

with the shortest Na–O bonds of Na1–O5 of 2.332(5) Å and Na2–O of 2.326(6) Å. The THF ligands with Na1–O10 of 2.383(8) Å and Na2-O11 of 3.395(9) Å coordinate as strongly as the water molecules Na1–O13 of 2.489(5) Å, Na1–O14 of 2.383(8) Å Na2–O13 of 3.360(5) Å and Na2–O14 of 2.484(5) Å. The last THF molecule is shared by the two sodium ions which explains the longer distances observed for Na1–O9 of 2.601(6) and Na2–O9 of 2.669(7) Å.

If the eight oxygen atoms of the 4-tert-butylcalix[8]arene are taken as a mean plane, the sodium ions are 0.935(4) and 0.973(4) Å for Na1 and Na2, respectively, out of this plane. The three coordinating THF ligands point, two above and the bridging one below, this mean plane. The bridging H_2O molecules are H-bonded to O2 and O6 of the 4-tert-butylcalix[8]arene with O–O distances of 2.792(3) Å.

Under the given reaction conditions, it was surprising not to observe complete deprotonation of the 4-tert-butylcalix[8]arene. As the sodium metal stopped reacting with the 4-tert-butylcalix[8]arene, passivation of the surface of Na metal by water molecules may occur, which stops in the end the deprotonation of the 4-tert-butylcalix[8]arene.

II.2.5. [K(4-tert-butylcalix[8]arene-H)(THF)$_4$(H_2O)$_7$] 12

Reaction of 4-tert-butylcalix[8]arene with potassium carbonate affords single crystals of [K(4-tert-butylcalix[8]arene-H)(THF)$_4$(H_2O)$_7$] **12** are isolated in quantitative yield.

12 crystallises in the monoclinic space group C2/c (No. 15) (Figure II.30). The asymmetric unit consists of one singly deprotonated 4-tert-butylcalix[8]arene ligand, one potassium cation, four THF molecules, and seven water molecules. In the asymmetric unit, the metal ion is bound to one side of the 4-tert-butylcalix[8]arene ligand, where it is coordinated by O7 and O8 of the macrocycle.

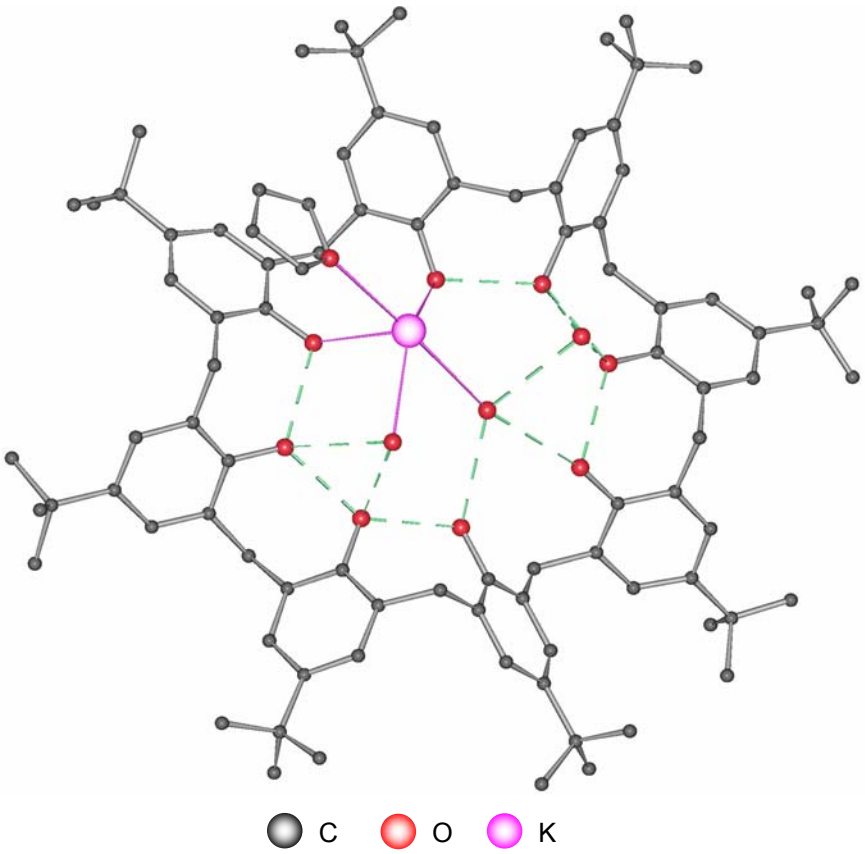


Figure II.30: Asymmetric unit of **12** (H-atoms and non-coordinating solvent omitted for clarity).

The distance O8–K1 is, at 2.717(6) Å, shorter than O7–K1 (2.894(6) Å), and O8 can therefore be attributed to the deprotonated oxygen atom of the macrocycle. No other clue such as the C–O distances differences can be found. The coordination sphere of the potassium cation is completed by three oxygen atoms of water molecules, O9, O9', and O10, as well as by one oxygen atom, O11, of a THF molecule to yield a distorted octahedral environment. The distance K1–O11 (THF) is 2.835(8) Å, which is in the normal range for terminally bonded neutral O-donor ligands [211]. The distances K1–O9 and K1–O9' are 2.844(6) and 2.749(7) Å, respectively. O9 and O9' form the bridge between K1 and its symmetry equivalent K1', related by an inversion centre in the middle of the rhombus so formed. The longest K–O bond is found for K1–O10, at 2.987(7) Å, O10 being positioned over the centre of the cavity in the 4-tert-butylcalix[8]arene, on the same side of the latter as

the potassium ion. It forms a hydrogen bond contact of 2.87(1) Å through the cavity of the calixarene, to the oxygen atom O15 of a water molecule on the opposite side of the organic ligand. The latter is involved in further hydrogen bonding to the water molecules of O16, O17, and O16' (2.88(1), 2.75(1), and 2.71(1) Å, respectively). The four water molecules O15, O16, O17, and O18 form, together with their symmetry equivalents, a distorted-cubane structure. A 2-fold axis is found passing through the middle of the distances between O18–O18' and O16–O16'. Together with O10 and its symmetry equivalent, a $(H_2O)_{10}$ cluster with C_2 symmetry is obtained. Even though the hydrogen atoms of the water molecules could not be located in the structure, the C_2 symmetry of this cluster indicates that 12 out of 16 H atoms in the cubane part of the cluster must connect their eight oxygen atoms to each other, whereas the other four point outward to the coordinated THF ligands. Furthermore, at least one H atom of O10 has to point toward O15. Such a structure for $(H_2O)_{10}$ has not been described before in the literature, but a similar one has been predicted as one possible, but not the most stable, structure in ab initio studies [212-216].

The eight oxygen atoms of the 4-tert-butylcalix[8]arene are all linked to each other via hydrogen bonding, the shortest contact being between O1 and O2 (2.45(1) Å). The longest bonds are (i) 3.44(1) Å for O7 and O8, as O7 is without its hydrogen atom and is coordinated to K1, and (ii) 3.29(1) Å for O3 and O4, as O3 is involved in weak H bonding to oxygen atoms O10 and O17 (ca. 2.9 Å). The O atoms (O1–O8) of the ligand are arranged almost perfectly in a plane, the maximum deviation from the mean plane being ca. 0.19 Å for O2 (Figure II.31).

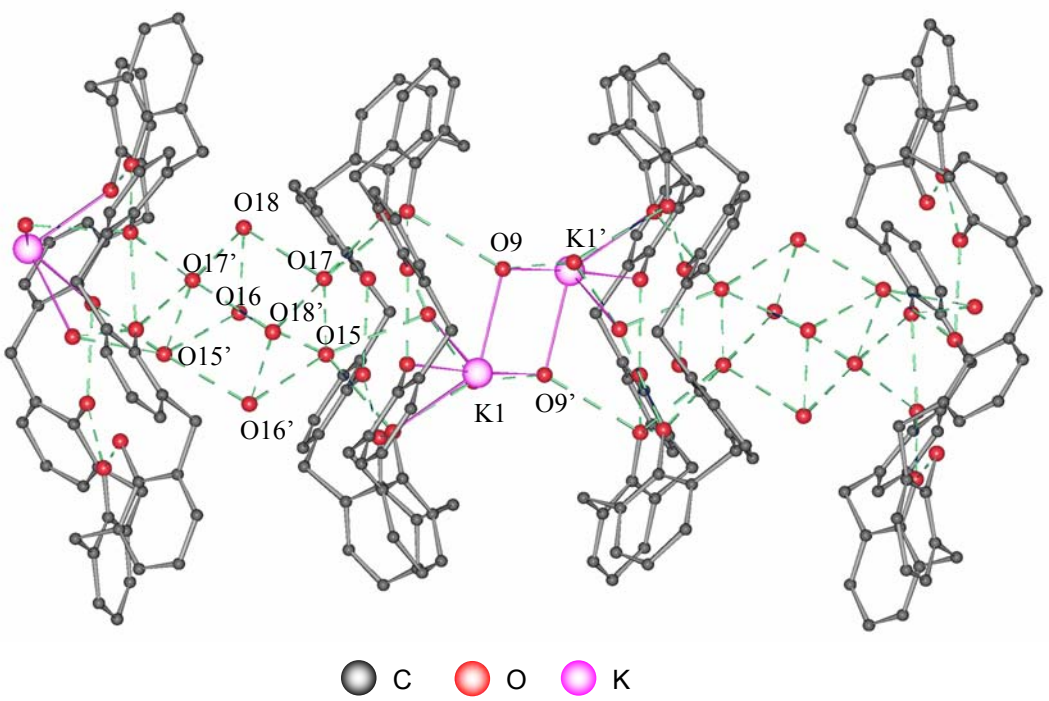
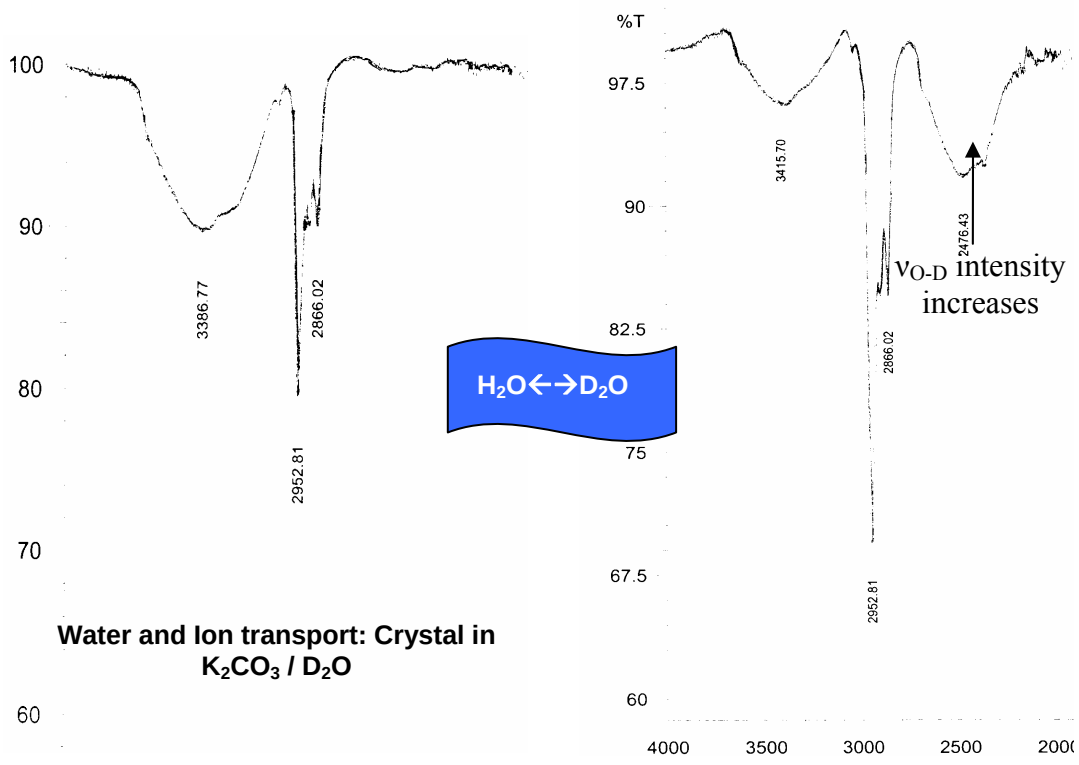


Figure II.31: One channel present in **12** with 4-tert-butylcalix[8]arene (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).

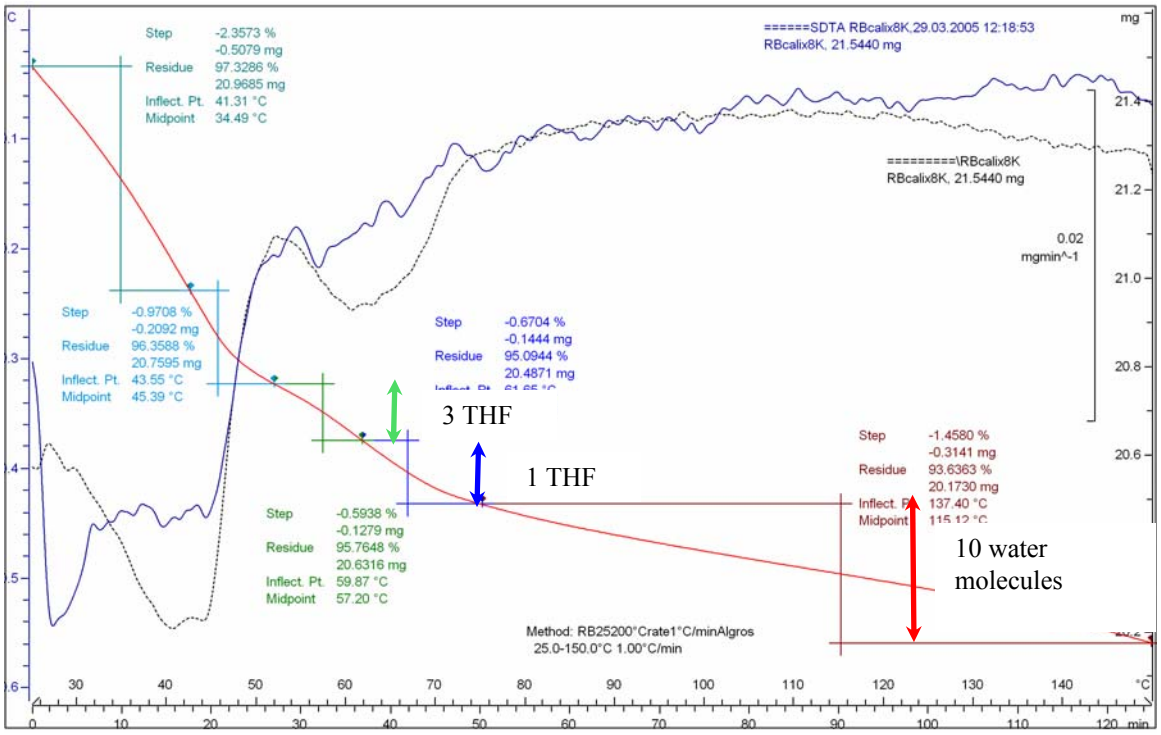
To the same side as O2 lies the potassium ion, ca. 2 Å above the mean plane formed by the oxygen atoms. The two 4-tert-butylcalix[8]arene ligands coordinating to the potassium dimer are ca. 0.4 Å closer to each other than the two macrocycles around the water cluster, which can be explained by the positive charge of the cations and the resulting electrostatic forces. The 4-tert-butylcalix[8]arene ligand of **12** adopts a "pleated loop" conformation, with the CH_2 groups pointing alternatingly above and below the mean plane of the eight oxygen atoms O1–O8. Thus, the mean planes formed by the phenyl rings attached to O1–O8 form dihedral angles between ca. 61° and 77°. Consequently, the *tert*-butyl groups also point in alternating directions, shielding both the potassium dimer and the water cluster from inclusion solvent. Both are additionally protected by THF ligands, the *tert*-butyl groups being at the outer rim of the channel, however. Slight interdigitation between the *tert*-butyl groups of adjacent calix[8]arene ligands in the channel allows for this perfect shielding (Figure II.31).

All in all, a one-dimensional channel is obtained. The 4-tert-butylcalix[8]arene can be considered as rings on a hydrogen bonding chain between the water cubane cluster and the potassium dimer unit. Furthermore, there are three more THF ligands and one additional water molecule in the asymmetric unit. The water molecule, O21, bridges the two water ligands O9 and O10. Two of the THF molecules and their symmetry equivalents are H-bonded to O16 and O18 as well as their symmetry equivalents and thus coordinate around the water cluster; the third one is linked to the bridging water molecule O21, and all act as terminal ligands on the water molecules. Thus, the THF ligands and the 4-tert-butylcalix[8]arene ligands together form a channel system with the nonpolar parts of the molecules pointing outward and the polar oxygen donors pointing toward the channel. The inside of the channel is filled with water molecules and potassium ions. The mobility of water molecules in the compound can be shown by immersion of a crystal of **12** into D_2O for 5 min. The ^1H NMR spectrum of **12** shows a dramatic decrease of the proton signals of the OH groups of 4-tert-butylcalix[8]arene as well as of the water molecules. The IR spectrum also reflects this exchange by a decrease of the OH stretching band of linked water molecules (Spectrum II.1) and an increase of the OD stretching band.



Spectrum II.1: Decrease of OH stretching band in IR when D_2O exchanged with H_2O molecules.

A TGA of **12** shows the loss of the four THF molecules per unit in two steps: three are lost in a broad step between 40 and 80 °C, whereas the last one is lost between 80 and 100 °C. The latter is suggested to be the one linked directly to the potassium cation, as this bond is expected to be stronger than the THF-water interactions. On further heating, water is lost quantitatively, starting at 110 °C up to 220 °C. Decomposition is finally observed at 280 °C (curve II.4).



Curve II.4: Loss of THF and water molecules from **12**.

This structure is therefore the first decameric water cluster stabilized by another solvent, THF, and it is the first one to possess a 2-fold axis as the only symmetry element. Both the water cluster and the potassium dimer form the knots on a string of pearls, where the pearls are the 4-tert-butylcalix[8]arene ligands and the string is formed by hydrogen bonds between water molecules across the center of the macrocycle. This compound is also a rare example of a solid-state structure containing a 4-tert-butylcalix[8]arene ligand.

II.2.6. [K_4(4-tert-butylcalix[8]arene-4H)$(THF)_6(H_2O)_{12}$] 13

Another channel structure with 4-tert-butylcalix[8]arene is obtained with potassium hydroxide in the aqueous phase. At this higher pH, four protons per 4-tert-butylcalix[8]arene are replaced by potassium to give single crystals of [K_4(4-tert-butylcalix[8]arene-4H)$(THF)_6(H_2O)_{12}$] **13** in quantitative yield.

13 crystallizes in the triclinic space group *P*-1 (N° 2) with one molecule per unit cell (Figure II.34). The molecule in **13** is formed by a 4-tert-butylcalix[8]arene in a chair-conformation which coordinates two potassium ions via the bonds O1–K1 O5–K2 at 2.83(1) and 2.85(1) Å, respectively. Each potassium ion (K1, K2) is situated on a different face of the 4-tert-butylcalix[8]arene and is hexa-coordinated by five water molecules and the cited O atoms of the 4-tert-butylcalix[8]arene. Two water molecules bridge K1 to K2, with an average K–O length of 2.819(7) Å, which results in a square. Other water molecules link K1 to K4 (O18, O19, O22) and K2' to K3 (O17, O20, O21) respectively to form a trigonal bipyramid. These outer potassium ions are also linked to the 4-tert-butylcalix[8]arene. K3 is bridged to the 4-tert-butylcalix[8]arene with K3–O4 (2.77(1) Å) and K4 with K4–O8 (2.82(1) Å). To the six water molecules, four water molecules have to be added to complete the coordination of the potassium ions K3 and K4. The water molecules (O23, O24, O25, O26) with an average K–O bond of 2.956(7) Å do not create water bridges but are in terminal positions and assure, here, the completion of the coordination sphere of both potassium ions. Finally, the coordinating THF molecules interact mainly with the oxygen atoms of water molecules. These interactions are characterized by an average distance of 2.73(1) Å. Only one THF molecule has a form of dipole-dipole interaction with another THF molecule at a distance of 3.474 Å.

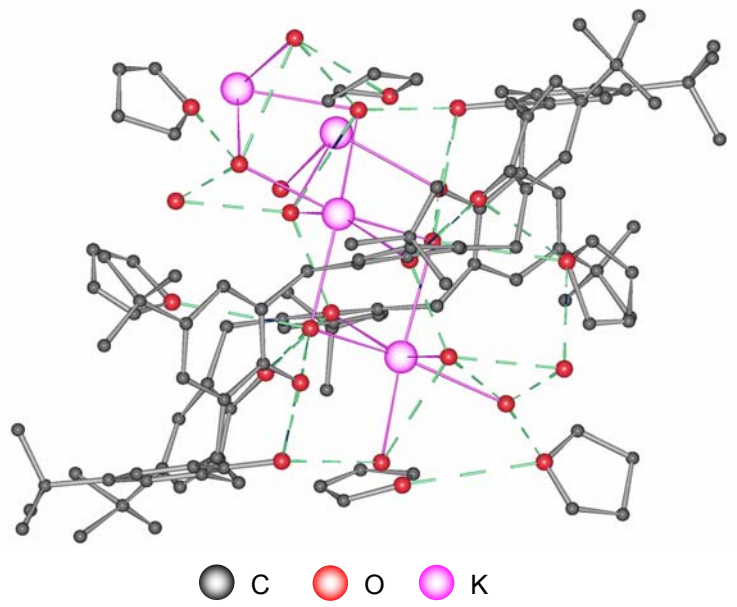


Figure II.34: molecule of **13** (H-atoms omitted for clarity).

In terms of the 4-tert-butylcalix[8]arene conformation, similar structures have already been reported [216, 217]. The originality of this structure, in comparison to the cited ones, is that instead of being a monomer, the K–O bonds create a chain inside the hydrophilic cavity of 4-tert-butylcalix[8]arene. The chain of water molecules and potassium cations is composed by two motifs: a square formed by K1, K2, O15 and O16 and a distorted heterocubane of water molecules and K4 and K3 cations on the opposite apices of the cube (Figure II.33 and scheme II.9).

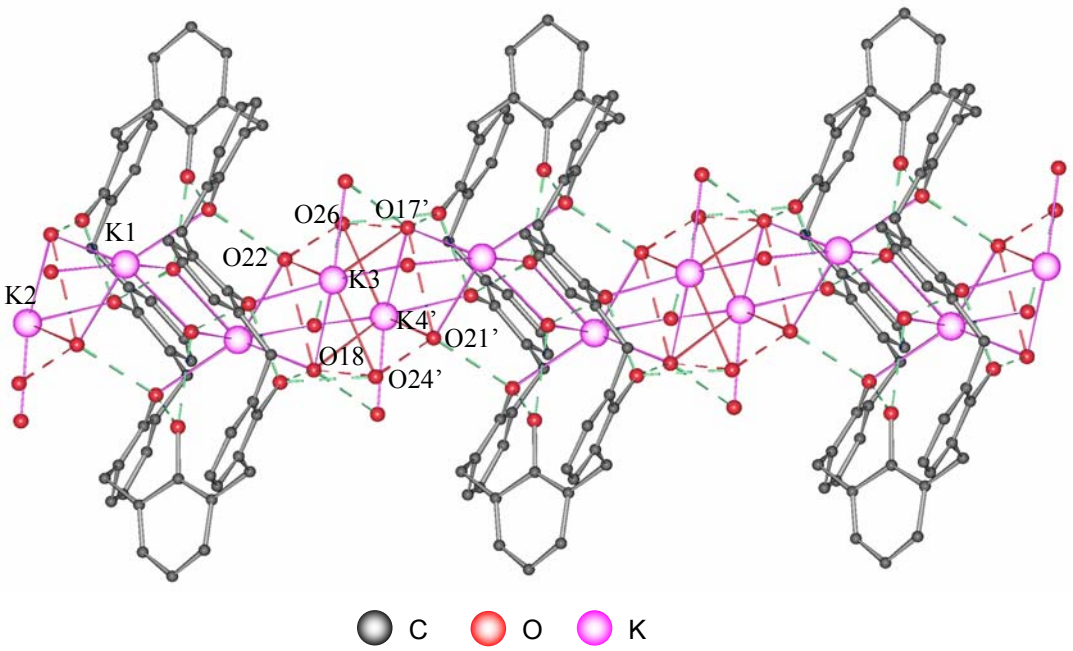
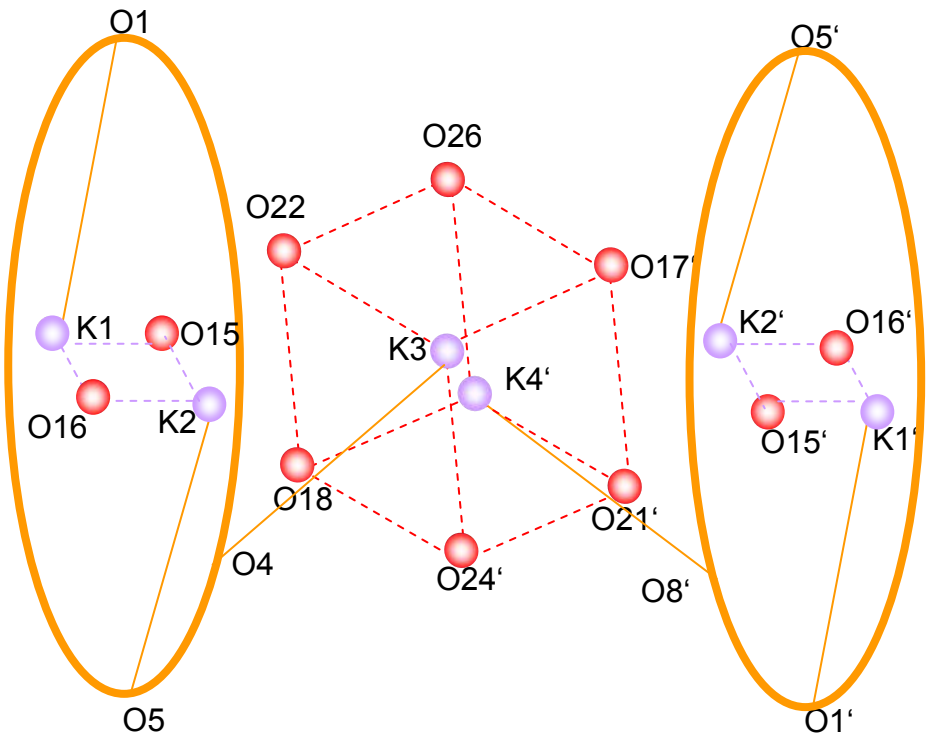


Figure II.33: Alternating squares and cubes inside and between 4-tert-butylcalix[8]arene units (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).



Scheme II.9: Main coordinating oxygen atoms of potassium ions.

II.2.7. $[Rb_2((4\text{-tert-butylcalix[8]arene-H}))_2(THF)_8(H_2O)_{14}]$ 14

The reaction of rubidium carbonate and 4-tert-butylcalix[8]arene at the water/THF interface yields single crystals of $[Rb_2(4\text{-tert-butylcalix[8]arene-H})(THF)_8(H_2O)_{14}]$ **14** in quantitative yield.

14 crystallises in the monoclinic space group *Cc* (No. 9) (Figure II.34). The asymmetric unit consists in two 4-tert-butylcalix[8]arenes, in two rubidium ions, two THF molecules linked to the Rb^+ ions while the six other THF ligands are coordinated to the water molecules, and thirteen water molecules. Finally, a non-coordinated water molecule (O38) is also present.

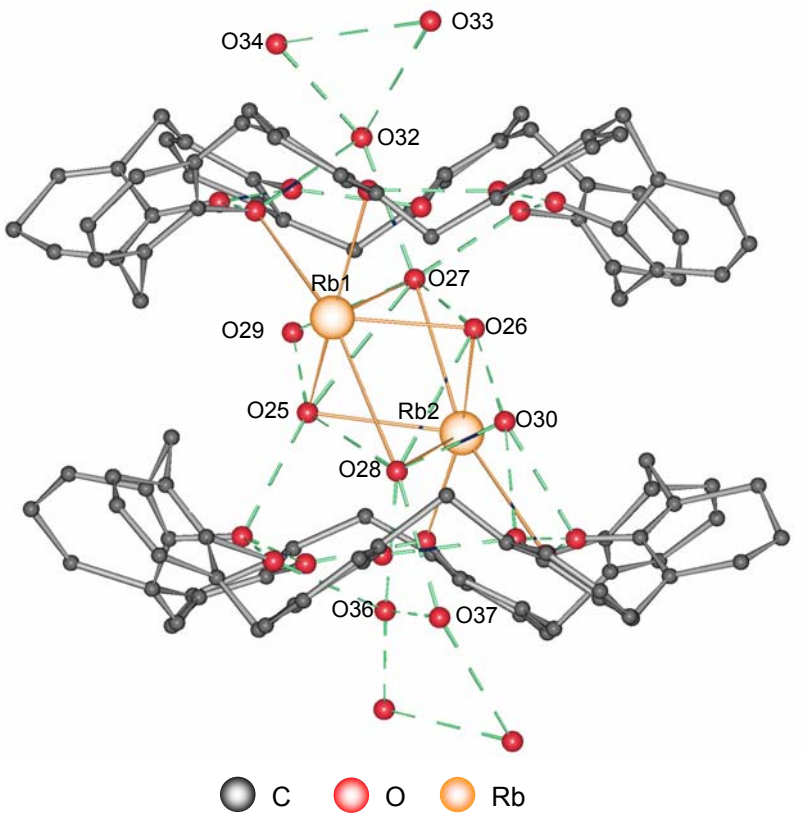


Figure II.34: Asymmetric unit of **14** (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).

The hepta-coordination of the Rb^{+} ions can be broken down into two interactions with 4-tert-butylcalix[8]arenes via Rb1–O3 (2.94(3) Å), Rb1–O4 (2.96(2) Å) and Rb2–O9 (2.69(4) Å), Rb2–O16 (3.15(2) Å) respectively, four interactions with water molecules O25, O26, O27 and O28 which bridge Rb atoms to form a distorted octahedron and finally two interactions with O17 and O18 from THF molecules which make with Rb1–O17 (2.94(3) Å) and Rb2–O18 2.89(2) Å contacts as strong as one of the water molecules Rb2–O26 (2.87(5) Å). To stabilize the hetero-octahedron of water molecules and rubidium ions, two water molecules O29 and O30 have to be mentioned. They have electrostatic interactions with O25 and O27 and with O26 and O28 respectively.

The distance Rb2–O9 is shorter by 0.2 Å compared to an interaction with Kryptofix 222 (azacrown ether) i.e. with non-charged oxygen atoms [218] but in accord with a charged oxygen atom such as in a *tert*-butoxide anion [219]. It can be

noticed the strong ability of 4-*tert*-butylcalix[8]arene to coordinate alkali metals ions thanks to its phenol group as a designed molecule for complexation [220].

The water/THF molecule organisation is rather easy to understand and involves a transition between two types of water cluster. The crystallographic data show the presence of two water aggregates stabilized by THF molecules (Figure II. 35). Once again, a distorted cube of water composed of eight water molecules (O31, O31', O35, O35', O36, O36', O37, O37') can be observed. It is stabilized by two THF molecules (O20 and O20' with a distance of 2.91(5)Å). The water molecule which was stabilizing the cube in **12** is, in **14**, more dependent on the alkali metal structure because it is now included in the hetero-octahedron. There is also a distorted octahedron made of six water molecules (O32, O32', O33, O33', O34, O34)' and coordinated by two THF molecules (O23, O23' with a distance of 2.87(5) Å).

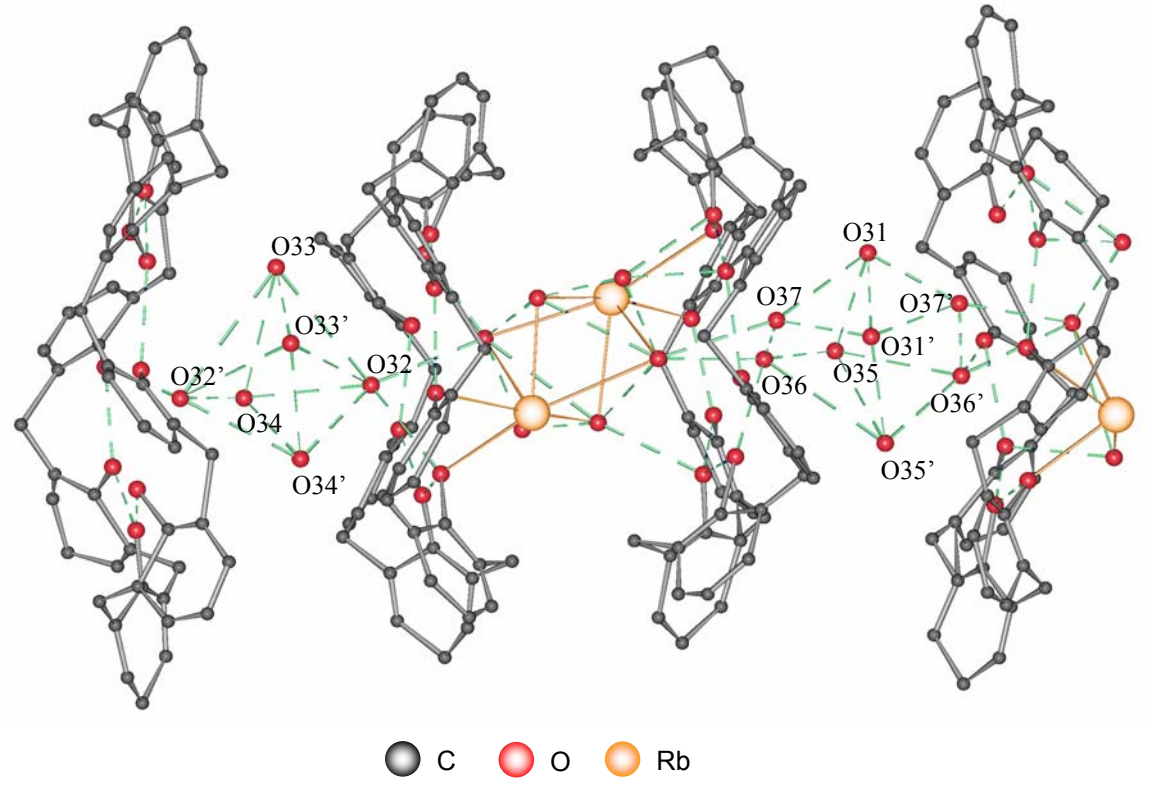
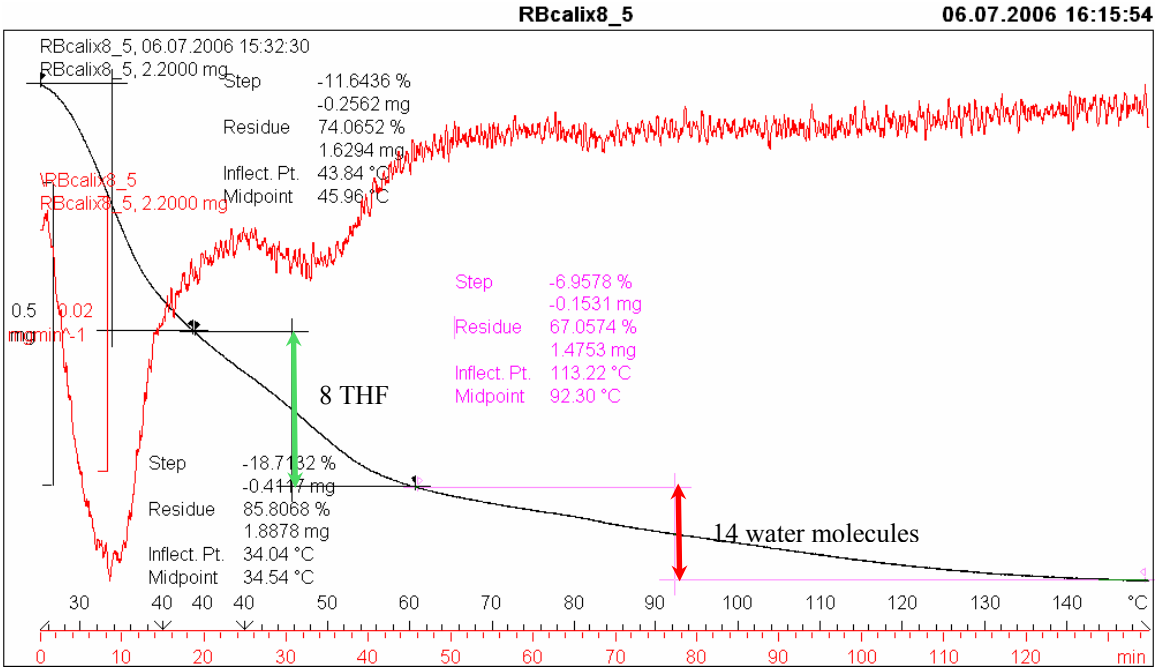


Figure II. 35: Alternating distorted octahedron and distorted cube between 4-tert-butylcalix[8]arene (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).

The mobility of water molecules in the compound can be shown by immersion of a crystal of **14** into D_2O for 5 min. The ^1H NMR spectrum of **14** shows a dramatic decrease of the proton signals of the OH groups of 4-tert-butylcalix[8]arene as well as of the water molecules as observed already in **12**. TGA was made on **14**. TGA of **14** shows the loss of free solvent during the plateau at 40 °C, whereas coordinated THF molecules are lost between 40 and 60 °C. On further heating, water is lost quantitatively between 60 °C to 140 °C (curve II.5). The loss of 7% at the end fits well with the percentage of water molecule in the structure.



Curve II.5: Loss of THF and water molecules in **14**.

II.2.8. [Cs(4-tert-butylcalix[8]arene-H)(THF)$_5$(H$_2$O)$_6$] 15

The reaction of caesium carbonate with 4-tert-butylcalix[8]arene at the water/THF interface yields single crystals of [Cs(4-tert-butylcalix[8]arene-H)(THF)$_5$(H$_2$O)$_6$] **15** in quantitative yield.

15 crystallises in the monoclinic space group *C*2/*c* (No. 15) (Figure II.37). The asymmetric unit consists of one singly deprotonated 4-tert-butylcalix[8]arene ligand, one caesium ion, five THF and six water molecules.

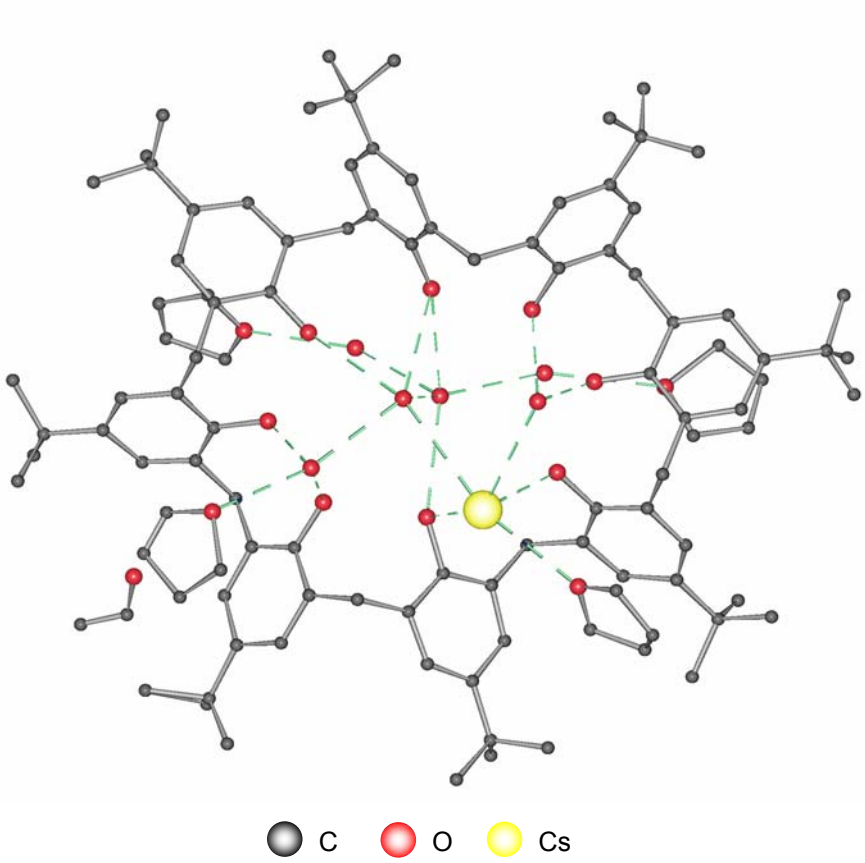


Figure II.36: Asymmetric unit of **15**, on the left THF molecule on the C_2-axis (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).

The hepta-coordination of the Cs1 ion can be described in term of two interactions with 4-tert-butylcalix[8]arene via Cs1–O3 (3.06(1) Å) and Cs1–O2 (3.29(1) Å) which is in good agreement with literature [222], four water molecules which bridge also to the symmetry equivalent Cs1' to form a distorted octahedron and finally an oxygen O9 from a THF molecule which makes with 3.06(1) Å a contact as strong as O3 (3.06(1) Å). For this side up, there is also a water molecules O17 which

interacts with O14 and O15 and a THF molecule O11 as well as a non-coordinating THF molecule O13.

On the other side of the 4-tert-butylcalix[8]arene, three water molecules (O18, O16, O20) and their symmetric equivalents (O18', O16', O20') organise themselves in another distorted octahedron. O16, O20 and O16', O20' are in onteraction with THF molecules O12, O10, and O12', O10'.

This is the third structure to feature channels of 4-tert-butylcalix[8]arenes which organizes themselves around alkali metal ions and water clusters, here a $(H_2O)_6$-cluster (Figure II. 38).

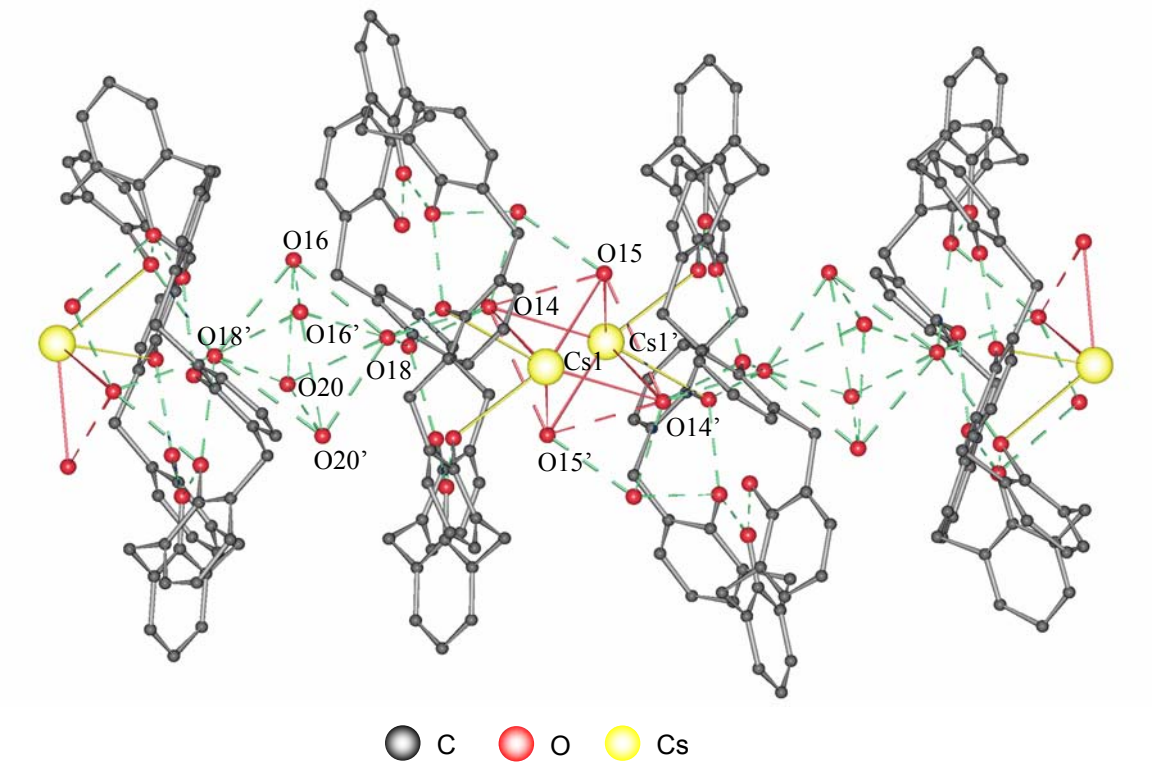
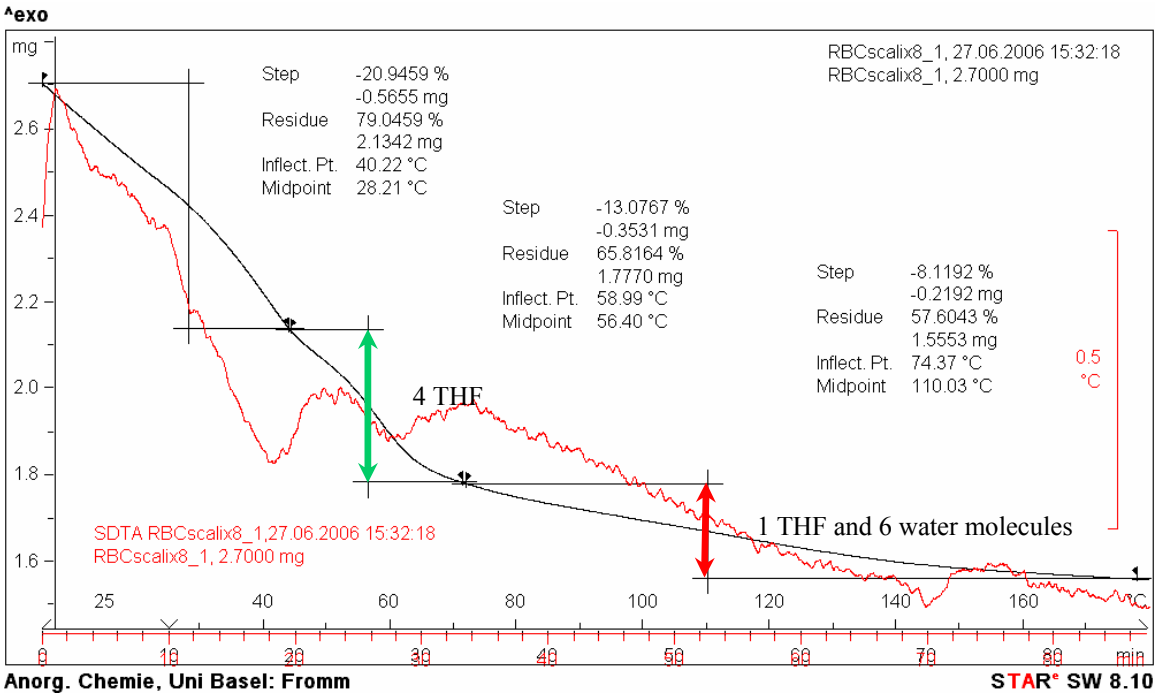


Figure II. 37: Alternating octahedra of water molecules and heterooctahedra in 4-tert-butylcalix[8]arene (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).

The mobility of water molecules in the compound can be shown by immersion of a crystal of **15** into D_2O for 5 min. The ^{1}H NMR spectrum shows a dramatic decrease of the proton signals of the OH groups of 4-tert-butylcalix[8]arene as well as of the

water molecules like in **12** and **14**. To determine the number of water molecules in this structure and to confirm the information of the crystal structure, TGA measurements were made on **15**. The TGA of **15** shows the loss of the five THF molecules per unit in two steps: four are lost in a broad step between 45 and 75 °C, whereas the last one is lost between 80 and 100 °C with the first evaporating water molecules. The latter THF molecule is suggested to be the one linked directly to the caesium cation, as this bond is expected to be stronger than the THF-water interactions. On further heating, water is lost quantitatively, starting at 110 °C up to 180 °C (curve II.6).



Curve II.6: Loss of THF and water molecules in **15**.

II.2.9. Formation of channels and influence of the alkali metal ion

In this part dedicated to 4-tert-butylcalix[n]arenes, the structures discussed show the possibility of trapping ionic species in a pocket (e. g. in **9)** or to generate a tubular organization (**8** and **10** to **15**). This last point is of interest because it seems that for

the first time, a series of channel structures with alkali metal ions has been made. In the literature, the 4-tert-butylcalix[8]arene ligand adopts a pleated loop or a chair conformation but none of them has this special characteristic of the formation of channels.

8 is the only example of crystallised 4-tert-butylcalix[6]arene reported here but its arrangement around a water channel is rather exceptional. The 4-tert-butylcalix[6]arene adopts a transannularly pinched conformation to form two circular hydrogen bonded arrays containing three OH groups respectively. This kind of channel was reported in membranes [65, 66, 222]. This structure could bring informations on transport mechanisms thanks to simulation of water and potassium translocation along this channel.

The organisation of 4-tert-butylcalix[6]arene in trimers is also reported in crystalline edifices [209, 223, 224], but none of the compounds possess any linkers to bridge or pack the trimeric units together. In the latter compound, the heart of the trimolecular complex is not strong enough to keep oxygen atoms of 4-tert-butylcalix[6]arene pointing inwards. The trimeric unit looks like a helix of windmills.

One detail is very important in this structure: the total absence of potassium ions inside the 4-tert-butylcalix[6]arene cavity. The cation-π interactions between potassium ions and aromatic rings are normally important for potassium ion complexation with such ligands. The crystallographic data reveal a THF molecule and two water molecules instead. Nevertheless calix[6]arene is normally able to complex K^+ cations when deprotonated [225]. The unexpected position of potassium ion is maybe the result of the low concentration of potassium ions and water molecules in the THF solvent when the organisation occurs. It means that the pH in the above solution is less basic (around 8) than the solution below (around 12). The 4-tert-butylcalix[6]arene are here soluble in THF solvent thanks to a hydrated structure more than by deprotonation. This idea also explains the presence of hydrogen carbonate in the crystal structure. Another aspect could be the weak basic character of K_2CO_3 not causing deprotonation of the OH groups of the 4-tert-butylcalix[6]arene ligand. By the way, K^+ cations are not able to cross the intramolecular hydrogen bond barrier of the 4-tert-butylcalix[6]arene like other bigger molecules can do by creation

of dipole-dipole interaction with hydrogen atoms of OH groups [226, 227]. K^+ cations are "obliged" to stay outside, in the position attributed. **8** cannot form channels as observed with 4-tert-butylcalix[8]arene in **10**-**15** because it cannot adopt a flattened ring conformation due to the number of monomers. A second reason to the absence of flat 4-tert-butylcalix[6]arene is that the deformation of CH_2 angles created to obtain flat 4-tert-butylcalix[6]arene is too high for the CH_2 groups [228].

9 is the archetype of the fully deprotonated 4-tert-butylcalix[8]arene and shows the great flexibility of the 4-tert-butylcalix[8]arene ligand by adopting the saddle conformation. Actually, cutting all the intramolecular hydrogen bonds is like freeing a puppet from its strings. To stabilize this fully deprotonated ligand in solid state, lithium ions are used because these cations are more restrictive than hydrogen atoms. In this example, the intramolecular cooperation described by Gutsche [207, 228] gives rise to the free rotation of phenol groups around the CH_2 bridges. To compensate this new freedom, 4-tert-butylcalix[8]arene has to find an equal force to fix its conformation: Ionic interactions as in **9** [206]. Another means is to substitute some hydrogen atoms in the structure by some alkyl chain to form some ether bridge like 1,5-3,7-calix[8]biscrown-3 or 1,5-bridged calix[8]arene [229, 230].

To end this part, the whole family of 4-tert-butylcalix[8]arene derivative crystal structures from **10** to **15** deals with the problem of the creation or not of dimers of 4-tert-butylcalix[8]arene around an alkali metal ion edifice and the linking of this unit thanks to water aggregates. As a result of these two conditions, the creation or not of a one dimensional channel occurs.

The first structure to be discussed is of course the monomer of **11** with two sodium ions per 4-tert-butylcalix[8]arene molecule. **11** is the most representative of precedent works with other metals ions [86]. To avoid dimerisation, chemical scissors are employed. On both sides, excess THF molecules which are stabilising the complex create a bulk which prevents intrusion of new water molecules in the sodium ion coordination sphere. Finally the low concentration of water molecules can not "break" this interaction. Nevertheless, 4-tert-butylcalix[8]arene molecules show a parallel organisation which evokes a dimeric unit but the distance between two

monomers is circa 10 Å (Figure II.38), with THF and uncoordinated solvents molecules in between.

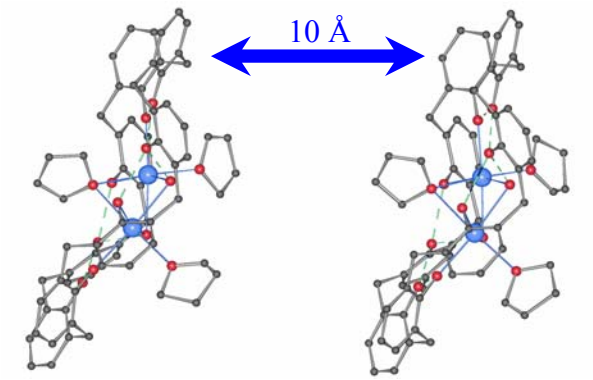


Figure II.38: Predimerisation of **11** (H-atoms, non-coordinating solvents, THF molecules and ^{t}butyl groups omitted for clarity).

If more water molecules are present in solution, the crystallisation of a dimer occurs like in **10**. This structure is another example of the high facility of calix[n]arenes to oligomerize [93, 98, 167, 224, 231, 232]. 4-tert-butylcalix[8]arene molecules need only a heteroladder of lithium ions and water molecules to self assemble. It is also the first example of a dimer of 4-tert-butylcalix[8]arene with such a small cation. The first difference in this structure is the shift of the Li^{+}-ion out of the plane defined by the eight oxygen atoms of the 4-tert-butylcalix[8]arene. The present water molecules interact strongly with the lithium cations and the 4-tert-butylcalix[8]arene restores as much as possible its intramolecular interactions between its oxygen atoms by intramolecular hydrogen bonds. To create an interaction between two dimers to form oligomers, a strong ionic force is certainly helpful. The embryonic structure of a heteroladder in **11** and the short bonds Li–O of around 2 Å are unsuitable for such realization because it does not promote a long distance order. In **10**, uncoordinated THF and water molecules build a "curtain" between each dimer.

Until now, dimeric species with sodium ions could not be isolated via this way of crystallisation. Considering structures **10** to **15**, the bridge between two dimers is formed by a hydrogen-bonded water cluster to which THF molecules are coordinated at the outside. In the case of sodium cation, this water edifice is maybe unstable in the

solid state. Such an absence may define a frontier between a dimeric and polymeric structure.

12 is finally the first structure with a stable polymeric structure. Water cubes seem to find a relatively good environment in which to be stabilised in the crystal structure by forming a sandwich with two 4-tert-butylcalix[8]arene ligands. THF molecules interact efficiently with the water molecules and the interactions induced by the cations creates suitable conditions for such water cluster. This seems to be confirmed when larger cations like in **14** or in **15** lead to a new water aggregate (cube and octahedron for **14** and octahedron for **15**).

One of the last points to explain is the role of a four cations coordinated by the same 4-tert-butylcalix[8]arene ligand like in **13**. The deprotonation of four OH^- groups involves a deformation of the 4-tert-butylcalix[8]arene which cannot be minimized by the intramolecular hydrogen bonds. The 4-tert-butylcalix[8]arene ligand is subjected to a deformation from a pleated loop (fully protonated) to a chair conformation (four times deprotonated). If Ph-CH_2–Ph angles are compared, **13** is the one with **14** which has the widest angles (table II.6).

Finally, other attempts were done with isopropyl and 2,2,4,4-tetramethylpentyl groups without success. It might be possible that tert-butyl groups play a role in crystallisation process of 4-tert-butylcalix[n]arene by weak interaction with host molecule. In **8**, a carbonate anion was isolated between two tert-butyl groups of 4-tert-butylcalix[6]arene.

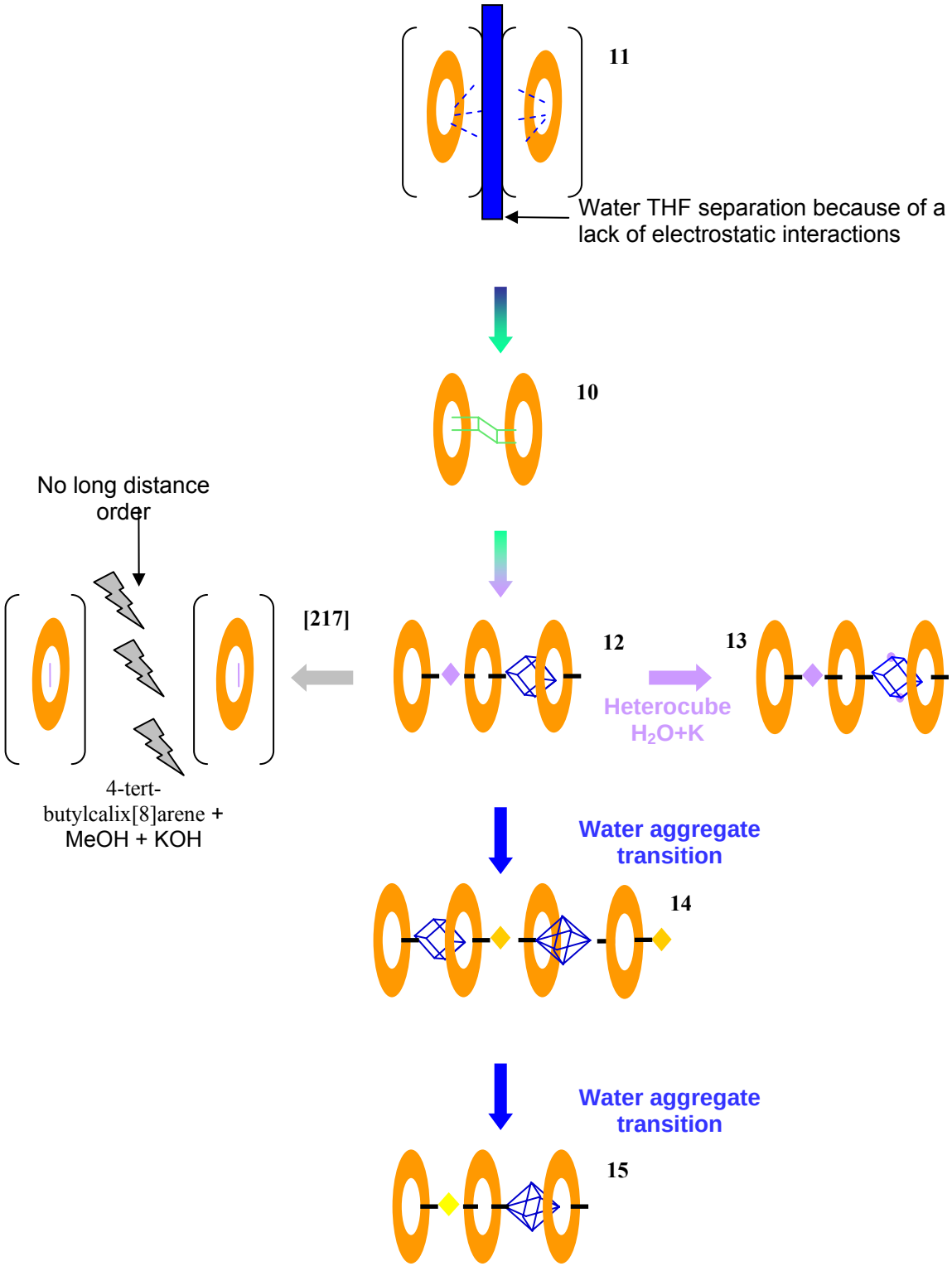
	Shortest Ph–CH_2–Ph angle (°)	Ph–CH_2–Ph angle (°)	Ph–CH_2–Ph angle (°)	Ph–CH_2–Ph angle (°)	Ph–CH_2–Ph angle (°)	Largest Ph–CH_2–Ph angle (°)	Ph–CH_2–Ph angle (°)	Ph–CH_2–Ph angle (°)
10	109.6(9)	111.2(9)	111(1)	114.6(9)	112(1)	116.5(9)	115.4(9)	112.4(7)
11	112.2(6)	114.8(7)	115.7(8)	115.4(8)	113.8(6)	117.0(8)	113.0(7)	115.5(7)
12	112.2(9)	112.9(9)	115.2(9)	113.9(8)	115.0(8)	117.0(8)	114.9(8)	113.7(8)
13	108(1)	115(1)	112(1)	115(1)	121(1)	118(1)	118(1)	116(1)
14(a)	107(3)	119(3)	111(3)	109(2)	126(2)	113(3)	122(4)	109(5)
14 (b)	96(3)	115(4)	127(3)	112(2)	112(4)	125(3)	105(3)	105(3)
15	110(1)	115(1)	114(1)	113(1)	114(1)	115(1)	115(1)	113(1)

Table II.6: Comparison of the Ph–CH_2–Ph angle of **10-15**

To summarize the progression from monomers to polymeric 4-tert-butylcalix[8]arene channels (scheme II.10),

- an ionic structure has to be created and this one must promote electrostatic interactions between ions, solvent molecules and 4-tert-butylcalix[8]arenes to create a dimer,
- the ionic interactions of the cation are important to create the necessary condition to stabilize water cluster.
- only water aggregates are so far able to propagate the one dimensional channel.

If more metal ions are present in the structure, 4-tert-butylcalix[8]arene adapts its shape to the new environment and the water cluster is replaced by a metal ions-water aggregate.

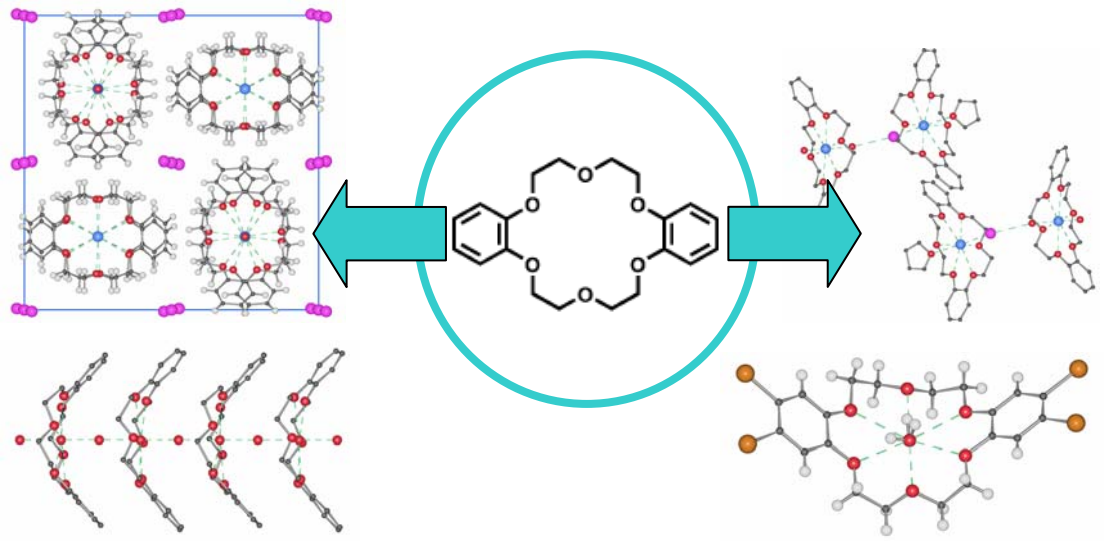


Scheme II. 10: Crystallisation as a function of the solvent and the base

III. Conclusion

This thesis gives an overview of seven crown ether compounds, their crystalline edifices, and their ability to act as ion and water channels. It reports also on 4-tert-butylcalix[n]arene compounds with n = 6 and 8, the special crystallization technique, their solid state structures forming from monomers to one-dimensional channels, as well as their properties in the solid state.

If the study of crown ether compounds is, by far, not exhausted, it brings nevertheless a lot of information on ionic transport in the solid state and on the pH-dependency in the crystallization process. For the first time is reported here the water and sodium ion transport in the solid state through single crystals of **1**. Recall that, $7.31*10^{-17}$ mol/channel/h of sodium ions are running through the crystal via osmotic process. It has been shown that the translocation was very probably due to a hopping process. The study of bromocrown ethers testifies to a pH dependency for the brominating mechanism and/or for crystallization (Scheme III.1).



Infinite 1D-Channel systems: **1** and **2**

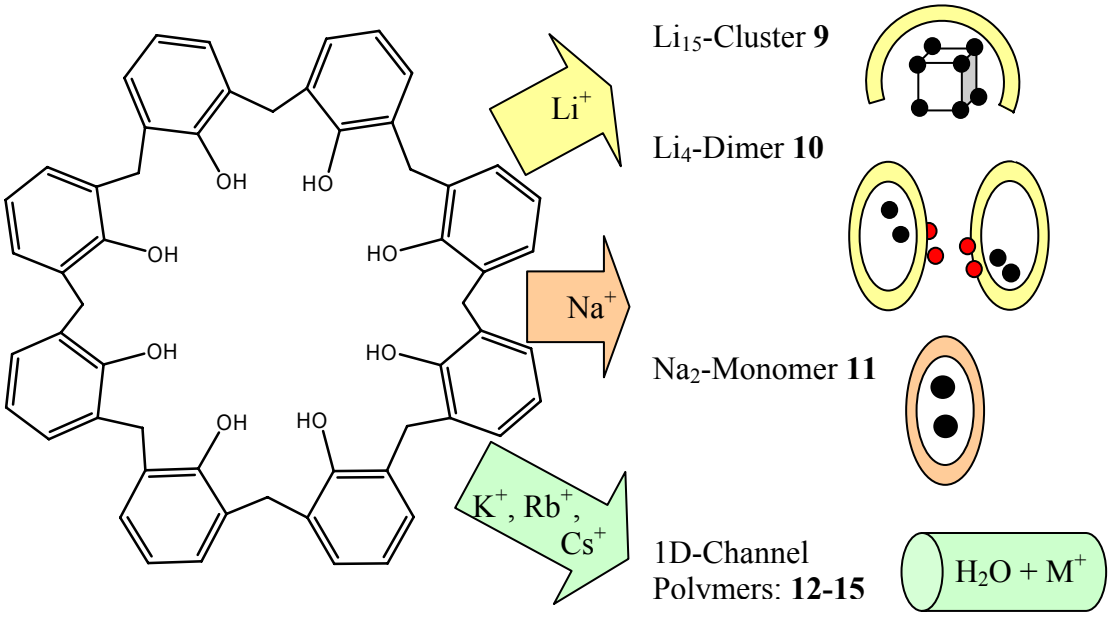
Dimers, Capsules, Offset stackings: **3-7**

Scheme III.1: Summary of crown ether structures.

The appearance of square holes in the largest crystals of **1** are not yet explained, and their further study could be interesting. An application of such a square tubular compound could be in microelectronic or optoelectronic compounds.

The pH to avoid bromination of DB18C6 still has to be found, and also a cation which will not fill the cavity and prevent stacking has to be found in order to make a Br_3-analogue compound to **1**. It will certainly be the solution to obtain the isostructural compound to **1** with tribromide anions.

4-tert-butylcalix[n]arenes are interesting for their inherent abilities to form channels. The thesis reports the evolution of water edifices from a distorted cube to a distorted octahedron as a function of alkali metal ions, as it seems from our results. Despite these nice structures, it was however not possible to keep the crystals at room temperature for more than some hours due to the loss of solvent molecules and subsequent destruction of the single crystals. However, it was seen for compound **12**, that H_2O can be exchanged by D_2O, and this exchange process has to be further investigated together with possible cation transport.



Scheme III.2: Summary of 4-tert-butylcalix[n]arene crystallization.

As seen in this study, the channels of **1** are not ion specific. To enhance the affinity of water, the oxygen crown of DB18C6 has to be modified by substitution of two O atoms by two NH groups. The hydrogen bonds developed inside the cavity could stop the sodium ions by repulsive forces.

To develop 4-tert-butylcalix[n]arene channels, an alternative to THF has to be found. This solvent must have a higher boiling point (heat of vaporization) and the same coordination ability. Furthermore, this new solvent has to keep the shielding property of THF mentioned above in the description of **12**.

Another way to free the channel structure of solvent-dependency is to substitute *tert*-butyl groups for other functions like carboxylic group which can create hydrogen bonds, or phenyl groups which can create π-stacking. By this way, the role of *tert*-butyl groups in the packing of our compounds could be clearly determined.

IV. Experimental part

IV.1. Reagents and solvents

The reagents were purchased from ACROS and directly used without further purification.

THF was purchased from VWR and directly used without further purification or drying if not mentioned.

For the synthesis of $[Li_{15}$(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4]$ **9** and $[Na_2$(4-tert-butylcalix[8]arene-2H)$(THF)_3(H_2O)_2]$ **11**, dried and freshly distilled THF (sodium/benzophenone) was used under N_2 atmosphere.

IV.2. Equipment

Elementary analyses were performed by W. Kirsch, from the Mikrolabor, University of Basel.

IR-spectra were collected on a Shimadzu FTIR-8400S instrument equipped with a Golden Gate ATR (attenuated total reflection) system at the University of Basel. Frequencies are given in cm^{-1}. Abbreviations used are: s (strong), m (medium) and w (weak).

^{1}H-NMR analysis were performed on a Bruker Advance Spectrometer 250 MHz, at 298 K. ^{13}C-NMR analysis were performed on a Bruker Advance Spectrometer 500 MHz, at 298 K. The chemical shifts are given in ppm relative to $CDCl_3$ as an internal standard or to DMSO for **7**. Abbreviations used are: s (singlet), d (doublet), t (triplet), m (multiplet).

IV.3. Experimental part

IV.3.1. 1∞[(H_2O)(DB18C6)$(\mu_2$-$H_2O)_{2/2}$]1∞[(H_3O)(DB18C6)$(\mu_2$-$H_2O)_{2/2}$](I_3) 1

Synthesis of **1**: A different procedure to that given in [1] is described here to obtain single crystals of **1**. 0.532 g (1.85 mmol) dibenzo-18-crown-6 were dissolved in 20 ml THF and 20 ml 0.05 M aqueous I_2-solution. The beaker was closed with Parafilm and the solution was allowed to stand at room temperature for several weeks. After evaporation of solvent, crystals of **1** in form of dark brown needles were isolated, and used for single crystal diffraction. Yield: *ca.* 90 % referring to iodide.

^{1}H-NMR: ($CDCl_3$, 250 MHz): δ (ppm) 6.94 (ArH), 6.89 (ArH), 4.98 (H_3O^+ .nH_2O), 4.16 (md, CH_2), 1.54 (H_2O)

IR cm^{-1}: 3649 (s), 2900(vs), 2724 (m), 2669 (m), 1592 (m), 1455 (s), 1376 (s), 1254 (m), 1217 (m), 1127 (m), 1069(sh), 951 (sh), 722 (m)

IV.3.2. 1∞[(DB18C6)Na(μ-OH)]1∞[DB18C6Na(μ-H_2O)] (I_3) 2

Synthesis of **2**: A single crystal of **1** was immersed into a 0.1 M NaOH-solution for one day. The crystal was used for diffraction measurements.

Yield 100%.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 6.94 (ArH), 6.89 (ArH), 4.16 (md, CH_2), 2.61 (s, OH^-), 1.64 (H_2O)

IR cm^{-1}: 3616(w), 2925 (w), 2871 (w), 1593 (m), 1504 (s), 1448 (s), 1335 (w), 1326 (m), 1247 (s), 1217(s), 1126 (s), 1060 (s), 1051(s), 941 (s), 910 (m), 854 (m), 781 (s), 738 (s), 599(w).

IV.3.3. [Na(DB18C6)I(THF)][Na(DB18C6)(H_2O)$_2$]I(THF)$_2$(CHI_3)] 3

Syntheses of **3**: 0.3478 g (3.66 mmol) of NaO*t*Bu were slowly added to a solution of 50 ml THF/H_2O (1:1). A two-phase system was formed. Acetone is added until disappearance of the interface. 10 ml of a diiode solution 0.1 M (1 mmol) were added. Finally, 0.3824 g (1.061 mmol) of dibenzo-18-crown-6 were dissolved in this

solution. After 12 h, the growth of needle-like crystals was observed. Yield: 1.510 g (0.938 mmol, 88 % with respect to DB18C6).

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 6.871 (d, 8H, ArH), 4.144 (d, 16H, CH_2), 4.784 (s, H, CHI_3), 4.737 (m, 2H, H_2O free), 3.744 (m, 4H, CH_2 THF), 1.718 (m, 4H, CH_2 THF), 1.250(s, 2H, H_2O).

IR cm^{-1}: 3064 (w), 2948 (w), 2874 (w), 1595 (m), 1508 (m), 1452 (m), 1418 (w), 1392 (w), 1368 (w), 1330 (m), 1291 (w), 1254 (s), 1228 (s), 1127 (s), 1117(s), 1079 (m), 1027 (m), 993 (s), 931 (s), 841 (w), 820 (m), 777 (m), 749 (s), 737 (s), 727(s).

IV.3.4. $[(H_5O_2)(Br4\text{-}DB18C6)_2][FeBr_4]$ 4

Synthesis of **4**: (a) 4, 4', 5, 5'-tetrabromodibenzo-18-crown-6 was prepared as follows: 1 g (2.77 mmol) of dibenzo-18-crown-6 is dissolved in 20 ml THF in a Teflon beaker. 0.215 g (3.85 mmol) of iron was added to this solution. An excess of 2 ml (38.919 mmol) of bromine was added. This mixture was stirred for 12 h. After evaporation of THF in air, the white solid was washed with water and filtered to yield 1.353 g (2.00 mmol, 72 % with respect to DB18C6).

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.704 (s, 5H, H_5O_2), 7.036 (s, 4H, ArH), 4.105 (s, 8H, CH_2), 3.967 (s, 8H, CH_2), 3.747 (s, 3H,H_3O^+), 1.928 (s, H_2O).

(b) 0.182 g (0.269 mmol) of 4, 4', 5, 5'-tetrabromodibenzo-18-crown-6 were dissolved in 20 ml THF in a Teflon beaker. 0.0215 g (0.385 mmol) of iron was added to this solution. Finally, 1 ml of bromine (19.459 mmol) was slowly added. After two weeks, we observed the growth of needle-like crystals. Yield: 0.160 g
(0.150 mmol, 56 % with respect to DB18C6)

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.021 (s, 4H, ArH), 4.087 (s, 8H, CH_2), 3.975 (s, 8H, CH_2), 3.747 (s, 3H, H_3O^+), 1.600 (s, H_2O).

IR cm^{-1}: 3359 (m, broad), 2921 (m), 2866 (m), 1668 (m), 1583(m), 1492 (s), 1444 (s), 1352 (s), 1321 (m), 1244 (s), 1195 (s), 1116 (s), 1062 (s), 1043 (s), 950 (s), 860 (m), 800 (s), 649 (s).

IV.3.5. [(H_3O)(Br4-DB18C6)(Br_3)Br_2] 5

Synthesis of **5**: 0.186 g (0.275 mmol) of 4, 4', 5, 5'-tetrabromodibenzo-18-crown-6 were dissolved in 10 ml THF in a Teflon beaker. 0.5 ml (9.73 mmol) of bromine was slowly added. After two weeks, we observed the growth of needle-like crystals. Yield: 0.182 g (0.167 mmol, 61 % with respect to DB18C6)

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.016 (s, 4H, ArH), 4.093 (s, 8H, CH_2), 3.972 (s, 8H, CH_2), 1.604 (s, H_2O)

IR cm^{-1}: 3350 (m, bright), 2921 (m), 2867 (m), 1668 (m), 1583(m), 1494 (s), 1444 (s), 1352 (s), 1323 (m), 1245 (s), 1239 (s), 1114 (s), 1062 (s), 1045 (s), 948 (s), 914 (m), 877 (s), 858 (s), 802 (m), 649 (s).

IV.3.6. [K(Br2-DB18C6)(Br_3)] 6

Synthesis of **6**: In a Teflon beaker, 1 mL of bromine under 10 mL water is slowly mixed with 1 M potassium hydroxide solution until neutralization (pH 7). This is an exothermic process and needs to be done slowly. 10 mL of THF are then added. After cooling of the solution at room temperature, 100 mg of dibenzo-18-crown-6 (0.278 mmol) are added. After two weeks fragile orange crystals are obtained in 72% yield with respect to DB18C6.

^{1}H NMR (DMSO, 250 MHz): δ (ppm) 7.163 (m, 4H, ArH), 6.974 (m, 2H, ArH), 4.149 (m, 8H, CH_2), 3.879 (m, 8H, CH_2).

IR cm^{-1}: 3413 (w, broad), 2923 (w), 2881 (w), 1766 (w), 1595 (m), 1504 (s), 1452 (m), 1328 (w), 1290 (w), 1249 (s), 1215 (m), 1126 (s), 1037 (m), 991 (m), 931 (s), 852 (w), 819 (w), 777 (m), 736 (s), 727(s), 594(m), 557 (w).

IV.3.7. [(Me_3NPh)(DB18C6)(Br_3)] 7

Synthesis of **7**: 0.1080 g (0.299 mmol) of dibenzo-18-crown-6 and 0.2007 g (0.536 mmol) of phenyltrimethylammonium tribromide were dissolved in 10 ml of THF. After two weeks, we observed the growth of needle-like crystals. Yield: 0.182 g (0.247 mmol, 82 % with respect to DB18C6)

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.816 (d, 2H, ArH), 7.704 (m, 3H, ArH), 7.140 to 7.031 (m, 8H, ArH), 4.090 (m, 4H, CH_2), 3.983 (m, 4H, CH_2), 3.928 (s, 9H, $(CH_3)_3$), 3.570 (m, 8H, CH_2).

IR cm^{-1}: 3411 (w, broad), 2952 (m), 2906 (m), 2864 (m), 1639 (w), 1600 (s), 1566 (m), 1494 (s), 1353 (s), 1249 (m), 1197 (m), 1134 (w), 1120 (w), 1089 (m), 1062 (m), 1043 (m), 952 (s), 844 (m), 761 (m), 658(m).

IV.3.8. [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$] 8

Synthesis of **8**: In a beaker, 10 mL of THF was slowly layered on top of 20 mL of an aqueous solution of 1 M potassium carbonate (Notice THF and water were no longer miscible due to lithium carbonate saturation of water). A 15 mg portion (0.015 mmol) of 4-tert-butylcalix[6]arene was powdered onto the liquid. The calixarene molecules arrange themselves at the THF-water interface. After evaporation (1-2 days) of the THF through Parafilm TM cover at a temperature of 23°C, needlelike colourless crystals grow in quantitative yield.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.129 (s, 8H, ArCH), 6.976 (s, 4H, ArCH), 5.010 (m, 12H, CH_2), 3.744 (s, 8H, THF), 1.851 (s, 8H, THF), 1.429 (s, H_2O), 1.249 (s, 54H, CH_3).

IR (cm^{-1}): 3618.21 (w), 3379.05 (m), 2954.74 (s), 2869.88 (m), 1751.24 (w), 1635.52 (m), 1473.51 (s), 1357.79 (m), 1296.08 (m), 1195.78 (s), 1157.21 (m), 1041.49 (w), 987.49 (w), 871.76 (s), 794.62 (w), 725.18 (w), 671.18 (w), 601.75 (w), 563.18 (w).

IV.3.9. [Li_{15}(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4$] 9

Synthesis of **9**: In a schlenk, 0.5 g of 4-tert-butylcalix[8]arene (0.386 mmol) and 0.0651 g of sodium ethoxide (1.41 mmol) were covered by 10 mL of dried THF under N_2 atmosphere. 10 mL of butyllithium (1M) were then slowly added. After 30 min, a dark red solution was obtained. After cooling, this solution is concentrated. In a day, colourless pyrophoric crystals were obtained in quantitative yield.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.064 (s, 16H, ArCH), 3.764 (16H, CH_2), 3.733 (s, 16H, THF), 3.72 (s, 2H, CH_2 (EtO)),1.840 (s, 16H, THF), 1.412 (s, CH_3(EtO)), 1.244 (s, 72H, CH_3).

IR (cm^{-1}): 2923.88 (s), 2852.62 (s), 1606.59 (s), 1460.01 (w), 1392.51 (w), 1359.72 (w), 1311.50 (s), 1209.28 (s), 1132.14 (w), 1047.27 (s), 973.99 (s), 896.84 (m), 819.69 (w), 762.62 (m), 711.68 (w), 655.40 (w), 553.53 (m).

IV.3.10. $[Li_2$(4-tert-butylcalix[8]arene-2H)$(THF)_7(H_2O)_{12}]$ 10

Synthesis of **10**: In a beaker, 10 mL of THF were slowly layered on top of 20 mL of an aqueous solution saturated in lithium carbonate (Notice THF and water were no longer miscible due to lithium carbonate saturation of water). A 20 mg portion (0.015 mmol) of 4-tert-butylcalix[8]arene was powdered onto the liquid. The calixarene molecules arrange themselves at the THF-water interface. After evaporation (1-2 days) of the THF through Parafilm TM cover at a temperature of 23°C, needle like colourless crystals grew in quantitative yield.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.064 (s, 16H, ArCH), 3.764 (8H, CH_2), 3.738 (s, 16H, THF), 3.711 (8H, CH_2), 1.846 (s, 16H, THF), 1.429 (s, H_2O), 1.216 (s, 54H, CH_3).

IR (cm^{-1}): 3305.76 (w), 2954.74 (s), 2866.02 (m), 1477.37 (s), 1361.65 (m), 1299.93 (m), 1184.21 (w), 1114.78 (w), 1049.20 (w), 871.76 (m), 817.76 (w), 790.76 (w), 702.04 (w), 651.89 (w), 594.03 (w), 574.75 (m), 551.60 (m), 524.60 (s).

IV.3.11. IV.3.4. $[Na_2$(4-tert-butyl calix[8]arene-2H)$(THF)_4(H_2O)_5]$ 11

Synthesis of **11**: 0.1975 g of 4-tert-butylcalix[8]arene (0.152 mmol) and 0.5 g of sodium (0.02 mol) were introduced in a Schlenk flask under N_2 atmosphere. 15 mL of dried THF were then added by cryodistillation. This suspension was stirred until total solubilisation (45 min). The yellow solution was then concentrated to 5 mL. 1 mL of toluene was layered upon the solution for a slow diffusion. After one day the solution was concentrated one more time. After few days, colorless crystals were obtained.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 7.164 (s, 16H, ArCH), 3.764 (8H, CH_2), 3.748 (s, 16H, THF), 3.711 (8H, CH_2), 1.866 (s, 16H, THF), 1.429 (s, H_2O), 1.29 (s, 54H, CH_3).

IR (cm^{-1}): 341.84 (w), 2952.81 (m), 2864.09 (w), 1770.53 (w), 1660.60 (w), 1566.09 (w), 1436.87 (s), 1359.72 (w), 1292.22 (m), 1201.57 (w), 1120.56 (w), 1049.20 (w), 879.48 (w), 815.83 (w), 790.76 (w), 752.19 (s).

IV.3.12. [K(4-tert-butylcalix[8]arene-H)(THF)$_4$(H_2O)$_7$] 12

Synthesis of **12**: In a Petri dish, 10 mL of THF was slowly layered on top of 20 mL of an aqueous solution saturated in potassium carbonate (Notice THF and water were no longer miscible due to potassium carbonate saturation of water). A 50 mg portion (0.038mmol) of 4-tert-butylcalix[8]arene was powdered onto the liquid. The calixarene molecules arranged themselves at the THF-water interface. After evaporation (1-2 days) of the THF, needle-like colourless crystals grew in quantitative yield.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 9.626 (s, 7H, OH), 7.175 (s, 16H, ArCH), 4.334 (d, 8H, CH_2), 3.747 (m, 16H, THF), 3.469 (d, 8H, CH_2), 1.853 (m, 16H, THF), 1.575 (s, H_2O), 1.247 (s, 72H, CH_3).

^{13}C NMR ($CDCl_3$, 500 MHz): δ (ppm) 157.03 (s, 8C, ArC(OH)), 146.18 (s, 16C, ArC(CH_2)), 130.53 (s, 16C, ArCH), 39.20 (s, 16C, ArCH_2), 38.60 (s, 8C, C(CH_3)), 37.02 (s, 24C, CH_3).

IR (cm^{-1}): 3380.02 (s), 3245.49 (s), 3150.50 (s), 2901.70 (m), 2865.54 (sh), 1643.24 (s), 1473.51 (s), 1388.65 (m), 1365.51 (m), 1288.36 (w), 1249.79 (s), 1203.50 (s),

1157.21 (vw), 1118.64 (vw), 1056.92 (w), 871.76 (w), 817.76 (w), 786.90 (w), 732.90 (w), 694.33 (m).

Anal. Found (calcd): C, 70.71% (71.3); H, 9.22% (9.03); O, 17.72% (17.37).

IV.3.13. [K_4(4-tert-butylcalix[8]arene-4H)$(THF)_6(H_2O)_{12}$] 13

Synthesis of **13**: In a Petri dish, 10 mL of THF was slowly layered on top of 20 mL of an aqueous solution of 1M potassium hydroxide (Notice THF and water were no longer miscible due to potassium hydroxide saturation of water). A 20 mg portion (0.015 mmol) of 4-tert-butylcalix[8]arene was powdered onto the liquid. The calixarene molecules arranged themselves at the THF-water interface. After evaporation (1-2 days) of the THF at a temperature of 23°C, needle-like colourless crystals grew in quantitative yield.

^{1}H NMR ($CDCl_3$, 500 MHz) **:** δ (ppm) 6.84 (s, 16H, ArH), 1.98 (s, 16H, $ArCH_2$), 1.04 (s, 72H, CH_3)

^{13}C NMR ($CDCl_3$, 500 MHz): 157.82 (s, 8C, ArC(OH)), 145.50 (s, 16C, $ArC(CH_2)$), 130.37 (s, 16C, ArCH), 39.17 (s, 16C, $ArCH_2$), 38.60 (s, 8C, $C(CH_3)$), 37.04 (s, 24C, CH_3)

IR cm^{-1}: 3413.77 (w), 2950.89 (s), 2866.02 (m), 1647.10 (w), 1477.37 (s), 1392.51 (w), 1361.65 (m), 1299.93 (m), 1249.79 (w), 1203.50 (m), 118.64 (w), 1053.06 (s), 910.34 (m), 871.76 (m), 817.76 (m), 790.76 (w), 732.90 (w), 648.04 (w), 586.32 (s), 516.89 (s).

IV.3.14. [Rb_2((4-tert-butylcalix[8]arene-H))$_2(THF)_8(H_2O)_{14}$] 14

Synthesis of **14**: In a beaker, 10 mL of THF was slowly layered on top of 20 mL of an aqueous solution of 0.25 M rubidium carbonate (Notice THF and water were no longer miscible due to rubidium carbonate saturation of water). A 20 mg portion (0.015 mmol) of 4-tert-butylcalix[8]arene was powdered onto the liquid. The

calixarene molecules arranged themselves at the THF-water interface. After evaporation of the THF through Parafilm TM cover at a temperature of 23°C (1-2 days), needle-like colourless crystals grew in quantitative yield.

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 9.627 (s, 7H, OH), 7.175 (s, 16H, ArCH), 4.338 (d, 8H, CH_2), 3.746 (m, 16H, THF), 3.743 (d, 8H, CH_2), 1.852 (m, 16H, THF), 1.430 (s, H_2O), 1.249 (s, 72H, CH_3).

IR cm^{-1}: 3409.91 (w), 2954.74 (m), 2862.17 (m), 1712.67 (w), 1643.24 (w), 1612.38 (w), 1581.52 (w), 1542.95 (w), 1473.51 (s), 1357.79 (w), 1288.36 (s), 1249.79 (s), 1203.50 (s), 1110.92 (w), 1056.92 (m), 871.76 (m), 817.76 (w), 786.90 (w), 732.90 (w), 686.61 (w), 586.32 (w), 570.89 (w).

IV.3.15. [Cs(4-tert-butylcalix[8]arene-H)$(THF)_5(H_2O)_6$] 15

Synthesis of **15**: In a beaker, 10 mL of THF was slowly layered on top of 20 mL of an aqueous solution of 0.20 M caesium carbonate (Notice THF and water were no longer miscible due to caesium carbonate saturation of water). A 20 mg portion (0.015 mmol) of 4-tert-butylcalix[8]arene was powdered onto the liquid. The calixarene molecules arranged themselves at the THF-water interface. After evaporation (1-2 days) of the THF through Parafilm TM cover at 23°, needle-like colourless crystals grew in quantitative yield

^{1}H NMR ($CDCl_3$, 250 MHz): δ (ppm) 9.633 (s, 7H, OH), 7.173 (s, 16H, ArCH), 4.334 (d, 8H, CH_2), 3.747 (m, 16H, THF), 3.524 (d, 8H, CH_2), 1.853 (m, 16H, THF), 1.575 (sh, H_2O), 1.247 (s, 72H, CH_3).

IR (cm^{-1}): 3589.28 (w), 3365.55 (m), 3161.11 (m), 2952.81 (s), 2906.53 (m), 2866.02 (m), 1633.59 (m), 1479.30 (s), 1390.58 (w), 1359.72 (m), 1290.29 (m), 1247.86 (m), 1201.57 (s), 1155.28 (w), 11118.64 (w), 1054.99 (m), 910.34 (w), 871.76 (m), 817.76 (m), 784.97 (w), 661.54 (s), 621.04 (s).

V. Crystallographic Data

V.1. Equipment

The intensities of reflection were collected on a STOE IPDS II diffractometer with monochromated graphite Mo Kα radiation, λ= 071073 Å, equipped with an Oxford Cryosystems open flow cryostat [232], with an absorption correction by analytical integration [233]. The structures were solved with direct methods and refined by full-matrix least-square on F^2 with the SHELX-99 package [234]. Disorder was observed for THF molecules as well as for the water molecules.

The different representations of the compounds were drawn with the Schakal program.

Definitions of the factors R_{int}, R_1 and R_2:

$$R(int)= \Sigma \left| F_o^2 - F_o^2(mean) \right| / \Sigma [F_o^2]$$

$$R_1= \Sigma \left| \left| F_o^2 \right| - \left| F_c \right| \right| / \Sigma \left| F_o \right|$$

$$wR_2= \{\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]\}^{1/2}$$

V.2. Crystal data and structure refinement for single crystal X-ray diffraction

V.2.1. Crystal data and structure refinement for 1∞ [(H_2O)(DB18C6)$(\mu_2$-$H_2O)_{2/2}$]1∞ [(H_3O)(DB18C6)$(\mu_2$-$H_2O)_{2/2}$] (I_3) 1

Empirical formula	$C_{40}\ H_{52}\ I_3\ O_{16}$	
Formula weight	1169.52	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pccn	
Unit cell dimensions	a = 22.047(2) Å	α= 90°.
	b = 22.0840(17) Å	β= 90°.
	c = 9.3800(6) Å	γ = 90°.
Volume	4567.1(6) $Å^3$	
Z	4	
Density (calculated)	1.701 Mg/m^3	
Absorption coefficient	2.120 mm^{-1}	
F(000)	2316	
Theta range for data collection	1.31 to 23.15°.	
Index ranges	-24<=h<=24, -24<=k<=24, -10<=l<=9	
Reflections collected	22204	
Independent reflections	3228 [R(int) = 0.1272]	
Completeness to theta = 23.15°	99.5 %	
Absorption correction	Spherical	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3228 / 0 / 278	
Goodness-of-fit on F^2	0.899	
Final R indices [I>2sigma(I)]	R1 = 0.0569, wR2 = 0.1292	
R indices (all data)	R1 = 0.1209, wR2 = 0.1634	
Largest diff. peak and hole	0.342 and -0.587 $e.Å^{-3}$	

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $1\infty[(H_2O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}]1\infty[(H_3O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}](I_3)$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	0	5000	5000	61(1)
I(2)	51(1)	4737(1)	1951(1)	93(1)
C(1)	1112(5)	8109(5)	6174(12)	54(3)
O(1)	1238(3)	7552(3)	5482(7)	48(2)
C(2)	1097(5)	7035(5)	6310(13)	54(3)
C(3)	1240(4)	6490(5)	5463(12)	49(3)
O(2)	1884(3)	6453(3)	5324(7)	49(2)
C(4)	2114(5)	6016(5)	4446(10)	44(3)
C(5)	1768(5)	5614(5)	3650(11)	53(3)
C(6)	2053(6)	5198(5)	2727(12)	57(3)
C(7)	2662(6)	5201(5)	2642(12)	62(3)
C(8)	3024(6)	5583(5)	3444(11)	57(3)
C(9)	2746(5)	6002(5)	4313(11)	52(3)
O(3)	3044(3)	6416(3)	5154(8)	55(2)
C(10)	3684(5)	6382(5)	5240(12)	52(3)
C(11)	-3262(5)	8809(5)	5323(12)	52(3)
O(4)	-2663(3)	8726(3)	5904(7)	44(2)
C(12)	-2200(5)	8962(5)	5014(12)	50(3)
C(13)	-1609(5)	8891(4)	5777(13)	54(3)
O(5)	-1483(3)	8248(3)	5856(8)	51(2)
C(14)	-1032(4)	8086(5)	6799(11)	46(3)
C(15)	-649(4)	8472(6)	7480(13)	59(3)
C(16)	-228(5)	8249(7)	8448(13)	66(4)
C(17)	-180(5)	7637(6)	8681(13)	65(3)
C(18)	-550(5)	7232(6)	7965(12)	58(3)
C(19)	-980(4)	7461(6)	7012(11)	47(3)
O(6)	-1381(3)	7109(3)	6276(8)	51(2)
C(20)	-1288(4)	6473(5)	6300(11)	47(3)
O(7)	2500	2500	3953(8)	20(2)
O(8)	2500	2500	1550(30)	129(7)

O(9)	2500	7500	2910(40)	165(13)
O(10)	2500	7500	5258(7)	20(2)

Bond lengths [Å] and angles [°] for $1\infty[(H_2O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}]1\infty[(H_3O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}](I_3)$.

I(1)-I(2)	2.9202(10)	C(13)-O(5)	1.447(11)
I(1)-I(2)#1	2.9202(10)	O(5)-C(14)	1.377(12)
C(1)-O(1)	1.417(12)	O(5)-O(10)	2.791(6)
C(1)-C(10)#2	1.495(15)	C(14)-C(15)	1.359(14)
O(1)-C(2)	1.417(12)	C(14)-C(19)	1.402(16)
O(1)-O(8)	2.793(6)	C(15)-C(16)	1.389(16)
C(2)-C(3)	1.475(14)	C(16)-C(17)	1.374(18)
C(3)-O(2)	1.429(11)	C(17)-C(18)	1.384(15)
O(2)-C(4)	1.367(12)	C(18)-C(19)	1.397(15)
O(2)-O(8)	2.681(6)	C(19)-O(6)	1.364(12)
C(4)-C(5)	1.387(14)	O(6)-C(20)	1.420(12)
C(4)-C(9)	1.401(14)	O(6)-O(10)	2.623(7)
C(5)-C(6)	1.410(15)	C(20)-C(11)#3	1.487(15)
C(6)-C(7)	1.345(16)	O(7)-O(7)#2	0.00(7)
C(7)-C(8)	1.382(16)	O(7)-O(8)	2.20(4)
C(8)-C(9)	1.377(15)	O(8)-O(7)#2	2.20(4)
C(9)-O(3)	1.374(13)	O(8)-O(3)#2	2.679(7)
O(3)-C(10)	1.416(12)	O(8)-O(2)#2	2.681(6)
O(3)-O(8)	2.679(7)	O(8)-O(1)#2	2.793(6)
C(10)-C(1)#2	1.495(15)	O(9)-O(10)	2.25(3)
C(11)-O(4)	1.441(12)	O(10)-O(9)#4	2.44(3)
C(11)-C(20)#3	1.487(15)	O(10)-O(6)#3	2.623(7)
O(4)-C(12)	1.417(11)	O(10)-O(4)#3	2.734(6)
O(4)-O(10)	2.734(6)	O(10)-O(5)#3	2.791(6)
C(12)-C(13)	1.496(15)		
		O(2)-C(3)-C(2)	108.0(8)
I(2)-I(1)-I(2)#1	180.000(8)	C(4)-O(2)-C(3)	117.6(8)
O(1)-C(1)-C(10)#2	109.0(8)	C(4)-O(2)-O(8)	114.1(6)
C(2)-O(1)-C(1)	113.9(8)	C(3)-O(2)-O(8)	117.1(6)
C(2)-O(1)-O(8)	103.1(5)	O(2)-C(4)-C(5)	125.0(10)
C(1)-O(1)-O(8)	105.4(6)	O(2)-C(4)-C(9)	115.9(9)
O(1)-C(2)-C(3)	108.4(9)	C(5)-C(4)-C(9)	119.1(11)

C(4)-C(5)-C(6)	120.1(11)	O(7)#2-O(8)-O(3)	87.9(2)
C(7)-C(6)-C(5)	118.6(11)	O(7)-O(8)-O(3)	87.9(2)
C(6)-C(7)-C(8)	123.1(11)	O(7)#2-O(8)-O(3)#2	87.9(2)
C(9)-C(8)-C(7)	118.5(11)	O(7)-O(8)-O(3)#2	87.9(2)
O(3)-C(9)-C(8)	125.1(10)	O(3)-O(8)-O(3)#2	175.9(4)
O(3)-C(9)-C(4)	114.2(10)	O(7)#2-O(8)-O(2)	91.3(2)
C(8)-C(9)-C(4)	120.6(11)	O(7)-O(8)-O(2)	91.3(2)
C(9)-O(3)-C(10)	118.2(8)	O(3)-O(8)-O(2)	57.13(19)
C(9)-O(3)-O(8)	113.7(6)	O(3)#2-O(8)-O(2)	122.99(19)
C(10)-O(3)-O(8)	119.5(6)	O(7)#2-O(8)-O(2)#2	91.3(2)
O(3)-C(10)-C(1)#2	107.0(9)	O(7)-O(8)-O(2)#2	91.3(2)
O(4)-C(11)-C(20)#3	109.0(8)	O(3)-O(8)-O(2)#2	122.99(19)
C(12)-O(4)-C(11)	113.0(8)	O(3)#2-O(8)-O(2)#2	57.13(19)
C(12)-O(4)-O(10)	107.4(5)	O(2)-O(8)-O(2)#2	177.3(4)
C(11)-O(4)-O(10)	105.4(5)	O(7)#2-O(8)-O(1)#2	94.3(2)
O(4)-C(12)-C(13)	107.9(8)	O(7)-O(8)-O(1)#2	94.3(2)
O(5)-C(13)-C(12)	107.2(8)	O(3)-O(8)-O(1)#2	61.3(2)
C(14)-O(5)-C(13)	115.2(8)	O(3)#2-O(8)-O(1)#2	119.1(2)
C(14)-O(5)-O(10)	112.6(6)	O(2)-O(8)-O(1)#2	117.8(2)
C(13)-O(5)-O(10)	115.5(5)	O(2)#2-O(8)-O(1)#2	61.9(2)
C(15)-C(14)-O(5)	126.0(11)	O(7)#2-O(8)-O(1)	94.3(2)
C(15)-C(14)-C(19)	120.0(10)	O(7)-O(8)-O(1)	94.3(2)
O(5)-C(14)-C(19)	114.0(9)	O(3)-O(8)-O(1)	119.1(2)
C(14)-C(15)-C(16)	120.0(12)	O(3)#2-O(8)-O(1)	61.3(2)
C(17)-C(16)-C(15)	120.3(11)	O(2)-O(8)-O(1)	61.9(2)
C(16)-C(17)-C(18)	120.8(11)	O(2)#2-O(8)-O(1)	117.8(2)
C(17)-C(18)-C(19)	118.6(11)	O(1)#2-O(8)-O(1)	171.4(4)
O(6)-C(19)-C(18)	123.9(11)	O(9)-O(10)-O(9)#4	180.000(15)
O(6)-C(19)-C(14)	115.9(9)	O(9)-O(10)-O(6)	85.3(2)
C(18)-C(19)-C(14)	120.1(10)	O(9)#4-O(10)-O(6)	94.7(2)
C(19)-O(6)-C(20)	117.5(8)	O(9)-O(10)-O(6)#3	85.3(2)
C(19)-O(6)-O(10)	117.6(6)	O(9)#4-O(10)-O(6)#3	94.7(2)
C(20)-O(6)-O(10)	117.6(5)	O(6)-O(10)-O(6)#3	170.6(5)
O(6)-C(20)-C(11)#3	107.9(8)	O(9)-O(10)-O(4)#3	92.8(2)
O(7)#2-O(7)-O(8)	0(10)	O(9)#4-O(10)-O(4)#3	87.2(2)
O(7)#2-O(8)-O(7)	0.000(7)	O(6)-O(10)-O(4)#3	63.53(19)

O(6)#3-O(10)-O(4)#3	116.99(19)	O(4)#3-O(10)-O(5)#3	61.05(19)
O(9)-O(10)-O(4)	92.8(2)	O(4)-O(10)-O(5)#3	118.53(19)
O(9)#4-O(10)-O(4)	87.2(2)	O(9)-O(10)-O(5)	93.7(2)
O(6)-O(10)-O(4)	116.99(19)	O(9)#4-O(10)-O(5)	86.3(2)
O(6)#3-O(10)-O(4)	63.53(19)	O(6)-O(10)-O(5)	56.3(2)
O(4)#3-O(10)-O(4)	174.3(4)	O(6)#3-O(10)-O(5)	124.4(2)
O(9)-O(10)-O(5)#3	93.7(2)	O(4)#3-O(10)-O(5)	118.53(19)
O(9)#4-O(10)-O(5)#3	86.3(2)	O(4)-O(10)-O(5)	61.05(19)
O(6)-O(10)-O(5)#3	124.4(2)	O(5)#3-O(10)-O(5)	172.6(4)
O(6)#3-O(10)-O(5)#3	56.3(2)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1 #2 -x+1/2,-y+3/2,z #3 -x-1/2,-y+3/2,z

#4 x,-y+3/2,z-1/2

Anisotropic displacement parameters ($Å^2 x\ 10^3$)for $1\infty[(H_2O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}]1\infty[(H_3O)(DB18C6)(\mu_2\text{-}H_2O)_{2/2}](I_3)$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	56(1)	53(1)	74(1)	9(1)	-3(1)	-4(1)
I(2)	103(1)	103(1)	72(1)	-4(1)	-7(1)	19(1)
C(1)	48(6)	66(8)	46(7)	-6(6)	13(5)	-4(6)
O(1)	48(4)	50(4)	47(4)	2(4)	9(3)	3(3)
C(2)	38(6)	61(8)	63(8)	9(6)	8(6)	-4(5)
C(3)	40(6)	53(7)	55(7)	1(6)	12(5)	-10(5)
O(2)	43(4)	47(4)	56(4)	1(4)	-1(3)	-6(3)
C(4)	53(7)	39(6)	40(6)	2(5)	3(5)	-8(5)
C(5)	72(7)	38(6)	49(7)	7(5)	-4(6)	-10(6)
C(6)	83(9)	43(7)	47(7)	-8(6)	-5(6)	-7(6)
C(7)	97(10)	39(6)	49(7)	-8(6)	10(6)	15(6)
C(8)	74(7)	50(7)	46(7)	-5(6)	-3(6)	4(6)
C(9)	62(7)	53(7)	40(7)	15(6)	-1(6)	-11(6)
O(3)	46(4)	49(4)	70(5)	-6(4)	-4(4)	-1(3)
C(10)	49(6)	43(6)	65(8)	12(6)	-7(5)	2(5)

C(11)	64(7)	41(6)	53(7)	-1(5)	-19(6)	0(5)
O(4)	47(4)	44(4)	41(4)	8(3)	-6(3)	-6(3)
C(12)	59(7)	45(6)	44(6)	1(6)	6(6)	-13(5)
C(13)	58(7)	36(6)	69(8)	4(6)	2(6)	-10(5)
O(5)	47(4)	43(4)	63(5)	-13(4)	-7(4)	-3(3)
C(14)	33(5)	60(8)	45(7)	-12(6)	0(5)	-3(5)
C(15)	33(6)	70(8)	74(8)	-27(7)	5(6)	-5(6)
C(16)	31(5)	90(10)	76(9)	-28(7)	7(6)	-10(6)
C(17)	51(7)	86(10)	57(7)	-14(7)	1(5)	18(6)
C(18)	54(7)	71(8)	48(7)	-8(6)	18(6)	-12(6)
C(19)	30(5)	69(8)	44(6)	-18(7)	10(5)	-10(5)
O(6)	50(4)	39(4)	65(5)	-1(4)	-13(4)	1(3)
C(20)	49(6)	48(7)	44(6)	8(5)	7(5)	2(5)
O(7)	18(4)	20(4)	24(4)	0	0	-1(3)
O(8)	85(11)	113(13)	190(20)	0	0	28(10)
O(9)	121(15)	82(16)	290(40)	0	0	8(13)
O(10)	18(3)	15(4)	26(5)	0	0	2(3)

V.2.2. Crystal data and structure refinement for 1∞[(DB18C6)Na(μ-OH)]1∞[DB18C6Na(μ-H_2O)](I_3) 2

In this structure the largest diffraction peak of the remaining electron density is centered on an oxygen atom delocalized on two position. It can be due to an equilibrium between ion pairs Na1–O7 and Na1'–O7.

Empirical formula	C_{40} H_{48} I_3 Na_2 O_{14}	
Formula weight	1179.46	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pccn	
Unit cell dimensions	a = 21.988(4) Å	α= 90°.
	b = 21.988(4) Å	β= 90°.

	c = 9.3928(19) Å γ = 90°.
Volume	4541.2(16) $Å^3$
Z	4
Density (calculated)	1.725 Mg/m^3
Absorption coefficient	2.146 mm^{-1}
F(000)	2324
Theta range for data collection	1.31 to 23.97°.
Index ranges	-22<=h<=24, -24<=k<=25, -10<=l<=10
Reflections collected	22110
Independent reflections	3415 [R(int) = 0.0775]
Completeness to theta = 23.97°	96.2 %
Absorption correction	Spherical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3415 / 0 / 270
Goodness-of-fit on F^2	1.117
Final R indices [I>2sigma(I)]	R1 = 0.0800, wR2 = 0.2208
R indices (all data)	R1 = 0.0816, wR2 = 0.2218
Largest diff. peak and hole	2.066 and -1.300 $e.Å^{-3}$

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1∞[(DB18C6)Na(μ-OH)]1∞[DB18C6Na(μ-H_2O)](I_3). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	0	5000	5000	46(1)
I(2)	256(1)	5049(1)	8049(1)	71(1)
Na(1)	2500	7500	6022(6)	43(1)
Na(2)	2500	2500	5281(7)	44(1)
C(1)	2960(5)	1083(5)	6325(10)	38(2)
O(1)	2443(3)	1233(3)	5467(7)	36(1)
C(2)	1888(5)	1106(5)	6173(10)	40(2)
C(3)	1368(4)	1307(4)	5232(11)	39(2)

O(2)	1407(3)	1952(3)	5174(7)	40(2)
C(4)	990(4)	2256(4)	4342(9)	33(2)
C(5)	583(4)	1981(5)	3454(10)	40(2)
C(6)	190(5)	2339(6)	2628(11)	49(3)
C(7)	210(5)	2946(5)	2723(12)	48(3)
C(8)	611(4)	3239(5)	3645(11)	40(2)
C(9)	1013(4)	2892(4)	4462(9)	32(2)
O(3)	1445(3)	3114(3)	5349(7)	36(2)
C(10)	1490(5)	3763(4)	5481(11)	38(2)
C(11)	1183(4)	-3255(4)	5319(10)	37(2)
O(4)	1263(3)	-2667(3)	5917(7)	34(1)
C(12)	1026(4)	-2199(4)	5032(9)	37(2)
C(13)	1112(4)	-1601(4)	5774(11)	39(2)
O(5)	1747(3)	-1476(3)	5855(7)	37(2)
C(14)	1922(4)	-1028(4)	6783(9)	32(2)
C(15)	1525(5)	-645(4)	7535(12)	45(3)
C(16)	1747(6)	-227(5)	8480(13)	53(3)
C(17)	2368(6)	-175(4)	8684(11)	48(3)
C(18)	2770(5)	-546(4)	7948(11)	42(2)
C(19)	2551(5)	-973(4)	7000(10)	35(2)
O(6)	2901(3)	-1370(3)	6246(7)	38(2)
C(20)	3538(4)	-1283(4)	6333(9)	36(2)
O(7)	2494(4)	2501(4)	2331(9)	165(12)
O(8)	2494(4)	7501(4)	8529(9)	160(11)

Bond lengths [Å] and angles [°] for $1\infty[(DB18C6)Na(\mu\text{-}OH)]1\infty[DB18C6Na(\mu\text{-}H_2O)](I_3)$

I(1)-I(2)#1	2.9209(11)	Na(1)-O(7)#3	2.771(10)
I(1)-I(2)	2.9209(11)	Na(1)-O(7)	2.771(10)
Na(1)-O(7)#2	1.925(10)	Na(2)-O(8)	2.355(10)
Na(1)-O(3)#3	2.684(6)	Na(2)-O(8)#4	2.342(10)
Na(1)-O(3)	2.684(6)	Na(2)-O(6)#3	2.645(6)
Na(1)-O(2)	2.690(7)	Na(2)-O(6)#5	2.645(6)
Na(1)-O(2)#3	2.690(7)	Na(2)-O(4)#5	2.747(6)
Na(1)-O(1)#3	2.795(6)	Na(2)-O(4)#3	2.747(6)
Na(1)-O(1)	2.795(6)	Na(2)-O(5)#5	2.800(6)

Na(2)-O(5)#3	2.800(6)	C(11)-C(20)#6	1.521(14)
Na(2)-O(6)	19.524(7)	O(4)-C(12)	1.421(11)
Na(2)-O(5)	19.806(7)	O(4)-Na(2)#7	2.747(6)
Na(2)-O(4)	22.520(7)	C(12)-C(13)	1.502(14)
C(1)-O(1)	1.431(12)	C(13)-O(5)	1.424(11)
C(1)-C(10)#3	1.487(14)	O(5)-C(14)	1.370(11)
O(1)-C(2)	1.416(12)	O(5)-Na(2)#7	2.800(6)
C(2)-C(3)	1.512(14)	C(14)-C(19)	1.403(15)
C(3)-O(2)	1.423(11)	C(14)-C(15)	1.404(13)
O(2)-C(4)	1.378(11)	C(15)-C(16)	1.367(16)
C(4)-C(5)	1.364(14)	C(16)-C(17)	1.383(18)
C(4)-C(9)	1.405(13)	C(17)-C(18)	1.388(15)
C(5)-C(6)	1.402(15)	C(18)-C(19)	1.380(14)
C(6)-C(7)	1.338(17)	C(19)-O(6)	1.360(12)
C(7)-C(8)	1.394(15)	O(6)-C(20)	1.416(11)
C(8)-C(9)	1.397(13)	O(6)-Na(2)#7	2.645(6)
C(9)-O(3)	1.356(11)	C(20)-C(11)#6	1.521(14)
O(3)-C(10)	1.435(11)	O(7)-O(7)#3	0.027(19)
C(10)-C(1)#3	1.487(14)	O(7)-Na(1)#8	1.925(10)
C(11)-O(4)	1.422(11)	O(8)-Na(2)#9	2.342(10)

I(2)#1-I(1)-I(2)	180.0	O(2)#3-Na(1)-O(1)#3	61.03(19)
O(7)#2-Na(1)-O(3)#3	89.0(3)	O(7)#2-Na(1)-O(1)	86.4(3)
O(7)#2-Na(1)-O(3)	88.3(3)	O(3)#3-Na(1)-O(1)	62.34(19)
O(3)#3-Na(1)-O(3)	177.3(4)	O(3)-Na(1)-O(1)	117.47(19)
O(7)#2-Na(1)-O(2)	91.8(3)	O(2)-Na(1)-O(1)	61.03(19)
O(3)#3-Na(1)-O(2)	123.18(18)	O(2)#3-Na(1)-O(1)	119.28(19)
O(3)-Na(1)-O(2)	56.94(18)	O(1)#3-Na(1)-O(1)	172.8(4)
O(7)#2-Na(1)-O(2)#3	92.5(3)	O(7)#2-Na(1)-O(7)#3	179.9(3)
O(3)#3-Na(1)-O(2)#3	56.94(18)	O(3)#3-Na(1)-O(7)#3	91.1(3)
O(3)-Na(1)-O(2)#3	123.18(18)	O(3)-Na(1)-O(7)#3	91.6(3)
O(2)-Na(1)-O(2)#3	175.7(4)	O(2)-Na(1)-O(7)#3	88.1(3)
O(7)#2-Na(1)-O(1)#3	86.5(3)	O(2)#3-Na(1)-O(7)#3	87.6(3)
O(3)#3-Na(1)-O(1)#3	117.47(19)	O(1)#3-Na(1)-O(7)#3	93.6(3)
O(3)-Na(1)-O(1)#3	62.34(19)	O(1)-Na(1)-O(7)#3	93.6(3)
O(2)-Na(1)-O(1)#3	119.28(19)	O(7)#2-Na(1)-O(7)	179.3(5)

O(3)#3-Na(1)-O(7)	91.6(3)	O(8)#4-Na(2)-O(6)	90.6(2)
O(3)-Na(1)-O(7)	91.1(3)	O(6)#3-Na(2)-O(6)	22.42(15)
O(2)-Na(1)-O(7)	87.6(3)	O(6)#5-Na(2)-O(6)	157.33(16)
O(2)#3-Na(1)-O(7)	88.1(3)	O(4)#5-Na(2)-O(6)	84.94(12)
O(1)#3-Na(1)-O(7)	93.6(3)	O(4)#3-Na(2)-O(6)	95.10(12)
O(1)-Na(1)-O(7)	93.6(3)	O(5)#5-Na(2)-O(6)	146.18(13)
O(7)#3-Na(1)-O(7)	0.6(4)	O(5)#3-Na(2)-O(6)	33.94(13)
O(8)-Na(2)-O(8)#4	179.9(5)	O(8)-Na(2)-O(5)	90.5(2)
O(8)-Na(2)-O(6)#3	85.4(3)	O(8)#4-Na(2)-O(5)	89.6(2)
O(8)#4-Na(2)-O(6)#3	94.7(3)	O(6)#3-Na(2)-O(5)	15.53(14)
O(8)-Na(2)-O(6)#5	85.5(3)	O(6)#5-Na(2)-O(5)	164.74(14)
O(8)#4-Na(2)-O(6)#5	94.4(3)	O(4)#5-Na(2)-O(5)	77.53(12)
O(6)#3-Na(2)-O(6)#5	170.9(4)	O(4)#3-Na(2)-O(5)	102.44(12)
O(8)-Na(2)-O(4)#5	91.7(3)	O(5)#5-Na(2)-O(5)	138.73(14)
O(8)#4-Na(2)-O(4)#5	88.3(3)	O(5)#3-Na(2)-O(5)	41.19(14)
O(6)#3-Na(2)-O(4)#5	63.12(18)	O(6)-Na(2)-O(5)	7.46(3)
O(6)#5-Na(2)-O(4)#5	117.25(18)	O(8)-Na(2)-O(4)	90.2(2)
O(8)-Na(2)-O(4)#3	92.4(3)	O(8)#4-Na(2)-O(4)	89.8(2)
O(8)#4-Na(2)-O(4)#3	87.6(3)	O(6)#3-Na(2)-O(4)	13.45(14)
O(6)#3-Na(2)-O(4)#3	117.25(18)	O(6)#5-Na(2)-O(4)	166.72(14)
O(6)#5-Na(2)-O(4)#3	63.12(18)	O(4)#5-Na(2)-O(4)	75.39(12)
O(4)#5-Na(2)-O(4)#3	175.9(4)	O(4)#3-Na(2)-O(4)	104.59(12)
O(8)-Na(2)-O(5)#5	93.0(3)	O(5)#5-Na(2)-O(4)	136.62(13)
O(8)#4-Na(2)-O(5)#5	87.0(3)	O(5)#3-Na(2)-O(4)	43.34(13)
O(6)#3-Na(2)-O(5)#5	124.29(19)	O(6)-Na(2)-O(4)	9.56(2)
O(6)#5-Na(2)-O(5)#5	56.33(19)	O(5)-Na(2)-O(4)	2.15(2)
O(4)#5-Na(2)-O(5)#5	61.28(18)	O(1)-C(1)-C(10)#3	107.1(8)
O(4)#3-Na(2)-O(5)#5	118.46(18)	C(2)-O(1)-C(1)	112.0(7)
O(8)-Na(2)-O(5)#3	93.4(3)	C(2)-O(1)-Na(1)	105.3(5)
O(8)#4-Na(2)-O(5)#3	86.6(3)	C(1)-O(1)-Na(1)	103.2(5)
O(6)#3-Na(2)-O(5)#3	56.33(19)	O(1)-C(2)-C(3)	108.6(7)
O(6)#5-Na(2)-O(5)#3	124.29(19)	O(2)-C(3)-C(2)	105.6(8)
O(4)#5-Na(2)-O(5)#3	118.46(18)	C(4)-O(2)-C(3)	117.7(7)
O(4)#3-Na(2)-O(5)#3	61.28(18)	C(4)-O(2)-Na(1)	113.6(5)
O(5)#5-Na(2)-O(5)#3	173.6(4)	C(3)-O(2)-Na(1)	119.9(5)
O(8)-Na(2)-O(6)	89.4(2)	C(5)-C(4)-O(2)	124.7(9)

C(5)-C(4)-C(9)	120.9(9)	C(14)-O(5)-Na(2)	41.9(4)
O(2)-C(4)-C(9)	114.4(8)	C(13)-O(5)-Na(2)	105.9(5)
C(4)-C(5)-C(6)	119.6(10)	Na(2)#7-O(5)-Na(2)	138.73(14)
C(7)-C(6)-C(5)	120.2(10)	O(5)-C(14)-C(19)	115.6(8)
C(6)-C(7)-C(8)	121.5(11)	O(5)-C(14)-C(15)	125.1(9)
C(9)-C(8)-C(7)	119.2(10)	C(19)-C(14)-C(15)	119.2(9)
O(3)-C(9)-C(8)	125.8(8)	C(16)-C(15)-C(14)	120.5(11)
O(3)-C(9)-C(4)	115.7(8)	C(15)-C(16)-C(17)	119.9(10)
C(8)-C(9)-C(4)	118.5(9)	C(16)-C(17)-C(18)	120.7(10)
C(9)-O(3)-C(10)	117.3(7)	C(17)-C(18)-C(19)	119.9(11)
C(9)-O(3)-Na(1)	114.2(5)	O(6)-C(19)-C(14)	115.2(8)
C(10)-O(3)-Na(1)	116.3(5)	O(6)-C(19)-C(18)	125.1(10)
O(3)-C(10)-C(1)#3	109.1(8)	C(14)-C(19)-C(18)	119.8(9)
O(4)-C(11)-C(20)#6	108.1(7)	C(19)-O(6)-C(20)	116.3(7)
C(11)-O(4)-C(12)	112.5(7)	C(19)-O(6)-Na(2)#7	117.1(5)
C(11)-O(4)-Na(2)#7	105.0(5)	C(20)-O(6)-Na(2)#7	117.4(5)
C(12)-O(4)-Na(2)#7	106.7(5)	C(19)-O(6)-Na(2)	48.7(5)
C(11)-O(4)-Na(2)	157.0(5)	C(20)-O(6)-Na(2)	84.9(5)
C(12)-O(4)-Na(2)	47.8(4)	Na(2)#7-O(6)-Na(2)	157.33(15)
Na(2)#7-O(4)-Na(2)	75.39(12)	O(6)-C(20)-C(11)#6	105.9(7)
O(4)-C(12)-C(13)	108.4(7)	O(7)#3-O(7)-Na(1)#8	89.6(3)
O(5)-C(13)-C(12)	108.5(8)	O(7)#3-O(7)-Na(1)	89.7(2)
C(14)-O(5)-C(13)	116.7(7)	Na(1)#8-O(7)-Na(1)	179.3(5)
C(14)-O(5)-Na(2)#7	112.1(5)	Na(2)-O(8)-Na(2)#9	179.3(5)
C(13)-O(5)-Na(2)#7	115.3(5)		

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1 #2 x,-y+1/2,z+1/2 #3 -x+1/2,-y+1/2,z

#4 -x+1/2,y,z-1/2 #5 x,y+1,z #6 -x+1/2,-y-1/2,z

#7 x,y-1,z #8 x,-y+1/2,z-1/2 #9 x,-y+3/2,z+1/2

Anisotropic displacement parameters ($Å^2$x 10^3)for $1\infty[(DB18C6)Na(\mu\text{-}OH)]1\infty[DB18C6Na(\mu\text{-}H_2O)](I_3)$.

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}

I(1)	41(1)	41(1)	55(1)	2(1)	6(1)	3(1)
I(2)	81(1)	80(1)	53(1)	5(1)	-2(1)	-17(1)
Na(1)	42(3)	42(3)	46(3)	0	0	-2(3)
Na(2)	41(3)	39(3)	53(3)	0	0	-3(3)
C(1)	45(6)	38(5)	32(5)	5(4)	0(4)	12(4)
O(1)	38(3)	37(3)	31(3)	9(3)	1(3)	3(3)
C(2)	48(6)	42(5)	30(5)	15(4)	5(4)	5(4)
C(3)	42(5)	39(5)	35(5)	-2(4)	10(4)	-3(4)
O(2)	43(4)	33(3)	45(4)	2(3)	-6(3)	0(3)
C(4)	32(5)	48(5)	18(4)	4(4)	5(4)	5(4)
C(5)	35(5)	46(6)	38(5)	0(4)	3(4)	-1(4)
C(6)	37(5)	79(8)	32(5)	1(5)	2(5)	-5(5)
C(7)	37(6)	65(7)	43(6)	3(5)	-4(5)	-2(5)
C(8)	33(5)	45(6)	41(5)	4(5)	-1(4)	10(4)
C(9)	36(5)	38(5)	21(4)	1(4)	7(4)	1(4)
O(3)	36(3)	30(3)	41(4)	2(3)	-4(3)	1(3)
C(10)	43(5)	32(5)	40(5)	-3(4)	3(4)	7(4)
C(11)	32(5)	50(6)	29(5)	-7(4)	-6(4)	-4(4)
O(4)	31(3)	34(3)	36(3)	-8(3)	-8(3)	0(3)
C(12)	36(5)	52(6)	22(4)	3(4)	-5(4)	5(4)
C(13)	32(5)	41(5)	45(6)	6(4)	1(4)	6(4)
O(5)	31(3)	39(3)	42(4)	-4(3)	7(3)	1(3)
C(14)	45(6)	26(4)	26(5)	4(4)	10(4)	7(4)
C(15)	55(6)	28(5)	52(6)	5(5)	20(5)	0(4)
C(16)	70(8)	29(5)	60(7)	2(5)	26(6)	7(5)
C(17)	76(9)	29(5)	39(6)	2(4)	10(5)	-9(5)
C(18)	55(6)	35(5)	36(5)	1(4)	4(5)	-5(4)
C(19)	49(6)	27(4)	30(5)	9(4)	3(4)	2(4)
O(6)	28(3)	36(3)	49(4)	-6(3)	0(3)	-1(3)
C(20)	35(5)	41(5)	31(5)	7(4)	-7(4)	-2(4)
O(7)	81(11)	56(9)	360(40)	0	0	-1(9)
O(8)	67(9)	60(9)	350(40)	0	0	-22(9)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 1∞[(DB18C6)Na(μ-OH)]1∞[DB18C6Na(μ-H_2O)](I_3).

	x	y	z	U(eq)
H(1A)	2957	654	6560	46
H(1B)	2953	1315	7204	46
H(2A)	1873	1322	7073	48
H(2B)	1857	674	6366	48
H(3A)	1408	1133	4287	46
H(3B)	982	1181	5633	46
H(5)	566	1559	3397	48
H(6)	-84	2154	2011	59
H(7)	-51	3177	2162	58
H(8)	611	3661	3715	48
H(10A)	1130	3921	5951	46
H(10B)	1516	3947	4544	46
H(11A)	1381	-3278	4398	45
H(11B)	753	-3340	5191	45
H(12A)	597	-2269	4854	44
H(12B)	1237	-2197	4125	44
H(13A)	908	-1280	5250	47
H(13B)	940	-1619	6724	47
H(15)	1108	-675	7389	54
H(16)	1481	22	8984	64
H(17)	2518	112	9321	58
H(18)	3187	-508	8094	50
H(20A)	3679	-1352	7297	43
H(20B)	3646	-873	6052	43

V.2.3. Crystal data and structure refinement for [Na(DB18C6)I(THF)][Na(DB18C6)(H_2O)$_2$]I(THF)$_2$(CHI_3)] 3

In this structure the largest diffraction peak of the remaining electron density is centered on an iodide atom I4 which is on a symmetry position. Thus, this atom could lead to a failure in remaining electron density.

Empirical formula	$C_{53}\ H_{77}\ I_5\ Na_2\ O_{17}$	
Formula weight	1666.67	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/m	
Unit cell dimensions	a = 13.760(3) Å	α= 90°.
	b = 19.021(3) Å	β= 117.726(15)°.
	c = 14.297(3) Å	γ = 90°.
Volume	3312.3(11) $Å^3$	
Z	2	
Density (calculated)	1.642 Mg/m^3	
Absorption coefficient	2.420 mm^{-1}	
F(000)	1578	
Theta range for data collection	2.01 to 32.27°.	
Index ranges	-20<=h<=20, -23<=k<=28, -21<=l<=21	
Reflections collected	40217	
Independent reflections	11814 [R(int) = 0.0788]	
Completeness to theta = 32.27°	97.7 %	
Absorption correction	Spherical	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11814 / 0 / 367	
Goodness-of-fit on F^2	1.016	
Final R indices [I>2sigma(I)]	R1 = 0.1075, wR2 = 0.3130	
R indices (all data)	R1 = 0.1572, wR2 = 0.3625	
Largest diff. peak and hole	4.387 and -2.475 $e.Å^{-3}$	

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Na(DB18C6)I(THF)][Na(DB18C6)$(H_2O)_2$]I$(THF)_2(CHI_3)$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I(1)	4075(1)	7500	1689(1)	73(1)
I(2)	0	5000	5000	88(1)
I(3)	1282(1)	7500	9076(1)	84(1)
I(4)	-82(1)	6551(1)	6623(1)	87(1)
Na(1)	6176(3)	7500	936(2)	29(1)
Na(2)	4327(4)	7500	5655(5)	56(1)
O(1)	6348(8)	7500	5684(7)	61(2)
C(1)	6384(9)	6893(6)	5154(8)	65(2)
C(2)	6331(9)	6235(6)	5723(9)	70(2)
O(2)	5307(6)	6232(3)	5738(6)	66(2)
C(3)	5060(8)	5640(5)	6132(7)	61(2)
C(4)	5714(11)	5051(6)	6503(9)	74(3)
C(5)	5389(13)	4473(6)	6889(8)	87(4)
C(6)	4381(13)	4482(6)	6858(9)	85(3)
C(7)	3670(12)	5075(6)	6477(10)	83(3)
C(8)	4035(9)	5643(5)	6116(8)	68(2)
O(3)	3417(6)	6239(4)	5711(7)	72(2)
C(9)	2347(9)	6245(7)	5657(11)	80(3)
C(10)	1773(8)	6890(7)	5039(11)	82(3)
O(4)	2302(8)	7500	5589(9)	76(3)
O(5)	4569(8)	7500	-1153(7)	67(2)
C(11)	3938(9)	6871(6)	-1189(8)	73(3)
C(12)	4609(9)	6236(6)	-1129(7)	71(2)
O(6)	5539(6)	6234(3)	-100(5)	67(2)
C(13)	6220(8)	5662(5)	158(8)	64(2)
C(14)	6094(11)	5099(6)	-506(10)	83(3)
C(15)	6791(11)	4529(6)	-154(11)	85(3)
C(16)	7683(13)	4527(6)	830(12)	93(4)
C(17)	7835(10)	5091(6)	1539(11)	79(3)
C(18)	7101(8)	5651(5)	1192(8)	63(2)
O(7)	7186(5)	6227(3)	1792(5)	61(1)
C(19)	8137(9)	6249(5)	2812(8)	70(2)
C(20)	8009(10)	6884(5)	3378(7)	71(3)
O(8)	8080(8)	7500	2858(6)	58(2)
O(9)	5320(40)	7500	7330(40)	300(20)
C(21)	-62(14)	7500	7481(13)	81(4)
O(10)	3268(16)	7500	3700(14)	151(8)
O(11)	7690(40)	7500	-50(30)	252(19)
C(22)	7790(50)	6930(20)	-510(60)	360(40)
C(23)	8090(30)	7110(20)	-1190(30)	280(30)
O(12)	1250(20)	5590(20)	2770(20)	281(16)
C(24)	1670(30)	6165(19)	2620(20)	204(17)
C(25)	1130(60)	6340(30)	1750(30)	330(40)
C(26)	880(50)	5850(30)	1140(30)	280(30)
C(27)	1040(30)	5097(17)	1850(30)	204(15)

Bond lengths [Å] and angles [°] for [Na(DB18C6)I(THF)][Na(DB18C6)$(H_2O)_2$]I$(THF)_2(CHI_3)$].

I(1)-Na(1)	3.523(4)	I(4)-C(21)	2.176(9)
I(3)-C(21)	2.167(17)	Na(1)-O(6)	2.748(6)

Na(1)-O(6)#1	2.748(6)	O(4)-C(10)#1	1.401(13)
Na(1)-O(5)	2.769(9)	O(5)-C(11)	1.465(12)
Na(1)-O(7)#1	2.777(6)	O(5)-C(11)#1	1.465(13)
Na(1)-O(7)	2.777(6)	C(11)-C(12)	1.498(15)
Na(1)-O(8)	2.778(9)	C(12)-O(6)	1.432(11)
Na(1)-O(11)	3.01(4)	O(6)-C(13)	1.371(12)
Na(2)-O(9)	2.13(5)	C(13)-C(14)	1.387(14)
Na(2)-O(10)	2.479(19)	C(13)-C(18)	1.410(14)
Na(2)-O(3)	2.725(7)	C(14)-C(15)	1.378(17)
Na(2)-O(3)#1	2.725(7)	C(15)-C(16)	1.37(2)
Na(2)-O(2)#1	2.739(7)	C(16)-C(17)	1.423(17)
Na(2)-O(2)	2.739(7)	C(17)-C(18)	1.391(15)
Na(2)-O(4)	2.744(10)	C(18)-O(7)	1.363(11)
Na(2)-O(1)	2.763(9)	O(7)-C(19)	1.437(11)
O(1)-C(1)	1.394(11)	C(19)-C(20)	1.510(15)
O(1)-C(1)#1	1.394(11)	C(20)-O(8)	1.416(10)
C(1)-C(2)	1.513(15)	O(8)-C(20)#1	1.416(10)
C(2)-O(2)	1.419(12)	C(21)-I(4)#1	2.176(9)
O(2)-C(3)	1.369(11)	O(11)-C(22)	1.31(5)
C(3)-C(4)	1.381(15)	O(11)-C(22)#1	1.31(5)
C(3)-C(8)	1.400(15)	C(22)-C(23)	1.27(6)
C(4)-C(5)	1.393(16)	C(23)-C(23)#1	1.49(9)
C(5)-C(6)	1.37(2)	O(12)-C(24)	1.29(4)
C(6)-C(7)	1.424(19)	O(12)-C(27)	1.53(4)
C(7)-C(8)	1.387(15)	C(24)-C(25)	1.16(5)
C(8)-O(3)	1.373(13)	C(24)-C(26)	1.96(5)
O(3)-C(9)	1.437(12)	C(25)-C(26)	1.20(5)
C(9)-C(10)	1.504(19)	C(26)-C(27)	1.71(5)
C(10)-O(4)	1.401(13)		

O(6)-Na(1)-O(6)#1	122.4(3)	O(8)-Na(1)-I(1)	103.2(2)
O(6)-Na(1)-O(5)	61.57(15)	O(11)-Na(1)-I(1)	171.3(9)
O(6)#1-Na(1)-O(5)	61.57(14)	O(9)-Na(2)-O(10)	176.9(10)
O(6)-Na(1)-O(7)#1	166.0(3)	O(9)-Na(2)-O(3)	91.7(5)
O(6)#1-Na(1)-O(7)#1	56.11(18)	O(10)-Na(2)-O(3)	89.8(3)
O(5)-Na(1)-O(7)#1	117.40(14)	O(9)-Na(2)-O(3)#1	91.7(5)
O(6)-Na(1)-O(7)	56.11(18)	O(10)-Na(2)-O(3)#1	89.8(3)
O(6)#1-Na(1)-O(7)	166.0(3)	O(3)-Na(2)-O(3)#1	123.4(3)
O(5)-Na(1)-O(7)	117.40(14)	O(9)-Na(2)-O(2)#1	84.7(5)
O(7)#1-Na(1)-O(7)	121.4(3)	O(10)-Na(2)-O(2)#1	93.9(3)
O(6)-Na(1)-O(8)	116.79(15)	O(3)-Na(2)-O(2)#1	176.3(3)
O(6)#1-Na(1)-O(8)	116.79(15)	O(3)#1-Na(2)-O(2)#1	56.4(2)
O(5)-Na(1)-O(8)	168.4(3)	O(9)-Na(2)-O(2)	84.7(5)
O(7)#1-Na(1)-O(8)	61.03(13)	O(10)-Na(2)-O(2)	93.9(3)
O(7)-Na(1)-O(8)	61.03(13)	O(3)-Na(2)-O(2)	56.4(2)
O(6)-Na(1)-O(11)	82.2(4)	O(3)#1-Na(2)-O(2)	176.3(3)
O(6)#1-Na(1)-O(11)	82.2(4)	O(2)#1-Na(2)-O(2)	123.4(3)
O(5)-Na(1)-O(11)	82.8(9)	O(9)-Na(2)-O(4)	98.4(10)
O(7)#1-Na(1)-O(11)	83.8(5)	O(10)-Na(2)-O(4)	84.6(5)
O(7)-Na(1)-O(11)	83.8(5)	O(3)-Na(2)-O(4)	61.82(16)
O(8)-Na(1)-O(11)	85.5(9)	O(3)#1-Na(2)-O(4)	61.82(16)
O(6)-Na(1)-I(1)	93.65(18)	O(2)#1-Na(2)-O(4)	118.24(16)
O(6)#1-Na(1)-I(1)	93.65(18)	O(2)-Na(2)-O(4)	118.24(16)
O(5)-Na(1)-I(1)	88.4(2)	O(9)-Na(2)-O(1)	82.6(10)
O(7)#1-Na(1)-I(1)	100.30(16)	O(10)-Na(2)-O(1)	94.4(5)
O(7)-Na(1)-I(1)	100.30(16)	O(3)-Na(2)-O(1)	118.22(16)

O(3)#1-Na(2)-O(1)	118.22(16)	C(12)-O(6)-Na(1)	117.4(6)
O(2)#1-Na(2)-O(1)	61.80(16)	O(6)-C(13)-C(14)	125.2(9)
O(2)-Na(2)-O(1)	61.80(16)	O(6)-C(13)-C(18)	116.1(8)
O(4)-Na(2)-O(1)	179.0(4)	C(14)-C(13)-C(18)	118.7(10)
C(1)-O(1)-C(1)#1	111.8(10)	C(15)-C(14)-C(13)	120.8(11)
C(1)-O(1)-Na(2)	106.4(6)	C(16)-C(15)-C(14)	121.1(11)
C(1)#1-O(1)-Na(2)	106.4(6)	C(15)-C(16)-C(17)	119.5(12)
O(1)-C(1)-C(2)	111.7(8)	C(18)-C(17)-C(16)	118.8(12)
O(2)-C(2)-C(1)	108.0(8)	O(7)-C(18)-C(17)	123.8(9)
C(3)-O(2)-C(2)	116.7(8)	O(7)-C(18)-C(13)	115.4(8)
C(3)-O(2)-Na(2)	122.0(5)	C(17)-C(18)-C(13)	120.8(9)
C(2)-O(2)-Na(2)	117.8(6)	C(18)-O(7)-C(19)	115.6(7)
O(2)-C(3)-C(4)	125.6(9)	C(18)-O(7)-Na(1)	123.0(5)
O(2)-C(3)-C(8)	115.6(9)	C(19)-O(7)-Na(1)	117.1(5)
C(4)-C(3)-C(8)	118.8(9)	O(7)-C(19)-C(20)	106.7(8)
C(3)-C(4)-C(5)	121.4(12)	O(8)-C(20)-C(19)	109.1(8)
C(6)-C(5)-C(4)	119.0(12)	C(20)-O(8)-C(20)#1	111.8(10)
C(5)-C(6)-C(7)	121.9(10)	C(20)-O(8)-Na(1)	104.8(6)
C(8)-C(7)-C(6)	117.2(12)	C(20)#1-O(8)-Na(1)	104.8(6)
O(3)-C(8)-C(7)	122.7(11)	I(3)-C(21)-I(4)	112.0(5)
O(3)-C(8)-C(3)	115.5(8)	I(3)-C(21)-I(4)#1	112.0(5)
C(7)-C(8)-C(3)	121.8(11)	I(4)-C(21)-I(4)#1	112.1(7)
C(8)-O(3)-C(9)	116.5(8)	C(22)-O(11)-C(22)#1	111(5)
C(8)-O(3)-Na(2)	122.4(6)	C(22)-O(11)-Na(1)	120(2)
C(9)-O(3)-Na(2)	117.6(6)	C(22)#1-O(11)-Na(1)	120(2)
O(3)-C(9)-C(10)	106.5(9)	C(23)-C(22)-O(11)	109(4)
O(4)-C(10)-C(9)	110.6(10)	C(22)-C(23)-C(23)#1	106(3)
C(10)-O(4)-C(10)#1	111.8(13)	C(24)-O(12)-C(27)	108(3)
C(10)-O(4)-Na(2)	105.9(6)	C(25)-C(24)-O(12)	107(4)
C(10)#1-O(4)-Na(2)	105.9(6)	C(25)-C(24)-C(26)	34(3)
C(11)-O(5)-C(11)#1	109.5(11)	O(12)-C(24)-C(26)	82(2)
C(11)-O(5)-Na(1)	101.5(5)	C(24)-C(25)-C(26)	113(6)
C(11)#1-O(5)-Na(1)	101.5(5)	C(25)-C(26)-C(27)	108(3)
O(5)-C(11)-C(12)	108.5(9)	C(25)-C(26)-C(24)	33(3)
O(6)-C(12)-C(11)	107.0(7)	C(27)-C(26)-C(24)	76.5(19)
C(13)-O(6)-C(12)	116.8(7)	O(12)-C(27)-C(26)	85(2)
C(13)-O(6)-Na(1)	123.0(5)		

Symmetry transformations used to generate equivalent atoms:
#1 x,-y+3/2,z

Anisotropic displacement parameters ($Å^2$x 10^3)for $[Na(DB18C6)I(THF)][Na(DB18C6)(H_2O)_2]I(THF)_2(CHI_3)$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I(1)	109(1)	54(1)	71(1)	0	55(1)	0
I(2)	66(1)	85(1)	79(1)	24(1)	6(1)	-26(1)
I(3)	103(1)	85(1)	63(1)	0	37(1)	0
I(4)	83(1)	100(1)	67(1)	-6(1)	26(1)	-16(1)
Na(1)	37(2)	22(1)	15(1)	0	1(1)	0
Na(2)	45(2)	36(2)	107(4)	0	52(2)	0
O(1)	64(5)	66(5)	63(5)	0	39(4)	0
C(1)	69(5)	74(6)	58(5)	-2(4)	34(4)	4(4)
C(2)	68(5)	77(6)	69(5)	5(5)	36(5)	9(5)
O(2)	68(4)	53(3)	87(4)	7(3)	43(3)	6(3)

C(3)	80(6)	50(4)	57(4)	-12(3)	36(4)	-10(4)
C(4)	93(7)	63(5)	67(5)	2(4)	38(5)	-2(5)
C(5)	132(11)	52(5)	57(5)	-2(4)	27(6)	0(6)
C(6)	128(11)	55(5)	75(6)	-3(5)	50(7)	-28(6)
C(7)	104(8)	66(6)	86(7)	-20(5)	50(7)	-29(6)
C(8)	88(7)	52(4)	68(5)	-14(4)	40(5)	-15(4)
O(3)	71(4)	57(3)	102(5)	-3(3)	52(4)	-8(3)
C(9)	61(5)	84(7)	108(8)	-16(6)	50(6)	-19(5)
C(10)	47(4)	102(8)	100(8)	-13(7)	37(5)	-18(5)
O(4)	52(5)	90(7)	89(7)	0	35(5)	0
O(5)	70(5)	71(6)	46(4)	0	16(4)	0
C(11)	73(6)	68(6)	56(5)	-9(4)	11(4)	-4(5)
C(12)	83(6)	66(5)	53(4)	-15(4)	23(4)	-6(5)
O(6)	80(4)	59(3)	47(3)	-13(3)	17(3)	4(3)
C(13)	71(5)	57(5)	61(5)	-6(4)	29(4)	0(4)
C(14)	98(8)	69(6)	73(6)	-21(5)	33(6)	-2(6)
C(15)	104(9)	55(5)	98(8)	-19(5)	49(7)	0(5)
C(16)	116(10)	55(5)	110(10)	-3(6)	55(9)	6(6)
C(17)	81(7)	58(5)	92(8)	0(5)	35(6)	2(5)
C(18)	72(5)	47(4)	66(5)	1(4)	30(4)	-4(4)
O(7)	70(4)	54(3)	46(3)	-2(2)	15(3)	2(3)
C(19)	72(6)	53(5)	64(5)	9(4)	14(4)	-2(4)
C(20)	90(7)	68(5)	43(4)	5(4)	20(4)	-12(5)
O(8)	73(5)	55(4)	41(3)	0	21(4)	0
O(9)	270(40)	430(70)	290(50)	0	200(40)	0
C(21)	72(9)	112(13)	68(8)	0	42(7)	0
O(10)	127(13)	250(30)	103(11)	0	80(11)	0
O(11)	370(60)	280(50)	180(30)	0	200(30)	0
C(22)	470(80)	170(30)	710(120)	20(50)	510(100)	-10(40)
C(23)	270(40)	370(80)	310(50)	-170(50)	230(40)	-130(40)
O(12)	250(30)	430(50)	200(20)	-30(30)	140(20)	-90(30)
C(24)	260(40)	230(30)	118(18)	-60(20)	90(20)	-150(30)
C(25)	480(90)	270(50)	100(20)	-10(30)	10(40)	-130(60)
C(26)	490(80)	230(40)	120(20)	20(30)	140(40)	20(50)
C(27)	170(30)	160(20)	190(30)	-30(20)	10(20)	-40(20)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)
for [Na(DB18C6)I(THF)][Na(DB18C6)$(H_2O)_2$]I$(THF)_2(CHI_3)$].

	x	y	z	U(eq)
H(1A)	7057	6890	5093	78
H(1B)	5772	6896	4445	78
H(2A)	6396	5821	5361	84
H(2B)	6928	6233	6440	84
H(4)	6386	5039	6496	89
H(5)	5851	4087	7163	105
H(6)	4152	4090	7093	102
H(7)	2992	5082	6471	99
H(9A)	2411	6264	6361	96
H(9B)	1942	5825	5306	96
H(10A)	1768	6883	4358	98
H(10B)	1017	6891	4915	98
H(11A)	3252	6864	-1840	88
H(11B)	3772	6873	-600	88
H(12A)	4179	5812	-1235	85

H(12B)	4847	6258	-1670	85
H(14)	5531	5106	-1197	99
H(15)	6655	4140	-591	102
H(16)	8186	4159	1033	111
H(17)	8415	5086	2221	95
H(19A)	8799	6288	2735	84
H(19B)	8184	5824	3206	84
H(20A)	7304	6867	3377	86
H(20B)	8581	6886	4106	86

V.2.4. Crystal data and structure refinement for $[(H_5O_2)(Br_4\text{-}DB18C6)_2][FeBr_4]$ 4

In this structure the largest diffraction peak of the remaining electron density is centered on an iron atom Fe1 which is on a symmetry position. The estimation of the remaining electron density may be overestimated due to this special fact.

Empirical formula	$C_{40}\ H_{45}\ Br_8\ Fe\ O_8$	
Formula weight	1067.4524	
Temperature	243(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 19.8188(15) Å	α= 90°.
	b = 19.2684(18) Å	β= 117.968(5)°.
	c = 16.4908(13) Å	γ = 90°.
Volume	5562.0(8) $Å^3$	
Z	4	
Density (calculated)	2.102 Mg/m^3	
Absorption coefficient	8.949 mm^{-1}	
F(000)	3356	
Theta range for data collection	1.57 to 27.03°.	
Index ranges	-25<=h<=25, -24<=k<=24, -21<=l<=20	
Reflections collected	19093	
Independent reflections	6049 [R(int) = 0.1333]	

Completeness to theta = 27.03°	99.0 %
Absorption correction	Spherical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6049 / 0 / 305
Goodness-of-fit on F^2	0.960
Final R indices [I>2sigma(I)]	R1 = 0.1001, wR2 = 0.2573
R indices (all data)	R1 = 0.1597, wR2 = 0.3085
Largest diff. peak and hole	1.663 and -1.988 e.Å^{-3}

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for [(H_5O_2)(Br_4-DB18C6)$_2$][$FeBr_4$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	2134(1)	1215(1)	157(1)	45(1)
Br(2)	981(1)	-92(1)	80(1)	65(1)
Br(3)	3955(1)	2362(1)	1549(1)	63(1)
Br(4)	8064(1)	-2861(1)	3436(2)	77(1)
Br(5)	4626(1)	3739(1)	3387(2)	80(1)
Fe(1)	5000	3068(1)	2500	40(1)
Br(7)	6800(1)	-4171(1)	2994(1)	79(1)
C(1)	3382(7)	167(7)	2612(8)	31(2)
C(2)	3147(7)	646(6)	1912(9)	33(3)
C(3)	2427(8)	578(6)	1134(9)	36(3)
C(4)	1959(7)	33(7)	1099(9)	38(3)
C(5)	2206(7)	-449(6)	1824(9)	32(3)
C(6)	2911(7)	-377(6)	2575(8)	28(2)
O(1)	3207(5)	-814(4)	3303(6)	32(2)
C(7)	2774(8)	-1438(6)	3206(8)	34(3)
C(8)	3272(8)	-1938(7)	3930(10)	39(3)
O(2)	3864(6)	-2174(5)	3741(7)	46(2)
C(9)	4330(10)	-2674(9)	4378(13)	58(4)
C(10)	4863(9)	-2996(7)	4076(12)	46(3)
O(3)	5322(6)	-2428(5)	4043(8)	50(3)
C(11)	5938(8)	-2575(8)	3900(9)	41(3)
C(12)	6059(10)	-3213(10)	3609(11)	55(4)
C(13)	6706(10)	-3288(8)	3465(10)	50(4)
C(14)	7181(10)	-2775(9)	3607(10)	54(4)
C(15)	7068(9)	-2119(8)	3921(9)	45(3)
C(16)	6423(8)	-2018(7)	4045(10)	40(3)
O(4)	6257(6)	-1427(5)	4350(7)	42(2)
C(17)	6702(8)	-832(8)	4432(9)	41(3)
C(18)	6380(8)	-249(7)	4766(9)	38(3)
O(5)	5628(5)	-77(5)	4051(6)	41(2)
C(19)	5366(7)	552(7)	4234(9)	36(3)
C(20)	4612(7)	722(7)	3439(10)	38(3)
O(6)	4075(5)	202(4)	3386(6)	35(2)
O(7)	344(6)	3847(5)	2056(7)	46(2)

Bond lengths [Å] and angles [°] for $[(H_5O_2)(Br_4\text{-}DB18C6)_2][FeBr_4]$.

Br(1)-C(3)	1.887(13)	C(8)-O(2)	1.423(15)
Br(2)-C(4)	1.894(14)	O(2)-C(9)	1.405(18)
Br(3)-Fe(1)	2.357(2)	C(9)-C(10)	1.50(2)
Br(4)-C(14)	1.905(16)	C(10)-O(3)	1.441(17)
Br(5)-Fe(1)	2.319(2)	O(3)-C(11)	1.377(15)
Fe(1)-Br(5)#1	2.319(2)	C(11)-C(16)	1.39(2)
Fe(1)-Br(3)#1	2.357(2)	C(11)-C(12)	1.38(2)
Br(7)-C(13)	1.917(15)	C(12)-C(13)	1.42(2)
C(1)-C(2)	1.377(17)	C(13)-C(14)	1.31(2)
C(1)-O(6)	1.370(15)	C(14)-C(15)	1.42(2)
C(1)-C(6)	1.386(16)	C(15)-C(16)	1.400(18)
C(2)-C(3)	1.407(19)	C(16)-O(4)	1.347(17)
C(3)-C(4)	1.384(19)	O(4)-C(17)	1.414(17)
C(4)-C(5)	1.409(18)	C(17)-C(18)	1.517(19)
C(5)-C(6)	1.371(18)	C(18)-O(5)	1.439(16)
C(6)-O(1)	1.355(14)	O(5)-C(19)	1.407(16)
O(1)-C(7)	1.442(15)	C(19)-C(20)	1.491(19)
C(7)-C(8)	1.491(18)	C(20)-O(6)	1.435(14)
Br(5)#1-Fe(1)-Br(5)	112.26(18)	O(2)-C(9)-C(10)	110.7(13)
Br(5)#1-Fe(1)-Br(3)	110.11(7)	O(3)-C(10)-C(9)	104.8(12)
Br(5)-Fe(1)-Br(3)	107.46(7)	C(11)-O(3)-C(10)	118.4(11)
Br(5)#1-Fe(1)-Br(3)#1	107.46(7)	C(16)-C(11)-O(3)	114.2(12)
Br(5)-Fe(1)-Br(3)#1	110.11(7)	C(16)-C(11)-C(12)	122.1(13)
Br(3)-Fe(1)-Br(3)#1	109.44(15)	O(3)-C(11)-C(12)	123.7(14)
C(2)-C(1)-O(6)	123.1(11)	C(11)-C(12)-C(13)	117.8(16)
C(2)-C(1)-C(6)	120.9(11)	C(14)-C(13)-C(12)	121.7(15)
O(6)-C(1)-C(6)	116.0(10)	C(14)-C(13)-Br(7)	123.0(12)
C(1)-C(2)-C(3)	120.1(11)	C(12)-C(13)-Br(7)	115.2(14)
C(4)-C(3)-C(2)	118.8(12)	C(13)-C(14)-C(15)	120.9(14)
C(4)-C(3)-Br(1)	121.7(11)	C(13)-C(14)-Br(4)	122.8(13)
C(2)-C(3)-Br(1)	119.5(10)	C(15)-C(14)-Br(4)	116.3(13)
C(3)-C(4)-C(5)	120.4(12)	C(16)-C(15)-C(14)	119.1(15)
C(3)-C(4)-Br(2)	121.4(10)	O(4)-C(16)-C(11)	116.6(11)
C(5)-C(4)-Br(2)	118.3(10)	O(4)-C(16)-C(15)	124.9(14)
C(6)-C(5)-C(4)	120.1(11)	C(11)-C(16)-C(15)	118.4(13)
O(1)-C(6)-C(5)	124.9(10)	C(16)-O(4)-C(17)	118.0(10)
O(1)-C(6)-C(1)	115.4(11)	O(4)-C(17)-C(18)	106.5(10)
C(5)-C(6)-C(1)	119.7(11)	O(5)-C(18)-C(17)	108.9(11)
C(6)-O(1)-C(7)	115.5(9)	C(19)-O(5)-C(18)	111.6(10)
O(1)-C(7)-C(8)	108.3(10)	O(5)-C(19)-C(20)	108.7(10)
O(2)-C(8)-C(7)	109.5(11)	O(6)-C(20)-C(19)	108.0(10)
C(9)-O(2)-C(8)	112.1(11)	C(1)-O(6)-C(20)	118.8(10)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,y,-z+1/2

Anisotropic displacement parameters ($Å^2x\ 10^3$)for $[(H_5O_2)(Br_4\text{-}DB18C6)_2][FeBr_4]$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	48(1)	46(1)	44(1)	14(1)	23(1)	11(1)
Br(2)	40(1)	78(1)	53(1)	16(1)	2(1)	-7(1)
Br(3)	76(1)	60(1)	48(1)	-9(1)	24(1)	-28(1)

Br(4)	80(1)	97(1)	84(1)	40(1)	63(1)	53(1)
Br(5)	58(1)	99(2)	78(1)	-35(1)	27(1)	14(1)
Fe(1)	40(2)	39(1)	40(1)	0	19(1)	0
Br(7)	102(2)	73(1)	67(1)	-3(1)	43(1)	39(1)
C(1)	22(6)	43(7)	25(5)	4(5)	9(5)	-4(5)
C(2)	36(7)	25(5)	44(7)	-14(5)	22(6)	-6(5)
C(3)	47(8)	34(6)	42(7)	2(5)	33(6)	10(5)
C(4)	28(6)	49(7)	39(7)	-1(6)	18(6)	10(6)
C(5)	29(6)	31(6)	41(7)	3(5)	20(6)	0(5)
C(6)	32(6)	23(5)	32(6)	2(4)	19(5)	3(4)
O(1)	33(5)	32(4)	27(4)	6(3)	12(4)	-2(3)
C(7)	44(7)	37(6)	26(6)	5(5)	22(6)	0(5)
C(8)	35(7)	36(7)	50(8)	-9(6)	24(6)	-4(5)
O(2)	50(6)	49(6)	51(6)	11(5)	34(5)	13(5)
C(9)	52(10)	59(10)	75(11)	33(8)	40(9)	19(8)
C(10)	46(8)	30(7)	65(9)	13(6)	28(7)	4(6)
O(3)	58(7)	35(5)	72(7)	4(5)	44(6)	6(4)
C(11)	39(7)	57(8)	38(7)	10(6)	28(6)	18(6)
C(12)	51(10)	76(11)	41(8)	11(7)	23(7)	17(8)
C(13)	64(10)	54(9)	32(7)	2(6)	22(7)	23(8)
C(14)	58(10)	75(11)	35(7)	25(7)	28(7)	27(9)
C(15)	51(9)	57(8)	39(7)	29(6)	32(7)	28(7)
C(16)	37(7)	47(8)	44(7)	20(6)	24(6)	17(6)
O(4)	44(6)	42(5)	55(6)	4(4)	35(5)	5(4)
C(17)	38(7)	60(9)	33(7)	0(6)	22(6)	-5(6)
C(18)	38(7)	44(7)	32(6)	6(5)	18(6)	8(6)
O(5)	36(5)	46(5)	35(5)	-4(4)	13(4)	5(4)
C(19)	34(7)	37(6)	37(7)	-2(5)	18(6)	-10(5)
C(20)	33(7)	30(6)	54(8)	1(5)	24(6)	-5(5)
O(6)	35(5)	31(4)	40(5)	0(4)	18(4)	-5(4)
O(7)	51(6)	44(5)	44(5)	-1(4)	23(5)	-4(4)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $[(H_5O_2)(Br_4$-DB18C6)$_2][FeBr_4]$.

	x	y	z	U(eq)
H(2)	3464	1015	1953	40
H(5)	1891	-817	1794	39
H(7A)	2610	-1638	2602	40
H(7B)	2324	-1332	3272	40
H(8A)	3493	-1713	4526	46
H(8B)	2970	-2329	3945	46
H(9A)	4012	-3032	4439	70
H(9B)	4623	-2457	4973	70
H(10A)	5180	-3346	4511	55
H(10B)	4581	-3209	3476	55
H(12)	5726	-3581	3511	66
H(15)	7418	-1761	4042	54
H(17A)	6670	-714	3843	50
H(17B)	7234	-915	4868	50
H(18A)	6353	-394	5313	45
H(18B)	6710	154	4918	45
H(19A)	5730	920	4326	43
H(19B)	5314	509	4788	43
H(20A)	4443	1177	3523	45

H(20B)	4654	729	2876	45
H(21)	0	320(80)	2500	10(30)

V.2.5. Crystal data and structure refinement for [$(H_3O)(Br_4$-DB18C6) $(Br_3)Br_2$] 5

In this structure the largest diffraction peak of the remaining electron density is centered on an bromide atom Br5 which is on a symmetry position. The estimation of the remaining electron density may be overestimated due to this special fact.

Empirical formula	C_{20} H_{23} Br_9 O_7	
Formula weight	1090.53	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.1379(12) Å	α= 80.412(11)°.
	b = 13.0758(19) Å	β= 87.039(11)°.
	c = 14.342(2) Å	γ = 77.757(11)°.
Volume	1470.4(4) $Å^3$	
Z	2	
Density (calculated)	2.283 Mg/m^3	
Absorption coefficient	10.945 mm^{-1}	
F(000)	950	
Theta range for data collection	1.44 to 27.16°.	
Index ranges	-10<=h<=10, -16<=k<=16, -18<=l<=17	
Reflections collected	11849	
Independent reflections	6049 [R(int) = 0.0878]	
Completeness to theta = 27.16°	92.5 %	
Absorption correction	Spherical	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6049 / 0 / 325	

Goodness-of-fit on F^2	1.036
Final R indices [I>2sigma(I)]	R1 = 0.0727, wR2 = 0.1782
R indices (all data)	R1 = 0.1378, wR2 = 0.2147
Largest diff. peak and hole	1.340 and -1.284 e.Å^{-3}

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for [(H_3O)(Br_4-DB18C6) (Br_3)Br_2]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	3510(2)	2802(1)	2251(1)	60(1)
Br(2)	1191(2)	1775(1)	3000(1)	61(1)
Br(3)	14036(2)	411(1)	4410(1)	64(1)
Br(4)	14027(2)	-1157(1)	2434(1)	73(1)
Br(5)	13680(2)	6082(1)	-6520(1)	62(1)
Br(6)	13649(2)	7472(1)	-4707(1)	64(1)
Br(7)	15495(2)	-2727(1)	801(1)	72(1)
Br(8)	5470(30)	3966(19)	1447(17)	103(6)
Br(8)	5680(30)	3684(19)	1578(15)	88(4)
C(1)	11770(16)	4302(8)	-4261(9)	44(3)
C(2)	12365(15)	4652(9)	-5116(9)	44(3)
C(3)	12884(16)	5611(9)	-5282(10)	50(3)
C(4)	12881(16)	6178(9)	-4549(9)	47(3)
C(5)	12259(16)	5840(9)	-3689(10)	50(3)
C(6)	11662(15)	4907(9)	-3526(8)	43(3)
O(1)	10967(11)	4481(6)	-2681(6)	50(2)
C(7)	10496(18)	5171(9)	-1999(10)	52(3)
C(8)	9345(17)	4696(9)	-1296(9)	50(3)
O(2)	10227(10)	3734(6)	-829(6)	47(2)
C(9)	9176(17)	3180(10)	-172(10)	53(3)
C(10)	10298(18)	2197(10)	328(9)	53(3)
O(3)	10819(11)	1492(6)	-355(5)	47(2)
C(11)	11894(16)	539(8)	-36(9)	45(3)
C(12)	12372(16)	211(9)	891(8)	47(3)
C(13)	13424(18)	-765(10)	1139(10)	54(3)
C(14)	14022(16)	-1405(9)	478(9)	47(3)
C(15)	13533(15)	-1087(9)	-456(9)	46(3)
C(16)	12441(15)	-133(9)	-717(8)	42(3)
O(4)	11816(11)	251(6)	-1586(6)	49(2)
C(17)	12398(18)	-364(9)	-2344(8)	47(3)
C(18)	11312(19)	167(9)	-3178(8)	52(3)
O(5)	11629(10)	1219(6)	-3457(5)	43(2)
C(19)	10613(17)	1761(9)	-4256(8)	46(3)
C(20)	11359(16)	2694(9)	-4719(8)	45(3)
O(6)	11178(11)	3378(6)	-4013(6)	46(2)
O(7)	10727(14)	2409(7)	-2211(6)	62(3)

Bond lengths [Å] and angles [°] for [(H_3O)(Br_4-DB18C6) (Br_3)Br_2].

Br(1)-Br(8)	2.39(2)	Br(5)-C(3)	1.908(14)
Br(1)-Br(8)	2.54(2)	Br(6)-C(4)	1.900(12)
Br(1)-Br(2)	2.627(2)	Br(7)-C(14)	1.884(12)
Br(3)-Br(3)#1	2.331(3)	C(1)-C(2)	1.339(17)
Br(4)-C(13)	1.904(13)	C(1)-O(6)	1.379(13)

C(1)-C(6)	1.409(16)	C(11)-C(12)	1.376(16)
C(2)-C(3)	1.386(16)	C(11)-C(16)	1.414(16)
C(3)-C(4)	1.383(17)	C(12)-C(13)	1.381(18)
C(4)-C(5)	1.349(18)	C(13)-C(14)	1.371(19)
C(5)-C(6)	1.386(16)	C(14)-C(15)	1.387(17)
C(6)-O(1)	1.392(15)	C(15)-C(16)	1.376(16)
O(1)-C(7)	1.423(14)	C(16)-O(4)	1.346(14)
C(7)-C(8)	1.490(19)	O(4)-C(17)	1.459(13)
C(8)-O(2)	1.390(14)	C(17)-C(18)	1.506(16)
O(2)-C(9)	1.448(15)	C(18)-O(5)	1.441(12)
C(9)-C(10)	1.505(18)	O(5)-C(19)	1.442(14)
C(10)-O(3)	1.443(14)	C(19)-C(20)	1.517(16)
O(3)-C(11)	1.384(13)	C(20)-O(6)	1.441(13)

Br(8)-Br(1)-Br(8)	8.5(12)	C(11)-O(3)-C(10)	116.3(9)
Br(8)-Br(1)-Br(2)	178.2(6)	C(12)-C(11)-O(3)	123.9(11)
Br(8)-Br(1)-Br(2)	173.2(6)	C(12)-C(11)-C(16)	120.1(11)
C(2)-C(1)-O(6)	125.8(11)	O(3)-C(11)-C(16)	116.0(10)
C(2)-C(1)-C(6)	120.2(11)	C(11)-C(12)-C(13)	119.2(12)
O(6)-C(1)-C(6)	114.0(11)	C(14)-C(13)-C(12)	121.3(12)
C(1)-C(2)-C(3)	120.2(11)	C(14)-C(13)-Br(4)	122.2(10)
C(4)-C(3)-C(2)	120.0(12)	C(12)-C(13)-Br(4)	116.5(10)
C(4)-C(3)-Br(5)	121.7(9)	C(13)-C(14)-C(15)	119.9(12)
C(2)-C(3)-Br(5)	118.3(10)	C(13)-C(14)-Br(7)	121.7(9)
C(5)-C(4)-C(3)	120.1(11)	C(15)-C(14)-Br(7)	118.3(10)
C(5)-C(4)-Br(6)	117.3(9)	C(16)-C(15)-C(14)	120.0(12)
C(3)-C(4)-Br(6)	122.5(10)	O(4)-C(16)-C(15)	126.1(11)
C(4)-C(5)-C(6)	120.4(12)	O(4)-C(16)-C(11)	114.5(10)
C(5)-C(6)-O(1)	126.1(11)	C(15)-C(16)-C(11)	119.4(11)
C(5)-C(6)-C(1)	119.0(12)	C(16)-O(4)-C(17)	118.0(9)
O(1)-C(6)-C(1)	114.9(10)	O(4)-C(17)-C(18)	105.8(9)
C(6)-O(1)-C(7)	116.2(9)	O(5)-C(18)-C(17)	107.9(9)
O(1)-C(7)-C(8)	107.6(10)	C(18)-O(5)-C(19)	110.0(8)
O(2)-C(8)-C(7)	108.7(11)	O(5)-C(19)-C(20)	108.2(9)
C(8)-O(2)-C(9)	112.6(10)	O(6)-C(20)-C(19)	104.6(10)
O(2)-C(9)-C(10)	107.2(11)	C(1)-O(6)-C(20)	116.3(9)
O(3)-C(10)-C(9)	107.3(10)		

Symmetry transformations used to generate equivalent atoms:
#1 -x+3,-y,-z+1

Anisotropic displacement parameters ($Å^2 x\ 10^3$)for $[(H_3O)(Br_4\text{-}DB18C6)\ (Br_3)Br_2]$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	54(1)	68(1)	61(1)	-26(1)	-6(1)	-5(1)
Br(2)	83(1)	49(1)	55(1)	-11(1)	-2(1)	-21(1)
Br(3)	83(1)	55(1)	60(1)	-20(1)	8(1)	-20(1)
Br(4)	109(1)	57(1)	51(1)	-2(1)	-22(1)	-16(1)
Br(5)	61(1)	62(1)	59(1)	5(1)	7(1)	-17(1)
Br(6)	63(1)	48(1)	85(1)	-6(1)	2(1)	-24(1)
Br(7)	76(1)	58(1)	75(1)	-11(1)	-17(1)	3(1)
Br(8)	63(4)	161(16)	108(8)	-53(9)	23(5)	-54(8)
Br(8)	90(9)	116(7)	84(5)	-48(6)	24(5)	-61(7)
C(1)	53(7)	32(6)	46(7)	-3(5)	3(6)	-8(5)
C(2)	42(7)	44(6)	45(7)	-11(5)	-1(6)	-2(5)

C(3)	45(7)	40(6)	60(8)	-5(6)	8(6)	-3(5)
C(4)	51(7)	37(6)	52(7)	-8(5)	-9(6)	-6(5)
C(5)	57(8)	33(6)	63(8)	-8(5)	-3(7)	-18(5)
C(6)	44(7)	43(6)	38(6)	-6(5)	-7(6)	2(5)
O(1)	65(6)	41(4)	47(5)	-18(4)	10(4)	-12(4)
C(7)	63(8)	31(6)	61(8)	-16(5)	12(7)	-8(5)
C(8)	55(8)	43(6)	54(7)	-17(6)	7(7)	-5(5)
O(2)	43(5)	39(4)	54(5)	-1(4)	11(4)	-6(3)
C(9)	57(8)	47(7)	61(8)	-14(6)	9(7)	-17(6)
C(10)	65(9)	50(7)	47(7)	-17(6)	12(7)	-18(6)
O(3)	67(6)	35(4)	37(4)	-6(3)	6(4)	-10(4)
C(11)	51(7)	30(5)	53(7)	-2(5)	0(6)	-11(5)
C(12)	62(8)	45(7)	37(6)	-5(5)	4(6)	-24(6)
C(13)	63(8)	49(7)	54(8)	1(6)	-6(7)	-24(6)
C(14)	56(8)	39(6)	48(7)	-5(5)	2(6)	-18(5)
C(15)	41(7)	45(6)	52(7)	-3(5)	-5(6)	-13(5)
C(16)	48(7)	47(6)	38(6)	-14(5)	14(6)	-22(5)
O(4)	66(6)	43(4)	40(4)	-13(4)	-5(4)	-6(4)
C(17)	72(9)	34(6)	37(6)	-10(5)	7(6)	-10(5)
C(18)	87(10)	34(6)	40(6)	-9(5)	0(7)	-25(6)
O(5)	53(5)	37(4)	42(4)	-6(3)	-3(4)	-14(3)
C(19)	59(8)	48(7)	40(6)	-16(5)	-3(6)	-22(6)
C(20)	52(7)	40(6)	44(6)	-6(5)	-10(6)	-13(5)
O(6)	61(5)	42(4)	43(4)	-16(4)	2(4)	-23(4)
O(7)	92(7)	51(5)	43(5)	-11(4)	0(5)	-16(5)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)for [$(H_3O)(Br_4$-DB18C6) $(Br_3)Br_2$].

	x	y	z	U(eq)
H(2)	12429	4252	-5600	53
H(5)	12231	6234	-3204	60
H(7A)	9930	5866	-2304	62
H(7B)	11485	5246	-1689	62
H(8A)	8931	5171	-845	60
H(8B)	8389	4585	-1614	60
H(9A)	8307	2993	-508	64
H(9B)	8640	3627	281	64
H(10A)	11271	2375	575	63
H(10B)	9693	1862	851	63
H(12)	11991	642	1345	56
H(17A)	12274	-1092	-2157	57
H(17B)	13572	-362	-2498	57
H(18A)	11582	-234	-3697	62
H(18B)	10135	203	-3007	62
H(19A)	9465	2008	-4049	56
H(19B)	10605	1282	-4704	56
H(20A)	12534	2463	-4886	53
H(20B)	10753	3055	-5286	53

V.2.6. Crystal data and structure refinement for [K(Br2-DB18C6) (Br_3)] 6

The goodness-of-fit on F^2 is rather high because Br2-DB18C6 are not as perfectly organized as described due to the unselectivity of bromination reaction. In reality, a third of the molecules is brominated in the other position.

Empirical formula	C_{20} H_{22} Br_5 K_1 O_7	
Formula weight	813.008	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.153(2) Å	α= 97.806(14)°.
	b = 12.041(2) Å	β= 99.785(18)°.
	c = 14.662(3) Å	γ = 105.341(17)°.
Volume	1343.0(5) $Å^3$	
Z	1	
Density (calculated)	2.011 Mg/m^3	
Absorption coefficient	7.673 mm^{-1}	
F(000)	784	
Theta range for data collection	1.79 to 20.60°.	
Index ranges	-7<=h<=5, -11<=k<=11, -14<=l<=13	
Reflections collected	2488	
Independent reflections	1823 [R(int) = 0.0465]	
Completeness to theta = 20.60°	66.9 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1823 / 0 / 315	
Goodness-of-fit on F^2	2.356	
Final R indices [I>2sigma(I)]	R1 = 0.0996, wR2 = 0.2892	
R indices (all data)	R1 = 0.1121, wR2 = 0.2961	
Largest diff. peak and hole	0.912 and -0.712 $e.Å^{-3}$	

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [K(Br2-DB18C6) (Br_3)]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	3730(5)	2804(2)	3028(2)	113(2)
Br(2)	3242(5)	695(2)	2385(2)	98(1)
Br(3)	2529(11)	-1482(4)	1641(3)	195(3)
Br(4)	7727(5)	4657(2)	5624(2)	102(1)
Br(5)	450(6)	5088(3)	-2072(3)	78(1)
Br(6)	461(15)	3480(8)	-746(11)	141(6)
K(1)	6469(15)	-1221(5)	2484(5)	131(4)
C(1)	7720(40)	10803(19)	4750(20)	67(8)
C(2)	7430(40)	11200(16)	5618(18)	60(7)
C(3)	7480(40)	12331(17)	5890(20)	79(9)
C(4)	7770(40)	13076(15)	5261(19)	75(9)
C(5)	8060(40)	12718(15)	4363(17)	57(7)
C(6)	7950(50)	11629(19)	4100(20)	91(12)
O(1)	8210(20)	11141(11)	3229(11)	67(5)
C(7)	8390(30)	11882(16)	2569(19)	65(7)
C(8)	8760(40)	11300(20)	1720(20)	87(10)
O(2)	7410(30)	10279(12)	1253(12)	72(5)
C(9)	7740(50)	9640(30)	470(20)	89(9)
C(10)	6120(50)	8602(19)	-70(20)	83(9)
O(3)	5880(30)	7786(12)	568(11)	71(6)
C(11)	4500(50)	6780(20)	260(20)	79(12)
C(12)	3470(50)	6480(20)	-654(18)	84(11)
C(13)	2190(50)	5443(19)	-913(17)	79(10)
C(14)	2040(60)	4750(30)	-270(50)	170(30)
C(15)	3020(60)	5040(30)	626(19)	89(16)
C(16)	4410(40)	6030(20)	870(20)	75(10)
O(4)	5420(30)	6360(11)	1723(12)	75(6)
C(17)	5080(40)	5641(15)	2438(18)	75(9)
C(18)	6480(40)	6135(16)	3286(18)	80(9)
O(5)	6390(20)	7244(10)	3699(11)	65(5)
C(19)	7640(40)	7744(16)	4536(17)	72(8)
C(20)	7410(40)	8831(17)	5015(16)	80(10)
O(6)	7650(30)	9641(10)	4358(12)	85(7)
O(7)	8810(160)	-1650(50)	2400(30)	570(100)

Bond lengths [Å] and angles [°] for [K(Br2-DB18C6) (Br_3)].

Br(1)-Br(2)	2.487(4)	C(1)-C(6)	1.47(3)
Br(2)-Br(3)	2.569(5)	C(2)-C(3)	1.35(3)
Br(3)-K(1)	3.156(14)	C(3)-C(4)	1.38(3)
Br(4)-C(4)#1	1.92(2)	C(4)-C(5)	1.41(3)
Br(5)-C(13)	1.94(3)	C(4)-Br(4)#2	1.92(2)
Br(6)-C(14)	1.69(4)	C(5)-C(6)	1.29(3)
K(1)-O(7)	2.13(13)	C(6)-O(1)	1.40(3)
K(1)-O(6)#1	2.712(19)	O(1)-C(7)	1.40(3)
K(1)-O(5)#1	2.731(14)	O(1)-K(1)#2	2.790(15)
K(1)-O(2)#1	2.780(14)	C(7)-C(8)	1.46(4)
K(1)-O(1)#1	2.790(15)	C(8)-O(2)	1.41(3)
K(1)-O(3)#1	2.806(17)	O(2)-C(9)	1.40(3)
K(1)-O(4)#1	2.816(14)	O(2)-K(1)#2	2.780(14)
K(1)-C(19)#1	3.49(2)	C(9)-C(10)	1.56(4)
K(1)-C(9)#1	3.50(3)	C(9)-K(1)#2	3.50(3)
C(1)-C(2)	1.38(3)	C(10)-O(3)	1.45(3)
C(1)-O(6)	1.42(3)	O(3)-C(11)	1.37(3)

O(3)-K(1)#2	2.806(17)	O(4)-K(1)#2	2.816(14)
C(11)-C(16)	1.36(4)	C(17)-C(18)	1.47(4)
C(11)-C(12)	1.40(5)	C(18)-O(5)	1.42(3)
C(12)-C(13)	1.35(4)	O(5)-C(19)	1.40(3)
C(13)-C(14)	1.35(7)	O(5)-K(1)#2	2.731(14)
C(14)-C(15)	1.36(8)	C(19)-C(20)	1.47(3)
C(15)-C(16)	1.37(4)	C(19)-K(1)#2	3.49(2)
C(16)-O(4)	1.31(4)	C(20)-O(6)	1.46(2)
O(4)-C(17)	1.46(3)	O(6)-K(1)#2	2.712(18)

Br(1)-Br(2)-Br(3)	175.8(2)	C(19)#1-K(1)-C(9)#1	145.9(9)
Br(2)-Br(3)-K(1)	86.1(3)	C(2)-C(1)-O(6)	128.1(18)
O(7)-K(1)-O(6)#1	91.0(15)	C(2)-C(1)-C(6)	117(2)
O(7)-K(1)-O(5)#1	79.6(12)	O(6)-C(1)-C(6)	114(3)
O(6)#1-K(1)-O(5)#1	61.1(4)	C(3)-C(2)-C(1)	121.7(19)
O(7)-K(1)-O(2)#1	85.7(13)	C(2)-C(3)-C(4)	118(3)
O(6)#1-K(1)-O(2)#1	117.6(5)	C(3)-C(4)-C(5)	122.8(19)
O(5)#1-K(1)-O(2)#1	165.1(7)	C(3)-C(4)-Br(4)#2	118(2)
O(7)-K(1)-O(1)#1	93.4(16)	C(5)-C(4)-Br(4)#2	119.0(14)
O(6)#1-K(1)-O(1)#1	56.7(4)	C(6)-C(5)-C(4)	118(2)
O(5)#1-K(1)-O(1)#1	117.3(5)	C(5)-C(6)-O(1)	125.2(19)
O(2)#1-K(1)-O(1)#1	61.4(5)	C(5)-C(6)-C(1)	121(3)
O(7)-K(1)-O(3)#1	77.7(15)	O(1)-C(6)-C(1)	113(2)
O(6)#1-K(1)-O(3)#1	168.6(8)	C(6)-O(1)-C(7)	115.8(17)
O(5)#1-K(1)-O(3)#1	115.7(5)	C(6)-O(1)-K(1)#2	118.3(11)
O(2)#1-K(1)-O(3)#1	62.3(5)	C(7)-O(1)-K(1)#2	115.6(15)
O(1)#1-K(1)-O(3)#1	123.4(5)	O(1)-C(7)-C(8)	110.3(18)
O(7)-K(1)-O(4)#1	74.6(15)	O(2)-C(8)-C(7)	113(2)
O(6)#1-K(1)-O(4)#1	122.6(4)	C(9)-O(2)-C(8)	116(2)
O(5)#1-K(1)-O(4)#1	61.7(4)	C(9)-O(2)-K(1)#2	109.0(13)
O(2)#1-K(1)-O(4)#1	116.2(5)	C(8)-O(2)-K(1)#2	111.2(16)
O(1)#1-K(1)-O(4)#1	168.1(7)	O(2)-C(9)-C(10)	112(3)
O(3)#1-K(1)-O(4)#1	54.5(4)	O(2)-C(9)-K(1)#2	48.7(10)
O(7)-K(1)-Br(3)	150.8(15)	C(10)-C(9)-K(1)#2	84.2(14)
O(6)#1-K(1)-Br(3)	116.8(6)	O(3)-C(10)-C(9)	105(3)
O(5)#1-K(1)-Br(3)	105.2(5)	C(11)-O(3)-C(10)	116(2)
O(2)#1-K(1)-Br(3)	88.7(5)	C(11)-O(3)-K(1)#2	115.2(13)
O(1)#1-K(1)-Br(3)	108.8(5)	C(10)-O(3)-K(1)#2	116.3(13)
O(3)#1-K(1)-Br(3)	74.4(5)	C(16)-C(11)-O(3)	113(3)
O(4)#1-K(1)-Br(3)	82.4(5)	C(16)-C(11)-C(12)	123(3)
O(7)-K(1)-C(19)#1	73.6(13)	O(3)-C(11)-C(12)	123(2)
O(6)#1-K(1)-C(19)#1	41.9(5)	C(13)-C(12)-C(11)	119(2)
O(5)#1-K(1)-C(19)#1	22.0(5)	C(14)-C(13)-C(12)	117(3)
O(2)#1-K(1)-C(19)#1	148.7(7)	C(14)-C(13)-Br(5)	121(3)
O(1)#1-K(1)-C(19)#1	96.1(5)	C(12)-C(13)-Br(5)	121(2)
O(3)#1-K(1)-C(19)#1	132.1(6)	C(13)-C(14)-C(15)	125(3)
O(4)#1-K(1)-C(19)#1	81.1(5)	C(13)-C(14)-Br(6)	109(5)
Br(3)-K(1)-C(19)#1	120.6(5)	C(15)-C(14)-Br(6)	126(5)
O(7)-K(1)-C(9)#1	72.7(14)	C(14)-C(15)-C(16)	119(3)
O(6)#1-K(1)-C(9)#1	133.9(7)	O(4)-C(16)-C(11)	120(3)
O(5)#1-K(1)-C(9)#1	147.8(8)	O(4)-C(16)-C(15)	122(2)
O(2)#1-K(1)-C(9)#1	22.3(6)	C(11)-C(16)-C(15)	116(4)
O(1)#1-K(1)-C(9)#1	80.9(6)	C(16)-O(4)-C(17)	119.9(19)
O(3)#1-K(1)-C(9)#1	42.8(6)	C(16)-O(4)-K(1)#2	116.3(13)
O(4)#1-K(1)-C(9)#1	94.9(6)	C(17)-O(4)-K(1)#2	112.3(13)
Br(3)-K(1)-C(9)#1	92.0(6)	O(4)-C(17)-C(18)	109(2)

O(5)-C(18)-C(17)	109(2)	C(20)-C(19)-K(1)#2	84.1(12)
C(19)-O(5)-C(18)	112.5(18)	O(6)-C(20)-C(19)	105.3(19)
C(19)-O(5)-K(1)#2	111.2(10)	C(1)-O(6)-C(20)	113.4(18)
C(18)-O(5)-K(1)#2	113.5(13)	C(1)-O(6)-K(1)#2	121.4(14)
O(5)-C(19)-C(20)	112(2)	C(20)-O(6)-K(1)#2	118.8(13)
O(5)-C(19)-K(1)#2	46.9(8)		

Symmetry transformations used to generate equivalent atoms:
#1 x,y-1,z #2 x,y+1,z

Anisotropic displacement parameters ($Å^2$x 10^3)for [K(Br2-DB18C6) (Br_3)]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	119(3)	98(2)	116(3)	50(2)	5(2)	20(2)
Br(2)	120(3)	128(2)	79(2)	60(2)	33(2)	63(2)
Br(3)	373(11)	151(3)	113(3)	46(3)	52(5)	153(4)
Br(4)	153(4)	66(2)	96(2)	25(1)	24(2)	42(2)
Br(5)	85(4)	82(2)	61(3)	9(2)	-6(2)	30(2)
Br(6)	70(8)	115(6)	190(14)	-76(7)	-39(8)	41(5)
K(1)	231(11)	76(3)	48(4)	23(3)	-15(5)	4(4)
C(1)	60(20)	88(15)	60(20)	41(13)	3(16)	32(12)
C(2)	70(20)	58(12)	62(18)	29(10)	8(14)	27(10)
C(3)	100(30)	56(13)	76(19)	29(12)	8(18)	10(12)
C(4)	80(20)	33(10)	90(20)	24(11)	-2(17)	-2(10)
C(5)	80(20)	43(12)	60(18)	17(9)	0(15)	35(10)
C(6)	120(30)	65(15)	60(20)	36(13)	-13(19)	-11(15)
O(1)	87(15)	75(8)	46(11)	28(8)	9(10)	32(7)
C(7)	58(18)	49(10)	90(19)	32(12)	16(15)	12(10)
C(8)	42(19)	83(15)	140(30)	67(17)	-7(18)	17(13)
O(2)	90(17)	91(10)	57(11)	35(8)	26(11)	47(9)
C(9)	70(30)	160(20)	60(20)	54(17)	2(19)	61(18)
C(10)	110(30)	95(16)	52(17)	25(12)	-10(18)	49(14)
O(3)	101(19)	75(9)	43(10)	25(7)	6(11)	33(9)
C(11)	120(30)	80(16)	60(20)	0(13)	60(20)	49(15)
C(12)	140(40)	99(19)	40(19)	9(13)	30(20)	87(19)
C(13)	110(30)	51(13)	50(16)	-12(11)	-34(16)	16(13)
C(14)	130(40)	80(20)	290(80)	-90(30)	130(60)	20(20)
C(15)	150(50)	69(19)	34(16)	-16(12)	0(20)	30(20)
C(16)	70(30)	95(18)	90(30)	51(17)	40(20)	48(15)
O(4)	117(18)	69(9)	38(10)	16(7)	0(12)	35(8)
C(17)	130(30)	50(10)	72(19)	21(11)	50(20)	51(12)
C(18)	120(30)	49(11)	63(17)	18(10)	-7(18)	24(12)
O(5)	83(14)	56(8)	55(10)	19(7)	-4(11)	28(7)
C(19)	80(20)	59(12)	75(19)	34(11)	2(17)	18(11)
C(20)	140(30)	66(13)	48(15)	40(11)	16(17)	48(14)
O(6)	150(20)	41(7)	69(11)	25(7)	32(12)	25(8)
O(7)	1000(200)	380(70)	90(30)	60(40)	110(80)	-180(100)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [K(Br2-DB18C6) (Br_3)].

	x	y	z	U(eq)
H(55)	2600(900)	5600(400)	-1600(500)	0(150)

H(66)	1200(300)	4570(180)	-300(150)	110(70)
H(2)	7180	10678	6024	72
H(3)	7330	12598	6488	95
H(5)	8317	13253	3969	69
H(7A)	9324	12596	2843	78
H(7B)	7319	12093	2404	78
H(8A)	8949	11840	1293	105
H(8B)	9822	11087	1896	105
H(9A)	8704	9340	668	107
H(9B)	8084	10168	39	107
H(10A)	5101	8870	-219	99
H(10B)	6333	8238	-655	99
H(12)	3656	6982	-1079	101
H(15)	2746	4561	1063	106
H(17A)	5048	4842	2198	90
H(17B)	3969	5630	2590	90
H(18A)	6359	5610	3734	96
H(18B)	7603	6219	3121	96
H(19A)	8791	7915	4395	86
H(19B)	7566	7184	4956	86
H(20A)	6251	8686	5147	96
H(20B)	8262	9148	5603	96

V.2.7. Crystal data and structure refinement for $[(Me_3NPh)(DB_{18}C_6)(Br_3)]$ 7

Empirical formula	C_{29} H_{38} Br3 N_1 O_6	
Formula weight	1472.67	
Temperature	240(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/m$	
Unit cell dimensions	a = 9.5196(9) Å	α= 90°.
	b = 16.124(2) Å	β= 106.457(8)°.
	c = 10.8074(11) Å	γ = 90°.
Volume	1590.9(3) $Å^3$	
Z	2	
Density (calculated)	1.537 Mg/m^3	
Absorption coefficient	3.843 mm^{-1}	
F(000)	744	
Theta range for data collection	1.96 to 27.21°.	
Index ranges	-12<=h<=10, -20<=k<=20, -13<=l<=13	

Reflections collected	12906
Independent reflections	3572 [R(int) = 0.1113]
Completeness to theta = 27.21°	97.2 %
Absorption correction	Spherical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3572 / 0 / 190
Goodness-of-fit on F^2	0.997
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.0864
R indices (all data)	R1 = 0.1141, wR2 = 0.1057
Largest diff. peak and hole	0.313 and -0.401 e.Å^{-3}

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for [(Me_3NPh)($DB_{18}C_6$)()]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	10000	0	10000	52(1)
Br(2)	12500(1)	425(1)	11451(1)	66(1)
C(1)	2794(6)	-2076(3)	11366(5)	63(2)
C(2)	3109(5)	-1644(3)	10366(5)	54(1)
C(3)	3385(5)	-2066(3)	9350(4)	41(1)
O(1)	3632(3)	-1686(2)	8292(3)	48(1)
C(4)	3549(6)	-800(3)	8241(5)	51(1)
C(5)	3611(6)	-527(3)	6938(5)	53(1)
O(2)	2279(4)	-730(2)	6007(3)	53(1)
C(6)	2306(6)	-547(3)	4741(5)	62(2)
C(7)	894(6)	-815(3)	3810(5)	61(2)
O(3)	847(4)	-1699(2)	3794(3)	54(1)
C(8)	-325(5)	-2062(3)	2907(5)	48(1)
C(9)	-1463(6)	-1649(4)	2065(5)	60(2)
C(10)	-2605(6)	-2081(4)	1236(6)	71(2)
C(11)	3592(7)	2500	3129(7)	44(2)
C(12)	5017(7)	2500	3921(7)	49(2)
C(13)	5324(8)	2500	5239(8)	63(2)
C(14)	4178(10)	2500	5769(8)	91(3)
C(15)	2757(9)	2500	5005(8)	77(3)
C(16)	2459(7)	2500	3678(7)	46(2)
N(1)	892(6)	2500	2884(5)	47(1)
C(17)	711(8)	2500	1468(7)	53(2)
C(18)	126(6)	1738(4)	3193(6)	70(2)

Bond lengths [Å] and angles [°] for [(Me_3NPh)($DB_{18}C_6$)(Br_3)].

Br(1)-Br(2)	2.5454(6)	C(3)-C(3)#2	1.401(9)
Br(1)-Br(2)#1	2.5454(6)	O(1)-C(4)	1.431(5)
C(1)-C(1)#2	1.367(11)	C(4)-C(5)	1.493(7)
C(1)-C(2)	1.388(8)	C(5)-O(2)	1.416(6)
C(2)-C(3)	1.380(7)	O(2)-C(6)	1.407(6)
C(3)-O(1)	1.374(6)	C(6)-C(7)	1.496(7)

C(7)-O(3)	1.427(6)	C(12)-C(13)	1.370(10)
O(3)-C(8)	1.378(6)	C(13)-C(14)	1.369(11)
C(8)-C(9)	1.373(7)	C(14)-C(15)	1.371(11)
C(8)-C(8)#2	1.413(9)	C(15)-C(16)	1.380(10)
C(9)-C(10)	1.385(7)	C(16)-N(1)	1.496(8)
C(10)-C(10)#2	1.353(12)	N(1)-C(17)	1.491(9)
C(11)-C(16)	1.371(9)	N(1)-C(18)	1.514(6)
C(11)-C(12)	1.384(9)	N(1)-C(18)#3	1.514(6)

Br(2)-Br(1)-Br(2)#1	180.0	C(8)-C(9)-C(10)	120.8(5)
C(1)#2-C(1)-C(2)	120.2(3)	C(10)#2-C(10)-C(9)	120.2(3)
C(3)-C(2)-C(1)	120.3(5)	C(16)-C(11)-C(12)	119.0(6)
O(1)-C(3)-C(2)	124.0(4)	C(13)-C(12)-C(11)	121.8(7)
O(1)-C(3)-C(3)#2	116.4(2)	C(14)-C(13)-C(12)	118.3(7)
C(2)-C(3)-C(3)#2	119.6(3)	C(13)-C(14)-C(15)	121.0(8)
C(3)-O(1)-C(4)	117.1(4)	C(14)-C(15)-C(16)	120.2(7)
O(1)-C(4)-C(5)	108.3(4)	C(11)-C(16)-C(15)	119.6(6)
O(2)-C(5)-C(4)	109.6(4)	C(11)-C(16)-N(1)	122.1(6)
C(6)-O(2)-C(5)	112.5(4)	C(15)-C(16)-N(1)	118.3(6)
O(2)-C(6)-C(7)	109.4(4)	C(17)-N(1)-C(16)	113.3(5)
O(3)-C(7)-C(6)	108.4(4)	C(17)-N(1)-C(18)	107.7(4)
C(8)-O(3)-C(7)	116.7(4)	C(16)-N(1)-C(18)	109.8(4)
C(9)-C(8)-O(3)	125.9(5)	C(17)-N(1)-C(18)#3	107.7(4)
C(9)-C(8)-C(8)#2	119.0(3)	C(16)-N(1)-C(18)#3	109.8(4)
O(3)-C(8)-C(8)#2	115.1(2)	C(18)-N(1)-C(18)#3	108.6(7)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y,-z+2 #2 x,-y-1/2,z #3 x,-y+1/2,z

Anisotropic displacement parameters ($Å^2$x 10^3)for [(Me_3NPh)($DB_{18}C_6$)(Br_3)]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	49(1)	49(1)	58(1)	1(1)	15(1)	-2(1)
Br(2)	52(1)	57(1)	82(1)	-9(1)	7(1)	-5(1)
C(1)	56(3)	94(4)	40(3)	-11(3)	14(3)	2(3)
C(2)	50(3)	68(4)	40(3)	-8(3)	6(3)	1(3)
C(3)	32(2)	59(3)	30(3)	-2(2)	6(2)	-2(2)
O(1)	50(2)	51(2)	46(2)	0(2)	18(2)	2(2)
C(4)	54(3)	47(3)	53(3)	-6(2)	14(3)	-6(2)
C(5)	59(3)	42(3)	57(3)	3(2)	14(3)	-3(2)
O(2)	53(2)	62(2)	41(2)	4(2)	10(2)	-4(2)
C(6)	69(4)	57(3)	58(4)	17(3)	14(3)	-5(3)
C(7)	69(4)	61(3)	45(3)	14(3)	5(3)	5(3)
O(3)	56(2)	55(2)	45(2)	6(2)	4(2)	-2(2)
C(8)	40(3)	68(3)	35(3)	2(2)	10(2)	0(2)
C(9)	54(3)	75(4)	52(3)	6(3)	16(3)	5(3)
C(10)	50(3)	103(5)	55(4)	9(3)	5(3)	5(3)
C(11)	44(4)	50(4)	37(4)	0	13(3)	0
C(12)	40(4)	60(5)	51(5)	0	18(4)	0
C(13)	41(4)	100(7)	46(5)	0	10(4)	0
C(14)	62(6)	183(11)	31(5)	0	15(5)	0
C(15)	40(4)	149(9)	47(5)	0	21(4)	0
C(16)	40(4)	57(4)	41(4)	0	13(3)	0
N(1)	35(3)	63(4)	43(4)	0	10(3)	0
C(17)	50(4)	60(5)	43(4)	0	5(4)	0

C(18)	53(3)	91(5)	62(4)	14(3)	13(3)	-23(3)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [(Me_3NPh)($DB_{18}C_6$)(Br_3)].

	x	y	z	U(eq)
H(1)	2583	-1788	12038	76
H(2)	3133	-1067	10382	65
H(4A)	4359	-562	8902	62
H(4B)	2641	-615	8391	62
H(5A)	3772	67	6939	64
H(5B)	4420	-800	6723	64
H(6A)	3117	-834	4553	74
H(6B)	2442	44	4655	74
H(7A)	74	-596	4074	73
H(7B)	830	-606	2955	73
H(9)	-1467	-1072	2052	72
H(10)	-3374	-1793	677	85
H(11)	3406	2500	2236	52
H(12)	5787	2500	3550	59
H(13)	6287	2500	5760	75
H(14)	4366	2500	6662	110
H(15)	1991	2500	5381	92
H(17A)	-313	2500	1011	80
H(17B)	1165	2014	1241	80
H(17C)	1165	2986	1241	80
H(18A)	-879	1740	2681	104
H(18B)	169	1741	4092	104
H(18C)	602	1249	3003	104

Due to the large amount of water molecules in the crystals, and the rotation of the tert-butyl groups of the 4-tert-butylcalix[n]arene as well as of the ligand itself, R_1 values are relatively huge in the following structures. In extreme case, it affects the goodness-of-fit on F^2 like in **15**.

V.2.8. Crystal data and structure refinement for [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$] 8

Empirical formula	C75 H133 K O27
Formula weight	1505.95
Temperature	240(2) K
Wavelength	0.71073 Å
Crystal system	Hexagonal
Space group	$P6_3/m$

Unit cell dimensions	a = 27.092(4) Å	α= 90°.
	b = 27.092(4) Å	β= 90°.
	c = 18.350(4) Å	γ = 120°.
Volume	11664(3) $Å^3$	
Z	2	
Density (calculated)	1.114 Mg/m^3	
Absorption coefficient	0.097 mm^{-1}	
F(000)	4190	
Theta range for data collection	1.41 to 26.99°.	
Index ranges	-34<=h<=26, -13<=k<=33, -23<=l<=14	
Reflections collected	14703	
Independent reflections	7879 [R(int) = 0.0953]	
Completeness to theta = 26.99°	89.9 %	
Absorption correction	Spherical	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7879 / 0 / 470	
Goodness-of-fit on F^2	0.878	
Final R indices [I>2sigma(I)]	R1 = 0.1202, wR2 = 0.3170	
R indices (all data)	R1 = 0.1540, wR2 = 0.3554	
Largest diff. peak and hole	0.701 and -0.563 $e.Å^{-3}$	

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	2999(2)	1483(2)	7500	68(1)
C(1)	3365(2)	2070(2)	7500	48(1)
C(2)	3532(2)	2359(2)	6837(2)	48(1)
C(3)	3901(2)	2950(2)	6852(2)	48(1)
C(4)	4099(2)	3253(2)	7500	47(1)

C(5)	4546(3)	3892(2)	7500	61(2)
C(6)	4529(5)	4186(3)	6828(4)	170(5)
C(7)	5121(4)	3942(5)	7500	207(10)
C(8)	3328(2)	2044(2)	6120(2)	54(1)
O(2)	2859(1)	1000(1)	5318(2)	53(1)
C(9)	3438(2)	1294(2)	5422(2)	46(1)
C(10)	3688(2)	1801(2)	5830(2)	49(1)
C(11)	4280(2)	2090(2)	5937(2)	56(1)
C(12)	4629(2)	1902(2)	5657(2)	54(1)
C(13)	4368(2)	1396(2)	5248(2)	51(1)
C(14)	3783(2)	1092(2)	5126(2)	47(1)
C(15)	5282(2)	2213(2)	5786(3)	68(1)
C(16)	5592(2)	2400(2)	5049(3)	80(2)
C(17)	5481(2)	2752(3)	6259(4)	109(2)
C(18)	5443(2)	1814(3)	6164(4)	91(2)
C(19)	3522(2)	547(2)	4675(2)	51(1)
O(3)	2399(1)	-63(1)	5305(2)	56(1)
C(20)	2756(2)	-275(2)	5418(2)	47(1)
C(21)	3313(2)	5(2)	5118(2)	48(1)
C(22)	3666(2)	-225(2)	5234(2)	52(1)
C(23)	3496(2)	-723(2)	5625(2)	54(1)
C(24)	2947(2)	-987(2)	5912(2)	56(1)
C(25)	2573(2)	-781(2)	5822(2)	51(1)
C(26)	3869(2)	-992(2)	5731(3)	72(1)
C(27)	4462(3)	-645(3)	5438(5)	117(3)
C(28)	3898(5)	-1120(7)	6526(6)	218(8)
C(29)	3576(4)	-1575(3)	5349(8)	171(5)
C(30)	1964(2)	-1107(2)	6121(2)	55(1)
O(4)	2053(2)	-583(2)	7500	69(1)
C(31)	1964(2)	-1133(2)	7500	48(1)
C(32)	1915(2)	-1407(2)	6835(2)	50(1)
C(33)	1832(2)	-1960(2)	6855(2)	52(1)
C(34)	1795(2)	-2242(2)	7500	51(1)
C(35)	1740(3)	-2835(3)	7500	68(2)
C(36)	1549(7)	-3133(4)	6837(5)	218(8)
C(37)	2330(8)	-2746(7)	7500	370(30)

O(5)	3426(4)	702(7)	7500	160(4)
C(38)	3981(6)	1022(5)	7500	237(13)
C(39)	4207(9)	533(11)	7500	290(16)
C(40)	3574(9)	54(7)	7500	217(10)
C(41)	3400(13)	401(11)	7500	520(50)
O(6)	2697(5)	329(4)	2500	241(9)
C(42)	2298(5)	247(5)	3133(6)	155(4)
C(43)	1844(4)	200(6)	2826(5)	177(5)
O(7)	2033(2)	1305(2)	5123(2)	78(1)
O(8)	2497(2)	446(1)	6719(1)	58(1)
O(9)	1767(6)	829(6)	7500	610(40)
O(10)	1157(5)	734(4)	6046(6)	232(5)
O(11)	749(5)	-495(5)	9031(7)	237(5)
O(12)	1026(9)	-490(7)	7500	258(7)
C(44)	6667	3333	2500	800(300)
O(13)	6542(11)	2823(18)	2500	660(50)
K(1)	0	0	5000	830(40)

Bond lengths [Å] and angles [°] for [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$].

O(1)-C(1)	1.391(6)	O(2)-C(9)	1.372(4)
O(1)-O(8)#1	2.825(4)	O(2)-O(3)	2.502(4)
O(1)-O(8)#2	2.825(4)	O(2)-O(7)	2.770(5)
O(1)-O(5)#1	2.875(15)	O(2)-O(8)#1	2.891(4)
O(1)-O(9)#1	2.891(15)	C(9)-C(10)	1.404(5)
C(1)-C(2)#2	1.395(5)	C(9)-C(14)	1.409(5)
C(1)-C(2)	1.395(5)	C(10)-C(11)	1.402(5)
C(2)-C(3)	1.400(5)	C(11)-C(12)	1.379(5)
C(2)-C(8)	1.514(5)	C(12)-C(13)	1.405(5)
C(3)-C(4)	1.391(5)	C(12)-C(15)	1.549(6)
C(4)-C(3)#2	1.391(5)	C(13)-C(14)	1.391(5)
C(4)-C(5)	1.539(7)	C(14)-C(19)	1.523(5)
C(5)-C(6)#2	1.481(8)	C(15)-C(18)	1.521(8)
C(5)-C(6)	1.481(8)	C(15)-C(16)	1.539(8)
C(5)-C(7)	1.495(14)	C(15)-C(17)	1.546(7)
C(8)-C(10)	1.521(5)	C(19)-C(21)	1.518(5)

O(3)-C(20)	1.364(4)	C(39)-C(40)	1.55(3)
O(3)-O(7)#3	2.703(5)	C(39)-C(41)	2.03(3)
O(3)-O(8)#1	2.887(4)	C(40)-C(41)	1.25(2)
C(20)-C(25)	1.412(5)	C(41)-O(5)#1	0.78(2)
C(20)-C(21)	1.418(5)	O(6)-C(42)#4	1.527(12)
C(21)-C(22)	1.394(5)	O(6)-C(42)	1.527(12)
C(22)-C(23)	1.389(5)	C(42)-C(43)	1.299(13)
C(23)-C(24)	1.393(6)	C(43)-C(43)#4	1.196(19)
C(23)-C(26)	1.524(6)	O(7)-O(7)#1	0.000(17)
C(24)-C(25)	1.391(5)	O(7)-O(11)#5	2.761(9)
C(25)-C(30)	1.530(5)	O(8)-O(8)#1	0.000(14)
C(26)-C(27)	1.498(8)	O(8)-O(8)#2	2.865(5)
C(26)-C(28)	1.511(11)	O(8)-O(9)#1	3.016(11)
C(26)-C(29)	1.538(10)	O(9)-O(9)#1	0.00(3)
C(30)-C(32)	1.513(5)	O(9)-O(8)#2	3.016(11)
O(4)-C(31)	1.385(6)	O(9)-O(10)#2	3.080(15)
O(4)-O(8)#2	2.812(4)	O(9)-O(12)#1	3.10(2)
O(4)-O(8)#1	2.812(4)	O(10)-O(7)#1	2.686(8)
O(4)-O(12)#1	2.916(19)	O(10)-O(9)#1	3.080(15)
C(31)-C(32)	1.399(5)	O(10)-K(1)	3.352(14)
C(31)-C(32)#2	1.399(5)	O(11)-O(10)#2	2.942(15)
C(32)-C(33)	1.401(5)	O(11)-K(1)#6	3.436(14)
C(33)-C(34)	1.385(5)	O(11)-O(12)#7	6.231(16)
C(34)-C(33)#2	1.385(5)	O(12)-O(12)#1	0.00(8)
C(34)-C(35)	1.538(8)	C(44)-O(13)	1.25(4)
C(35)-C(36)	1.408(8)	C(44)-O(13)#8	1.25(4)
C(35)-C(36)#2	1.408(8)	C(44)-O(13)#9	1.25(4)
C(35)-C(37)	1.492(19)	K(1)-O(10)#10	3.352(14)
O(5)-O(5)#1	0.00(2)	K(1)-O(10)#3	3.352(14)
O(5)-C(41)	0.78(2)	K(1)-O(10)#11	3.352(14)
O(5)-C(38)	1.307(15)	K(1)-O(10)#12	3.352(14)
O(5)-O(8)#1	2.668(8)	K(1)-O(10)#13	3.352(14)
O(5)-O(8)#2	2.668(8)	K(1)-O(11)#14	3.436(14)
C(38)-O(5)#1	1.307(15)	K(1)-O(11)#15	3.436(14)
C(38)-C(41)	1.63(3)	K(1)-O(11)#7	3.436(14)
C(38)-C(39)	1.72(2)	K(1)-O(11)#2	3.436(14)

K(1)-O(11)#5	3.436(14)	K(1)-O(11)#16	3.436(14)
C(1)-O(1)-O(8)#1	148.27(11)	O(2)-C(9)-C(14)	120.5(3)
C(1)-O(1)-O(8)#2	148.27(11)	C(10)-C(9)-C(14)	119.7(3)
O(8)#1-O(1)-O(8)#2	60.95(13)	C(9)-C(10)-C(11)	118.7(3)
C(1)-O(1)-O(5)#1	121.4(4)	C(9)-C(10)-C(8)	120.5(3)
O(8)#1-O(1)-O(5)#1	55.8(2)	C(11)-C(10)-C(8)	120.7(3)
O(8)#2-O(1)-O(5)#1	55.8(2)	C(12)-C(11)-C(10)	123.1(4)
C(1)-O(1)-O(9)#1	130.2(4)	C(11)-C(12)-C(13)	117.1(3)
O(8)#1-O(1)-O(9)#1	63.7(2)	C(11)-C(12)-C(15)	123.4(4)
O(8)#2-O(1)-O(9)#1	63.7(2)	C(13)-C(12)-C(15)	119.6(4)
O(5)#1-O(1)-O(9)#1	108.4(4)	C(14)-C(13)-C(12)	122.3(4)
O(1)-C(1)-C(2)#2	119.2(2)	C(13)-C(14)-C(9)	119.2(3)
O(1)-C(1)-C(2)	119.2(2)	C(13)-C(14)-C(19)	120.1(3)
C(2)#2-C(1)-C(2)	121.5(5)	C(9)-C(14)-C(19)	120.7(3)
C(1)-C(2)-C(3)	118.0(4)	C(18)-C(15)-C(16)	109.3(4)
C(1)-C(2)-C(8)	121.1(4)	C(18)-C(15)-C(12)	109.8(4)
C(3)-C(2)-C(8)	120.9(4)	C(16)-C(15)-C(12)	109.4(4)
C(4)-C(3)-C(2)	122.4(4)	C(18)-C(15)-C(17)	109.0(5)
C(3)-C(4)-C(3)#2	117.4(5)	C(16)-C(15)-C(17)	107.9(5)
C(3)-C(4)-C(5)	121.2(2)	C(12)-C(15)-C(17)	111.3(4)
C(3)#2-C(4)-C(5)	121.2(2)	C(21)-C(19)-C(14)	114.5(3)
C(6)#2-C(5)-C(6)	112.8(9)	C(20)-O(3)-O(2)	115.7(2)
C(6)#2-C(5)-C(7)	105.2(6)	C(20)-O(3)-O(7)#3	122.4(2)
C(6)-C(5)-C(7)	105.2(6)	O(2)-O(3)-O(7)#3	121.20(15)
C(6)#2-C(5)-C(4)	112.7(4)	C(20)-O(3)-O(8)#1	98.6(2)
C(6)-C(5)-C(4)	112.7(4)	O(2)-O(3)-O(8)#1	64.41(11)
C(7)-C(5)-C(4)	107.5(6)	O(7)#3-O(3)-O(8)#1	112.83(15)
C(2)-C(8)-C(10)	114.5(3)	O(3)-C(20)-C(25)	120.6(3)
C(9)-O(2)-O(3)	115.9(2)	O(3)-C(20)-C(21)	120.3(3)
C(9)-O(2)-O(7)	134.8(2)	C(25)-C(20)-C(21)	119.1(3)
O(3)-O(2)-O(7)	109.32(13)	C(22)-C(21)-C(20)	119.1(3)
C(9)-O(2)-O(8)#1	99.8(2)	C(22)-C(21)-C(19)	120.4(3)
O(3)-O(2)-O(8)#1	64.26(11)	C(20)-C(21)-C(19)	120.5(3)
O(7)-O(2)-O(8)#1	98.94(14)	C(23)-C(22)-C(21)	123.0(4)
O(2)-C(9)-C(10)	119.7(3)	C(22)-C(23)-C(24)	116.4(4)

C(22)-C(23)-C(26)	123.6(4)	O(5)#1-O(5)-C(38)	0(10)
C(24)-C(23)-C(26)	120.0(4)	C(41)-O(5)-C(38)	100(3)
C(25)-C(24)-C(23)	123.7(4)	O(5)#1-O(5)-O(8)#1	0(10)
C(24)-C(25)-C(20)	118.7(3)	C(41)-O(5)-O(8)#1	98(2)
C(24)-C(25)-C(30)	121.1(3)	C(38)-O(5)-O(8)#1	142.7(6)
C(20)-C(25)-C(30)	120.2(3)	O(5)#1-O(5)-O(8)#2	0(10)
C(27)-C(26)-C(28)	109.2(7)	C(41)-O(5)-O(8)#2	98(2)
C(27)-C(26)-C(23)	114.5(4)	C(38)-O(5)-O(8)#2	142.7(6)
C(28)-C(26)-C(23)	110.8(5)	O(8)#1-O(5)-O(8)#2	64.9(2)
C(27)-C(26)-C(29)	108.9(6)	O(5)#1-C(38)-O(5)	0.0(19)
C(28)-C(26)-C(29)	105.3(9)	O(5)#1-C(38)-C(41)	28.2(9)
C(23)-C(26)-C(29)	107.8(5)	O(5)-C(38)-C(41)	28.2(9)
C(32)-C(30)-C(25)	113.6(3)	O(5)#1-C(38)-C(39)	103.0(13)
C(31)-O(4)-O(8)#2	145.50(17)	O(5)-C(38)-C(39)	103.0(13)
C(31)-O(4)-O(8)#1	145.50(17)	C(41)-C(38)-C(39)	74.8(12)
O(8)#2-O(4)-O(8)#1	61.24(14)	C(40)-C(39)-C(38)	88.4(10)
C(31)-O(4)-O(12)#1	115.5(5)	C(40)-C(39)-C(41)	37.8(9)
O(8)#2-O(4)-O(12)#1	82.4(3)	C(38)-C(39)-C(41)	50.7(9)
O(8)#1-O(4)-O(12)#1	82.4(3)	C(41)-C(40)-C(39)	92.7(19)
O(4)-C(31)-C(32)	119.4(2)	O(5)#1-C(41)-O(5)	0.0(13)
O(4)-C(31)-C(32)#2	119.4(2)	O(5)#1-C(41)-C(40)	156(4)
C(32)-C(31)-C(32)#2	121.3(5)	O(5)-C(41)-C(40)	156(4)
C(31)-C(32)-C(33)	117.9(4)	O(5)#1-C(41)-C(38)	52(2)
C(31)-C(32)-C(30)	120.8(4)	O(5)-C(41)-C(38)	52(2)
C(33)-C(32)-C(30)	121.3(4)	C(40)-C(41)-C(38)	104(2)
C(34)-C(33)-C(32)	122.7(4)	O(5)#1-C(41)-C(39)	107(3)
C(33)#2-C(34)-C(33)	117.5(5)	O(5)-C(41)-C(39)	107(3)
C(33)#2-C(34)-C(35)	121.2(3)	C(40)-C(41)-C(39)	49.6(15)
C(33)-C(34)-C(35)	121.2(3)	C(38)-C(41)-C(39)	54.5(13)
C(36)-C(35)-C(36)#2	119.6(10)	C(42)#4-O(6)-C(42)	99.1(10)
C(36)-C(35)-C(37)	98.7(8)	C(43)-C(42)-O(6)	104.6(10)
C(36)#2-C(35)-C(37)	98.7(8)	C(43)#4-C(43)-C(42)	115.7(7)
C(36)-C(35)-C(34)	114.5(4)	O(7)#1-O(7)-O(11)#5	0(10)
C(36)#2-C(35)-C(34)	114.5(4)	O(7)#1-O(7)-O(2)	0(10)
C(37)-C(35)-C(34)	107.1(8)	O(11)#5-O(7)-O(2)	116.8(3)
O(5)#1-O(5)-C(41)	0(10)	O(8)#1-O(8)-O(8)#2	0(10)

O(8)#1-O(8)-O(9)#1	0(10)	O(10)#12-K(1)-O(11)#14	66.1(2)
O(8)#2-O(8)-O(9)#1	61.64(12)	O(10)#13-K(1)-O(11)#14	113.9(2)
O(9)#1-O(9)-O(8)#2	0(10)	O(10)-K(1)-O(11)#15	48.0(2)
O(9)#1-O(9)-O(10)#2	0(10)	O(10)#10-K(1)-O(11)#15	132.0(2)
O(8)#2-O(9)-O(10)#2	88.7(2)	O(10)#3-K(1)-O(11)#15	128.6(3)
O(9)#1-O(9)-O(12)#1	0(10)	O(10)#11-K(1)-O(11)#15	51.4(3)
O(8)#2-O(9)-O(12)#1	76.2(4)	O(10)#12-K(1)-O(11)#15	113.9(2)
O(10)#2-O(9)-O(12)#1	83.9(4)	O(10)#13-K(1)-O(11)#15	66.1(2)
O(7)#1-O(10)-O(9)#1	102.4(5)	O(11)#14-K(1)-O(11)#15	180.0(4)
O(7)#1-O(10)-K(1)	106.0(4)	O(10)-K(1)-O(11)#7	113.9(2)
O(9)#1-O(10)-K(1)	148.1(4)	O(10)#10-K(1)-O(11)#7	66.1(2)
O(10)#2-O(11)-K(1)#6	62.8(3)	O(10)#3-K(1)-O(11)#7	132.0(2)
O(10)#2-O(11)-O(12)#7	90.5(3)	O(10)#11-K(1)-O(11)#7	48.0(2)
K(1)#6-O(11)-O(12)#7	67.49(18)	O(10)#12-K(1)-O(11)#7	51.4(3)
O(13)-C(44)-O(13)#8	120.000(5)	O(10)#13-K(1)-O(11)#7	128.6(3)
O(13)-C(44)-O(13)#9	120.000(10)	O(11)#14-K(1)-O(11)#7	84.34(19)
O(13)#8-C(44)-O(13)#9	120.000(9)	O(11)#15-K(1)-O(11)#7	95.66(19)
O(10)-K(1)-O(10)#10	180.0(3)	O(10)-K(1)-O(11)#2	51.4(3)
O(10)-K(1)-O(10)#3	89.55(19)	O(10)#10-K(1)-O(11)#2	128.6(3)
O(10)#10-K(1)-O(10)#3	90.45(19)	O(10)#3-K(1)-O(11)#2	66.1(2)
O(10)-K(1)-O(10)#11	90.45(19)	O(10)#11-K(1)-O(11)#2	113.9(2)
O(10)#10-K(1)-O(10)#11	89.55(19)	O(10)#12-K(1)-O(11)#2	48.0(2)
O(10)#3-K(1)-O(10)#11	180.000(1)	O(10)#13-K(1)-O(11)#2	132.0(2)
O(10)-K(1)-O(10)#12	90.45(19)	O(11)#14-K(1)-O(11)#2	84.34(19)
O(10)#10-K(1)-O(10)#12	89.55(19)	O(11)#15-K(1)-O(11)#2	95.66(19)
O(10)#3-K(1)-O(10)#12	89.55(19)	O(11)#7-K(1)-O(11)#2	95.66(19)
O(10)#11-K(1)-O(10)#12	90.45(19)	O(10)-K(1)-O(11)#5	66.1(2)
O(10)-K(1)-O(10)#13	89.55(19)	O(10)#10-K(1)-O(11)#5	113.9(2)
O(10)#10-K(1)-O(10)#13	90.45(19)	O(10)#3-K(1)-O(11)#5	48.0(2)
O(10)#3-K(1)-O(10)#13	90.45(19)	O(10)#11-K(1)-O(11)#5	132.0(2)
O(10)#11-K(1)-O(10)#13	89.55(19)	O(10)#12-K(1)-O(11)#5	128.6(3)
O(10)#12-K(1)-O(10)#13	180.0(3)	O(10)#13-K(1)-O(11)#5	51.4(3)
O(10)-K(1)-O(11)#14	132.0(2)	O(11)#14-K(1)-O(11)#5	95.66(19)
O(10)#10-K(1)-O(11)#14	48.0(2)	O(11)#15-K(1)-O(11)#5	84.34(19)
O(10)#3-K(1)-O(11)#14	51.4(3)	O(11)#7-K(1)-O(11)#5	180.0(3)
O(10)#11-K(1)-O(11)#14	128.6(3)	O(11)#2-K(1)-O(11)#5	84.34(19)

O(10)-K(1)-O(11)#16	128.6(3)	O(11)#14-K(1)-O(11)#16	95.66(19)
O(10)#10-K(1)-O(11)#16	51.4(3)	O(11)#15-K(1)-O(11)#16	84.34(19)
O(10)#3-K(1)-O(11)#16	113.9(2)	O(11)#7-K(1)-O(11)#16	84.34(19)
O(10)#11-K(1)-O(11)#16	66.1(2)	O(11)#2-K(1)-O(11)#16	180.0(3)
O(10)#12-K(1)-O(11)#16	132.0(2)	O(11)#5-K(1)-O(11)#16	95.66(19)
O(10)#13-K(1)-O(11)#16	48.0(2)		

Symmetry transformations used to generate equivalent atoms:

#1 x,y,z #2 x,y,-z+3/2 #3 y,-x+y,-z+1 #4 x,y,-z+1/2

#5 x-y,x,z-1/2 #6 -x,-y,z+1/2 #7 -x+y,-x,-z+3/2

#8 -y+1,x-y,z #9 -x+y+1,-x+1,z #10 -x,-y,-z+1

#11 -y,x-y,z #12 -x+y,-x,z #13 x-y,x,-z+1

#14 y,-x+y,z-1/2 #15 -y,x-y,-z+3/2 #16 -x,-y,z-1/2

Anisotropic displacement parameters ($Å^2 x\ 10^3$)for [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	60(2)	40(2)	99(3)	0	0	22(2)
C(1)	40(2)	36(2)	72(3)	0	0	22(2)
C(2)	44(2)	45(2)	61(2)	-5(2)	-3(2)	28(2)
C(3)	48(2)	47(2)	54(2)	3(2)	4(2)	27(2)
C(4)	45(3)	38(2)	59(3)	0	0	21(2)
C(5)	63(4)	43(3)	66(4)	0	0	19(3)
C(6)	266(12)	44(3)	108(6)	18(3)	-55(6)	8(5)
C(7)	58(6)	86(8)	430(30)	0	0	1(5)
C(8)	50(2)	55(2)	65(2)	-11(2)	-8(2)	32(2)
O(2)	45(1)	43(1)	70(2)	-4(1)	-8(1)	21(1)
C(9)	44(2)	40(2)	52(2)	1(1)	-6(1)	20(2)
C(10)	49(2)	43(2)	60(2)	-5(2)	-4(2)	27(2)
C(11)	46(2)	45(2)	76(3)	-11(2)	-5(2)	23(2)
C(12)	45(2)	50(2)	65(2)	-8(2)	-3(2)	23(2)
C(13)	48(2)	44(2)	61(2)	-1(2)	4(2)	23(2)
C(14)	51(2)	41(2)	48(2)	2(1)	2(2)	24(2)
C(15)	45(2)	62(3)	94(3)	-16(2)	-2(2)	25(2)

C(16)	51(2)	70(3)	113(4)	4(3)	10(2)	26(2)
C(17)	53(3)	110(5)	153(6)	-71(4)	-26(3)	33(3)
C(18)	66(3)	105(4)	110(5)	1(3)	-15(3)	50(3)
C(19)	61(2)	43(2)	46(2)	2(1)	5(2)	25(2)
O(3)	50(2)	47(1)	70(2)	5(1)	-2(1)	22(1)
C(20)	49(2)	41(2)	51(2)	-2(1)	-2(2)	21(2)
C(21)	56(2)	39(2)	47(2)	-1(1)	4(2)	22(2)
C(22)	51(2)	43(2)	57(2)	-3(2)	7(2)	21(2)
C(23)	60(2)	48(2)	59(2)	3(2)	10(2)	32(2)
C(24)	58(2)	43(2)	66(2)	7(2)	8(2)	25(2)
C(25)	50(2)	40(2)	58(2)	1(2)	1(2)	21(2)
C(26)	72(3)	65(3)	95(4)	16(2)	20(2)	47(2)
C(27)	81(4)	110(5)	176(8)	32(5)	29(4)	60(4)
C(28)	248(12)	396(19)	169(10)	168(12)	109(9)	279(15)
C(29)	108(6)	77(4)	347(16)	-19(6)	41(7)	60(5)
C(30)	51(2)	51(2)	58(2)	5(2)	-2(2)	21(2)
O(4)	67(3)	44(2)	94(3)	0	0	26(2)
C(31)	37(2)	37(2)	64(3)	0	0	13(2)
C(32)	39(2)	44(2)	59(2)	4(2)	-2(2)	14(2)
C(33)	48(2)	44(2)	54(2)	-4(2)	0(2)	15(2)
C(34)	50(3)	38(2)	56(3)	0	0	16(2)
C(35)	85(4)	51(3)	72(4)	0	0	38(3)
C(36)	500(20)	104(6)	93(5)	-31(4)	-67(9)	180(11)
C(37)	144(15)	99(11)	880(80)	0	0	81(12)
O(5)	101(6)	275(14)	97(5)	0	0	88(8)
C(38)	80(8)	68(7)	550(40)	0	0	28(6)
C(39)	123(14)	230(20)	560(50)	0	0	123(16)
C(40)	176(16)	104(10)	400(30)	0	0	90(12)
C(41)	180(20)	134(17)	1310(160)	0	0	132(19)
O(6)	109(8)	105(7)	510(30)	0	0	56(6)
C(42)	170(11)	147(9)	140(9)	16(6)	-23(7)	74(8)
C(43)	94(6)	293(15)	131(8)	-9(7)	4(5)	87(8)
O(7)	53(2)	64(2)	111(3)	0(2)	-4(2)	25(2)
O(8)	84(2)	51(2)	37(1)	2(1)	-5(1)	33(2)
O(9)	112(9)	138(11)	1610(110)	0	0	87(9)
O(10)	252(10)	165(7)	288(12)	76(7)	164(9)	111(7)

O(11)	227(9)	267(11)	226(11)	4(8)	-105(8)	131(9)
O(12)	390(20)	243(15)	198(13)	0	0	202(17)
C(44)	260(80)	260(80)	2000(800)	0	0	130(40)
O(13)	180(20)	390(60)	1370(170)	0	0	110(30)
K(1)	410(20)	410(20)	1680(150)	0	0	207(11)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [K(4-tert-butylcalix[6]arene)$(THF)_2(H_2O)_{16}(HCO_3)$].

	x	y	z	U(eq)
H(3)	4018	3146	6413	58
H(6A)	4815	4581	6854	255
H(6B)	4160	4152	6781	255
H(6C)	4600	4015	6413	255
H(7A)	5410	4337	7500	310
H(7B)	5161	3760	7073	310
H(7C)	5161	3760	7927	310
H(8A)	3322	2302	5759	65
H(8B)	2940	1734	6182	65
H(2)	2730(30)	580(30)	5210(40)	100(19)
H(11)	4444	2424	6211	67
H(13)	4593	1259	5052	61
H(16A)	5997	2593	5128	120
H(16B)	5492	2653	4809	120
H(16C)	5482	2071	4749	120
H(17A)	5885	2934	6333	163
H(17B)	5291	2646	6722	163
H(17C)	5391	3011	6017	163
H(18A)	5847	2010	6246	136
H(18B)	5337	1487	5862	136
H(18C)	5247	1694	6622	136
H(19A)	3204	520	4400	61
H(19B)	3804	572	4328	61
H(22)	4033	-35	5041	62

H(24)	2823	-1320	6180	67
H(27A)	4644	-273	5659	175
H(27B)	4448	-607	4920	175
H(27C)	4676	-831	5549	175
H(28A)	4087	-770	6796	327
H(28B)	4106	-1319	6576	327
H(28C)	3519	-1352	6712	327
H(29A)	3194	-1800	5531	257
H(29B)	3785	-1768	5445	257
H(29C)	3565	-1522	4833	257
H(30A)	1825	-843	6188	66
H(30B)	1722	-1388	5763	66
H(33)	1801	-2146	6416	63
H(36A)	1751	-2882	6440	328
H(36B)	1148	-3272	6783	328
H(36C)	1617	-3448	6835	328
H(37A)	2526	-2543	7933	549
H(37B)	2532	-2528	7079	549
H(37C)	2313	-3108	7489	549
H(42A)	2207	-94	3407	185
H(42B)	2469	572	3459	185
H(43A)	1517	-151	2992	213
H(43B)	1794	512	2992	213
H(7Z)	2280(30)	1270(30)	5130(40)	90(20)
H(8Z)	2125(12)	327(11)	6626(14)	7(6)
H(11Z)	618(17)	-365(17)	9650(20)	42(10)

V.2.9. Crystal data and structure refinement for $[Li_{15}$(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4]$ 9

Empirical formula	C144 H197 Li15 O26
Formula weight	2448.2211
Temperature	240(2) K
Wavelength	0.71073 Å

Crystal system	Orthorhombic
Space group	Pmmn
Unit cell dimensions	a = 22.093(4) Å α= 90°.
b = 22.859(5) Å	β= 90°.
c = 15.769(3) Å	γ = 90°.
Volume	7964(3) $Å^3$
Z	2
Density (calculated)	1.076 Mg/m^3
Absorption coefficient	0.071 mm^{-1}
F(000)	2746
Theta range for data collection	1.28 to 24.97°.
Index ranges	-26<=h<=25, -26<=k<=26, -18<=l<=18
Reflections collected	39268
Independent reflections	7219 [R(int) = 0.1271]
Completeness to theta = 24.97°	97.8 %
Absorption correction	Spherical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7219 / 0 / 484
Goodness-of-fit on F^2	1.126
Final R indices [I>2sigma(I)]	R1 = 0.1297, wR2 = 0.3403
R indices (all data)	R1 = 0.1929, wR2 = 0.3887
Extinction coefficient	0.0060(12)
Largest diff. peak and hole	0.854 and -0.806 $e.Å^{-3}$

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

For $[Li_{15}$(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4]$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

x	y	z	U(eq)	
Li(1)	1937(4)	3019(4)	2448(6)	65(2)
Li(2)	890(6)	2500	3223(9)	70(3)

Li(3)	1968(4)	3036(4)	3998(7)	66(2)
Li(4)	2500	2500	5765(11)	65(5)
Li(5)	2500	4067(7)	3024(11)	102(6)
Li(6)	1833(11)	2500	654(14)	214(19)
O(1)	2500	3668(2)	4099(3)	64(1)
C(1)	2500	4131(3)	4640(5)	58(2)
C(2)	1953(2)	4373(2)	4911(4)	62(1)
C(3)	1970(3)	4899(2)	5373(4)	76(2)
C(4)	2500	5179(4)	5587(6)	74(2)
C(5)	2500	5761(5)	6060(9)	103(3)
C(6)	1964(6)	6125(5)	5856(11)	238(9)
C(7)	2500	5648(8)	6943(13)	350(30)
O(2)	1319(1)	3253(1)	3267(2)	59(1)
C(8)	1359(2)	4053(2)	4753(3)	61(1)
C(9)	1059(2)	3794(2)	3222(3)	57(1)
C(10)	1056(2)	4179(2)	3920(4)	60(1)
C(11)	743(2)	4714(2)	3857(4)	68(2)
C(12)	452(3)	4890(2)	3125(4)	70(2)
C(13)	485(2)	4515(2)	2441(4)	72(2)
C(14)	792(2)	3981(2)	2457(4)	61(1)
C(15)	72(3)	5455(3)	3071(5)	89(2)
C(16)	180(4)	5849(3)	3864(6)	125(3)
C(17)	-592(3)	5288(4)	3049(7)	137(3)
C(18)	240(5)	5794(4)	2268(7)	159(4)
C(19)	843(3)	3617(2)	1651(4)	67(2)
O(3)	1472(2)	2500	1694(3)	66(1)
C(20)	852(3)	2500	1684(5)	62(2)
C(21)	523(2)	3030(2)	1676(3)	64(1)
C(22)	-104(2)	3020(3)	1655(4)	70(2)
C(23)	-433(4)	2500	1631(6)	77(2)
C(24)	-1130(4)	2500	1553(7)	91(3)
C(25)	-1400(4)	3035(6)	1928(11)	220(8)
C(26)	-1277(6)	2500	640(11)	213(9)
O(4)	2500	2500	3197(4)	55(2)
O(5)	121(2)	2500	3791(4)	78(2)
C(27)	-184(3)	3008(3)	4096(6)	102(2)

C(28)	-631(4)	2801(3)	4712(7)	140(4)
O(6)	2500	3153(4)	6656(5)	123(3)
C(29)	2058(7)	3567(6)	6877(9)	210(6)
C(30)	2215(5)	3834(8)	7654(11)	283(12)
O(7)	2500	4878(3)	2757(5)	101(2)
C(31)	2034(5)	5228(6)	2697(15)	269(12)
C(32)	2190(8)	5794(7)	2810(20)	383(19)
C(33)	1258(3)	2500	5332(5)	64(2)
C(34)	1073(6)	2500	6048(9)	186(9)
O(8)	1334(4)	2500	9659(5)	132(3)
C(35)	1014(8)	2993(6)	9385(9)	188(5)
C(36)	443(8)	2810(6)	9065(15)	285(11)
O(9)	1796(2)	2500	4963(3)	59(1)
O(10)	2500	3591(2)	2053(4)	67(1)
C(37)	2500	3747(5)	1253(7)	86(3)
C(38)	2500	3434(7)	545(7)	119(4)
O(11)	807(13)	5471(15)	9530(20)	442(15)
C(39)	890(30)	4890(20)	9870(30)	390(20)
C(40)	1430(30)	4830(20)	9510(30)	360(20)
C(41)	1675(17)	5390(30)	9330(40)	390(20)
C(42)	1380(30)	5500(30)	10210(20)	400(20)
O(12)	1750(30)	7500	2950(60)	720(70)
C(43)	1420(20)	7212(12)	1816(19)	440(30)
C(44)	1099(18)	7212(16)	2840(30)	440(30)
O(13)	2500	5845(10)	260(12)	447(9)

Bond lengths [Å] and angles [°] for $[Li_{15}$(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4]$.

Li(1)-O(10)	1.909(10)	Li(1)-Li(2)	2.872(15)
Li(1)-O(2)	1.955(10)	Li(1)-Li(6)	3.07(2)
Li(1)-O(3)	1.968(10)	Li(1)-Li(3)#1	3.435(13)
Li(1)-O(4)	2.085(10)	Li(1)-Li(1)#3	3.436(17)
Li(1)-Li(1)#1	2.371(18)	Li(2)-O(5)	1.922(14)
Li(1)-Li(3)	2.446(14)	Li(2)-O(2)	1.966(7)
Li(1)-Li(1)#2	2.487(18)	Li(2)-O(2)#1	1.966(7)
Li(1)-Li(5)	2.848(18)	Li(2)-C(20)	2.428(16)

Li(2)-Li(1)#1	2.872(15)	Li(6)-O(3)#3	4.09(2)
Li(2)-Li(3)	2.944(15)	O(1)-C(1)	1.359(8)
Li(2)-Li(3)#1	2.944(15)	O(1)-Li(3)#2	1.869(9)
Li(2)-Li(3)#2	5.039(16)	O(1)-O(2)#2	3.071(4)
Li(3)-O(1)	1.869(9)	C(1)-C(2)#2	1.397(6)
Li(3)-O(2)	1.905(10)	C(1)-C(2)	1.397(6)
Li(3)-O(9)	1.990(10)	C(2)-C(3)	1.406(7)
Li(3)-O(4)	2.117(10)	C(2)-C(8)	1.523(7)
Li(3)-Li(3)#2	2.351(17)	C(3)-C(4)	1.376(7)
Li(3)-Li(3)#1	2.452(17)	C(4)-C(3)#2	1.376(7)
Li(3)-Li(5)	3.048(17)	C(4)-C(5)	1.527(12)
Li(3)-Li(4)	3.263(18)	C(5)-C(7)	1.42(2)
Li(3)-Li(3)#3	3.397(17)	C(5)-C(6)#2	1.482(12)
Li(3)-Li(1)#1	3.435(13)	C(5)-C(6)	1.482(12)
Li(4)-O(9)	2.004(12)	O(2)-C(9)	1.365(6)
Li(4)-O(9)#3	2.004(12)	C(8)-C(10)	1.501(7)
Li(4)-O(6)	2.050(15)	C(9)-C(10)	1.409(7)
Li(4)-O(6)#3	2.050(15)	C(9)-C(14)	1.409(7)
Li(4)-Li(3)#1	3.263(18)	C(10)-C(11)	1.409(7)
Li(4)-Li(3)#3	3.263(18)	C(11)-C(12)	1.381(8)
Li(4)-Li(3)#2	3.263(18)	C(12)-C(13)	1.380(8)
Li(5)-O(10)	1.878(18)	C(12)-C(15)	1.543(8)
Li(5)-O(7)	1.903(17)	C(13)-C(14)	1.397(7)
Li(5)-O(1)	1.925(17)	C(14)-C(19)	1.524(8)
Li(5)-C(1)	2.552(18)	C(15)-C(16)	1.560(11)
Li(5)-Li(1)#2	2.848(18)	C(15)-C(17)	1.515(11)
Li(5)-Li(3)#2	3.048(17)	C(15)-C(18)	1.530(12)
Li(5)-O(2)#2	3.227(10)	C(19)-C(21)	1.517(8)
Li(6)-O(3)	1.82(3)	O(3)-C(20)	1.371(9)
Li(6)-O(8)#4	1.92(2)	O(3)-Li(1)#1	1.968(10)
Li(6)-C(38)	2.601(19)	O(3)-O(2)#1	3.039(6)
Li(6)-C(38)#3	2.601(19)	C(20)-C(21)#1	1.412(7)
Li(6)-C(20)	2.71(3)	C(20)-C(21)	1.412(7)
Li(6)-Li(6)#3	2.95(5)	C(21)-C(22)	1.387(8)
Li(6)-Li(1)#1	3.07(2)	C(22)-C(23)	1.394(7)
Li(6)-O(10)#3	3.640(16)	C(23)-C(22)#1	1.394(7)

C(23)-C(24)	1.544(12)	C(32)-C(32)#2	1.37(4)
C(24)-C(26)	1.476(18)	C(33)-C(34)	1.201(14)
C(24)-C(25)	1.484(12)	C(33)-O(9)	1.325(8)
C(24)-C(25)#1	1.484(12)	O(8)-C(35)#1	1.397(14)
O(4)-Li(1)#1	2.085(10)	O(8)-C(35)	1.397(14)
O(4)-Li(1)#2	2.085(10)	O(8)-Li(6)#5	1.92(2)
O(4)-Li(1)#3	2.085(10)	C(35)-C(36)	1.423(17)
O(4)-Li(3)#2	2.117(10)	C(36)-C(36)#1	1.42(3)
O(4)-Li(3)#3	2.117(10)	O(9)-Li(3)#1	1.990(10)
O(4)-Li(3)#1	2.117(10)	O(10)-C(37)	1.310(10)
O(4)-O(1)#3	3.026(6)	O(10)-Li(1)#2	1.909(10)
O(4)-O(10)#3	3.077(6)	C(37)-C(38)	1.326(15)
O(4)-O(2)#2	3.128(3)	C(38)-Li(6)#3	2.601(19)
O(4)-O(2)#1	3.128(3)	O(11)-C(39)	1.45(4)
O(5)-C(27)#1	1.426(7)	O(11)-C(42)	1.65(4)
O(5)-C(27)	1.426(7)	C(39)-C(40)	1.34(5)
C(27)-C(28)	1.465(11)	C(39)-C(42)	1.85(6)
C(28)-C(28)#1	1.376(16)	C(40)-C(41)	1.43(5)
O(6)-C(29)	1.404(13)	C(40)-C(42)	1.89(6)
O(6)-C(29)#2	1.404(14)	C(41)-C(42)	1.56(5)
C(29)-C(30)	1.412(17)	O(12)-C(44)#6	1.58(6)
C(29)-C(29)#2	1.96(3)	O(12)-C(44)	1.58(6)
C(30)-C(30)#2	1.26(2)	C(43)-C(43)#6	1.32(6)
O(7)-C(31)	1.306(12)	C(43)-C(44)	1.76(4)
O(7)-C(31)#2	1.306(12)	C(44)-C(44)#6	1.32(7)
C(31)-C(32)	1.35(2)		

O(10)-Li(1)-O(2)	118.9(5)	O(4)-Li(1)-Li(1)#1	55.4(3)
O(10)-Li(1)-O(3)	123.7(5)	O(10)-Li(1)-Li(3)	107.2(5)
O(2)-Li(1)-O(3)	101.5(4)	O(2)-Li(1)-Li(3)	49.8(3)
O(10)-Li(1)-O(4)	100.7(4)	O(3)-Li(1)-Li(3)	128.9(5)
O(2)-Li(1)-O(4)	101.4(4)	O(4)-Li(1)-Li(3)	55.0(3)
O(3)-Li(1)-O(4)	108.1(4)	Li(1)#1-Li(1)-Li(3)	91.0(3)
O(10)-Li(1)-Li(1)#1	133.2(3)	O(10)-Li(1)-Li(1)#2	49.3(3)
O(2)-Li(1)-Li(1)#1	105.9(3)	O(2)-Li(1)-Li(1)#2	134.3(3)
O(3)-Li(1)-Li(1)#1	53.0(3)	O(3)-Li(1)-Li(1)#2	121.4(3)

O(4)-Li(1)-Li(1)#2	53.4(3)	Li(6)-Li(1)-Li(3)#1	112.7(3)
Li(1)#1-Li(1)-Li(1)#2	90.000(1)	O(10)-Li(1)-Li(1)#3	90.1(4)
Li(3)-Li(1)-Li(1)#2	88.4(3)	O(2)-Li(1)-Li(1)#3	134.0(3)
O(10)-Li(1)-Li(5)	40.8(4)	O(3)-Li(1)-Li(1)#3	87.8(4)
O(2)-Li(1)-Li(5)	82.2(4)	O(4)-Li(1)-Li(1)#3	34.5(3)
O(3)-Li(1)-Li(5)	158.0(5)	Li(1)#1-Li(1)-Li(1)#3	46.4(3)
O(4)-Li(1)-Li(5)	92.1(4)	Li(3)-Li(1)-Li(1)#3	89.5(3)
Li(1)#1-Li(1)-Li(5)	147.3(3)	Li(1)#2-Li(1)-Li(1)#3	43.6(3)
Li(3)-Li(1)-Li(5)	69.8(4)	Li(5)-Li(1)-Li(1)#3	105.3(4)
Li(1)#2-Li(1)-Li(5)	64.1(2)	Li(2)-Li(1)-Li(1)#3	107.3(4)
O(10)-Li(1)-Li(2)	161.1(5)	Li(6)-Li(1)-Li(1)#3	77.8(4)
O(2)-Li(1)-Li(2)	43.1(3)	Li(3)#1-Li(1)-Li(1)#3	60.1(2)
O(3)-Li(1)-Li(2)	65.7(4)	O(5)-Li(2)-O(2)	114.2(4)
O(4)-Li(1)-Li(2)	90.3(4)	O(5)-Li(2)-O(2)#1	114.2(4)
Li(1)#1-Li(1)-Li(2)	65.6(2)	O(2)-Li(2)-O(2)#1	122.2(7)
Li(3)-Li(1)-Li(2)	66.7(4)	O(5)-Li(2)-C(20)	115.8(7)
Li(1)#2-Li(1)-Li(2)	143.6(3)	O(2)-Li(2)-C(20)	93.0(4)
Li(5)-Li(1)-Li(2)	124.3(4)	O(2)#1-Li(2)-C(20)	93.0(4)
O(10)-Li(1)-Li(6)	90.8(5)	O(5)-Li(2)-Li(1)	155.6(2)
O(2)-Li(1)-Li(6)	131.4(6)	O(2)-Li(2)-Li(1)	42.8(3)
O(3)-Li(1)-Li(6)	34.2(5)	O(2)#1-Li(2)-Li(1)	89.3(5)
O(4)-Li(1)-Li(6)	110.3(5)	C(20)-Li(2)-Li(1)	66.6(4)
Li(1)#1-Li(1)-Li(6)	67.3(2)	O(5)-Li(2)-Li(1)#1	155.6(2)
Li(3)-Li(1)-Li(6)	158.1(5)	O(2)-Li(2)-Li(1)#1	89.3(5)
Li(1)#2-Li(1)-Li(6)	94.3(5)	O(2)#1-Li(2)-Li(1)#1	42.8(3)
Li(5)-Li(1)-Li(6)	130.6(5)	C(20)-Li(2)-Li(1)#1	66.6(4)
Li(2)-Li(1)-Li(6)	99.9(5)	Li(1)-Li(2)-Li(1)#1	48.8(4)
O(10)-Li(1)-Li(3)#1	134.4(4)	O(5)-Li(2)-Li(3)	121.4(6)
O(2)-Li(1)-Li(3)#1	74.7(3)	O(2)-Li(2)-Li(3)	39.7(3)
O(3)-Li(1)-Li(3)#1	91.0(4)	O(2)#1-Li(2)-Li(3)	87.7(5)
O(4)-Li(1)-Li(3)#1	35.5(2)	C(20)-Li(2)-Li(3)	116.3(5)
Li(1)#1-Li(1)-Li(3)#1	45.4(2)	Li(1)-Li(2)-Li(3)	49.7(3)
Li(3)-Li(1)-Li(3)#1	45.5(4)	Li(1)#1-Li(2)-Li(3)	72.4(4)
Li(1)#2-Li(1)-Li(3)#1	88.9(2)	O(5)-Li(2)-Li(3)#1	121.4(6)
Li(5)-Li(1)-Li(3)#1	110.8(5)	O(2)-Li(2)-Li(3)#1	87.7(5)
Li(2)-Li(1)-Li(3)#1	54.8(3)	O(2)#1-Li(2)-Li(3)#1	39.7(3)

C(20)-Li(2)-Li(3)#1	116.3(5)	O(4)-Li(3)-Li(2)	87.7(4)
Li(1)-Li(2)-Li(3)#1	72.4(4)	Li(3)#2-Li(3)-Li(2)	144.0(3)
Li(1)#1-Li(2)-Li(3)#1	49.7(3)	Li(3)#1-Li(3)-Li(2)	65.4(2)
Li(3)-Li(2)-Li(3)#1	49.2(4)	Li(1)-Li(3)-Li(2)	63.6(4)
O(5)-Li(2)-Li(3)#2	135.8(5)	O(1)-Li(3)-Li(5)	37.2(4)
O(2)-Li(2)-Li(3)#2	47.6(3)	O(2)-Li(3)-Li(5)	77.5(4)
O(2)#1-Li(2)-Li(3)#2	75.7(4)	O(9)-Li(3)-Li(5)	159.2(5)
C(20)-Li(2)-Li(3)#2	106.0(4)	O(4)-Li(3)-Li(5)	86.1(4)
Li(1)-Li(2)-Li(3)#2	41.1(3)	Li(3)#2-Li(3)-Li(5)	67.3(2)
Li(1)#1-Li(2)-Li(3)#2	56.5(3)	Li(3)#1-Li(3)-Li(5)	140.6(3)
Li(3)-Li(2)-Li(3)#2	15.93(18)	Li(1)-Li(3)-Li(5)	61.3(4)
Li(3)#1-Li(2)-Li(3)#2	40.6(3)	Li(2)-Li(3)-Li(5)	115.1(4)
O(1)-Li(3)-O(2)	108.9(5)	O(1)-Li(3)-Li(4)	89.5(4)
O(1)-Li(3)-O(9)	122.0(6)	O(2)-Li(3)-Li(4)	152.3(5)
O(2)-Li(3)-O(9)	118.6(5)	O(9)-Li(3)-Li(4)	35.4(2)
O(1)-Li(3)-O(4)	98.6(4)	O(4)-Li(3)-Li(4)	95.3(4)
O(2)-Li(3)-O(4)	102.0(5)	Li(3)#2-Li(3)-Li(4)	68.88(19)
O(9)-Li(3)-O(4)	101.9(4)	Li(3)#1-Li(3)-Li(4)	67.93(19)
O(1)-Li(3)-Li(3)#2	51.0(3)	Li(1)-Li(3)-Li(4)	149.0(4)
O(2)-Li(3)-Li(3)#2	138.8(3)	Li(2)-Li(3)-Li(4)	119.3(3)
O(9)-Li(3)-Li(3)#2	101.0(3)	Li(5)-Li(3)-Li(4)	125.6(4)
O(4)-Li(3)-Li(3)#2	56.3(3)	O(1)-Li(3)-Li(3)#3	97.0(4)
O(1)-Li(3)-Li(3)#1	140.6(3)	O(2)-Li(3)-Li(3)#3	135.1(4)
O(2)-Li(3)-Li(3)#1	105.1(3)	O(9)-Li(3)-Li(3)#3	71.8(3)
O(9)-Li(3)-Li(3)#1	52.0(3)	O(4)-Li(3)-Li(3)#3	36.7(3)
O(4)-Li(3)-Li(3)#1	54.6(2)	Li(3)#2-Li(3)-Li(3)#3	46.2(3)
Li(3)#2-Li(3)-Li(3)#1	90.000(1)	Li(3)#1-Li(3)-Li(3)#3	43.8(3)
O(1)-Li(3)-Li(1)	96.6(5)	Li(1)-Li(3)-Li(3)#3	90.4(3)
O(2)-Li(3)-Li(1)	51.6(3)	Li(2)-Li(3)-Li(3)#3	105.0(4)
O(9)-Li(3)-Li(1)	138.5(5)	Li(5)-Li(3)-Li(3)#3	106.9(4)
O(4)-Li(3)-Li(1)	53.8(3)	Li(4)-Li(3)-Li(3)#3	58.6(2)
Li(3)#2-Li(3)-Li(1)	91.6(3)	O(1)-Li(3)-Li(1)#1	128.0(5)
Li(3)#1-Li(3)-Li(1)	89.0(3)	O(2)-Li(3)-Li(1)#1	74.8(3)
O(1)-Li(3)-Li(2)	150.0(5)	O(9)-Li(3)-Li(1)#1	96.2(3)
O(2)-Li(3)-Li(2)	41.3(2)	O(4)-Li(3)-Li(1)#1	34.9(2)
O(9)-Li(3)-Li(2)	84.6(4)	Li(3)#2-Li(3)-Li(1)#1	91.1(2)

Li(3)#1-Li(3)-Li(1)#1	45.4(2)	O(7)-Li(5)-O(1)	131.0(10)
Li(1)-Li(3)-Li(1)#1	43.6(4)	O(10)-Li(5)-C(1)	147.9(8)
Li(2)-Li(3)-Li(1)#1	52.8(3)	O(7)-Li(5)-C(1)	99.5(8)
Li(5)-Li(3)-Li(1)#1	101.0(5)	O(1)-Li(5)-C(1)	31.5(3)
Li(4)-Li(3)-Li(1)#1	110.6(3)	O(10)-Li(5)-Li(1)	41.6(4)
Li(3)#3-Li(3)-Li(1)#1	60.5(2)	O(7)-Li(5)-Li(1)	138.6(7)
O(9)-Li(4)-O(9)#3	101.8(8)	O(1)-Li(5)-Li(1)	83.3(6)
O(9)-Li(4)-O(6)	115.61(18)	C(1)-Li(5)-Li(1)	111.5(6)
O(9)#3-Li(4)-O(6)	115.61(18)	O(10)-Li(5)-Li(1)#2	41.6(4)
O(9)-Li(4)-O(6)#3	115.61(18)	O(7)-Li(5)-Li(1)#2	138.6(7)
O(9)#3-Li(4)-O(6)#3	115.61(18)	O(1)-Li(5)-Li(1)#2	83.3(6)
O(6)-Li(4)-O(6)#3	93.5(9)	C(1)-Li(5)-Li(1)#2	111.5(6)
O(9)-Li(4)-Li(3)#1	35.1(3)	Li(1)-Li(5)-Li(1)#2	51.8(5)
O(9)#3-Li(4)-Li(3)#1	75.0(5)	O(10)-Li(5)-Li(3)	87.9(6)
O(6)-Li(4)-Li(3)#1	149.2(4)	O(7)-Li(5)-Li(3)	149.9(6)
O(6)#3-Li(4)-Li(3)#1	108.2(4)	O(1)-Li(5)-Li(3)	35.9(3)
O(9)-Li(4)-Li(3)#3	75.0(5)	C(1)-Li(5)-Li(3)	62.7(4)
O(9)#3-Li(4)-Li(3)#3	35.1(3)	Li(1)-Li(5)-Li(3)	48.9(4)
O(6)-Li(4)-Li(3)#3	149.2(4)	Li(1)#2-Li(5)-Li(3)	71.3(4)
O(6)#3-Li(4)-Li(3)#3	108.2(4)	O(10)-Li(5)-Li(3)#2	87.9(6)
Li(3)#1-Li(4)-Li(3)#3	42.2(4)	O(7)-Li(5)-Li(3)#2	149.9(6)
O(9)-Li(4)-Li(3)	35.1(3)	O(1)-Li(5)-Li(3)#2	35.9(3)
O(9)#3-Li(4)-Li(3)	75.0(5)	C(1)-Li(5)-Li(3)#2	62.7(4)
O(6)-Li(4)-Li(3)	108.2(4)	Li(1)-Li(5)-Li(3)#2	71.3(4)
O(6)#3-Li(4)-Li(3)	149.2(4)	Li(1)#2-Li(5)-Li(3)#2	48.9(4)
Li(3)#1-Li(4)-Li(3)	44.1(4)	Li(3)-Li(5)-Li(3)#2	45.4(4)
Li(3)#3-Li(4)-Li(3)	62.8(5)	O(10)-Li(5)-O(2)#2	76.3(4)
O(9)-Li(4)-Li(3)#2	75.0(5)	O(7)-Li(5)-O(2)#2	126.0(2)
O(9)#3-Li(4)-Li(3)#2	35.1(3)	O(1)-Li(5)-O(2)#2	67.8(3)
O(6)-Li(4)-Li(3)#2	108.2(4)	C(1)-Li(5)-O(2)#2	85.1(4)
O(6)#3-Li(4)-Li(3)#2	149.2(4)	Li(1)-Li(5)-O(2)#2	84.6(4)
Li(3)#1-Li(4)-Li(3)#2	62.8(5)	Li(1)#2-Li(5)-O(2)#2	36.9(2)
Li(3)#3-Li(4)-Li(3)#2	44.1(4)	Li(3)-Li(5)-O(2)#2	78.9(4)
Li(3)-Li(4)-Li(3)#2	42.2(4)	Li(3)#2-Li(5)-O(2)#2	35.2(2)
O(10)-Li(5)-O(7)	112.6(9)	O(3)-Li(6)-O(8)#4	119.0(13)
O(10)-Li(5)-O(1)	116.4(9)	O(3)-Li(6)-C(38)	107.9(7)

O(8)#4-Li(6)-C(38)	105.8(7)	C(38)-Li(6)-O(3)#3	60.5(5)
O(3)-Li(6)-C(38)#3	107.9(7)	C(38)#3-Li(6)-O(3)#3	60.5(5)
O(8)#4-Li(6)-C(38)#3	105.8(7)	C(20)-Li(6)-O(3)#3	119.5(7)
C(38)-Li(6)-C(38)#3	110.4(9)	Li(6)#3-Li(6)-O(3)#3	23.6(3)
O(3)-Li(6)-C(20)	27.3(4)	Li(1)-Li(6)-O(3)#3	64.1(4)
O(8)#4-Li(6)-C(20)	91.8(9)	Li(1)#1-Li(6)-O(3)#3	64.1(4)
C(38)-Li(6)-C(20)	119.6(6)	O(10)#3-Li(6)-O(3)#3	52.1(3)
C(38)#3-Li(6)-C(20)	119.6(6)	C(1)-O(1)-Li(3)	131.0(4)
O(3)-Li(6)-Li(6)#3	115.9(7)	C(1)-O(1)-Li(3)#2	131.0(4)
O(8)#4-Li(6)-Li(6)#3	125.1(8)	Li(3)-O(1)-Li(3)#2	78.0(6)
C(38)-Li(6)-Li(6)#3	55.5(5)	C(1)-O(1)-Li(5)	100.6(7)
C(38)#3-Li(6)-Li(6)#3	55.5(5)	Li(3)-O(1)-Li(5)	106.9(5)
C(20)-Li(6)-Li(6)#3	143.1(5)	Li(3)#2-O(1)-Li(5)	106.9(5)
O(3)-Li(6)-Li(1)	37.4(4)	C(1)-O(1)-O(2)#2	120.57(12)
O(8)#4-Li(6)-Li(1)	142.7(9)	Li(3)-O(1)-O(2)#2	105.0(3)
C(38)-Li(6)-Li(1)	72.6(5)	Li(3)#2-O(1)-O(2)#2	35.9(3)
C(38)#3-Li(6)-Li(1)	109.6(7)	Li(5)-O(1)-O(2)#2	76.7(3)
C(20)-Li(6)-Li(1)	60.6(5)	O(1)-C(1)-C(2)#2	120.0(3)
Li(6)#3-Li(6)-Li(1)	85.7(5)	O(1)-C(1)-C(2)	120.0(3)
O(3)-Li(6)-Li(1)#1	37.4(4)	C(2)#2-C(1)-C(2)	119.9(6)
O(8)#4-Li(6)-Li(1)#1	142.7(9)	O(1)-C(1)-Li(5)	47.9(5)
C(38)-Li(6)-Li(1)#1	109.6(7)	C(2)#2-C(1)-Li(5)	109.2(4)
C(38)#3-Li(6)-Li(1)#1	72.6(5)	C(2)-C(1)-Li(5)	109.2(4)
C(20)-Li(6)-Li(1)#1	60.6(5)	C(1)-C(2)-C(3)	118.2(5)
Li(6)#3-Li(6)-Li(1)#1	85.7(5)	C(1)-C(2)-C(8)	120.3(5)
Li(1)-Li(6)-Li(1)#1	45.4(5)	C(3)-C(2)-C(8)	121.3(5)
O(3)-Li(6)-O(10)#3	68.4(5)	C(4)-C(3)-C(2)	123.3(5)
O(8)#4-Li(6)-O(10)#3	136.8(2)	C(3)-C(4)-C(3)#2	116.6(7)
C(38)-Li(6)-O(10)#3	111.9(7)	C(3)-C(4)-C(5)	121.7(3)
C(38)#3-Li(6)-O(10)#3	41.2(4)	C(3)#2-C(4)-C(5)	121.7(3)
C(20)-Li(6)-O(10)#3	87.8(5)	C(7)-C(5)-C(6)#2	108.4(10)
Li(6)#3-Li(6)-O(10)#3	66.1(4)	C(7)-C(5)-C(6)	108.4(10)
Li(1)-Li(6)-O(10)#3	71.1(4)	C(6)#2-C(5)-C(6)	106.0(15)
Li(1)#1-Li(6)-O(10)#3	31.6(2)	C(7)-C(5)-C(4)	108.7(11)
O(3)-Li(6)-O(3)#3	92.3(8)	C(6)#2-C(5)-C(4)	112.5(7)
O(8)#4-Li(6)-O(3)#3	148.7(11)	C(6)-C(5)-C(4)	112.5(7)

C(9)-O(2)-Li(3)	125.7(4)	Li(1)-O(3)-O(2)#1	84.6(3)
C(9)-O(2)-Li(1)	120.5(4)	Li(1)#1-O(3)-O(2)#1	39.1(3)
Li(3)-O(2)-Li(1)	78.6(4)	O(3)-C(20)-C(21)#1	121.0(3)
C(9)-O(2)-Li(2)	126.1(4)	O(3)-C(20)-C(21)	121.0(3)
Li(3)-O(2)-Li(2)	99.0(5)	C(21)#1-C(20)-C(21)	118.1(7)
Li(1)-O(2)-Li(2)	94.2(5)	O(3)-C(20)-Li(2)	87.4(5)
C(10)-C(8)-C(2)	115.8(4)	C(21)#1-C(20)-Li(2)	91.5(4)
O(2)-C(9)-C(10)	121.8(5)	C(21)-C(20)-Li(2)	91.5(4)
O(2)-C(9)-C(14)	119.7(5)	O(3)-C(20)-Li(6)	37.5(5)
C(10)-C(9)-C(14)	118.5(5)	C(21)#1-C(20)-Li(6)	114.0(4)
C(9)-C(10)-C(11)	119.2(5)	C(21)-C(20)-Li(6)	114.0(4)
C(9)-C(10)-C(8)	124.1(4)	Li(2)-C(20)-Li(6)	124.8(6)
C(11)-C(10)-C(8)	116.6(5)	C(22)-C(21)-C(20)	120.1(5)
C(12)-C(11)-C(10)	122.8(5)	C(22)-C(21)-C(19)	118.6(5)
C(11)-C(12)-C(13)	116.6(5)	C(20)-C(21)-C(19)	121.3(5)
C(11)-C(12)-C(15)	122.9(6)	C(21)-C(22)-C(23)	122.4(6)
C(13)-C(12)-C(15)	120.4(6)	C(22)-C(23)-C(22)#1	117.0(7)
C(12)-C(13)-C(14)	123.6(6)	C(22)-C(23)-C(24)	121.5(4)
C(13)-C(14)-C(9)	119.0(5)	C(22)#1-C(23)-C(24)	121.5(4)
C(13)-C(14)-C(19)	119.8(5)	C(26)-C(24)-C(25)	107.5(8)
C(9)-C(14)-C(19)	121.2(5)	C(26)-C(24)-C(25)#1	107.5(8)
C(16)-C(15)-C(17)	108.2(7)	C(25)-C(24)-C(25)#1	111.0(14)
C(16)-C(15)-C(18)	109.5(7)	C(26)-C(24)-C(23)	107.3(10)
C(17)-C(15)-C(18)	110.2(8)	C(25)-C(24)-C(23)	111.6(6)
C(16)-C(15)-C(12)	110.8(6)	C(25)#1-C(24)-C(23)	111.6(6)
C(17)-C(15)-C(12)	108.5(6)	Li(1)#1-O(4)-Li(1)	69.3(5)
C(18)-C(15)-C(12)	109.7(6)	Li(1)#1-O(4)-Li(1)#2	111.0(6)
C(14)-C(19)-C(21)	115.3(5)	Li(1)-O(4)-Li(1)#2	73.2(5)
C(20)-O(3)-Li(6)	115.3(8)	Li(1)#1-O(4)-Li(1)#3	73.2(5)
C(20)-O(3)-Li(1)	121.9(4)	Li(1)-O(4)-Li(1)#3	111.0(6)
Li(6)-O(3)-Li(1)	108.4(6)	Li(1)#2-O(4)-Li(1)#3	69.3(5)
C(20)-O(3)-Li(1)#1	121.9(4)	Li(1)#1-O(4)-Li(3)#2	177.0(4)
Li(6)-O(3)-Li(1)#1	108.4(6)	Li(1)-O(4)-Li(3)#2	109.9(3)
Li(1)-O(3)-Li(1)#1	74.1(5)	Li(1)#2-O(4)-Li(3)#2	71.2(4)
C(20)-O(3)-O(2)#1	84.1(3)	Li(1)#3-O(4)-Li(3)#2	109.6(3)
Li(6)-O(3)-O(2)#1	141.6(3)	Li(1)#1-O(4)-Li(3)#3	109.9(3)

Li(1)-O(4)-Li(3)#3	177.0(4)	Li(1)#2-O(4)-O(2)#2	37.8(3)
Li(1)#2-O(4)-Li(3)#3	109.6(3)	Li(1)#3-O(4)-O(2)#2	80.5(3)
Li(1)#3-O(4)-Li(3)#3	71.2(4)	Li(3)#2-O(4)-O(2)#2	36.6(3)
Li(3)#2-O(4)-Li(3)#3	70.8(5)	Li(3)#3-O(4)-O(2)#2	80.5(2)
Li(1)#1-O(4)-Li(3)#1	71.2(4)	Li(3)#1-O(4)-O(2)#2	139.5(4)
Li(1)-O(4)-Li(3)#1	109.6(3)	Li(3)-O(4)-O(2)#2	97.1(3)
Li(1)#2-O(4)-Li(3)#1	177.0(4)	O(1)#3-O(4)-O(2)#2	117.97(13)
Li(1)#3-O(4)-Li(3)#1	109.9(3)	O(10)#3-O(4)-O(2)#2	117.82(13)
Li(3)#2-O(4)-Li(3)#1	106.7(6)	Li(1)#1-O(4)-O(2)#1	37.8(3)
Li(3)#3-O(4)-Li(3)#1	67.5(5)	Li(1)-O(4)-O(2)#1	80.5(3)
Li(1)#1-O(4)-Li(3)	109.6(3)	Li(1)#2-O(4)-O(2)#1	146.2(4)
Li(1)-O(4)-Li(3)	71.2(4)	Li(1)#3-O(4)-O(2)#1	101.8(3)
Li(1)#2-O(4)-Li(3)	109.9(3)	Li(3)#2-O(4)-O(2)#1	139.5(4)
Li(1)#3-O(4)-Li(3)	177.0(4)	Li(3)#3-O(4)-O(2)#1	97.1(3)
Li(3)#2-O(4)-Li(3)	67.5(5)	Li(3)#1-O(4)-O(2)#1	36.6(3)
Li(3)#3-O(4)-Li(3)	106.7(6)	Li(3)-O(4)-O(2)#1	80.5(2)
Li(3)#1-O(4)-Li(3)	70.8(5)	O(1)#3-O(4)-O(2)#1	59.84(7)
Li(1)#1-O(4)-O(1)#3	76.4(3)	O(10)#3-O(4)-O(2)#1	64.84(9)
Li(1)-O(4)-O(1)#3	140.2(3)	O(2)#2-O(4)-O(2)#1	175.9(3)
Li(1)#2-O(4)-O(1)#3	140.2(3)	C(27)#1-O(5)-C(27)	109.1(7)
Li(1)#3-O(4)-O(1)#3	76.4(3)	C(27)#1-O(5)-Li(2)	125.1(3)
Li(3)#2-O(4)-O(1)#3	103.3(3)	C(27)-O(5)-Li(2)	125.1(3)
Li(3)#3-O(4)-O(1)#3	37.6(2)	O(5)-C(27)-C(28)	106.2(6)
Li(3)#1-O(4)-O(1)#3	37.6(2)	C(28)#1-C(28)-C(27)	108.8(4)
Li(3)-O(4)-O(1)#3	103.3(3)	C(29)-O(6)-C(29)#2	88.3(15)
Li(1)#1-O(4)-O(10)#3	37.6(3)	C(29)-O(6)-Li(4)	131.4(7)
Li(1)-O(4)-O(10)#3	97.4(3)	C(29)#2-O(6)-Li(4)	131.4(7)
Li(1)#2-O(4)-O(10)#3	97.4(3)	O(6)-C(29)-C(30)	109.6(12)
Li(1)#3-O(4)-O(10)#3	37.6(3)	O(6)-C(29)-C(29)#2	45.9(8)
Li(3)#2-O(4)-O(10)#3	145.0(2)	C(30)-C(29)-C(29)#2	75.8(8)
Li(3)#3-O(4)-O(10)#3	83.2(3)	C(30)#2-C(30)-C(29)	104.2(8)
Li(3)#1-O(4)-O(10)#3	83.2(3)	C(31)-O(7)-C(31)#2	104.0(12)
Li(3)-O(4)-O(10)#3	145.0(2)	C(31)-O(7)-Li(5)	127.8(6)
O(1)#3-O(4)-O(10)#3	63.95(15)	C(31)#2-O(7)-Li(5)	127.8(6)
Li(1)#1-O(4)-O(2)#2	146.2(4)	O(7)-C(31)-C(32)	112.1(13)
Li(1)-O(4)-O(2)#2	101.8(3)	C(32)#2-C(32)-C(31)	104.8(9)

C(34)-C(33)-O(9)	135.9(10)	Li(6)-C(38)-Li(6)#3	69.1(9)
C(35)#1-O(8)-C(35)	107.4(13)	C(39)-O(11)-C(42)	73(3)
C(35)#1-O(8)-Li(6)#5	122.9(7)	C(40)-C(39)-O(11)	92(3)
C(35)-O(8)-Li(6)#5	122.9(7)	C(40)-C(39)-C(42)	71(4)
O(8)-C(35)-C(36)	108.7(11)	O(11)-C(39)-C(42)	59(2)
C(36)#1-C(36)-C(35)	107.1(8)	C(39)-C(40)-C(41)	110(4)
C(33)-O(9)-Li(3)#1	120.5(4)	C(39)-C(40)-C(42)	67(3)
C(33)-O(9)-Li(3)	120.5(4)	C(41)-C(40)-C(42)	54(3)
Li(3)#1-O(9)-Li(3)	76.0(6)	C(40)-C(41)-C(42)	78(3)
C(33)-O(9)-Li(4)	114.8(6)	C(41)-C(42)-O(11)	75(2)
Li(3)#1-O(9)-Li(4)	109.5(4)	C(41)-C(42)-C(39)	83(3)
Li(3)-O(9)-Li(4)	109.5(4)	O(11)-C(42)-C(39)	49(2)
C(37)-O(10)-Li(5)	128.7(8)	C(41)-C(42)-C(40)	48(2)
C(37)-O(10)-Li(1)	120.1(5)	O(11)-C(42)-C(40)	69(2)
Li(5)-O(10)-Li(1)	97.5(5)	C(39)-C(42)-C(40)	42(2)
C(37)-O(10)-Li(1)#2	120.1(5)	C(44)#6-O(12)-C(44)	49(3)
Li(5)-O(10)-Li(1)#2	97.5(5)	C(43)#6-C(43)-C(44)	90.0(15)
Li(1)-O(10)-Li(1)#2	81.3(6)	C(44)#6-C(44)-O(12)	65.4(17)
C(38)-C(37)-O(10)	131.5(10)	C(44)#6-C(44)-C(43)	90.0(15)
C(37)-C(38)-Li(6)	112.8(8)	O(12)-C(44)-C(43)	75(4)
C(37)-C(38)-Li(6)#3	112.8(8)		

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+1/2,z #2 -x+1/2,y,z #3 -x+1/2,-y+1/2,z

#4 x,y,z-1 #5 x,y,z+1 #6 x,-y+3/2,z

Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $[Li_{15}$(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4]$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Li(1)	63(5)	56(5)	77(6)	2(4)	-4(4)	2(4)
Li(2)	63(7)	67(8)	79(8)	0	4(6)	0
Li(3)	57(5)	47(4)	95(7)	0(4)	3(5)	2(4)
Li(4)	62(10)	74(12)	60(11)	0	0	0
Li(5)	159(17)	65(9)	81(10)	11(8)	0	0

Li(6)	94(14)	460(60)	88(14)	0	-19(11)	0
O(1)	55(3)	48(3)	89(4)	-12(3)	0	0
C(1)	64(4)	43(4)	66(5)	-4(3)	0	0
C(2)	56(3)	50(3)	79(4)	-3(3)	5(3)	7(2)
C(3)	66(3)	54(3)	108(5)	-21(3)	7(3)	7(3)
C(4)	67(5)	57(5)	98(6)	-24(4)	0	0
C(5)	82(6)	81(7)	148(10)	-38(7)	0	0
C(6)	208(12)	115(8)	390(20)	-132(12)	-109(14)	82(8)
C(7)	800(80)	111(13)	146(16)	-72(12)	0	0
O(2)	50(2)	45(2)	81(2)	1(2)	-7(2)	7(2)
C(8)	58(3)	49(3)	75(3)	-3(2)	2(3)	0(2)
C(9)	45(2)	48(3)	79(4)	7(3)	1(2)	-2(2)
C(10)	46(3)	47(3)	86(4)	0(3)	9(3)	-2(2)
C(11)	51(3)	50(3)	102(4)	-2(3)	3(3)	5(2)
C(12)	57(3)	50(3)	102(5)	1(3)	8(3)	5(3)
C(13)	52(3)	58(3)	106(5)	19(3)	-2(3)	3(3)
C(14)	48(3)	48(3)	86(4)	7(3)	0(3)	-4(2)
C(15)	83(4)	66(4)	118(5)	13(4)	1(4)	27(3)
C(16)	116(6)	64(4)	193(9)	-21(5)	-20(6)	38(4)
C(17)	75(5)	128(7)	210(10)	-35(7)	-23(5)	49(5)
C(18)	199(11)	85(6)	193(10)	41(6)	30(8)	72(7)
C(19)	65(3)	57(3)	80(4)	10(3)	-7(3)	6(3)
O(3)	45(3)	65(3)	88(4)	0	-7(3)	0
C(20)	59(4)	63(5)	64(5)	0	-8(4)	0
C(21)	61(3)	63(3)	69(3)	6(3)	-8(3)	1(3)
C(22)	55(3)	71(4)	84(4)	8(3)	-12(3)	2(3)
C(23)	58(4)	91(6)	82(6)	0	-12(4)	0
C(24)	55(5)	92(7)	127(9)	0	-17(5)	0
C(25)	54(4)	231(14)	370(20)	-124(14)	0(7)	30(7)
C(26)	80(8)	380(30)	175(15)	0	-54(9)	0
O(4)	57(4)	43(3)	65(4)	0	0	0
O(5)	61(3)	62(3)	110(5)	0	10(3)	0
C(27)	86(5)	72(4)	148(7)	-3(4)	15(5)	15(4)
C(28)	131(7)	98(5)	192(9)	-7(5)	78(7)	15(5)
O(6)	80(4)	151(8)	136(7)	-58(6)	0	0
C(29)	254(16)	182(12)	195(13)	1(10)	-14(11)	91(11)

C(30)	168(12)	370(20)	307(19)	-260(19)	17(11)	-2(12)
O(7)	103(5)	57(3)	143(6)	8(4)	0	0
C(31)	97(7)	142(11)	570(40)	155(17)	23(13)	11(7)
C(32)	240(20)	120(11)	790(60)	30(20)	40(20)	58(12)
C(33)	59(4)	56(4)	78(5)	0	13(4)	0
C(34)	84(8)	390(30)	82(9)	0	4(7)	0
O(8)	128(7)	169(9)	98(6)	0	-17(5)	0
C(35)	233(16)	139(10)	191(12)	10(9)	-52(12)	-40(10)
C(36)	240(16)	164(13)	450(30)	20(14)	-186(19)	19(11)
O(9)	45(3)	53(3)	77(3)	0	3(2)	0
O(10)	66(3)	58(3)	77(4)	11(3)	0	0
C(37)	77(6)	95(7)	86(7)	29(6)	0	0
C(38)	80(7)	198(13)	78(7)	-6(8)	0	0
O(11)	390(30)	410(30)	530(40)	130(30)	-150(30)	40(30)
C(39)	550(70)	260(30)	350(40)	140(30)	150(50)	-40(40)
C(40)	400(60)	250(30)	430(60)	20(40)	120(40)	150(40)
C(41)	260(30)	480(60)	430(60)	120(50)	90(30)	10(40)
C(42)	470(70)	500(60)	230(30)	-70(30)	-10(40)	-80(60)
O(12)	520(80)	530(80)	1100(200)	0	-400(110)	0
C(43)	720(80)	320(50)	270(30)	-30(30)	-40(40)	20(40)
C(44)	540(50)	480(80)	310(30)	0(30)	-20(30)	-220(50)
O(13)	540(20)	450(20)	355(18)	1(15)	0	0

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [Li_{15}(4-tert-butylcalix[8]arene-8H)$(THF)_{12}O(OH)(EtO)_4$].

x	y	z	U(eq)
H(3)	1606	5066	5543	91
H(6A)	1602	5907	5971	357
H(6B)	1975	6232	5267	357
H(6C)	1969	6473	6198	357
H(7A)	2853	5424	7089	529
H(7B)	2143	5431	7091	529
H(7C)	2504	6011	7248	529

H(8A)	1435	3636	4787	73
H(8B)	1080	4152	5205	73
H(11)	731	4959	4328	81
H(13)	291	4623	1941	87
H(16A)	599	5958	3891	187
H(16B)	73	5636	4368	187
H(16C)	-65	6194	3823	187
H(17A)	-666	5038	2571	206
H(17B)	-834	5634	3001	206
H(17C)	-695	5085	3562	206
H(18A)	181	5549	1781	238
H(18B)	657	5911	2299	238
H(18C)	-12	6135	2222	238
H(19A)	1268	3550	1533	81
H(19B)	678	3843	1183	81
H(22)	-313	3373	1656	84
H(25A)	-1225	3375	1666	329
H(25B)	-1319	3043	2526	329
H(25C)	-1829	3034	1834	329
H(26A)	-1115	2846	381	320
H(26B)	-1708	2493	568	320
H(26C)	-1103	2161	377	320
H(27A)	-382	3212	3633	122
H(27B)	101	3273	4364	122
H(28A)	-531	2945	5273	168
H(28B)	-1030	2945	4562	168
H(29A)	1667	3376	6931	252
H(29B)	2027	3861	6437	252
H(30A)	2058	4230	7684	339
H(30B)	2058	3611	8130	339
H(31A)	1736	5120	3120	323
H(31B)	1850	5181	2142	323
H(32A)	2034	6034	2349	459
H(32B)	2034	5941	3341	459
H(33A)	1059	2837	5082	77
H(33B)	1059	2163	5082	77

H(35A)	1240	3192	8944	225
H(35B)	957	3262	9854	225
H(36A)	119	2957	9422	342
H(36B)	383	2957	8494	342
H(37A)	2150	4001	1199	104
H(37B)	2850	4001	1199	104
H(43A)	1820	7042	1793	522
H(43B)	1157	7042	1387	522

V.2.10. Crystal data and structure refinement for $[Li_2$(4-tert-butylcalix[8]arene-2H)$(THF)_7(H_2O)_{12}]$ 10

Empirical formula	C116 H220 Li2 O27	
Formula weight	2060.86	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 20.413(4) Å	α= 90°.
b = 32.402(7) Å	β= 114.84(3)°.	
c = 21.638(4) Å	γ = 90°.	
Volume	12987(5) $Å^3$	
Z	4	
Density (calculated)	0.993 Mg/m^3	
Absorption coefficient	0.070 mm^{-1}	
F(000)	4076	
Theta range for data collection	1.91 to 26.92°.	
Index ranges	-25<=h<=23, 0<=k<=24, 0<=l<=27	
Reflections collected	12222	
Completeness to theta = 26.92°	43.5 %	
Absorption correction	Spherical	

Refinement method	Full-matrix-block least-squares on F^2
Data / restraints / parameters	12222 / 0 / 1306
Goodness-of-fit on F^2	1.500
Final R indices [I>2sigma(I)]	R1 = 0.1442, wR2 = 0.2865
R indices (all data)	R1 = 0.2438, wR2 = 0.3279
Largest diff. peak and hole	0.436 and -0.297 e.Å^{-3}

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for [Li_2(4-tert-butylcalix[8]arene-2H)$(THF)_7(H_2O)_{12}$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Li(1)	-526(6)	9178(8)	-977(6)	91
Li(2)	-29(6)	9102(7)	685(6)	50
O(1)	1017(2)	9070(3)	-1523(2)	64
C(1)	1131(4)	9200(4)	-2067(4)	58
C(2)	1736(3)	9061(4)	-2152(4)	44
C(3)	1873(4)	9204(4)	-2695(4)	57
C(4)	1415(4)	9481(4)	-3176(4)	71
C(5)	812(4)	9598(4)	-3072(4)	68
C(6)	656(4)	9477(4)	-2545(4)	48
C(7)	1573(5)	9666(5)	-3757(5)	102
C(8)	905(6)	9747(6)	-4391(6)	145
C(9)	2078(13)	10013(11)	-3505(10)	358
C(10)	2004(12)	9336(10)	-3959(10)	266
C(11)	-14(4)	9649(4)	-2472(4)	63
O(2)	-260(2)	8982(3)	-1682(2)	66
C(12)	-747(3)	9027(4)	-2363(4)	56
C(13)	-644(4)	9330(4)	-2763(4)	45
C(14)	-1132(4)	9364(4)	-3444(4)	54
C(15)	-1724(4)	9096(4)	-3741(4)	53
C(16)	-1802(4)	8796(4)	-3325(4)	49
C(17)	-1334(3)	8737(4)	-2633(3)	45
C(18)	-2233(5)	9130(5)	-4514(4)	93
C(19)	-1804(10)	8988(8)	-4909(6)	229

C(20)	-2387(8)	9565(6)	-4724(6)	173
C(21)	-2908(8)	8901(9)	-4668(7)	306
C(22)	-1459(4)	8408(4)	-2210(4)	52
O(3)	-1226(2)	8774(2)	-931(2)	59
C(23)	-1915(3)	8678(3)	-1350(4)	44
C(24)	-2057(3)	8485(4)	-1986(3)	54
C(25)	-2775(4)	8382(4)	-2415(4)	49
C(26)	-3355(3)	8462(4)	-2255(4)	56
C(27)	-3191(4)	8625(4)	-1610(4)	48
C(28)	-2488(3)	8742(4)	-1160(4)	46
C(29)	-4135(4)	8320(5)	-2720(4)	80
C(30)	-4235(5)	8245(5)	-3428(5)	127
C(31)	-4649(5)	8719(5)	-2782(6)	129
C(32)	-4389(7)	8008(7)	-2370(8)	207
C(33)	-2345(4)	8931(4)	-472(3)	48
O(4)	-833(2)	8755(3)	296(2)	58
C(34)	-1320(3)	8565(4)	490(4)	50
C(35)	-2065(3)	8631(3)	134(3)	33
C(36)	-2545(4)	8439(4)	343(4)	42
C(37)	-2304(4)	8168(4)	906(4)	62
C(38)	-1557(4)	8107(4)	1251(4)	56
C(39)	-1064(3)	8307(4)	1063(4)	52
C(40)	-2831(4)	7908(6)	1127(5)	126
C(41)	-3588(6)	7973(8)	637(8)	218
C(42)	-2637(9)	7647(14)	1570(30)	675
C(43)	-2740(20)	8139(13)	1780(16)	385
C(44)	-249(3)	8240(4)	1484(4)	57
O(5)	708(2)	8911(2)	1549(2)	50
C(45)	554(3)	8854(4)	2099(4)	58
C(46)	74(4)	8545(4)	2081(4)	55
C(47)	-100(4)	8478(4)	2634(4)	73
C(48)	181(4)	8743(4)	3213(4)	71
C(49)	653(4)	9038(4)	3221(4)	78
C(50)	852(3)	9108(4)	2673(4)	56
C(51)	-4(6)	8632(8)	3837(5)	155
C(52)	-320(14)	9038(10)	3943(12)	259

C(53)	669(10)	8626(8)	4469(7)	203
C(54)	-503(19)	8356(15)	3742(13)	520
C(55)	1409(4)	9444(4)	2736(4)	52
O(6)	2034(2)	9025(3)	1932(2)	65
C(56)	2442(4)	9066(4)	2631(4)	47
C(57)	2169(3)	9271(4)	3033(4)	46
C(58)	2610(4)	9320(4)	3726(4)	51
C(59)	3309(4)	9142(4)	4034(4)	72
C(60)	3549(4)	8921(4)	3622(4)	65
C(61)	3131(4)	8877(4)	2913(4)	58
C(62)	3781(6)	9221(7)	4803(5)	154
C(63)	4492(10)	9316(12)	4926(8)	395
C(64)	3446(12)	9377(10)	5155(7)	346
C(65)	3863(19)	8755(9)	5111(8)	358
C(66)	3422(4)	8621(4)	2480(4)	56
O(7)	2710(2)	9027(3)	1172(3)	68
C(67)	3452(3)	9099(4)	1533(4)	54
C(68)	3820(3)	8901(4)	2164(4)	47
C(69)	4574(4)	8959(4)	2509(4)	68
C(70)	4947(4)	9196(4)	2257(4)	74
C(71)	4562(4)	9412(4)	1619(4)	68
C(72)	3813(3)	9341(4)	1256(4)	49
C(73)	5776(4)	9279(6)	2650(5)	122
C(74)	6132(5)	9384(7)	2192(6)	176
C(75)	6135(7)	8884(8)	3044(13)	276
C(76)	5901(8)	9582(11)	3168(11)	331
C(77)	3416(4)	9564(4)	578(4)	45
O(8)	2210(2)	9138(3)	-442(3)	70
C(78)	2846(3)	9142(4)	-534(4)	52
C(79)	3451(3)	9353(4)	-21(4)	51
C(80)	4104(4)	9353(4)	-111(4)	47
C(81)	4188(3)	9157(4)	-640(4)	64
C(83)	2905(4)	8964(4)	-1072(4)	57
C(82)	3576(3)	8967(4)	-1125(4)	52
C(84)	4903(4)	9158(5)	-720(5)	69
C(85)	5533(4)	9323(5)	-89(6)	126

C(86)	4815(6)	9423(7)	-1334(7)	145
C(87)	5081(5)	8695(5)	-832(6)	97
C(88)	2259(4)	8749(4)	-1639(5)	81
O(9)	1360(4)	4646(4)	3722(6)	140
C(89)	1298(8)	4396(7)	3178(10)	150
C(90)	1988(9)	4363(9)	3127(10)	200
C(91)	2468(7)	4661(8)	3643(10)	181
C(92)	2058(6)	4821(6)	4025(8)	122
O(10)	1525(6)	2610(6)	691(7)	200
C(93)	2006(12)	2496(11)	405(12)	260
C(94)	2704(10)	2614(11)	865(11)	254
C(95)	2662(13)	2794(11)	1420(12)	262
C(96)	1971(11)	2727(10)	1380(12)	235
O(11)	3647(13)	7940(11)	550(20)	464
C(97)	4450(30)	7690(20)	850(50)	1154
C(98)	4162(16)	7782(14)	1326(18)	334
C(99)	4647(15)	8281(16)	1040(30)	424
C(100)	4170(30)	8158(16)	441(16)	428
O(12)	2564(19)	10364(13)	2530(20)	515
C(101)	2986(11)	10235(13)	2150(20)	371
C(102)	3526(16)	10155(9)	2378(10)	212
C(103)	3999(17)	10110(13)	3100(30)	419
C(104)	3357(19)	10308(10)	3170(20)	258
O(13)	414(12)	1928(10)	3739(19)	416
C(105)	-20(20)	1832(18)	3150(20)	411
C(106)	-610(20)	1926(14)	3043(17)	411
C(107)	-545(12)	1908(12)	3844(16)	273
C(108)	-90(20)	1611(16)	3920(20)	374
O(14)	1593(14)	7602(12)	3455(17)	386
C(109)	2200(20)	7735(14)	3192(16)	292
C(110)	2260(30)	7879(15)	4126(17)	425
C(111)	2013(17)	8134(10)	3540(20)	247
C(112)	2330(30)	7491(18)	4040(20)	484
O(15)	2419(10)	7255(9)	1388(14)	357
C(113)	2460(30)	6880(20)	1020(20)	431
C(114)	3187(17)	6748(14)	1600(30)	391

C(115)	3210(60)	6830(30)	2210(40)	992
C(116)	3088(17)	7205(14)	2105(11)	288
O(16)	-389(4)	9674(3)	724(4)	96
O(17)	1954(5)	9821(5)	1404(6)	155
O(18)	-731(4)	9745(3)	-973(5)	107
O(19)	347(3)	9069(3)	-43(3)	68
O(20)	1392(3)	9629(3)	108(4)	92
O(21)	872(10)	7557(9)	401(12)	411
O(22)	1634(8)	8099(6)	1600(13)	285
O(23)	1794(19)	8371(9)	314(10)	409
O(24)	403(8)	2044(7)	491(9)	246
O(25)	795(10)	8230(8)	-947(12)	342
O(26)	4983(15)	7736(11)	3664(10)	442
O(27)	5128(15)	7973(10)	4432(11)	456

Bond lengths [Å] and angles [°] for $[Li_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_7(H_2O)_{12}]$.

Li(1)-O(18)	1.88(3)	O(1)-C(1)	1.359(10)
Li(1)-O(2)	1.929(16)	O(1)-O(2)	2.499(7)
Li(1)-O(3)	1.97(2)	O(1)-O(8)	2.581(5)
Li(1)-O(19)	2.087(13)	O(1)-O(25)	3.11(3)
Li(1)-Li(2)	3.310(18)	C(1)-C(2)	1.397(12)
Li(1)-O(1)	3.818(14)	C(1)-C(6)	1.405(14)
Li(1)-O(20)#1	3.905(13)	C(2)-C(3)	1.397(12)
Li(1)-O(25)	4.07(2)	C(2)-C(88)	1.547(11)
Li(1)-O(16)#2	4.09(2)	C(3)-C(4)	1.396(15)
Li(1)-O(24)#3	4.08(3)	C(4)-C(5)	1.395(13)
Li(2)-O(4)	1.872(17)	C(4)-C(7)	1.544(14)
Li(2)-O(5)	1.944(13)	C(5)-C(6)	1.364(12)
Li(2)-O(16)#1	2.01(2)	C(6)-C(11)	1.545(13)
Li(2)-O(16)	2.01(2)	C(7)-C(9)	1.47(2)
Li(2)-O(19)	2.026(13)	C(7)-C(8)	1.496(14)
Li(2)-O(6)	3.942(12)	C(7)-C(10)	1.56(2)
Li(2)-O(18)#2	3.99(2)	C(11)-C(13)	1.560(14)
Li(2)-O(20)#1	3.993(14)	O(2)-C(12)	1.397(8)
Li(2)-O(9)	15.62(2)	O(2)-O(25)	3.20(2)

C(12)-C(13)	1.383(14)	C(40)-C(42)	1.22(2)
C(12)-C(17)	1.440(14)	C(40)-C(41)	1.476(14)
C(13)-C(14)	1.394(10)	C(40)-C(43)	1.54(3)
C(14)-C(15)	1.404(14)	C(42)-C(43)	1.69(6)
C(15)-C(16)	1.378(13)	C(44)-C(46)	1.538(14)
C(15)-C(18)	1.560(10)	O(5)-C(45)	1.367(9)
C(16)-C(17)	1.411(10)	O(5)-O(6)	2.504(7)
C(17)-C(22)	1.494(13)	O(5)-O(22)	3.213(18)
C(18)-C(21)	1.474(18)	C(45)-C(46)	1.388(14)
C(18)-C(20)	1.47(2)	C(45)-C(50)	1.398(13)
C(18)-C(19)	1.529(19)	C(46)-C(47)	1.400(12)
C(22)-C(24)	1.512(11)	C(47)-C(48)	1.424(15)
O(3)-C(23)	1.351(8)	C(48)-C(49)	1.352(15)
O(3)-O(4)	2.431(7)	C(48)-C(51)	1.587(15)
O(3)-O(24)	22.08(2)	C(49)-C(50)	1.426(12)
C(23)-C(28)	1.410(11)	C(50)-C(55)	1.539(14)
C(23)-C(24)	1.426(12)	C(51)-C(54)	1.31(2)
C(24)-C(25)	1.406(9)	C(51)-C(53)	1.477(17)
C(25)-C(26)	1.392(11)	C(51)-C(52)	1.53(3)
C(26)-C(27)	1.393(12)	C(55)-C(57)	1.516(11)
C(26)-C(29)	1.552(10)	O(6)-C(56)	1.392(9)
C(27)-C(28)	1.407(10)	O(6)-O(7)	2.552(8)
C(28)-C(33)	1.520(12)	O(6)-O(22)	3.11(2)
C(29)-C(32)	1.482(18)	O(6)-O(17)#2	9.109(11)
C(29)-C(30)	1.478(14)	C(56)-C(57)	1.385(12)
C(29)-C(31)	1.635(19)	C(56)-C(61)	1.415(12)
C(33)-C(35)	1.537(13)	C(57)-C(58)	1.397(11)
O(4)-C(34)	1.376(10)	C(58)-C(59)	1.419(12)
C(34)-C(35)	1.402(9)	C(59)-C(60)	1.384(14)
C(34)-C(39)	1.404(13)	C(59)-C(62)	1.555(13)
C(35)-C(36)	1.386(11)	C(60)-C(61)	1.416(11)
C(36)-C(37)	1.413(14)	C(61)-C(66)	1.545(14)
C(37)-C(38)	1.403(10)	C(62)-C(64)	1.32(2)
C(37)-C(40)	1.590(14)	C(62)-C(63)	1.397(19)
C(38)-C(39)	1.393(12)	C(62)-C(65)	1.63(3)
C(39)-C(44)	1.540(10)	C(66)-C(68)	1.555(13)

O(7)-C(67)	1.402(8)	O(10)-C(93)	1.41(2)
O(7)-O(23)	2.924(18)	O(10)-C(96)	1.43(2)
O(7)-O(8)	3.221(8)	O(10)-O(24)	2.82(3)
O(7)-O(17)#2	9.541(10)	C(93)-C(94)	1.41(2)
C(67)-C(72)	1.374(13)	C(94)-C(95)	1.37(3)
C(67)-C(68)	1.406(12)	C(95)-C(96)	1.39(3)
C(68)-C(69)	1.412(10)	O(11)-C(100)	1.38(4)
C(69)-C(70)	1.349(13)	O(11)-C(98)	1.64(4)
C(70)-C(71)	1.449(13)	O(11)-C(97)	1.69(5)
C(70)-C(73)	1.564(11)	C(97)-C(98)	1.41(8)
C(71)-C(72)	1.413(10)	C(97)-C(100)	1.72(6)
C(72)-C(77)	1.527(12)	C(97)-C(99)	1.95(10)
C(73)-C(76)	1.43(3)	C(99)-C(100)	1.32(4)
C(73)-C(74)	1.494(15)	O(12)-C(101)	1.49(4)
C(73)-C(75)	1.54(2)	O(12)-C(104)	1.64(5)
C(77)-C(79)	1.495(12)	O(12)-O(17)	2.83(5)
O(8)-C(78)	1.393(9)	C(101)-C(102)	1.03(3)
O(8)-O(23)	3.28(2)	C(101)-C(104)	2.04(5)
O(8)-O(20)#2	8.663(8)	C(102)-C(103)	1.46(5)
C(78)-C(83)	1.349(12)	C(102)-C(104)	1.96(3)
C(78)-C(79)	1.439(12)	C(103)-C(104)	1.53(5)
C(79)-C(80)	1.428(11)	O(13)-C(105)	1.25(4)
C(80)-C(81)	1.380(13)	O(13)-C(108)	1.60(4)
C(81)-C(82)	1.392(11)	C(105)-C(106)	1.17(5)
C(81)-C(84)	1.539(11)	C(105)-C(108)	1.87(5)
C(83)-C(82)	1.421(11)	C(106)-C(107)	1.68(5)
C(83)-C(88)	1.538(12)	C(106)-C(108)	2.01(4)
C(84)-C(86)	1.528(17)	C(107)-C(108)	1.31(4)
C(84)-C(85)	1.525(13)	O(14)-C(112)	1.54(4)
C(84)-C(87)	1.588(18)	O(14)-C(109)	1.62(3)
O(9)-C(89)	1.39(2)	O(14)-C(110)	1.77(4)
O(9)-C(92)	1.412(14)	O(14)-C(111)	1.90(4)
O(9)-O(16)#1	17.342(18)	C(109)-C(111)	1.61(4)
C(89)-C(90)	1.46(2)	C(109)-C(112)	1.91(6)
C(90)-C(91)	1.49(3)	C(109)-C(110)	2.02(5)
C(91)-C(92)	1.50(2)	C(110)-C(112)	1.29(6)

C(110)-C(111)	1.42(3)	O(17)-O(20)#1	2.621(14)
O(15)-C(113)	1.47(5)	O(17)-O(18)#2	2.669(14)
O(15)-C(116)	1.59(3)	O(19)-O(20)#1	2.714(10)
O(15)-O(21)	3.13(2)	O(20)-O(20)#1	0.00(3)
C(113)-C(114)	1.54(6)	O(21)-O(22)	2.97(4)
C(114)-C(115)	1.33(7)	O(21)-O(23)	3.29(3)
C(114)-C(116)	1.90(5)	O(21)-O(24)	17.89(3)
C(115)-C(116)	1.24(8)	O(22)-O(23)	3.06(3)
O(16)-O(16)#1	0.00(3)	O(23)-O(25)	2.67(3)
O(16)-O(18)#2	2.834(13)	O(24)-O(25)	20.35(3)
O(16)-O(20)#1	4.369(11)	O(26)-O(27)	1.74(3)

O(18)-Li(1)-O(2)	117.5(11)	O(19)-Li(1)-O(25)	64.3(6)
O(18)-Li(1)-O(3)	118.7(8)	Li(2)-Li(1)-O(25)	91.6(6)
O(2)-Li(1)-O(3)	105.7(11)	O(1)-Li(1)-O(25)	46.2(4)
O(18)-Li(1)-O(19)	105.2(8)	O(20)#1-Li(1)-O(25)	77.4(4)
O(2)-Li(1)-O(19)	107.5(8)	O(18)-Li(1)-O(16)#2	37.2(4)
O(3)-Li(1)-O(19)	100.5(9)	O(2)-Li(1)-O(16)#2	98.1(7)
O(18)-Li(1)-Li(2)	92.0(7)	O(3)-Li(1)-O(16)#2	153.7(9)
O(2)-Li(1)-Li(2)	140.8(10)	O(19)-Li(1)-O(16)#2	82.5(6)
O(3)-Li(1)-Li(2)	78.1(6)	Li(2)-Li(1)-O(16)#2	90.5(5)
O(19)-Li(1)-Li(2)	35.8(3)	O(1)-Li(1)-O(16)#2	74.7(3)
O(18)-Li(1)-O(1)	107.7(7)	O(20)#1-Li(1)-O(16)#2	45.1(2)
O(2)-Li(1)-O(1)	35.1(3)	O(25)-Li(1)-O(16)#2	115.0(4)
O(3)-Li(1)-O(1)	131.6(10)	O(18)-Li(1)-O(24)#3	159.3(6)
O(19)-Li(1)-O(1)	78.7(4)	O(2)-Li(1)-O(24)#3	82.9(9)
Li(2)-Li(1)-O(1)	114.5(4)	O(3)-Li(1)-O(24)#3	46.3(6)
O(18)-Li(1)-O(20)#1	78.5(5)	O(19)-Li(1)-O(24)#3	69.6(7)
O(2)-Li(1)-O(20)#1	93.1(4)	Li(2)-Li(1)-O(24)#3	72.0(6)
O(3)-Li(1)-O(20)#1	141.6(7)	O(1)-Li(1)-O(24)#3	91.2(5)
O(19)-Li(1)-O(20)#1	41.3(3)	O(20)#1-Li(1)-O(24)#3	105.7(4)
Li(2)-Li(1)-O(20)#1	66.6(3)	O(25)-Li(1)-O(24)#3	45.1(5)
O(1)-Li(1)-O(20)#1	58.06(19)	O(16)#2-Li(1)-O(24)#3	150.8(3)
O(18)-Li(1)-O(25)	152.0(6)	O(4)-Li(2)-O(5)	114.7(10)
O(2)-Li(1)-O(25)	50.2(6)	O(4)-Li(2)-O(16)#1	107.9(7)
O(3)-Li(1)-O(25)	89.3(9)	O(5)-Li(2)-O(16)#1	113.8(8)

O(4)-Li(2)-O(16)	107.9(7)	O(16)-Li(2)-O(9)	147.2(4)
O(5)-Li(2)-O(16)	113.8(8)	O(19)-Li(2)-O(9)	102.5(6)
O(16)#1-Li(2)-O(16)	0.0(6)	Li(1)-Li(2)-O(9)	116.4(6)
O(4)-Li(2)-O(19)	99.5(7)	O(6)-Li(2)-O(9)	72.6(3)
O(5)-Li(2)-O(19)	109.7(7)	O(18)#2-Li(2)-O(9)	144.0(2)
O(16)#1-Li(2)-O(19)	110.3(8)	O(20)#1-Li(2)-O(9)	120.0(3)
O(16)-Li(2)-O(19)	110.3(8)	C(1)-O(1)-O(2)	117.7(4)
O(4)-Li(2)-Li(1)	75.3(5)	C(1)-O(1)-O(8)	108.6(4)
O(5)-Li(2)-Li(1)	145.9(7)	O(2)-O(1)-O(8)	131.8(2)
O(16)#1-Li(2)-Li(1)	91.5(7)	C(1)-O(1)-O(25)	136.6(7)
O(16)-Li(2)-Li(1)	91.5(7)	O(2)-O(1)-O(25)	68.6(4)
O(19)-Li(2)-Li(1)	37.0(3)	O(8)-O(1)-O(25)	87.2(4)
O(4)-Li(2)-O(6)	138.7(9)	C(1)-O(1)-Li(1)	134.7(6)
O(5)-Li(2)-O(6)	31.6(2)	O(2)-O(1)-Li(1)	26.4(3)
O(16)#1-Li(2)-O(6)	109.5(6)	O(8)-O(1)-Li(1)	107.4(2)
O(16)-Li(2)-O(6)	109.5(6)	O(25)-O(1)-Li(1)	71.2(5)
O(19)-Li(2)-O(6)	83.3(4)	O(1)-C(1)-C(2)	119.8(9)
Li(1)-Li(2)-O(6)	120.0(4)	O(1)-C(1)-C(6)	121.2(8)
O(4)-Li(2)-O(18)#2	147.4(8)	C(2)-C(1)-C(6)	119.0(9)
O(5)-Li(2)-O(18)#2	93.2(6)	C(1)-C(2)-C(3)	120.3(9)
O(16)#1-Li(2)-O(18)#2	41.8(4)	C(1)-C(2)-C(88)	119.9(7)
O(16)-Li(2)-O(18)#2	41.8(4)	C(3)-C(2)-C(88)	119.8(7)
O(19)-Li(2)-O(18)#2	85.5(6)	C(4)-C(3)-C(2)	122.2(8)
Li(1)-Li(2)-O(18)#2	91.2(6)	C(5)-C(4)-C(3)	114.5(9)
O(6)-Li(2)-O(18)#2	73.7(3)	C(5)-C(4)-C(7)	121.8(10)
O(4)-Li(2)-O(20)#1	136.9(6)	C(3)-C(4)-C(7)	123.7(8)
O(5)-Li(2)-O(20)#1	93.8(4)	C(6)-C(5)-C(4)	126.1(10)
O(16)#1-Li(2)-O(20)#1	86.8(5)	C(5)-C(6)-C(1)	117.9(8)
O(16)-Li(2)-O(20)#1	86.8(5)	C(5)-C(6)-C(11)	120.6(9)
O(19)-Li(2)-O(20)#1	38.4(3)	C(1)-C(6)-C(11)	121.5(8)
Li(1)-Li(2)-O(20)#1	63.9(3)	C(9)-C(7)-C(8)	115.7(19)
O(6)-Li(2)-O(20)#1	62.16(18)	C(9)-C(7)-C(4)	110.3(11)
O(18)#2-Li(2)-O(20)#1	50.4(2)	C(8)-C(7)-C(4)	113.2(8)
O(4)-Li(2)-O(9)	66.6(6)	C(9)-C(7)-C(10)	103.9(17)
O(5)-Li(2)-O(9)	51.0(6)	C(8)-C(7)-C(10)	105.6(12)
O(16)#1-Li(2)-O(9)	147.2(4)	C(4)-C(7)-C(10)	107.3(14)

C(6)-C(11)-C(13)	109.6(9)	C(28)-C(23)-C(24)	119.3(6)
C(12)-O(2)-Li(1)	119.3(7)	C(25)-C(24)-C(23)	118.3(7)
C(12)-O(2)-O(1)	111.7(5)	C(25)-C(24)-C(22)	120.5(7)
Li(1)-O(2)-O(1)	118.5(4)	C(23)-C(24)-C(22)	121.1(6)
C(12)-O(2)-O(25)	129.5(6)	C(26)-C(25)-C(24)	123.4(8)
Li(1)-O(2)-O(25)	102.2(7)	C(27)-C(26)-C(25)	116.6(6)
O(1)-O(2)-O(25)	64.7(5)	C(27)-C(26)-C(29)	120.7(7)
C(13)-C(12)-O(2)	120.4(8)	C(25)-C(26)-C(29)	122.0(8)
C(13)-C(12)-C(17)	122.0(6)	C(26)-C(27)-C(28)	122.8(7)
O(2)-C(12)-C(17)	117.5(9)	C(27)-C(28)-C(23)	119.2(8)
C(12)-C(13)-C(14)	119.2(9)	C(27)-C(28)-C(33)	120.5(7)
C(12)-C(13)-C(11)	122.3(7)	C(23)-C(28)-C(33)	120.3(6)
C(14)-C(13)-C(11)	118.5(10)	C(32)-C(29)-C(30)	119.4(13)
C(13)-C(14)-C(15)	122.1(10)	C(32)-C(29)-C(26)	110.8(8)
C(16)-C(15)-C(14)	116.8(7)	C(30)-C(29)-C(26)	112.3(8)
C(16)-C(15)-C(18)	122.8(9)	C(32)-C(29)-C(31)	103.4(12)
C(14)-C(15)-C(18)	120.3(10)	C(30)-C(29)-C(31)	103.9(10)
C(15)-C(16)-C(17)	125.2(8)	C(26)-C(29)-C(31)	105.5(10)
C(16)-C(17)-C(12)	114.7(9)	C(28)-C(33)-C(35)	115.4(9)
C(16)-C(17)-C(22)	122.3(8)	C(34)-O(4)-Li(2)	137.7(6)
C(12)-C(17)-C(22)	123.0(6)	C(34)-O(4)-O(3)	113.3(4)
C(21)-C(18)-C(20)	110.7(14)	Li(2)-O(4)-O(3)	107.5(5)
C(21)-C(18)-C(19)	115.0(17)	O(4)-C(34)-C(35)	121.4(8)
C(20)-C(18)-C(19)	102.4(15)	O(4)-C(34)-C(39)	119.1(6)
C(21)-C(18)-C(15)	110.2(10)	C(35)-C(34)-C(39)	119.4(7)
C(20)-C(18)-C(15)	111.0(11)	C(36)-C(35)-C(34)	120.3(8)
C(19)-C(18)-C(15)	107.3(8)	C(36)-C(35)-C(33)	120.0(6)
C(17)-C(22)-C(24)	116.5(9)	C(34)-C(35)-C(33)	119.6(7)
C(23)-O(3)-Li(1)	133.8(7)	C(35)-C(36)-C(37)	121.5(7)
C(23)-O(3)-O(4)	120.0(5)	C(38)-C(37)-C(36)	116.9(8)
Li(1)-O(3)-O(4)	99.1(4)	C(38)-C(37)-C(40)	119.2(9)
C(23)-O(3)-O(24)	85.4(5)	C(36)-C(37)-C(40)	123.8(7)
Li(1)-O(3)-O(24)	126.4(7)	C(39)-C(38)-C(37)	122.5(8)
O(4)-O(3)-O(24)	82.0(3)	C(38)-C(39)-C(34)	119.3(7)
O(3)-C(23)-C(28)	121.9(7)	C(38)-C(39)-C(44)	119.8(8)
O(3)-C(23)-C(24)	118.8(7)	C(34)-C(39)-C(44)	120.9(7)

C(42)-C(40)-C(41)	124.2(15)	C(56)-O(6)-O(5)	116.3(5)
C(42)-C(40)-C(43)	75(3)	C(56)-O(6)-O(7)	117.5(4)
C(41)-C(40)-C(43)	106(2)	O(5)-O(6)-O(7)	126.1(2)
C(42)-C(40)-C(37)	124.8(12)	C(56)-O(6)-O(22)	108.7(6)
C(41)-C(40)-C(37)	110.2(10)	O(5)-O(6)-O(22)	68.8(3)
C(43)-C(40)-C(37)	100.6(13)	O(7)-O(6)-O(22)	90.8(4)
C(40)-C(42)-C(43)	61(2)	C(56)-O(6)-Li(2)	135.8(5)
C(40)-C(43)-C(42)	43.9(16)	O(5)-O(6)-Li(2)	24.0(2)
C(39)-C(44)-C(46)	112.4(7)	O(7)-O(6)-Li(2)	105.68(19)
C(45)-O(5)-Li(2)	120.8(4)	O(22)-O(6)-Li(2)	78.6(3)
C(45)-O(5)-O(6)	110.1(4)	C(56)-O(6)-O(17)#2	138.5(5)
Li(2)-O(5)-O(6)	124.3(4)	O(5)-O(6)-O(17)#2	46.8(2)
C(45)-O(5)-O(22)	101.9(6)	O(7)-O(6)-O(17)#2	91.89(18)
Li(2)-O(5)-O(22)	120.1(6)	O(22)-O(6)-O(17)#2	98.7(4)
O(6)-O(5)-O(22)	64.6(3)	Li(2)-O(6)-O(17)#2	24.4(3)
O(5)-C(45)-C(46)	119.4(8)	C(57)-C(56)-O(6)	120.8(7)
O(5)-C(45)-C(50)	121.3(9)	C(57)-C(56)-C(61)	121.7(7)
C(46)-C(45)-C(50)	119.3(8)	O(6)-C(56)-C(61)	117.4(8)
C(45)-C(46)-C(47)	121.1(9)	C(56)-C(57)-C(58)	118.8(7)
C(45)-C(46)-C(44)	122.3(8)	C(56)-C(57)-C(55)	121.5(6)
C(47)-C(46)-C(44)	116.5(9)	C(58)-C(57)-C(55)	119.7(8)
C(46)-C(47)-C(48)	120.3(10)	C(57)-C(58)-C(59)	121.6(9)
C(49)-C(48)-C(47)	117.4(9)	C(60)-C(59)-C(58)	117.9(7)
C(49)-C(48)-C(51)	124.7(10)	C(60)-C(59)-C(62)	123.1(8)
C(47)-C(48)-C(51)	117.4(11)	C(58)-C(59)-C(62)	119.0(10)
C(48)-C(49)-C(50)	123.5(9)	C(59)-C(60)-C(61)	122.3(8)
C(45)-C(50)-C(49)	118.3(9)	C(60)-C(61)-C(56)	117.5(9)
C(45)-C(50)-C(55)	121.8(8)	C(60)-C(61)-C(66)	120.0(7)
C(49)-C(50)-C(55)	119.9(8)	C(56)-C(61)-C(66)	122.4(7)
C(54)-C(51)-C(53)	120(2)	C(64)-C(62)-C(63)	124(2)
C(54)-C(51)-C(52)	105(2)	C(64)-C(62)-C(59)	116.3(10)
C(53)-C(51)-C(52)	99.2(17)	C(63)-C(62)-C(59)	111.5(11)
C(54)-C(51)-C(48)	118.1(13)	C(64)-C(62)-C(65)	96(2)
C(53)-C(51)-C(48)	109.3(10)	C(63)-C(62)-C(65)	101.9(19)
C(52)-C(51)-C(48)	101.6(15)	C(59)-C(62)-C(65)	101.4(15)
C(57)-C(55)-C(50)	111.2(9)	C(61)-C(66)-C(68)	111.0(10)

C(67)-O(7)-O(6)	113.0(5)	O(7)-O(8)-O(23)	53.5(3)
C(67)-O(7)-O(23)	136.7(8)	C(78)-O(8)-O(20)#2	151.9(6)
O(6)-O(7)-O(23)	91.8(5)	O(1)-O(8)-O(20)#2	66.68(15)
C(67)-O(7)-O(8)	111.2(5)	O(7)-O(8)-O(20)#2	81.60(16)
O(6)-O(7)-O(8)	133.5(2)	O(23)-O(8)-O(20)#2	84.3(7)
O(23)-O(7)-O(8)	64.3(5)	C(83)-C(78)-O(8)	123.0(7)
C(67)-O(7)-O(17)#2	147.1(6)	C(83)-C(78)-C(79)	120.6(7)
O(6)-O(7)-O(17)#2	72.60(16)	O(8)-C(78)-C(79)	116.4(8)
O(23)-O(7)-O(17)#2	72.5(8)	C(80)-C(79)-C(78)	116.3(8)
O(8)-O(7)-O(17)#2	62.57(13)	C(80)-C(79)-C(77)	120.4(7)
C(72)-C(67)-O(7)	120.3(7)	C(78)-C(79)-C(77)	123.3(7)
C(72)-C(67)-C(68)	121.1(6)	C(81)-C(80)-C(79)	124.2(8)
O(7)-C(67)-C(68)	118.5(8)	C(80)-C(81)-C(82)	116.3(7)
C(67)-C(68)-C(69)	118.3(8)	C(80)-C(81)-C(84)	123.0(8)
C(67)-C(68)-C(66)	121.9(6)	C(82)-C(81)-C(84)	120.7(9)
C(69)-C(68)-C(66)	119.9(7)	C(78)-C(83)-C(82)	120.3(7)
C(70)-C(69)-C(68)	122.4(8)	C(78)-C(83)-C(88)	121.6(7)
C(69)-C(70)-C(71)	119.2(7)	C(82)-C(83)-C(88)	118.1(8)
C(69)-C(70)-C(73)	122.9(8)	C(81)-C(82)-C(83)	122.2(9)
C(71)-C(70)-C(73)	117.7(9)	C(86)-C(84)-C(85)	109.2(12)
C(72)-C(71)-C(70)	118.5(9)	C(86)-C(84)-C(81)	109.2(9)
C(67)-C(72)-C(71)	120.3(7)	C(85)-C(84)-C(81)	112.6(9)
C(67)-C(72)-C(77)	121.6(6)	C(86)-C(84)-C(87)	110.4(11)
C(71)-C(72)-C(77)	118.0(8)	C(85)-C(84)-C(87)	107.8(10)
C(76)-C(73)-C(74)	112.8(17)	C(81)-C(84)-C(87)	107.6(10)
C(76)-C(73)-C(75)	104.4(16)	C(83)-C(88)-C(2)	112.3(10)
C(74)-C(73)-C(75)	107.7(15)	C(89)-O(9)-C(92)	110.1(11)
C(76)-C(73)-C(70)	109.9(12)	C(89)-O(9)-Li(2)	104.3(12)
C(74)-C(73)-C(70)	113.3(7)	C(92)-O(9)-Li(2)	78.1(9)
C(75)-C(73)-C(70)	108.2(12)	C(89)-O(9)-O(16)#1	107.4(12)
C(79)-C(77)-C(72)	114.6(9)	C(92)-O(9)-O(16)#1	78.8(9)
C(78)-O(8)-O(1)	116.9(5)	Li(2)-O(9)-O(16)#1	3.59(5)
C(78)-O(8)-O(7)	105.4(4)	O(9)-C(89)-C(90)	111.0(14)
O(1)-O(8)-O(7)	136.2(2)	C(89)-C(90)-C(91)	104.2(18)
C(78)-O(8)-O(23)	122.0(7)	C(90)-C(91)-C(92)	107.2(13)
O(1)-O(8)-O(23)	92.5(5)	O(9)-C(92)-C(91)	107.1(13)

C(93)-O(10)-C(96)	105.7(15)	C(102)-C(104)-C(101)	29.9(11)
C(93)-O(10)-O(24)	115.6(17)	C(105)-O(13)-C(108)	81(3)
C(96)-O(10)-O(24)	116.8(16)	C(106)-C(105)-O(13)	111(4)
C(94)-C(93)-O(10)	107.6(18)	C(106)-C(105)-C(108)	79(3)
C(95)-C(94)-C(93)	109(2)	O(13)-C(105)-C(108)	58(3)
C(96)-C(95)-C(94)	108(2)	C(105)-C(106)-C(107)	99(3)
C(95)-C(96)-O(10)	107(2)	C(105)-C(106)-C(108)	66(3)
C(100)-O(11)-C(98)	97(2)	C(107)-C(106)-C(108)	40.2(14)
C(100)-O(11)-C(97)	67(3)	C(108)-C(107)-C(106)	84(3)
C(98)-O(11)-C(97)	50(3)	C(107)-C(108)-O(13)	90(4)
C(98)-C(97)-O(11)	63(3)	C(107)-C(108)-C(105)	86(2)
C(98)-C(97)-C(100)	92(5)	O(13)-C(108)-C(105)	41.4(15)
O(11)-C(97)-C(100)	48(2)	C(107)-C(108)-C(106)	56(2)
C(98)-C(97)-C(99)	76(6)	O(13)-C(108)-C(106)	66.1(16)
O(11)-C(97)-C(99)	73(3)	C(105)-C(108)-C(106)	35.0(18)
C(100)-C(97)-C(99)	41(2)	C(112)-O(14)-C(109)	74(3)
C(97)-C(98)-O(11)	67(3)	C(112)-O(14)-C(110)	45(2)
C(100)-C(99)-C(97)	60(3)	C(109)-O(14)-C(110)	73(2)
C(99)-C(100)-O(11)	107(4)	C(112)-O(14)-C(111)	84(3)
C(99)-C(100)-C(97)	79(5)	C(109)-O(14)-C(111)	53.9(17)
O(11)-C(100)-C(97)	65(2)	C(110)-O(14)-C(111)	45.4(14)
C(101)-O(12)-C(104)	81(2)	C(111)-C(109)-O(14)	72.0(19)
C(101)-O(12)-O(17)	60(2)	C(111)-C(109)-C(112)	81(2)
C(104)-O(12)-O(17)	125.6(19)	O(14)-C(109)-C(112)	51(2)
C(102)-C(101)-O(12)	123(4)	C(111)-C(109)-C(110)	44.1(15)
C(102)-C(101)-C(104)	71(3)	O(14)-C(109)-C(110)	57(2)
O(12)-C(101)-C(104)	53(2)	C(112)-C(109)-C(110)	38.0(18)
C(101)-C(102)-C(103)	130(4)	C(112)-C(110)-C(111)	117(5)
C(101)-C(102)-C(104)	79(3)	C(112)-C(110)-O(14)	58(3)
C(103)-C(102)-C(104)	51(2)	C(111)-C(110)-O(14)	72(2)
C(102)-C(103)-C(104)	82(2)	C(112)-C(110)-C(109)	66(4)
C(103)-C(104)-O(12)	123(4)	C(111)-C(110)-C(109)	52(2)
C(103)-C(104)-C(102)	47(2)	O(14)-C(110)-C(109)	50.0(15)
O(12)-C(104)-C(102)	76(2)	C(110)-C(111)-C(109)	83(3)
C(103)-C(104)-C(101)	77(3)	C(110)-C(111)-O(14)	62(2)
O(12)-C(104)-C(101)	46.1(16)	C(109)-C(111)-O(14)	54.1(17)

C(110)-C(112)-O(14)	77(3)	O(21)-O(22)-O(23)	66.0(8)
C(110)-C(112)-C(109)	76(4)	O(21)-O(22)-O(6)	139.5(10)
O(14)-C(112)-C(109)	54.5(17)	O(23)-O(22)-O(6)	79.4(6)
C(113)-O(15)-C(116)	103(3)	O(21)-O(22)-O(5)	111.6(8)
C(113)-O(15)-O(21)	100(3)	O(23)-O(22)-O(5)	91.1(9)
C(116)-O(15)-O(21)	155(2)	O(6)-O(22)-O(5)	46.6(3)
O(15)-C(113)-C(114)	93(3)	O(25)-O(23)-O(7)	141.5(13)
C(115)-C(114)-C(113)	112(4)	O(25)-O(23)-O(22)	123.5(12)
C(115)-C(114)-C(116)	41(3)	O(7)-O(23)-O(22)	85.2(6)
C(113)-C(114)-C(116)	87(2)	O(25)-O(23)-O(8)	82.3(8)
C(116)-C(115)-C(114)	95(4)	O(7)-O(23)-O(8)	62.3(4)
C(115)-C(116)-O(15)	107(6)	O(22)-O(23)-O(8)	145.1(8)
C(115)-C(116)-C(114)	44(3)	O(25)-O(23)-O(21)	73.3(8)
O(15)-C(116)-C(114)	76.9(19)	O(7)-O(23)-O(21)	140.8(8)
O(16)#1-O(16)-Li(2)	0(10)	O(22)-O(23)-O(21)	55.7(8)
O(16)#1-O(16)-O(18)#2	0(10)	O(8)-O(23)-O(21)	155.6(8)
Li(2)-O(16)-O(18)#2	110.0(4)	O(10)-O(24)-O(21)	46.3(5)
O(16)#1-O(16)-O(20)#1	0(10)	O(10)-O(24)-O(25)	45.8(4)
Li(2)-O(16)-O(20)#1	65.9(4)	O(21)-O(24)-O(25)	7.87(12)
O(18)#2-O(16)-O(20)#1	51.1(3)	O(10)-O(24)-O(3)	56.3(4)
O(20)#1-O(17)-O(18)#2	80.0(4)	O(21)-O(24)-O(3)	10.93(5)
O(20)#1-O(17)-O(12)	154.8(10)	O(25)-O(24)-O(3)	11.25(7)
O(18)#2-O(17)-O(12)	89.0(7)	O(23)-O(25)-O(1)	94.8(9)
Li(2)-O(19)-Li(1)	107.2(5)	O(23)-O(25)-O(2)	116.3(12)
Li(2)-O(19)-O(20)#1	114.0(5)	O(1)-O(25)-O(2)	46.7(3)
Li(1)-O(19)-O(20)#1	108.2(6)	O(23)-O(25)-Li(1)	96.0(11)
O(22)-O(21)-O(15)	65.3(7)	O(1)-O(25)-Li(1)	62.6(4)
O(22)-O(21)-O(23)	58.3(7)	O(2)-O(25)-Li(1)	27.6(2)
O(15)-O(21)-O(23)	81.5(10)	O(23)-O(25)-O(24)	94.3(8)
O(22)-O(21)-O(24)	126.2(7)	O(1)-O(25)-O(24)	161.1(6)
O(15)-O(21)-O(24)	73.9(7)	O(2)-O(25)-O(24)	139.9(5)
O(23)-O(21)-O(24)	146.6(9)	Li(1)-O(25)-O(24)	132.7(4)

Symmetry transformations used to generate equivalent atoms:

#1 x,y,z #2 -x,-y+2,-z #3 -x,-y+1,-z

Anisotropic displacement parameters ($Å^2$x 10^3)for $[Li_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_7(H_2O)_{12}]$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Li(1)	31	204	37	-21	13	-28
Li(2)	54	60	41	5	27	-12
O(1)	42	109	37	6	13	-5
C(1)	51	75	42	-7	13	-16
C(2)	37	46	47	-11	15	-6
C(3)	45	75	54	-23	22	-9
C(4)	41	115	57	-21	21	-18
C(5)	44	104	44	2	7	-21
C(6)	46	43	56	1	23	-14
C(7)	67	203	50	-1	36	-21
C(8)	124	244	63	15	36	-9
C(9)	340	606	167	-149	144	-399
C(10)	318	410	167	120	198	194
C(11)	45	90	54	-7	21	-23
O(2)	45	120	27	0	9	-12
C(12)	34	89	34	3	4	14
C(13)	45	54	39	0	19	5
C(14)	46	78	40	5	18	3
C(15)	56	72	31	2	17	2
C(16)	46	54	43	-4	14	-21
C(17)	32	69	32	0	10	-9
C(18)	70	142	37	7	-8	0
C(19)	216	391	46	-14	23	112
C(20)	178	171	67	24	-51	20
C(21)	157	535	86	162	-85	-197
C(22)	43	65	44	-4	16	-13
O(3)	32	95	41	-11	8	-22
C(23)	39	39	43	6	5	-16
C(24)	28	100	28	3	6	-8
C(25)	48	60	37	-4	17	-21
C(26)	23	85	45	-1	0	-6

C(27)	40	57	41	2	12	-4
C(28)	34	49	51	4	15	2
C(29)	37	145	49	-20	8	-4
C(30)	50	223	83	-68	4	-26
C(31)	59	181	105	-58	-6	24
C(32)	113	273	159	52	-17	-161
C(33)	44	62	38	3	17	-4
O(4)	49	78	42	4	13	-20
C(34)	34	77	36	-4	11	-7
C(35)	45	18	34	-3	14	-10
C(36)	35	39	48	8	13	-11
C(37)	52	84	53	16	25	-6
C(38)	48	74	41	19	14	-15
C(39)	38	75	41	3	15	-16
C(40)	49	269	59	67	21	-37
C(41)	77	381	166	113	23	-75
C(42)	95	637	1158	825	132	65
C(43)	584	450	351	-235	422	-419
C(44)	40	79	43	12	8	-3
O(5)	48	69	34	7	17	-11
C(45)	29	98	34	0	2	-5
C(46)	38	72	36	8	-4	7
C(47)	54	123	40	9	17	-21
C(48)	65	102	47	-1	25	-32
C(49)	48	137	45	-16	14	-4
C(50)	35	85	43	-12	11	0
C(51)	84	331	55	4	34	-35
C(52)	391	289	244	136	279	190
C(53)	280	274	79	48	100	-8
C(54)	632	859	240	-303	352	-664
C(55)	50	55	47	-5	16	-3
O(6)	34	114	38	-2	7	-2
C(56)	48	48	42	6	18	-7
C(57)	38	62	34	5	12	0
C(58)	64	37	52	-9	24	-8
C(59)	42	120	43	1	7	0

C(60)	44	92	46	12	7	21
C(61)	39	89	42	16	14	-3
C(62)	76	343	25	15	5	72
C(63)	166	890	87	-112	11	-290
C(64)	278	599	45	-93	-44	262
C(65)	604	218	60	6	-48	142
C(66)	45	62	60	28	20	11
O(7)	35	108	51	-4	7	-5
C(67)	33	65	54	-5	9	2
C(68)	37	59	44	4	15	-2
C(69)	40	114	40	19	8	0
C(70)	33	134	45	25	6	-3
C(71)	37	117	43	1	10	-7
C(72)	38	69	36	6	11	-7
C(73)	43	247	52	43	-1	-41
C(74)	42	363	96	47	2	-50
C(75)	72	265	358	203	-41	16
C(76)	98	655	212	-252	38	-139
C(77)	34	42	50	2	8	2
O(8)	36	119	55	-13	18	-9
C(78)	36	68	53	0	19	9
C(79)	38	76	36	-9	12	4
C(80)	41	51	41	3	11	-4
C(81)	30	105	46	7	6	-8
C(83)	38	75	51	-17	13	2
C(82)	42	64	53	-10	23	-4
C(84)	53	92	64	-6	28	-6
C(85)	41	193	129	-44	22	-24
C(86)	92	236	127	52	66	12
C(87)	69	104	124	-27	46	18
C(88)	45	130	64	-18	19	-7
O(9)	110	155	172	-53	76	-37
C(89)	126	132	170	0	39	-18
C(90)	127	293	151	-56	31	38
C(91)	75	289	183	-37	59	-29
C(92)	94	115	149	-27	44	-31

O(10)	129	265	177	-53	35	39
C(93)	185	396	207	-158	90	23
C(94)	119	456	160	-9	33	-24
C(95)	233	407	156	-144	91	-23
C(96)	140	363	175	-47	40	51
O(11)	298	270	618	102	-11	-60
C(97)	1200	932	2000	1419	1330	635
C(98)	277	452	335	267	191	45
C(99)	218	538	553	-394	198	-151
C(100)	657	404	191	53	148	-332
O(12)	477	598	676	250	444	298
C(101)	156	503	538	390	229	192
C(102)	313	179	135	-101	87	-48
C(103)	237	145	750	118	86	-8
C(104)	346	131	473	-80	344	-56
O(13)	264	322	464	42	-41	-69
C(105)	318	638	359	-246	222	50
C(106)	410	364	216	130	-106	-206
C(107)	198	380	320	5	185	83
C(108)	498	356	496	313	431	267
O(14)	333	399	396	-10	124	-84
C(109)	462	285	262	-100	280	-47
C(110)	733	162	195	53	15	-7
C(111)	414	0	378	108	216	29
C(112)	633	291	293	37	-37	146
O(15)	243	346	346	-27	-8	107
C(113)	477	617	260	-185	216	-87
C(114)	228	336	684	-121	266	41
C(115)	1913	854	618	545	934	1136
C(116)	308	392	130	-70	56	-115
O(16)	92	58	114	18	20	-9
O(17)	136	183	150	18	63	20
O(18)	112	57	148	-27	51	6
O(19)	51	106	46	4	18	-13
O(20)	68	91	123	-26	47	-20
O(21)	254	398	329	70	-124	-77

O(22)	255	65	600	28	243	17
O(23)	848	217	246	-149	312	-286
O(24)	241	206	268	43	83	88
O(25)	283	277	371	94	46	78
O(26)	539	500	221	-57	95	343
O(27)	531	449	247	-64	25	361

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [Li_2(4-tert-butylcalix[8]arene-2H)$(THF)_7(H_2O)_{12}$].

	x	y	z	U(eq)
H(3)	2283	9111	-2738	69
H(5)	486	9776	-3391	81
H(8A)	603	9507	-4507	217
H(8B)	1036	9812	-4758	217
H(8C)	648	9976	-4316	217
H(9A)	2493	9929	-3106	537
H(9B)	1844	10239	-3391	537
H(9C)	2226	10099	-3851	537
H(10A)	1706	9098	-4144	399
H(10B)	2427	9257	-3563	399
H(10C)	2146	9449	-4295	399
H(11A)	-161	9907	-2718	75
H(11B)	103	9701	-1996	75
H(14)	-1063	9569	-3711	65
H(16)	-2194	8619	-3517	59
H(19A)	-1671	8703	-4806	343
H(19B)	-1377	9153	-4780	343
H(19C)	-2096	9017	-5389	343
H(20A)	-1943	9707	-4628	260
H(20B)	-2629	9692	-4477	260
H(20C)	-2691	9578	-5203	260
H(21A)	-2797	8619	-4529	459
H(21B)	-3207	8913	-5148	459
H(21C)	-3160	9024	-4426	459

H(22A)	-1013	8368	-1806	62
H(22B)	-1563	8152	-2467	62
H(25)	-2867	8253	-2827	58
H(27)	-3562	8657	-1472	57
H(30A)	-4045	8474	-3582	190
H(30B)	-4740	8215	-3716	190
H(30C)	-3985	7998	-3445	190
H(31A)	-4621	8788	-2340	193
H(31B)	-5139	8655	-3083	193
H(31C)	-4488	8950	-2960	193
H(32A)	-4289	8102	-1918	311
H(32B)	-4144	7752	-2345	311
H(32C)	-4900	7969	-2620	311
H(33A)	-2790	9054	-500	57
H(33B)	-1996	9151	-382	57
H(36)	-3036	8491	107	51
H(38)	-1384	7927	1620	67
H(41A)	-3638	8243	441	326
H(41B)	-3897	7949	869	326
H(41C)	-3721	7769	283	326
H(42A)	-2130	7678	1852	1012
H(42B)	-2729	7378	1370	1012
H(42C)	-2900	7680	1847	1012
H(44A)	-2	8272	1189	68
H(44B)	-167	7961	1660	68
H(47)	-400	8260	2624	88
H(49)	858	9204	3606	94
H(52A)	21	9257	4010	388
H(52B)	-421	9016	4337	388
H(52C)	-758	9097	3550	388
H(53A)	954	8864	4484	304
H(53B)	938	8381	4480	304
H(53C)	553	8628	4856	304
H(54A)	-912	8410	3319	780
H(54B)	-649	8369	4110	780
H(54C)	-316	8086	3728	780

H(55A)	1300	9560	2289	62
H(55B)	1378	9665	3025	62
H(58)	2440	9473	3993	61
H(60)	4001	8796	3819	78
H(63A)	4617	9177	4599	593
H(63B)	4810	9228	5377	593
H(63C)	4538	9608	4888	593
H(64A)	2961	9273	4978	518
H(64B)	3437	9672	5118	518
H(64C)	3698	9298	5624	518
H(65A)	4118	8586	4920	537
H(65B)	3393	8640	4997	537
H(65C)	4127	8764	5597	537
H(66A)	3024	8482	2119	67
H(66B)	3753	8413	2764	67
H(69)	4822	8828	2925	81
H(71)	4802	9594	1452	81
H(74A)	6038	9170	1859	265
H(74B)	6643	9409	2457	265
H(74C)	5944	9641	1966	265
H(75A)	6084	8666	2727	414
H(75B)	5907	8805	3334	414
H(75C)	6638	8935	3316	414
H(76A)	5662	9500	3448	497
H(76B)	5714	9844	2960	497
H(76C)	6410	9606	3444	497
H(77A)	2914	9595	496	54
H(77B)	3619	9839	616	54
H(80)	4499	9494	206	56
H(82)	3607	8839	-1496	62
H(85A)	5966	9314	-160	188
H(85B)	5593	9156	298	188
H(85C)	5436	9603	-8	188
H(86A)	5256	9419	-1392	218
H(86B)	4704	9702	-1260	218
H(86C)	4430	9315	-1735	218

H(87A)	5523	8687	-888	145
H(87B)	4696	8586	-1232	145
H(87C)	5133	8532	-444	145
H(88A)	1999	8590	-1436	98
H(88B)	2436	8560	-1881	98

V.2.11. Crystal data and structure refinement for $[Na_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_4(H_2O)_5]$ 11

Empirical formula	C104 H176 Na2 O17	
Formula weight	1744.63	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 22.668 Å	α= 90°.
	b = 25.508 Å	β= 113.57°.
	c = 23.938 Å	γ = 90°.
Volume	12686.4 $Å^3$	
Z	4	
Density (calculated)	0.806 Mg/m^3	
Absorption coefficient	0.062 mm^{-1}	
F(000)	3228	
Theta range for data collection	1.26 to 24.84°.	
Index ranges	-26<=h<=24, 0<=k<=30, 0<=l<=28	
Reflections collected	21561	
Completeness to theta = 24.84°	98.3 %	
Absorption correction	Spherical	
Refinement method	Full-matrix-block least-squares on F^2	
Data / restraints / parameters	21561 / 0 / 1142	

Goodness-of-fit on F^2	0.918
Final R indices [I>2sigma(I)]	R1 = 0.1337, wR2 = 0.3426
R indices (all data)	R1 = 0.2945, wR2 = 0.4153
Largest diff. peak and hole	0.808 and -0.436 e.$Å^{-3}$

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) For [Na_2(4-tert-butylcalix[8]arene-2H)$(THF)_4(H_2O)_5$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Na(1)	3840(1)	1101(2)	2119(1)	49
Na(2)	2416(1)	1030(2)	1012(1)	51
O(1)	2453(2)	1449(3)	164(2)	48
C(1)	1938(3)	1292(4)	-362(3)	41
C(2)	1343(4)	1535(4)	-472(4)	44
C(3)	815(4)	1367(4)	-989(4)	53
C(4)	862(4)	981(4)	-1375(4)	63
C(5)	1464(4)	761(4)	-1237(4)	54
C(6)	1994(4)	895(4)	-726(3)	43
C(7)	244(6)	808(7)	-1936(6)	131
C(8)	-193(10)	1237(10)	-2202(10)	140
C(9)	-131(16)	453(15)	-1705(17)	218
C(10)	332(13)	570(20)	-2299(15)	590
C(11)	2636(4)	610(4)	-584(4)	49
O(2)	3564(2)	1338(3)	174(2)	48
C(12)	3551(4)	1250(4)	-384(3)	45
C(13)	3111(4)	897(4)	-773(4)	46
C(14)	3110(4)	811(4)	-1356(4)	57
C(15)	3508(4)	1077(5)	-1560(4)	62
C(16)	3934(4)	1435(4)	-1172(4)	55
C(17)	3968(4)	1526(4)	-588(4)	50
C(18)	3452(6)	964(6)	-2219(5)	99
C(19)	2779(8)	1112(11)	-2674(6)	81
C(20)	3989(8)	1261(9)	-2348(6)	165
C(21)	3510(10)	377(7)	-2322(7)	151
C(22)	4418(4)	1949(4)	-184(4)	51

O(3)	4649(2)	1441(3)	976(2)	56
C(23)	5158(3)	1533(4)	811(3)	43
C(24)	5060(4)	1761(4)	253(4)	47
C(25)	5619(4)	1853(4)	135(4)	65
C(26)	6239(4)	1726(5)	559(4)	67
C(27)	6295(4)	1480(4)	1085(4)	57
C(28)	5769(4)	1381(4)	1227(4)	46
C(29)	6849(6)	1894(6)	429(7)	111
C(30)	6679(7)	2182(9)	-148(8)	69
C(31)	7260(7)	2250(9)	956(8)	62
C(32)	7220(8)	1373(8)	463(10)	172
C(33)	5864(4)	1142(3)	1835(4)	46
O(4)	4798(2)	1473(3)	2128(2)	49
C(34)	5409(3)	1675(3)	2465(3)	39
C(35)	5925(3)	1529(4)	2325(3)	43
C(36)	6519(4)	1755(4)	2661(4)	60
C(37)	6618(4)	2119(4)	3129(4)	58
C(38)	6085(4)	2234(4)	3259(4)	58
C(39)	5487(4)	2006(4)	2953(4)	44
C(40)	7258(5)	2402(6)	3487(6)	103
C(41)	7116(7)	3029(6)	3320(7)	127
C(42)	7470(6)	2351(8)	4153(6)	53
C(43)	7777(6)	2243(8)	3260(8)	62
C(44)	4946(4)	2119(4)	3151(4)	45
O(5)	3807(2)	1557(2)	2951(2)	43
C(45)	4326(3)	1431(3)	3475(3)	34
C(46)	4899(3)	1713(3)	3586(3)	40
C(47)	5431(3)	1579(3)	4117(3)	43
C(48)	5419(3)	1204(3)	4531(3)	41
C(49)	4850(4)	946(4)	4404(4)	44
C(50)	4292(3)	1051(3)	3870(3)	35
C(51)	6019(5)	1079(5)	5115(5)	77
C(52)	6473(9)	1497(9)	5315(10)	136
C(53)	6297(14)	620(12)	5039(13)	235
C(54)	5809(10)	1004(19)	5624(7)	206
C(55)	3678(4)	732(3)	3735(4)	43

O(6)	2700(2)	1417(2)	2946(2)	46
C(56)	2728(3)	1329(3)	3517(3)	37
C(57)	3176(3)	991(3)	3919(3)	37
C(58)	3182(4)	907(4)	4483(4)	47
C(59)	2746(4)	1140(4)	4682(4)	52
C(60)	2321(4)	1491(4)	4287(4)	46
C(61)	2299(3)	1589(3)	3715(3)	39
C(62)	2786(5)	1042(6)	5341(4)	89
C(63)	2889(11)	478(8)	5499(9)	90
C(64)	2163(7)	1191(7)	5389(6)	47
C(65)	3319(12)	1379(11)	5768(6)	108
C(66)	1825(4)	1992(4)	3295(3)	46
O(7)	1587(2)	1474(3)	2148(2)	52
C(67)	1084(3)	1529(4)	2330(4)	46
C(68)	1165(4)	1766(4)	2868(4)	47
C(69)	649(4)	1829(4)	3027(4)	62
C(70)	46(4)	1630(5)	2655(4)	70
C(71)	-10(4)	1388(4)	2128(4)	60
C(72)	491(3)	1329(4)	1945(3)	42
C(73)	-529(6)	1713(8)	2817(7)	57
C(74)	-403(9)	2061(15)	3367(12)	130
C(75)	-703(19)	1370(20)	2950(20)	484
C(76)	-1079(9)	1993(19)	2295(10)	184
C(77)	396(4)	1074(4)	1341(4)	49
O(8)	1450(2)	1371(2)	1009(2)	47
C(78)	835(3)	1557(3)	661(3)	38
C(79)	322(4)	1438(3)	816(3)	42
C(80)	-275(3)	1659(3)	483(3)	40
C(81)	-382(4)	1983(4)	-3(4)	51
C(82)	140(4)	2089(4)	-164(4)	49
C(83)	741(3)	1862(3)	153(3)	40
C(84)	-1027(4)	2261(4)	-361(4)	61
C(85)	-1548(6)	2098(7)	-168(8)	55
C(86)	-922(6)	2848(5)	-237(6)	99
C(87)	-1232(5)	2208(7)	-1044(5)	110
C(88)	1275(3)	1961(3)	-60(4)	41

O(9)	3113(3)	1877(3)	1527(3)	64
C(89)	2770(6)	2215(5)	1796(6)	88
C(90)	3011(14)	2740(8)	1826(16)	117
C(91)	3360(20)	2724(8)	1377(19)	179
C(92)	3425(6)	2220(5)	1240(5)	85
O(10)	4438(4)	340(3)	2598(4)	88
C(93)	5126(6)	389(6)	2927(7)	107
C(94)	5217(15)	-147(10)	3342(7)	242
C(95)	4470(20)	-347(13)	3030(20)	158
C(96)	4224(10)	-137(7)	2665(13)	78
O(11)	1862(4)	226(4)	611(4)	103
C(97)	2109(10)	-241(6)	450(12)	74
C(98)	1482(19)	-428(10)	-63(18)	289
C(99)	934(14)	-144(17)	59(16)	301
C(100)	1156(9)	248(9)	367(13)	113
O(12)	757(16)	2065(12)	6492(15)	382
C(109)	900(20)	1628(18)	6090(20)	338
C(110)	320(30)	1630(20)	5690(30)	531
C(111)	-50(40)	1820(30)	5780(50)	678
C(112)	-120(20)	1990(30)	6140(30)	430
O(13)	2778(3)	767(3)	2041(2)	52
O(14)	3506(3)	742(3)	1130(2)	53
O(15)	6009(10)	228(9)	9103(9)	260
O(16)	7597(17)	320(15)	7626(15)	426
O(17)	6266(16)	1917(14)	7152(14)	355
O(18)	5262(15)	2132(13)	6681(14)	346
O(19)	5665(13)	1754(10)	7549(12)	299

Bond lengths [Å] and angles [°] for $[Na_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_4(H_2O)_5]$.

Na(1)-O(5)	2.332(5)	Na(1)-Na(2)	3.260(4)
Na(1)-O(4)	2.363(6)	Na(1)-O(6)	3.917(5)
Na(1)-O(14)	2.364(6)	Na(1)-O(3)	3.953(6)
Na(1)-O(10)	2.383(8)	Na(1)-O(2)	4.484(6)
Na(1)-O(13)	2.489(5)	Na(2)-O(1)	2.326(6)
Na(1)-O(9)	2.601(6)	Na(2)-O(8)	2.352(5)
Na(1)-C(45)	3.097(7)	Na(2)-O(13)	2.360(5)

Na(2)-O(11)	2.395(9)	O(3)-C(23)	1.381(9)
Na(2)-O(14)	2.484(5)	O(3)-O(4)	2.644(7)
Na(2)-O(9)	2.669(7)	O(3)-O(14)	3.286(6)
Na(2)-C(1)	3.096(8)	C(23)-C(24)	1.388(11)
Na(2)-O(2)	3.947(6)	C(23)-C(28)	1.399(11)
Na(2)-O(7)	4.036(5)	C(24)-C(25)	1.424(11)
O(1)-C(1)	1.390(9)	C(25)-C(26)	1.403(13)
O(1)-O(2)	2.523(7)	C(26)-C(27)	1.365(13)
C(1)-C(6)	1.375(11)	C(26)-C(29)	1.594(13)
C(1)-C(2)	1.409(11)	C(27)-C(28)	1.389(11)
C(2)-C(3)	1.400(11)	C(28)-C(33)	1.512(11)
C(2)-C(88)	1.517(9)	C(29)-C(30)	1.47(2)
C(3)-C(4)	1.383(13)	C(29)-C(31)	1.53(2)
C(4)-C(5)	1.388(13)	C(29)-C(32)	1.56(2)
C(4)-C(7)	1.567(12)	C(33)-C(35)	1.497(12)
C(5)-C(6)	1.372(11)	O(4)-C(34)	1.393(9)
C(6)-C(11)	1.537(11)	C(34)-C(35)	1.389(11)
C(7)-C(10)	1.14(3)	C(34)-C(39)	1.395(11)
C(7)-C(8)	1.44(2)	C(35)-C(36)	1.387(11)
C(7)-C(9)	1.49(3)	C(36)-C(37)	1.402(13)
C(11)-C(13)	1.514(12)	C(37)-C(38)	1.393(12)
O(2)-C(12)	1.343(9)	C(37)-C(40)	1.538(12)
O(2)-O(3)	2.455(7)	C(38)-C(39)	1.386(11)
O(2)-O(14)	2.795(6)	C(39)-C(44)	1.508(10)
C(12)-C(13)	1.389(12)	C(40)-C(42)	1.477(19)
C(12)-C(17)	1.412(12)	C(40)-C(43)	1.534(15)
C(13)-C(14)	1.412(11)	C(40)-C(41)	1.65(2)
C(14)-C(15)	1.367(13)	C(44)-C(46)	1.503(12)
C(15)-C(16)	1.384(14)	O(5)-C(45)	1.371(8)
C(15)-C(18)	1.558(13)	O(5)-O(6)	2.532(7)
C(16)-C(17)	1.388(11)	C(45)-C(50)	1.377(11)
C(17)-C(22)	1.533(13)	C(45)-C(46)	1.415(10)
C(18)-C(19)	1.525(19)	C(46)-C(47)	1.400(10)
C(18)-C(21)	1.53(2)	C(47)-C(48)	1.385(11)
C(18)-C(20)	1.565(19)	C(48)-C(49)	1.370(11)
C(22)-C(24)	1.492(12)	C(48)-C(51)	1.544(11)

C(49)-C(50)	1.419(10)	O(8)-C(78)	1.391(9)
C(50)-C(55)	1.532(11)	C(78)-C(83)	1.386(11)
C(51)-C(53)	1.37(2)	C(78)-C(79)	1.388(10)
C(51)-C(52)	1.426(19)	C(79)-C(80)	1.387(10)
C(51)-C(54)	1.49(2)	C(80)-C(81)	1.365(11)
C(55)-C(57)	1.524(11)	C(81)-C(82)	1.410(11)
O(6)-C(56)	1.361(8)	C(81)-C(84)	1.541(11)
O(6)-O(7)	2.485(7)	C(82)-C(83)	1.392(11)
O(6)-O(13)	2.791(6)	C(83)-C(88)	1.512(10)
C(56)-C(57)	1.385(11)	C(84)-C(85)	1.489(15)
C(56)-C(61)	1.406(10)	C(84)-C(87)	1.517(15)
C(57)-C(58)	1.361(11)	C(84)-C(86)	1.528(16)
C(58)-C(59)	1.393(12)	O(9)-C(92)	1.457(13)
C(59)-C(60)	1.376(12)	O(9)-C(89)	1.473(12)
C(59)-C(62)	1.562(12)	C(89)-C(90)	1.44(2)
C(60)-C(61)	1.373(10)	C(90)-C(91)	1.56(3)
C(61)-C(66)	1.536(11)	C(91)-C(92)	1.35(2)
C(62)-C(63)	1.48(2)	O(10)-C(96)	1.344(18)
C(62)-C(65)	1.50(2)	O(10)-C(93)	1.445(14)
C(62)-C(64)	1.511(16)	C(93)-C(94)	1.66(3)
C(66)-C(68)	1.546(11)	C(94)-C(95)	1.63(4)
O(7)-C(67)	1.382(9)	C(95)-C(96)	0.99(5)
O(7)-O(8)	2.632(7)	O(11)-C(97)	1.432(18)
C(67)-C(68)	1.365(11)	O(11)-C(100)	1.47(2)
C(67)-C(72)	1.389(11)	C(97)-C(98)	1.54(4)
C(68)-C(69)	1.379(11)	C(98)-C(99)	1.56(4)
C(69)-C(70)	1.396(13)	C(99)-C(100)	1.23(5)
C(70)-C(71)	1.364(13)	O(12)-C(109)	1.59(4)
C(70)-C(73)	1.515(13)	O(12)-C(112)	1.83(6)
C(71)-C(72)	1.380(11)	C(109)-C(110)	1.29(5)
C(72)-C(77)	1.521(12)	C(110)-C(111)	1.05(10)
C(73)-C(75)	1.07(5)	C(110)-C(112)	1.94(8)
C(73)-C(74)	1.52(3)	C(111)-C(112)	1.03(13)
C(73)-C(76)	1.54(3)	O(13)-O(16)#1	3.10(4)
C(74)-C(75)	2.02(6)	O(14)-O(15)#1	2.85(2)
C(77)-C(79)	1.517(11)	O(17)-O(18)#2	2.17(4)

O(17)-O(18)#1	13.32(5)	O(19)-O(18)#2	2.14(4)
O(18)-O(18)#2	0.00(9)		
O(5)-Na(1)-O(4)	97.74(18)	O(9)-Na(1)-O(6)	73.59(15)
O(5)-Na(1)-O(14)	160.42(15)	C(45)-Na(1)-O(6)	56.26(14)
O(4)-Na(1)-O(14)	95.14(17)	Na(2)-Na(1)-O(6)	77.51(8)
O(5)-Na(1)-O(10)	101.6(2)	O(5)-Na(1)-O(3)	131.60(16)
O(4)-Na(1)-O(10)	88.8(2)	O(4)-Na(1)-O(3)	40.43(16)
O(14)-Na(1)-O(10)	93.3(2)	O(14)-Na(1)-O(3)	56.11(14)
O(5)-Na(1)-O(13)	82.84(13)	O(10)-Na(1)-O(3)	100.3(2)
O(4)-Na(1)-O(13)	175.0(2)	O(13)-Na(1)-O(3)	136.51(13)
O(14)-Na(1)-O(13)	83.08(18)	O(9)-Na(1)-O(3)	79.84(15)
O(10)-Na(1)-O(13)	96.0(2)	C(45)-Na(1)-O(3)	126.65(13)
O(5)-Na(1)-O(9)	81.81(15)	Na(2)-Na(1)-O(3)	91.83(11)
O(4)-Na(1)-O(9)	94.52(19)	O(6)-Na(1)-O(3)	152.59(14)
O(14)-Na(1)-O(9)	82.5(2)	O(5)-Na(1)-O(2)	141.31(15)
O(10)-Na(1)-O(9)	174.9(2)	O(4)-Na(1)-O(2)	72.63(16)
O(13)-Na(1)-O(9)	80.6(2)	O(14)-Na(1)-O(2)	32.47(15)
O(5)-Na(1)-C(45)	24.42(18)	O(10)-Na(1)-O(2)	115.2(2)
O(4)-Na(1)-C(45)	86.60(16)	O(13)-Na(1)-O(2)	103.81(12)
O(14)-Na(1)-C(45)	172.80(16)	O(9)-Na(1)-O(2)	62.37(15)
O(10)-Na(1)-C(45)	79.7(2)	C(45)-Na(1)-O(2)	153.49(15)
O(13)-Na(1)-C(45)	95.74(13)	Na(2)-Na(1)-O(2)	58.78(9)
O(9)-Na(1)-C(45)	104.32(15)	O(6)-Na(1)-O(2)	130.61(10)
O(5)-Na(1)-Na(2)	111.29(12)	O(3)-Na(1)-O(2)	33.08(10)
O(4)-Na(1)-Na(2)	129.61(18)	O(1)-Na(2)-O(8)	100.72(18)
O(14)-Na(1)-Na(2)	49.33(13)	O(1)-Na(2)-O(13)	157.3(2)
O(10)-Na(1)-Na(2)	122.22(19)	O(8)-Na(2)-O(13)	92.45(14)
O(13)-Na(1)-Na(2)	46.09(13)	O(1)-Na(2)-O(11)	103.8(2)
O(9)-Na(1)-Na(2)	52.72(15)	O(8)-Na(2)-O(11)	88.7(2)
C(45)-Na(1)-Na(2)	133.52(12)	O(13)-Na(2)-O(11)	94.8(3)
O(5)-Na(1)-O(6)	38.12(14)	O(1)-Na(2)-O(14)	81.59(16)
O(4)-Na(1)-O(6)	134.64(19)	O(8)-Na(2)-O(14)	172.73(16)
O(14)-Na(1)-O(6)	125.11(13)	O(13)-Na(2)-O(14)	83.25(18)
O(10)-Na(1)-O(6)	106.7(2)	O(11)-Na(2)-O(14)	97.5(3)
O(13)-Na(1)-O(6)	45.16(14)	O(1)-Na(2)-O(9)	79.04(18)

O(8)-Na(2)-O(9)	94.66(15)	Na(2)-O(1)-O(2)	108.9(2)
O(13)-Na(2)-O(9)	81.6(2)	C(6)-C(1)-O(1)	121.9(7)
O(11)-Na(2)-O(9)	175.1(3)	C(6)-C(1)-C(2)	122.0(7)
O(14)-Na(2)-O(9)	78.9(2)	O(1)-C(1)-C(2)	115.9(7)
O(1)-Na(2)-C(1)	24.91(19)	C(6)-C(1)-Na(2)	115.7(6)
O(8)-Na(2)-C(1)	89.24(17)	O(1)-C(1)-Na(2)	44.8(3)
O(13)-Na(2)-C(1)	175.9(2)	C(2)-C(1)-Na(2)	101.3(5)
O(11)-Na(2)-C(1)	81.6(2)	C(3)-C(2)-C(1)	116.5(8)
O(14)-Na(2)-C(1)	95.43(16)	C(3)-C(2)-C(88)	121.3(7)
O(9)-Na(2)-C(1)	101.95(19)	C(1)-C(2)-C(88)	122.2(6)
O(1)-Na(2)-Na(1)	108.33(17)	C(4)-C(3)-C(2)	122.8(8)
O(8)-Na(2)-Na(1)	126.82(13)	C(3)-C(4)-C(5)	117.4(8)
O(13)-Na(2)-Na(1)	49.45(13)	C(3)-C(4)-C(7)	119.5(9)
O(11)-Na(2)-Na(1)	124.3(2)	C(5)-C(4)-C(7)	123.1(10)
O(14)-Na(2)-Na(1)	46.19(13)	C(6)-C(5)-C(4)	122.6(9)
O(9)-Na(2)-Na(1)	50.84(14)	C(5)-C(6)-C(1)	118.5(8)
C(1)-Na(2)-Na(1)	131.59(17)	C(5)-C(6)-C(11)	119.6(8)
O(1)-Na(2)-O(2)	37.20(15)	C(1)-C(6)-C(11)	121.9(7)
O(8)-Na(2)-O(2)	136.57(16)	C(10)-C(7)-C(8)	111(3)
O(13)-Na(2)-O(2)	124.21(13)	C(10)-C(7)-C(9)	105(3)
O(11)-Na(2)-O(2)	108.3(2)	C(8)-C(7)-C(9)	103(2)
O(14)-Na(2)-O(2)	44.68(14)	C(10)-C(7)-C(4)	115.8(17)
O(9)-Na(2)-O(2)	71.60(15)	C(8)-C(7)-C(4)	112.6(13)
C(1)-Na(2)-O(2)	55.89(15)	C(9)-C(7)-C(4)	107.5(16)
Na(1)-Na(2)-O(2)	76.28(10)	C(13)-C(11)-C(6)	115.4(8)
O(1)-Na(2)-O(7)	130.43(19)	C(12)-O(2)-O(3)	114.2(4)
O(8)-Na(2)-O(7)	38.31(14)	C(12)-O(2)-O(1)	112.6(4)
O(13)-Na(2)-O(7)	55.79(14)	O(3)-O(2)-O(1)	132.8(3)
O(11)-Na(2)-O(7)	101.5(2)	C(12)-O(2)-O(14)	137.2(6)
O(14)-Na(2)-O(7)	135.75(13)	O(3)-O(2)-O(14)	77.19(19)
O(9)-Na(2)-O(7)	79.13(15)	O(1)-O(2)-O(14)	72.33(18)
C(1)-Na(2)-O(7)	126.55(17)	C(12)-O(2)-Na(2)	135.7(4)
Na(1)-Na(2)-O(7)	90.73(9)	O(3)-O(2)-Na(2)	106.4(2)
O(2)-Na(2)-O(7)	149.91(14)	O(1)-O(2)-Na(2)	33.88(15)
C(1)-O(1)-Na(2)	110.3(5)	O(14)-O(2)-Na(2)	38.70(11)
C(1)-O(1)-O(2)	116.8(4)	C(12)-O(2)-Na(1)	161.5(5)

O(3)-O(2)-Na(1)	61.50(16)	O(3)-C(23)-C(28)	116.7(7)
O(1)-O(2)-Na(1)	75.52(17)	C(24)-C(23)-C(28)	122.2(7)
O(14)-O(2)-Na(1)	27.00(12)	C(23)-C(24)-C(25)	116.6(8)
Na(2)-O(2)-Na(1)	44.94(7)	C(23)-C(24)-C(22)	123.0(7)
O(2)-C(12)-C(13)	120.0(7)	C(25)-C(24)-C(22)	120.2(8)
O(2)-C(12)-C(17)	120.6(8)	C(26)-C(25)-C(24)	122.0(9)
C(13)-C(12)-C(17)	119.3(7)	C(27)-C(26)-C(25)	118.1(8)
C(12)-C(13)-C(14)	118.9(8)	C(27)-C(26)-C(29)	122.4(9)
C(12)-C(13)-C(11)	121.2(7)	C(25)-C(26)-C(29)	119.4(10)
C(14)-C(13)-C(11)	119.9(8)	C(26)-C(27)-C(28)	122.4(8)
C(15)-C(14)-C(13)	122.3(9)	C(27)-C(28)-C(23)	118.5(8)
C(14)-C(15)-C(16)	118.1(8)	C(27)-C(28)-C(33)	120.3(8)
C(14)-C(15)-C(18)	118.2(10)	C(23)-C(28)-C(33)	121.2(7)
C(16)-C(15)-C(18)	123.7(9)	C(30)-C(29)-C(31)	108.8(15)
C(15)-C(16)-C(17)	122.0(9)	C(30)-C(29)-C(32)	114.1(14)
C(16)-C(17)-C(12)	119.4(9)	C(31)-C(29)-C(32)	109.0(14)
C(16)-C(17)-C(22)	120.4(8)	C(30)-C(29)-C(26)	113.3(10)
C(12)-C(17)-C(22)	120.1(7)	C(31)-C(29)-C(26)	106.7(11)
C(19)-C(18)-C(21)	104.6(15)	C(32)-C(29)-C(26)	104.6(11)
C(19)-C(18)-C(15)	109.3(10)	C(35)-C(33)-C(28)	114.8(7)
C(21)-C(18)-C(15)	111.4(11)	C(34)-O(4)-Na(1)	148.4(5)
C(19)-C(18)-C(20)	112.0(14)	C(34)-O(4)-O(3)	107.3(4)
C(21)-C(18)-C(20)	108.5(13)	Na(1)-O(4)-O(3)	104.2(2)
C(15)-C(18)-C(20)	110.8(11)	C(35)-C(34)-O(4)	120.1(7)
C(24)-C(22)-C(17)	115.7(8)	C(35)-C(34)-C(39)	121.8(7)
C(23)-O(3)-O(2)	119.0(5)	O(4)-C(34)-C(39)	118.1(7)
C(23)-O(3)-O(4)	121.4(4)	C(36)-C(35)-C(34)	117.7(8)
O(2)-O(3)-O(4)	119.3(2)	C(36)-C(35)-C(33)	119.5(7)
C(23)-O(3)-O(14)	155.7(5)	C(34)-C(35)-C(33)	122.8(7)
O(2)-O(3)-O(14)	56.03(17)	C(35)-C(36)-C(37)	123.1(8)
O(4)-O(3)-O(14)	71.15(16)	C(38)-C(37)-C(36)	116.2(8)
C(23)-O(3)-Na(1)	155.2(4)	C(38)-C(37)-C(40)	119.0(9)
O(2)-O(3)-Na(1)	85.42(18)	C(36)-C(37)-C(40)	124.8(8)
O(4)-O(3)-Na(1)	35.42(13)	C(39)-C(38)-C(37)	123.1(9)
O(14)-O(3)-Na(1)	36.67(10)	C(38)-C(39)-C(34)	117.8(7)
O(3)-C(23)-C(24)	121.0(7)	C(38)-C(39)-C(44)	120.0(8)

C(34)-C(39)-C(44)	122.2(7)	C(56)-O(6)-O(5)	111.7(4)
C(42)-C(40)-C(43)	113.1(13)	O(7)-O(6)-O(5)	134.0(3)
C(42)-C(40)-C(37)	112.8(10)	C(56)-O(6)-O(13)	133.6(5)
C(43)-C(40)-C(37)	111.4(9)	O(7)-O(6)-O(13)	78.29(19)
C(42)-C(40)-C(41)	107.2(12)	O(5)-O(6)-O(13)	73.50(18)
C(43)-C(40)-C(41)	105.9(12)	C(56)-O(6)-Na(1)	134.2(4)
C(37)-C(40)-C(41)	105.8(11)	O(7)-O(6)-Na(1)	107.15(19)
C(46)-C(44)-C(39)	112.2(6)	O(5)-O(6)-Na(1)	34.65(14)
C(45)-O(5)-Na(1)	110.9(4)	O(13)-O(6)-Na(1)	39.23(11)
C(45)-O(5)-O(6)	117.2(4)	O(6)-C(56)-C(57)	121.8(7)
Na(1)-O(5)-O(6)	107.2(2)	O(6)-C(56)-C(61)	119.9(7)
O(5)-C(45)-C(50)	122.0(6)	C(57)-C(56)-C(61)	118.3(7)
O(5)-C(45)-C(46)	116.1(7)	C(58)-C(57)-C(56)	120.0(7)
C(50)-C(45)-C(46)	121.9(7)	C(58)-C(57)-C(55)	120.4(7)
O(5)-C(45)-Na(1)	44.7(3)	C(56)-C(57)-C(55)	119.6(7)
C(50)-C(45)-Na(1)	115.7(5)	C(57)-C(58)-C(59)	123.1(8)
C(46)-C(45)-Na(1)	104.5(4)	C(60)-C(59)-C(58)	116.1(7)
C(47)-C(46)-C(45)	116.1(7)	C(60)-C(59)-C(62)	122.7(8)
C(47)-C(46)-C(44)	121.6(7)	C(58)-C(59)-C(62)	121.0(8)
C(45)-C(46)-C(44)	122.3(7)	C(61)-C(60)-C(59)	122.6(8)
C(48)-C(47)-C(46)	124.1(7)	C(60)-C(61)-C(56)	119.8(7)
C(49)-C(48)-C(47)	117.4(7)	C(60)-C(61)-C(66)	120.7(7)
C(49)-C(48)-C(51)	120.8(8)	C(56)-C(61)-C(66)	119.5(7)
C(47)-C(48)-C(51)	121.8(8)	C(63)-C(62)-C(65)	112.3(17)
C(48)-C(49)-C(50)	122.0(8)	C(63)-C(62)-C(64)	106.4(13)
C(45)-C(50)-C(49)	118.5(7)	C(65)-C(62)-C(64)	108.9(16)
C(45)-C(50)-C(55)	121.6(6)	C(63)-C(62)-C(59)	110.7(11)
C(49)-C(50)-C(55)	119.9(7)	C(65)-C(62)-C(59)	107.6(10)
C(53)-C(51)-C(52)	112(2)	C(64)-C(62)-C(59)	111.0(9)
C(53)-C(51)-C(54)	108(3)	C(61)-C(66)-C(68)	115.0(7)
C(52)-C(51)-C(54)	104(2)	C(67)-O(7)-O(6)	118.3(5)
C(53)-C(51)-C(48)	110.0(11)	C(67)-O(7)-O(8)	124.6(5)
C(52)-C(51)-C(48)	113.9(10)	O(6)-O(7)-O(8)	117.0(2)
C(54)-C(51)-C(48)	108.2(10)	C(67)-O(7)-Na(2)	155.0(5)
C(57)-C(55)-C(50)	115.5(7)	O(6)-O(7)-Na(2)	84.22(17)
C(56)-O(6)-O(7)	114.1(4)	O(8)-O(7)-Na(2)	33.65(12)

C(68)-C(67)-O(7)	121.7(7)	C(83)-C(82)-C(81)	121.0(8)
C(68)-C(67)-C(72)	121.2(7)	C(78)-C(83)-C(82)	119.1(7)
O(7)-C(67)-C(72)	117.1(7)	C(78)-C(83)-C(88)	121.8(7)
C(67)-C(68)-C(69)	120.3(8)	C(82)-C(83)-C(88)	119.1(7)
C(67)-C(68)-C(66)	121.3(7)	C(85)-C(84)-C(87)	111.8(11)
C(69)-C(68)-C(66)	118.4(8)	C(85)-C(84)-C(86)	107.1(12)
C(68)-C(69)-C(70)	120.2(8)	C(87)-C(84)-C(86)	104.7(10)
C(71)-C(70)-C(69)	117.4(8)	C(85)-C(84)-C(81)	113.2(8)
C(71)-C(70)-C(73)	121.9(10)	C(87)-C(84)-C(81)	111.9(8)
C(69)-C(70)-C(73)	120.6(10)	C(86)-C(84)-C(81)	107.7(8)
C(70)-C(71)-C(72)	124.0(8)	C(83)-C(88)-C(2)	113.8(6)
C(71)-C(72)-C(67)	116.8(8)	C(92)-O(9)-C(89)	107.1(8)
C(71)-C(72)-C(77)	121.9(7)	C(92)-O(9)-Na(1)	113.2(6)
C(67)-C(72)-C(77)	121.3(7)	C(89)-O(9)-Na(1)	122.5(6)
C(75)-C(73)-C(70)	115(2)	C(92)-O(9)-Na(2)	125.3(6)
C(75)-C(73)-C(74)	101(3)	C(89)-O(9)-Na(2)	110.9(6)
C(70)-C(73)-C(74)	114.9(12)	Na(1)-O(9)-Na(2)	76.44(17)
C(75)-C(73)-C(76)	110(3)	C(90)-C(89)-O(9)	108.1(11)
C(70)-C(73)-C(76)	110.8(13)	C(89)-C(90)-C(91)	103.1(15)
C(74)-C(73)-C(76)	104(2)	C(92)-C(91)-C(90)	109.3(15)
C(73)-C(74)-C(75)	31.2(13)	C(91)-C(92)-O(9)	109.4(11)
C(73)-C(75)-C(74)	47(3)	C(96)-O(10)-C(93)	112.1(12)
C(79)-C(77)-C(72)	117.0(8)	C(96)-O(10)-Na(1)	129.3(10)
C(78)-O(8)-Na(2)	146.8(4)	C(93)-O(10)-Na(1)	118.3(8)
C(78)-O(8)-O(7)	105.1(4)	O(10)-C(93)-C(94)	95.9(14)
Na(2)-O(8)-O(7)	108.0(2)	C(95)-C(94)-C(93)	97.2(15)
C(83)-C(78)-C(79)	120.4(7)	C(96)-C(95)-C(94)	111(2)
C(83)-C(78)-O(8)	118.9(6)	C(95)-C(96)-O(10)	119(3)
C(79)-C(78)-O(8)	120.8(7)	C(97)-O(11)-C(100)	113.9(13)
C(80)-C(79)-C(78)	119.1(7)	C(97)-O(11)-Na(2)	128.4(9)
C(80)-C(79)-C(77)	119.0(7)	C(100)-O(11)-Na(2)	116.1(9)
C(78)-C(79)-C(77)	121.9(7)	O(11)-C(97)-C(98)	97.9(17)
C(81)-C(80)-C(79)	122.4(7)	C(97)-C(98)-C(99)	105(2)
C(80)-C(81)-C(82)	117.8(7)	C(100)-C(99)-C(98)	109(3)
C(80)-C(81)-C(84)	123.9(7)	C(99)-C(100)-O(11)	109(2)
C(82)-C(81)-C(84)	118.2(8)	C(109)-O(12)-C(112)	95(3)

C(110)-C(109)-O(12)	92(4)	O(6)-O(13)-O(16)#1	102.9(7)
C(111)-C(110)-C(109)	121(10)	Na(1)-O(14)-Na(2)	84.48(18)
C(111)-C(110)-C(112)	21(8)	Na(1)-O(14)-O(2)	120.5(3)
C(109)-C(110)-C(112)	101(5)	Na(2)-O(14)-O(2)	96.62(19)
C(112)-C(111)-C(110)	138(10)	Na(1)-O(14)-O(15)#1	122.6(5)
C(111)-C(112)-O(12)	87(6)	Na(2)-O(14)-O(15)#1	132.7(5)
C(111)-C(112)-C(110)	21(5)	O(2)-O(14)-O(15)#1	99.4(4)
O(12)-C(112)-C(110)	67.1(19)	Na(1)-O(14)-O(3)	87.22(17)
Na(2)-O(13)-Na(1)	84.46(18)	Na(2)-O(14)-O(3)	128.3(2)
Na(2)-O(13)-O(6)	122.6(3)	O(2)-O(14)-O(3)	46.78(14)
Na(1)-O(13)-O(6)	95.61(19)	O(15)#1-O(14)-O(3)	93.8(4)
Na(2)-O(13)-O(16)#1	120.4(7)	O(18)#2-O(17)-O(18)#1	84.7(12)
Na(1)-O(13)-O(16)#1	130.1(7)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1 #2 x,y,z

Anisotropic displacement parameters ($Å^2$x 10^3)for $[Na_2(4\text{-tert-butylcalix[8]arene-2H})(THF)_4(H_2O)_5]$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Na(1)	34	70	43	-2	16	-5
Na(2)	36	74	43	0	17	6
O(1)	29	76	37	-5	11	1
C(1)	26	62	36	-3	14	-6
C(2)	38	55	43	8	19	-1
C(3)	38	66	46	3	7	-3
C(4)	33	78	62	-11	2	-12
C(5)	50	65	42	-13	13	-5
C(6)	39	59	32	-5	14	1
C(7)	65	177	95	-89	-26	-17
C(8)	80	129	79	-65	-108	71
C(9)	141	182	191	79	-79	-166
C(10)	197	954	343	-519	-182	189
C(11)	39	62	39	-10	10	1
O(2)	33	82	33	-4	17	-6

C(12)	33	62	38	1	13	8
C(13)	43	54	46	-5	21	13
C(14)	52	77	39	-13	15	11
C(15)	52	94	43	0	23	19
C(16)	52	87	37	2	29	18
C(17)	43	62	49	12	22	12
C(18)	105	143	68	-11	56	3
C(19)	47	183	0	8	-4	44
C(20)	154	302	67	-19	73	-51
C(21)	233	146	114	-53	111	-10
C(22)	42	60	62	13	31	12
O(3)	31	100	44	3	23	-3
C(23)	31	64	40	-12	21	-4
C(24)	38	64	52	-7	31	-10
C(25)	56	90	61	3	37	-15
C(26)	36	108	66	-7	31	0
C(27)	41	80	58	-2	29	-4
C(28)	38	52	51	-12	22	-7
C(29)	69	140	161	33	85	14
C(30)	25	139	69	55	44	-12
C(31)	14	104	62	14	9	-34
C(32)	125	206	269	57	166	41
C(33)	41	45	52	3	20	-2
O(4)	25	73	48	-2	15	-11
C(34)	30	48	36	5	10	-7
C(35)	30	61	42	3	18	-6
C(36)	31	91	67	-21	29	-12
C(37)	31	84	62	-24	20	-18
C(38)	45	76	64	-18	34	-10
C(39)	33	53	46	1	17	-2
C(40)	53	134	150	-83	69	-66
C(41)	103	132	163	-57	71	-78
C(42)	6	128	18	-36	-1	-18
C(43)	0	106	96	-73	35	-37
C(44)	37	53	52	5	27	8
O(5)	25	72	27	8	5	4

C(45)	31	47	28	-5	14	1
C(46)	26	54	43	-2	19	5
C(47)	21	52	50	1	9	-1
C(48)	31	54	30	-1	3	5
C(49)	37	50	48	3	20	1
C(50)	32	37	35	-2	14	3
C(51)	56	88	59	25	-6	14
C(52)	70	94	113	104	-103	-72
C(53)	179	170	140	-88	-164	183
C(54)	51	528	2	92	-29	-8
C(55)	38	39	49	0	15	2
O(6)	29	79	33	5	14	3
C(56)	21	57	32	0	11	-4
C(57)	35	36	44	1	20	-1
C(58)	35	62	48	15	20	7
C(59)	57	69	33	12	23	1
C(60)	43	60	43	-2	24	2
C(61)	24	61	35	5	13	0
C(62)	74	157	49	24	39	20
C(63)	157	82	88	100	109	101
C(64)	68	87	12	28	44	34
C(65)	147	179	0	-20	30	-100
C(66)	51	52	32	5	14	13
O(7)	29	90	36	-2	12	8
C(67)	28	69	47	4	22	12
C(68)	38	61	39	9	12	7
C(69)	43	102	50	-10	30	10
C(70)	42	124	51	6	27	22
C(71)	29	97	50	20	11	5
C(72)	31	56	42	9	19	4
C(73)	1	146	50	-15	39	-24
C(74)	32	259	122	-115	53	-6
C(75)	390	552	815	173	561	154
C(76)	24	462	62	-32	11	115
C(77)	31	53	56	8	10	-9
O(8)	23	72	46	0	13	9

C(78)	27	50	34	-2	11	-6
C(79)	37	44	38	6	7	0
C(80)	30	52	42	7	17	-3
C(81)	28	60	65	10	17	7
C(82)	37	61	52	25	21	6
C(83)	23	59	39	-4	14	-3
C(84)	38	79	69	40	26	15
C(85)	0	78	87	67	19	18
C(86)	78	82	129	25	31	29
C(87)	49	183	86	34	13	31
C(88)	25	55	45	0	15	-4
O(9)	83	56	55	8	28	5
C(89)	100	83	104	-14	65	21
C(90)	172	19	253	-61	183	-35
C(91)	363	0	374	23	358	17
C(92)	103	87	88	-1	61	-4
O(10)	74	93	87	6	21	11
C(93)	68	112	130	31	27	24
C(94)	363	220	90	70	33	237
C(95)	240	72	246	21	184	-44
C(96)	74	7	175	43	72	0
O(11)	94	98	114	-15	38	-17
C(97)	89	2	161	-23	82	-9
C(98)	410	101	406	-82	216	-48
C(99)	183	414	339	61	140	135
C(100)	40	73	176	-104	-8	-42
O(12)	347	253	415	-3	14	-1
C(109)	279	305	440	-89	154	67
C(110)	408	269	588	-50	-145	70
C(111)	624	401	1189	592	549	325
C(112)	301	562	574	257	331	207
O(13)	43	69	43	0	15	-6
O(14)	43	69	46	-2	19	6
O(15)	260	260	260	0	104	0
O(16)	426	426	426	0	170	0
O(17)	355	355	355	0	142	0

O(18)	346	346	346	0	138	0
O(19)	299	299	299	0	119	0

Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for [Na_2(4-tert-butylcalix[8]arene-2H)$(THF)_4(H_2O)_5$].

	x	y	z	U(eq)
H(3)	417	1521	-1076	63
H(5)	1509	512	-1501	65
H(8A)	-278	1416	-1888	210
H(8B)	-590	1104	-2501	210
H(8C)	-4	1477	-2392	210
H(9A)	-238	635	-1407	326
H(9B)	122	149	-1521	326
H(9C)	-519	346	-2037	326
H(10A)	640	302	-2109	885
H(10B)	494	802	-2524	885
H(10C)	-65	419	-2572	885
H(11A)	2838	546	-149	58
H(11B)	2545	272	-786	58
H(14)	2827	564	-1611	68
H(16)	4205	1621	-1306	66
H(19A)	2705	1478	-2634	122
H(19B)	2465	909	-2594	122
H(19C)	2743	1042	-3081	122
H(20A)	4405	1151	-2059	248
H(20B)	3943	1632	-2309	248
H(20C)	3950	1182	-2753	248
H(21A)	3927	255	-2051	226
H(21B)	3455	319	-2736	226
H(21C)	3184	190	-2244	226
H(22A)	4485	2211	-446	62
H(22B)	4204	2119	45	62
H(25)	5572	2002	-235	78
H(27)	6701	1376	1358	68

H(30A)	6417	1963	-481	104
H(30B)	7064	2277	-198	104
H(30C)	6444	2493	-140	104
H(31A)	7372	2066	1335	93
H(31B)	7020	2560	958	93
H(31C)	7645	2347	907	93
H(32A)	7330	1214	855	258
H(32B)	7606	1445	402	258
H(32C)	6952	1137	151	258
H(33A)	5502	913	1777	55
H(33B)	6248	927	1973	55
H(36)	6867	1661	2571	72
H(38)	6134	2476	3564	70
H(41A)	6977	3078	2888	191
H(41B)	7503	3227	3529	191
H(41C)	6787	3149	3445	191
H(42A)	7869	2534	4355	79
H(42B)	7530	1987	4264	79
H(42C)	7150	2497	4275	79
H(43A)	7612	2284	2826	93
H(43B)	7893	1883	3364	93
H(43C)	8149	2461	3450	93
H(44A)	5015	2460	3345	53
H(44B)	4543	2131	2794	53
H(47)	5817	1753	4198	51
H(49)	4829	693	4676	53
H(52A)	6256	1814	5340	204
H(52B)	6670	1545	5031	204
H(52C)	6798	1415	5710	204
H(53A)	5978	348	4911	353
H(53B)	6638	523	5418	353
H(53C)	6468	670	4735	353
H(54A)	5633	1327	5698	309
H(54B)	6171	903	5985	309
H(54C)	5486	736	5517	309
H(55A)	3481	660	3301	51

H(55B)	3796	398	3944	51
H(58)	3493	684	4747	57
H(60)	2038	1670	4413	55
H(63A)	2531	279	5223	134
H(63B)	2925	427	5909	134
H(63C)	3278	362	5467	134
H(64A)	2078	1557	5296	70
H(64B)	2198	1125	5796	70
H(64C)	1817	986	5106	70
H(65A)	3235	1740	5646	162
H(65B)	3720	1274	5757	162
H(65C)	3341	1340	6175	162
H(66A)	2022	2157	3048	55
H(66B)	1753	2262	3546	55
H(69)	703	2006	3385	74
H(71)	-411	1254	1877	72
H(77A)	761	846	1409	59
H(77B)	17	854	1217	59
H(80)	-615	1584	594	48
H(82)	82	2314	-487	59
H(85A)	-1628	1729	-239	82
H(85B)	-1934	2290	-398	82
H(85C)	-1419	2170	259	82
H(86A)	-586	2967	-354	149
H(86B)	-802	2914	190	149
H(86C)	-1313	3033	-468	149
H(87A)	-888	2320	-1153	165
H(87B)	-1605	2422	-1252	165
H(87C)	-1334	1848	-1161	165
H(88A)	1679	1993	293	50
H(88B)	1195	2292	-277	50
H(1)	5010(40)	1920(40)	6980(40)	61
H(2)	6090(30)	1710(30)	7410(30)	29
H(3)	5600(30)	2190(20)	6470(30)	13
H(4)	6110(30)	2090(30)	6810(30)	17

V.2.12. Crystal data and structure refinement for [K(4-tert-butylcalix[8]arene-H)(THF)$_4$(H_2O)$_7$] 12

Empirical formula	C104 H157 K O19
Formula weight	1750.45
Temperature	243(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 35.828(7) Å α= 90°.
b = 22.515(5) Å	β= 109.00(3)°.
c = 28.649(6) Å	γ = 90°.
Volume	21851(8) Å^3
Z	8
Density (calculated)	1.050 Mg/m^3
Absorption coefficient	0.108 mm^{-1}
F(000)	7432
Theta range for data collection	1.09 to 19.56°.
Index ranges	-33<=h<=31, -21<=k<=20, -24<=l<=26
Reflections collected	28900
Independent reflections	8989 [R(int) = 0.1217]
Completeness to theta = 19.56°	94.0 %
Absorption correction	Spherical
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8989 / 0 / 1123
Goodness-of-fit on F^2	1.485
Final R indices [I>2sigma(I)]	R1 = 0.1255, wR2 = 0.2634
R indices (all data)	R1 = 0.1853, wR2 = 0.2878
Largest diff. peak and hole	1.280 and -0.281 e.Å^{-3}

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [K (4-tert-butylcalix[8]arene-H$(THF)_4(H_2O)_7$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
K(1)	460(1)	5244(1)	5591(1)	75(1)
O(1)	445(2)	3730(3)	6393(3)	54(2)
C(1)	645(4)	3213(5)	6568(4)	39(3)
C(2)	1014(3)	3128(5)	6527(4)	37(3)
C(3)	1207(3)	2598(5)	6708(4)	45(3)
C(4)	1037(3)	2150(4)	6908(3)	36(3)
C(5)	667(3)	2260(5)	6944(3)	36(3)
C(6)	463(3)	2783(5)	6769(3)	34(3)
C(7)	1259(4)	1560(5)	7079(4)	50(3)
C(8)	1679(4)	1672(6)	7422(6)	121(7)
C(9)	1303(5)	1223(6)	6638(5)	105(5)
C(10)	1034(5)	1143(7)	7333(7)	134(7)
C(11)	55(3)	2880(5)	6811(3)	40(3)
O(2)	-278(2)	3812(3)	6098(2)	44(2)
C(12)	-446(3)	3263(5)	6010(4)	34(3)
C(13)	-284(3)	2796(5)	6336(4)	36(3)
C(14)	-459(3)	2237(5)	6225(4)	44(3)
C(15)	-788(3)	2131(5)	5803(4)	46(3)
C(16)	-944(3)	2610(5)	5513(4)	41(3)
C(17)	-787(3)	3168(5)	5607(4)	34(3)
C(18)	-958(4)	1494(5)	5719(4)	57(3)
C(19)	-635(5)	1039(6)	5733(7)	118(6)
C(20)	-1268(4)	1440(6)	5199(5)	103(5)
C(21)	-1151(5)	1349(6)	6095(6)	123(7)
C(22)	-1000(3)	3681(5)	5290(4)	43(3)
O(3)	-805(2)	4616(3)	6019(3)	55(2)
C(23)	-1194(4)	4439(5)	5834(4)	39(3)
C(24)	-1294(3)	3984(5)	5483(4)	37(3)
C(25)	-1686(4)	3807(5)	5303(4)	43(3)
C(26)	-1983(3)	4075(5)	5452(4)	39(3)
C(27)	-1858(4)	4517(5)	5809(4)	42(3)

C(28)	-1464(4)	4701(5)	6007(4)	39(3)
C(29)	-2425(3)	3884(5)	5245(4)	47(3)
C(30)	-2474(5)	3233(7)	5113(8)	148(8)
C(31)	-2642(5)	3995(9)	5617(6)	147(8)
C(32)	-2620(5)	4222(9)	4776(6)	168(10)
C(33)	-1379(3)	5184(5)	6402(4)	49(3)
O(4)	-1074(2)	6039(3)	5911(3)	59(2)
C(34)	-1439(3)	6155(5)	5946(4)	38(3)
C(35)	-1593(4)	5759(5)	6206(4)	45(3)
C(36)	-1954(4)	5882(5)	6253(4)	50(3)
C(37)	-2177(3)	6376(6)	6045(4)	49(3)
C(38)	-2011(4)	6748(5)	5780(4)	51(3)
C(39)	-1649(4)	6647(5)	5727(4)	44(3)
C(40)	-2577(4)	6498(7)	6107(5)	79(4)
C(41)	-2766(7)	7052(12)	5886(13)	280(20)
C(42)	-2527(6)	6521(14)	6657(7)	236(15)
C(43)	-2844(6)	5993(12)	5917(11)	239(16)
C(44)	-1484(4)	7091(5)	5435(4)	57(3)
O(5)	-686(2)	7022(3)	6015(3)	58(2)
C(45)	-867(4)	7545(5)	6038(4)	48(3)
C(46)	-1263(4)	7595(5)	5745(4)	48(3)
C(47)	-1453(4)	8119(6)	5772(4)	59(4)
C(48)	-1261(5)	8609(6)	6071(5)	69(4)
C(49)	-872(5)	8534(5)	6340(5)	59(4)
C(50)	-667(4)	8009(5)	6333(4)	47(3)
C(51)	-1498(5)	9204(6)	6071(7)	101(5)
C(52)	-1450(7)	9586(7)	5675(9)	175(10)
C(53)	-1935(6)	9104(7)	5946(11)	204(13)
C(54)	-1308(8)	9540(10)	6549(9)	230(15)
C(55)	-234(4)	7951(5)	6647(4)	54(3)
O(6)	33(2)	7011(3)	6150(3)	46(2)
C(56)	187(3)	7571(5)	6147(4)	41(3)
C(57)	61(3)	8039(5)	6371(4)	45(3)
C(58)	218(4)	8582(5)	6351(4)	56(3)
C(59)	499(4)	8701(5)	6130(4)	58(3)
C(60)	620(4)	8220(6)	5909(4)	55(3)

C(61)	465(3)	7659(5)	5911(4)	41(3)
C(62)	693(6)	9311(6)	6135(7)	101(6)
C(63)	401(8)	9787(9)	6069(15)	320(20)
C(64)	974(11)	9387(10)	5897(13)	300(20)
C(65)	952(10)	9453(11)	6666(10)	280(20)
C(66)	627(3)	7143(4)	5693(3)	42(3)
O(7)	566(2)	6165(3)	6328(2)	43(2)
C(67)	935(4)	6383(4)	6367(4)	37(3)
C(68)	978(4)	6840(4)	6057(3)	38(3)
C(69)	1360(4)	7048(5)	6108(4)	42(3)
C(70)	1693(4)	6804(5)	6447(4)	41(3)
C(71)	1640(3)	6356(5)	6740(4)	40(3)
C(72)	1273(4)	6130(4)	6717(4)	36(3)
C(73)	2105(4)	7043(5)	6506(4)	54(3)
C(74)	2291(5)	7300(8)	7013(6)	119(6)
C(75)	2374(4)	6527(7)	6455(6)	110(6)
C(76)	2104(5)	7496(9)	6111(7)	168(10)
C(77)	1241(3)	5635(5)	7067(4)	46(3)
O(8)	834(2)	4702(3)	6456(3)	48(2)
C(78)	1231(3)	4616(5)	6709(4)	36(3)
C(79)	1437(4)	5070(5)	7017(4)	34(3)
C(80)	1832(4)	4980(5)	7258(4)	46(3)
C(81)	2051(3)	4477(5)	7208(3)	38(3)
C(82)	1829(4)	4037(5)	6892(4)	40(3)
C(83)	1423(4)	4092(5)	6646(4)	35(3)
C(84)	2491(4)	4416(5)	7476(4)	54(3)
C(85)	2557(5)	4367(10)	8031(6)	149(8)
C(86)	2722(4)	4956(7)	7380(7)	121(6)
C(87)	2669(5)	3849(7)	7311(6)	114(6)
C(88)	1203(3)	3593(4)	6305(4)	41(3)
O(9)	-16(2)	4218(3)	5268(3)	65(2)
O(10)	-327(3)	5483(4)	5711(4)	103(3)
O(11)	1212(3)	4975(5)	5514(4)	106(3)
C(89)	1583(6)	5260(7)	5780(5)	99(6)
C(90)	1898(6)	4882(10)	5716(7)	126(6)
C(91)	1729(8)	4397(10)	5389(9)	155(9)

C(92)	1304(7)	4519(8)	5222(5)	111(7)
O(12)	1095(4)	6164(6)	2879(5)	131(4)
C(93)	1487(6)	6294(8)	2916(6)	109(6)
C(94)	1601(6)	6816(9)	3252(9)	145(8)
C(95)	1207(9)	7049(8)	3288(8)	124(7)
C(96)	930(6)	6594(10)	3083(7)	102(6)
O(13)	974(5)	3009(6)	2694(6)	144(5)
C(97)	806(8)	2563(13)	2382(7)	175(12)
C(98)	1078(11)	2167(12)	2335(10)	215(16)
C(99)	1457(8)	2327(11)	2742(11)	172(9)
C(100)	1368(6)	2871(9)	2947(6)	119(6)
O(14)	631(5)	2438(6)	5352(4)	131(4)
C(101)	317(6)	2124(12)	5473(7)	129(7)
C(102)	323(8)	1534(13)	5301(12)	174(10)
C(103)	650(12)	1471(16)	5099(11)	207(13)
C(104)	882(9)	2022(16)	5264(12)	187(13)
O(15)	89(2)	4058(2)	3291(2)	19(2)
O(16)	-517(3)	3987(6)	2338(5)	146(5)
O(17)	83(5)	5259(7)	3147(5)	231(9)
O(18)	584(3)	5254(5)	2601(5)	147(5)
O(19)	-392(3)	6559(4)	5149(3)	91(3)

Bond lengths [Å] and angles [°] for [K (4-tert-butylcalix[8]arene-H$(THF)_4(H_2O)_7$].

K(1)-O(8)	2.694(7)	C(2)-C(88)	1.497(13)
K(1)-O(9)#1	2.739(8)	C(3)-C(4)	1.392(14)
K(1)-O(11)	2.840(11)	C(3)-H(3)	0.9300
K(1)-O(9)	2.842(8)	C(4)-C(5)	1.384(14)
K(1)-O(7)	2.894(7)	C(4)-C(7)	1.546(14)
K(1)-O(10)	2.998(10)	C(5)-C(6)	1.392(13)
K(1)-C(67)	3.456(10)	C(5)-H(5)	0.9300
K(1)-K(1)#1	4.035(6)	C(6)-C(11)	1.520(14)
O(1)-C(1)	1.371(12)	C(7)-C(8)	1.525(17)
O(1)-H(1)	0.8201	C(7)-C(10)	1.563(18)
C(1)-C(2)	1.378(14)	C(7)-C(9)	1.523(16)
C(1)-C(6)	1.392(14)	C(8)-H(8A)	0.9600
C(2)-C(3)	1.393(14)	C(8)-H(8B)	0.9600

C(8)-H(8C)	0.9600	C(22)-H(22B)	0.9700
C(9)-H(9A)	0.9600	O(3)-C(23)	1.379(12)
C(9)-H(9B)	0.9600	O(3)-H(3A)	0.8200
C(9)-H(9C)	0.9600	C(23)-C(24)	1.399(14)
C(10)-H(10A)	0.9600	C(23)-C(28)	1.359(14)
C(10)-H(10B)	0.9600	C(24)-C(25)	1.388(14)
C(10)-H(10C)	0.9600	C(25)-C(26)	1.402(14)
C(11)-C(13)	1.515(14)	C(25)-H(25)	0.9300
C(11)-H(11A)	0.9700	C(26)-C(27)	1.392(14)
C(11)-H(11B)	0.9700	C(26)-C(29)	1.559(15)
O(2)-C(12)	1.362(12)	C(27)-C(28)	1.403(14)
O(2)-H(2)	0.8201	C(27)-H(27)	0.9300
C(12)-C(17)	1.397(14)	C(28)-C(33)	1.527(14)
C(12)-C(13)	1.400(14)	C(29)-C(32)	1.503(17)
C(13)-C(14)	1.394(14)	C(29)-C(30)	1.509(16)
C(14)-C(15)	1.407(15)	C(29)-C(31)	1.531(17)
C(14)-H(14)	0.9300	C(30)-H(30A)	0.9600
C(15)-C(16)	1.364(14)	C(30)-H(30B)	0.9600
C(15)-C(18)	1.546(15)	C(30)-H(30C)	0.9600
C(16)-C(17)	1.367(13)	C(31)-H(31A)	0.9600
C(16)-H(16)	0.9300	C(31)-H(31B)	0.9600
C(17)-C(22)	1.515(14)	C(31)-H(31C)	0.9600
C(18)-C(21)	1.494(16)	C(32)-H(32A)	0.9600
C(18)-C(20)	1.546(17)	C(32)-H(32B)	0.9600
C(18)-C(19)	1.536(17)	C(32)-H(32C)	0.9600
C(19)-H(19A)	0.9600	C(33)-C(35)	1.515(15)
C(19)-H(19B)	0.9600	C(33)-H(33A)	0.9700
C(19)-H(19C)	0.9600	C(33)-H(33B)	0.9700
C(20)-H(20A)	0.9600	O(4)-C(34)	1.370(12)
C(20)-H(20B)	0.9600	O(4)-H(4)	0.8209
C(20)-H(20C)	0.9600	C(34)-C(39)	1.371(14)
C(21)-H(21A)	0.9600	C(34)-C(35)	1.387(14)
C(21)-H(21B)	0.9600	C(35)-C(36)	1.370(15)
C(21)-H(21C)	0.9600	C(36)-C(37)	1.388(15)
C(22)-C(24)	1.503(14)	C(36)-H(36)	0.9300
C(22)-H(22A)	0.9700	C(37)-C(38)	1.387(15)

C(37)-C(40)	1.523(17)	C(52)-H(52C)	0.9600
C(38)-C(39)	1.374(15)	C(53)-H(53A)	0.9600
C(38)-H(38)	0.9300	C(53)-H(53B)	0.9600
C(39)-C(44)	1.540(15)	C(53)-H(53C)	0.9600
C(40)-C(41)	1.46(2)	C(54)-H(54A)	0.9600
C(40)-C(43)	1.47(2)	C(54)-H(54B)	0.9600
C(40)-C(42)	1.53(2)	C(54)-H(54C)	0.9600
C(41)-H(41A)	0.9600	C(55)-C(57)	1.526(15)
C(41)-H(41B)	0.9600	C(55)-H(55A)	0.9700
C(41)-H(41C)	0.9600	C(55)-H(55B)	0.9700
C(42)-H(42A)	0.9600	O(6)-C(56)	1.379(11)
C(42)-H(42B)	0.9600	O(6)-H(6)	0.8200
C(42)-H(42C)	0.9600	C(56)-C(61)	1.389(14)
C(43)-H(43A)	0.9600	C(56)-C(57)	1.383(14)
C(43)-H(43B)	0.9600	C(57)-C(58)	1.355(14)
C(43)-H(43C)	0.9600	C(58)-C(59)	1.379(16)
C(44)-C(46)	1.498(16)	C(58)-H(58)	0.9300
C(44)-H(44A)	0.9700	C(59)-C(60)	1.395(15)
C(44)-H(44B)	0.9700	C(59)-C(62)	1.537(17)
O(5)-C(45)	1.357(12)	C(60)-C(61)	1.380(14)
O(5)-H(5A)	0.8202	C(60)-H(60)	0.9300
C(45)-C(50)	1.387(16)	C(61)-C(66)	1.521(14)
C(45)-C(46)	1.398(16)	C(62)-C(64)	1.40(2)
C(46)-C(47)	1.375(15)	C(62)-C(63)	1.47(3)
C(47)-C(48)	1.429(17)	C(62)-C(65)	1.54(3)
C(47)-H(47)	0.9300	C(63)-H(63A)	0.9600
C(48)-C(49)	1.366(17)	C(63)-H(63B)	0.9600
C(48)-C(51)	1.586(18)	C(63)-H(63C)	0.9600
C(49)-C(50)	1.396(16)	C(64)-H(64A)	0.9600
C(49)-H(49)	0.9300	C(64)-H(64B)	0.9600
C(50)-C(55)	1.524(16)	C(64)-H(64C)	0.9600
C(51)-C(54)	1.52(3)	C(65)-H(65A)	0.9600
C(51)-C(52)	1.48(2)	C(65)-H(65B)	0.9600
C(51)-C(53)	1.50(2)	C(65)-H(65C)	0.9600
C(52)-H(52A)	0.9600	C(66)-C(68)	1.509(14)
C(52)-H(52B)	0.9600	C(66)-H(66A)	0.9700

C(66)-H(66B)	0.9700	C(82)-H(82)	0.9300
O(7)-C(67)	1.380(12)	C(83)-C(88)	1.529(14)
C(67)-C(72)	1.416(14)	C(84)-C(86)	1.546(17)
C(67)-C(68)	1.399(13)	C(84)-C(87)	1.564(17)
C(68)-C(69)	1.407(14)	C(84)-C(85)	1.533(18)
C(69)-C(70)	1.384(14)	C(85)-H(85A)	0.9600
C(69)-H(69)	0.9300	C(85)-H(85B)	0.9600
C(70)-C(71)	1.365(13)	C(85)-H(85C)	0.9600
C(70)-C(73)	1.530(15)	C(86)-H(86A)	0.9600
C(71)-C(72)	1.392(14)	C(86)-H(86B)	0.9600
C(71)-H(71)	0.9300	C(86)-H(86C)	0.9600
C(72)-C(77)	1.530(13)	C(87)-H(87A)	0.9600
C(73)-C(74)	1.504(17)	C(87)-H(87B)	0.9600
C(73)-C(76)	1.521(17)	C(87)-H(87C)	0.9600
C(73)-C(75)	1.545(18)	C(88)-H(88A)	0.9700
C(74)-H(74A)	0.9600	C(88)-H(88B)	0.9700
C(74)-H(74B)	0.9600	O(9)-K(1)#1	2.739(8)
C(74)-H(74C)	0.9600	O(9)-O(19)#1	2.785(12)
C(75)-H(75A)	0.9600	O(11)-C(89)	1.449(17)
C(75)-H(75B)	0.9600	O(11)-C(92)	1.428(17)
C(75)-H(75C)	0.9600	C(89)-C(90)	1.47(2)
C(76)-H(76A)	0.9600	C(89)-H(89A)	0.9700
C(76)-H(76B)	0.9600	C(89)-H(89B)	0.9700
C(76)-H(76C)	0.9600	C(90)-C(91)	1.44(2)
C(77)-C(79)	1.482(14)	C(90)-H(90A)	0.9700
C(77)-H(77A)	0.9700	C(90)-H(90B)	0.9700
C(77)-H(77B)	0.9700	C(91)-C(92)	1.47(2)
O(8)-C(78)	1.384(12)	C(91)-H(91A)	0.9700
C(78)-C(79)	1.393(14)	C(91)-H(91B)	0.9700
C(78)-C(83)	1.408(14)	C(92)-H(92A)	0.9700
C(79)-C(80)	1.374(14)	C(92)-H(92B)	0.9700
C(80)-C(81)	1.411(15)	O(12)-O(12)#2	0.00(4)
C(80)-H(80)	0.9300	O(12)-C(96)	1.363(18)
C(81)-C(82)	1.401(14)	O(12)-C(93)	1.405(18)
C(81)-C(84)	1.519(15)	C(93)-O(12)#2	1.405(18)
C(82)-C(83)	1.398(14)	C(93)-C(94)	1.49(2)

C(94)-C(95)	1.54(3)	C(101)-H(10F)	0.9700
C(95)-C(96)	1.42(2)	C(101)-H(10G)	0.9700
C(96)-O(12)#2	1.363(18)	C(102)-C(103)	1.47(3)
O(13)-C(97)	1.35(2)	C(102)-H(10H)	0.9700
O(13)-C(100)	1.398(19)	C(102)-H(10I)	0.9700
C(97)-C(98)	1.36(3)	C(103)-C(104)	1.48(3)
C(97)-H(97A)	0.9700	C(103)-H(10J)	0.9700
C(97)-H(97B)	0.9700	C(103)-H(10K)	0.9700
C(98)-C(99)	1.52(3)	C(104)-H(10L)	0.9700
C(98)-H(98A)	0.9700	C(104)-H(10M)	0.9700
C(98)-H(98B)	0.9700	O(15)-O(17)	2.736(18)
C(99)-C(100)	1.44(2)	O(15)-O(10)#1	2.896(12)
C(99)-H(99A)	0.9700	O(15)-O(16)	2.888(14)
C(99)-H(99B)	0.9700	O(16)-O(13)#3	2.726(17)
C(100)-H(10D)	0.9700	O(16)-O(18)	4.725(16)
C(100)-H(10E)	0.9700	O(17)-O(18)	2.74(2)
O(14)-C(104)	1.38(2)	O(18)-O(12)#2	2.690(17)
O(14)-C(101)	1.46(2)	O(19)-O(14)#1	2.663(17)
C(101)-C(102)	1.42(3)		

O(8)-K(1)-O(9)#1	174.6(3)	O(9)#1-K(1)-C(67)	105.7(3)
O(8)-K(1)-O(11)	77.0(3)	O(11)-K(1)-C(67)	85.8(3)
O(9)#1-K(1)-O(11)	108.2(3)	O(9)-K(1)-C(67)	158.5(2)
O(8)-K(1)-O(9)	89.8(2)	O(7)-K(1)-C(67)	23.0(2)
O(9)#1-K(1)-O(9)	87.4(2)	O(10)-K(1)-C(67)	94.5(3)
O(11)-K(1)-O(9)	106.5(3)	O(8)-K(1)-K(1)#1	132.4(2)
O(8)-K(1)-O(7)	75.3(2)	O(9)#1-K(1)-K(1)#1	44.73(17)
O(9)#1-K(1)-O(7)	103.8(2)	O(11)-K(1)-K(1)#1	114.4(3)
O(11)-K(1)-O(7)	107.9(3)	O(9)-K(1)-K(1)#1	42.70(15)
O(9)-K(1)-O(7)	138.0(2)	O(7)-K(1)-K(1)#1	133.2(2)
O(8)-K(1)-O(10)	100.4(3)	O(10)-K(1)-K(1)#1	66.6(2)
O(9)#1-K(1)-O(10)	74.4(3)	C(67)-K(1)-K(1)#1	147.0(2)
O(11)-K(1)-O(10)	177.2(3)	C(1)-O(1)-H(1)	109.5
O(9)-K(1)-O(10)	72.3(3)	O(1)-C(1)-C(2)	119.1(10)
O(7)-K(1)-O(10)	72.1(3)	O(1)-C(1)-C(6)	118.8(11)
O(8)-K(1)-C(67)	75.7(2)	C(2)-C(1)-C(6)	122.0(10)

C(1)-C(2)-C(3)	117.4(10)	C(7)-C(10)-H(10C)	109.5
C(1)-C(2)-C(88)	121.2(10)	H(10A)-C(10)-H(10C)	109.5
C(3)-C(2)-C(88)	121.3(10)	H(10B)-C(10)-H(10C)	109.5
C(4)-C(3)-C(2)	122.9(11)	C(13)-C(11)-C(6)	114.9(8)
C(4)-C(3)-H(3)	118.5	C(13)-C(11)-H(11A)	108.5
C(2)-C(3)-H(3)	118.5	C(6)-C(11)-H(11A)	108.6
C(3)-C(4)-C(5)	117.3(10)	C(13)-C(11)-H(11B)	108.5
C(3)-C(4)-C(7)	120.3(11)	C(6)-C(11)-H(11B)	108.6
C(5)-C(4)-C(7)	122.5(10)	H(11A)-C(11)-H(11B)	107.5
C(6)-C(5)-C(4)	121.8(9)	C(12)-O(2)-H(2)	109.5
C(6)-C(5)-H(5)	119.1	O(2)-C(12)-C(17)	120.4(10)
C(4)-C(5)-H(5)	119.1	O(2)-C(12)-C(13)	119.7(10)
C(5)-C(6)-C(1)	118.4(10)	C(17)-C(12)-C(13)	119.8(10)
C(5)-C(6)-C(11)	119.9(9)	C(14)-C(13)-C(12)	117.8(10)
C(1)-C(6)-C(11)	121.7(10)	C(14)-C(13)-C(11)	119.2(10)
C(8)-C(7)-C(10)	110.4(12)	C(12)-C(13)-C(11)	122.9(10)
C(8)-C(7)-C(4)	111.1(9)	C(15)-C(14)-C(13)	122.5(10)
C(10)-C(7)-C(4)	111.9(11)	C(15)-C(14)-H(14)	118.7
C(8)-C(7)-C(9)	105.8(12)	C(13)-C(14)-H(14)	118.7
C(10)-C(7)-C(9)	107.1(11)	C(14)-C(15)-C(16)	116.8(10)
C(4)-C(7)-C(9)	110.2(9)	C(14)-C(15)-C(18)	118.0(11)
C(7)-C(8)-H(8A)	109.5	C(16)-C(15)-C(18)	125.1(11)
C(7)-C(8)-H(8B)	109.5	C(15)-C(16)-C(17)	123.0(11)
H(8A)-C(8)-H(8B)	109.5	C(15)-C(16)-H(16)	118.5
C(7)-C(8)-H(8C)	109.4	C(17)-C(16)-H(16)	118.4
H(8A)-C(8)-H(8C)	109.5	C(12)-C(17)-C(16)	119.8(10)
H(8B)-C(8)-H(8C)	109.5	C(12)-C(17)-C(22)	120.8(10)
C(7)-C(9)-H(9A)	109.6	C(16)-C(17)-C(22)	119.3(10)
C(7)-C(9)-H(9B)	109.4	C(21)-C(18)-C(20)	108.8(12)
H(9A)-C(9)-H(9B)	109.5	C(21)-C(18)-C(19)	110.9(13)
C(7)-C(9)-H(9C)	109.4	C(20)-C(18)-C(19)	105.7(11)
H(9A)-C(9)-H(9C)	109.5	C(21)-C(18)-C(15)	110.0(9)
H(9B)-C(9)-H(9C)	109.5	C(20)-C(18)-C(15)	110.3(10)
C(7)-C(10)-H(10A)	109.5	C(19)-C(18)-C(15)	111.1(11)
C(7)-C(10)-H(10B)	109.5	C(18)-C(19)-H(19A)	109.5
H(10A)-C(10)-H(10B)	109.5	C(18)-C(19)-H(19B)	109.4

H(19A)-C(19)-H(19B)	109.5	C(28)-C(27)-H(27)	118.4
C(18)-C(19)-H(19C)	109.5	C(26)-C(27)-H(27)	118.4
H(19A)-C(19)-H(19C)	109.5	C(27)-C(28)-C(23)	117.8(10)
H(19B)-C(19)-H(19C)	109.5	C(27)-C(28)-C(33)	116.3(10)
C(18)-C(20)-H(20A)	109.5	C(23)-C(28)-C(33)	125.9(11)
C(18)-C(20)-H(20B)	109.5	C(32)-C(29)-C(30)	106.5(14)
H(20A)-C(20)-H(20B)	109.5	C(32)-C(29)-C(26)	108.7(9)
C(18)-C(20)-H(20C)	109.5	C(30)-C(29)-C(26)	112.6(10)
H(20A)-C(20)-H(20C)	109.5	C(32)-C(29)-C(31)	110.1(14)
H(20B)-C(20)-H(20C)	109.5	C(30)-C(29)-C(31)	106.7(12)
C(18)-C(21)-H(21A)	109.4	C(26)-C(29)-C(31)	111.9(10)
C(18)-C(21)-H(21B)	109.5	C(29)-C(30)-H(30A)	109.5
H(21A)-C(21)-H(21B)	109.5	C(29)-C(30)-H(30B)	109.5
C(18)-C(21)-H(21C)	109.5	H(30A)-C(30)-H(30B)	109.5
H(21A)-C(21)-H(21C)	109.5	C(29)-C(30)-H(30C)	109.5
H(21B)-C(21)-H(21C)	109.5	H(30A)-C(30)-H(30C)	109.5
C(17)-C(22)-C(24)	113.7(8)	H(30B)-C(30)-H(30C)	109.5
C(17)-C(22)-H(22A)	108.8	C(29)-C(31)-H(31A)	109.5
C(24)-C(22)-H(22A)	108.8	C(29)-C(31)-H(31B)	109.4
C(17)-C(22)-H(22B)	108.9	H(31A)-C(31)-H(31B)	109.5
C(24)-C(22)-H(22B)	108.8	C(29)-C(31)-H(31C)	109.5
H(22A)-C(22)-H(22B)	107.7	H(31A)-C(31)-H(31C)	109.5
C(23)-O(3)-H(3A)	109.4	H(31B)-C(31)-H(31C)	109.5
O(3)-C(23)-C(24)	118.6(10)	C(29)-C(32)-H(32A)	109.5
O(3)-C(23)-C(28)	119.0(11)	C(29)-C(32)-H(32B)	109.5
C(24)-C(23)-C(28)	122.4(11)	H(32A)-C(32)-H(32B)	109.5
C(25)-C(24)-C(23)	117.9(10)	C(29)-C(32)-H(32C)	109.5
C(25)-C(24)-C(22)	118.8(10)	H(32A)-C(32)-H(32C)	109.5
C(23)-C(24)-C(22)	123.4(11)	H(32B)-C(32)-H(32C)	109.5
C(24)-C(25)-C(26)	122.7(10)	C(28)-C(33)-C(35)	112.2(9)
C(24)-C(25)-H(25)	118.7	C(28)-C(33)-H(33A)	109.1
C(26)-C(25)-H(25)	118.6	C(35)-C(33)-H(33A)	109.2
C(27)-C(26)-C(25)	116.0(11)	C(28)-C(33)-H(33B)	109.2
C(27)-C(26)-C(29)	121.2(10)	C(35)-C(33)-H(33B)	109.1
C(25)-C(26)-C(29)	122.8(10)	H(33A)-C(33)-H(33B)	107.9
C(28)-C(27)-C(26)	123.2(10)	C(34)-O(4)-H(4)	109.5

O(4)-C(34)-C(39)	121.1(11)	C(40)-C(43)-H(43A)	109.8
O(4)-C(34)-C(35)	118.2(11)	C(40)-C(43)-H(43B)	109.3
C(39)-C(34)-C(35)	120.7(11)	H(43A)-C(43)-H(43B)	109.5
C(36)-C(35)-C(34)	118.4(11)	C(40)-C(43)-H(43C)	109.3
C(36)-C(35)-C(33)	121.0(11)	H(43A)-C(43)-H(43C)	109.5
C(34)-C(35)-C(33)	120.5(11)	H(43B)-C(43)-H(43C)	109.5
C(35)-C(36)-C(37)	123.5(11)	C(46)-C(44)-C(39)	112.9(9)
C(35)-C(36)-H(36)	118.2	C(46)-C(44)-H(44A)	109.0
C(37)-C(36)-H(36)	118.3	C(39)-C(44)-H(44A)	109.0
C(38)-C(37)-C(36)	115.3(11)	C(46)-C(44)-H(44B)	109.0
C(38)-C(37)-C(40)	122.8(12)	C(39)-C(44)-H(44B)	109.0
C(36)-C(37)-C(40)	121.8(12)	H(44A)-C(44)-H(44B)	107.8
C(39)-C(38)-C(37)	123.4(11)	C(45)-O(5)-H(5A)	109.6
C(39)-C(38)-H(38)	118.3	C(50)-C(45)-O(5)	121.4(13)
C(37)-C(38)-H(38)	118.3	C(50)-C(45)-C(46)	122.0(11)
C(38)-C(39)-C(34)	118.7(10)	O(5)-C(45)-C(46)	116.6(11)
C(38)-C(39)-C(44)	119.8(11)	C(47)-C(46)-C(45)	117.2(12)
C(34)-C(39)-C(44)	121.4(11)	C(47)-C(46)-C(44)	120.6(13)
C(41)-C(40)-C(43)	110(2)	C(45)-C(46)-C(44)	122.1(11)
C(41)-C(40)-C(42)	107.4(19)	C(46)-C(47)-C(48)	123.1(13)
C(43)-C(40)-C(42)	104.6(18)	C(46)-C(47)-H(47)	118.5
C(41)-C(40)-C(37)	115.0(13)	C(48)-C(47)-H(47)	118.4
C(43)-C(40)-C(37)	109.9(14)	C(49)-C(48)-C(47)	116.5(12)
C(42)-C(40)-C(37)	109.1(12)	C(49)-C(48)-C(51)	123.6(15)
C(40)-C(41)-H(41A)	109.7	C(47)-C(48)-C(51)	119.9(16)
C(40)-C(41)-H(41B)	109.2	C(48)-C(49)-C(50)	122.8(12)
H(41A)-C(41)-H(41B)	109.5	C(48)-C(49)-H(49)	118.6
C(40)-C(41)-H(41C)	109.5	C(50)-C(49)-H(49)	118.6
H(41A)-C(41)-H(41C)	109.5	C(45)-C(50)-C(49)	118.4(13)
H(41B)-C(41)-H(41C)	109.5	C(45)-C(50)-C(55)	121.4(11)
C(40)-C(42)-H(42A)	109.5	C(49)-C(50)-C(55)	120.2(12)
C(40)-C(42)-H(42B)	109.4	C(54)-C(51)-C(52)	105.8(18)
H(42A)-C(42)-H(42B)	109.5	C(54)-C(51)-C(53)	114.6(18)
C(40)-C(42)-H(42C)	109.5	C(52)-C(51)-C(53)	106.0(18)
H(42A)-C(42)-H(42C)	109.5	C(54)-C(51)-C(48)	109.8(16)
H(42B)-C(42)-H(42C)	109.5	C(52)-C(51)-C(48)	107.0(14)

C(53)-C(51)-C(48)	113.0(14)	C(58)-C(59)-C(62)	124.4(12)
C(51)-C(52)-H(52A)	109.3	C(60)-C(59)-C(62)	119.5(12)
C(51)-C(52)-H(52B)	109.5	C(59)-C(60)-C(61)	121.5(11)
H(52A)-C(52)-H(52B)	109.5	C(59)-C(60)-H(60)	119.2
C(51)-C(52)-H(52C)	109.5	C(61)-C(60)-H(60)	119.2
H(52A)-C(52)-H(52C)	109.5	C(56)-C(61)-C(60)	119.2(10)
H(52B)-C(52)-H(52C)	109.5	C(56)-C(61)-C(66)	121.3(10)
C(51)-C(53)-H(53A)	109.5	C(60)-C(61)-C(66)	119.3(10)
C(51)-C(53)-H(53B)	109.5	C(64)-C(62)-C(63)	117(2)
H(53A)-C(53)-H(53B)	109.5	C(64)-C(62)-C(65)	99(2)
C(51)-C(53)-H(53C)	109.4	C(63)-C(62)-C(65)	99(2)
H(53A)-C(53)-H(53C)	109.5	C(64)-C(62)-C(59)	120.0(14)
H(53B)-C(53)-H(53C)	109.5	C(63)-C(62)-C(59)	110.5(16)
C(51)-C(54)-H(54A)	109.3	C(65)-C(62)-C(59)	108.6(14)
C(51)-C(54)-H(54B)	109.6	C(62)-C(63)-H(63A)	109.7
H(54A)-C(54)-H(54B)	109.5	C(62)-C(63)-H(63B)	109.2
C(51)-C(54)-H(54C)	109.5	H(63A)-C(63)-H(63B)	109.5
H(54A)-C(54)-H(54C)	109.5	C(62)-C(63)-H(63C)	109.6
H(54B)-C(54)-H(54C)	109.5	H(63A)-C(63)-H(63C)	109.5
C(50)-C(55)-C(57)	115.2(9)	H(63B)-C(63)-H(63C)	109.5
C(50)-C(55)-H(55A)	108.5	C(62)-C(64)-H(64A)	109.5
C(57)-C(55)-H(55A)	108.4	C(62)-C(64)-H(64B)	109.6
C(50)-C(55)-H(55B)	108.5	H(64A)-C(64)-H(64B)	109.5
C(57)-C(55)-H(55B)	108.5	C(62)-C(64)-H(64C)	109.4
H(55A)-C(55)-H(55B)	107.5	H(64A)-C(64)-H(64C)	109.5
C(56)-O(6)-H(6)	109.5	H(64B)-C(64)-H(64C)	109.5
O(6)-C(56)-C(61)	119.2(10)	C(62)-C(65)-H(65A)	109.5
O(6)-C(56)-C(57)	120.1(10)	C(62)-C(65)-H(65B)	109.5
C(61)-C(56)-C(57)	120.7(10)	H(65A)-C(65)-H(65B)	109.5
C(58)-C(57)-C(56)	117.7(11)	C(62)-C(65)-H(65C)	109.4
C(58)-C(57)-C(55)	120.5(10)	H(65A)-C(65)-H(65C)	109.5
C(56)-C(57)-C(55)	121.8(10)	H(65B)-C(65)-H(65C)	109.5
C(59)-C(58)-C(57)	124.7(10)	C(68)-C(66)-C(61)	113.9(8)
C(59)-C(58)-H(58)	117.6	C(68)-C(66)-H(66A)	108.8
C(57)-C(58)-H(58)	117.7	C(61)-C(66)-H(66A)	108.8
C(58)-C(59)-C(60)	116.1(11)	C(68)-C(66)-H(66B)	108.7

C(61)-C(66)-H(66B)	108.8	C(73)-C(75)-H(75A)	109.4
H(66A)-C(66)-H(66B)	107.7	C(73)-C(75)-H(75B)	109.4
C(67)-O(7)-K(1)	102.0(5)	H(75A)-C(75)-H(75B)	109.5
O(7)-C(67)-C(72)	119.3(9)	C(73)-C(75)-H(75C)	109.5
O(7)-C(67)-C(68)	120.7(10)	H(75A)-C(75)-H(75C)	109.5
C(72)-C(67)-C(68)	120.0(10)	H(75B)-C(75)-H(75C)	109.5
O(7)-C(67)-K(1)	55.0(4)	C(73)-C(76)-H(76A)	109.5
C(72)-C(67)-K(1)	106.5(6)	C(73)-C(76)-H(76B)	109.5
C(68)-C(67)-K(1)	105.7(6)	H(76A)-C(76)-H(76B)	109.5
C(69)-C(68)-C(67)	118.8(10)	C(73)-C(76)-H(76C)	109.4
C(69)-C(68)-C(66)	119.0(9)	H(76A)-C(76)-H(76C)	109.5
C(67)-C(68)-C(66)	122.0(10)	H(76B)-C(76)-H(76C)	109.5
C(70)-C(69)-C(68)	121.9(10)	C(79)-C(77)-C(72)	115.0(8)
C(70)-C(69)-H(69)	119.1	C(79)-C(77)-H(77A)	108.5
C(68)-C(69)-H(69)	119.0	C(72)-C(77)-H(77A)	108.5
C(69)-C(70)-C(71)	117.7(10)	C(79)-C(77)-H(77B)	108.5
C(69)-C(70)-C(73)	121.3(10)	C(72)-C(77)-H(77B)	108.6
C(71)-C(70)-C(73)	121.0(11)	H(77A)-C(77)-H(77B)	107.5
C(72)-C(71)-C(70)	123.9(10)	C(78)-O(8)-K(1)	131.7(5)
C(72)-C(71)-H(71)	118.0	O(8)-C(78)-C(79)	118.1(10)
C(70)-C(71)-H(71)	118.0	O(8)-C(78)-C(83)	120.8(10)
C(71)-C(72)-C(67)	117.6(9)	C(79)-C(78)-C(83)	121.1(11)
C(71)-C(72)-C(77)	120.4(10)	C(80)-C(79)-C(78)	117.1(10)
C(67)-C(72)-C(77)	122.0(10)	C(80)-C(79)-C(77)	121.4(11)
C(74)-C(73)-C(76)	110.7(13)	C(78)-C(79)-C(77)	121.5(11)
C(74)-C(73)-C(70)	110.3(10)	C(79)-C(80)-C(81)	125.7(10)
C(76)-C(73)-C(70)	112.7(10)	C(79)-C(80)-H(80)	117.2
C(74)-C(73)-C(75)	106.7(13)	C(81)-C(80)-H(80)	117.1
C(76)-C(73)-C(75)	106.7(12)	C(82)-C(81)-C(80)	114.5(11)
C(70)-C(73)-C(75)	109.5(10)	C(82)-C(81)-C(84)	122.7(11)
C(73)-C(74)-H(74A)	109.4	C(80)-C(81)-C(84)	122.8(11)
C(73)-C(74)-H(74B)	109.5	C(83)-C(82)-C(81)	122.8(10)
H(74A)-C(74)-H(74B)	109.5	C(83)-C(82)-H(82)	118.6
C(73)-C(74)-H(74C)	109.5	C(81)-C(82)-H(82)	118.6
H(74A)-C(74)-H(74C)	109.5	C(82)-C(83)-C(78)	118.7(10)
H(74B)-C(74)-H(74C)	109.5	C(82)-C(83)-C(88)	119.6(10)

C(78)-C(83)-C(88)	121.7(11)	C(92)-O(11)-K(1)	127.2(11)
C(81)-C(84)-C(86)	111.5(10)	O(11)-C(89)-C(90)	106.6(13)
C(81)-C(84)-C(87)	112.3(11)	O(11)-C(89)-H(89A)	110.4
C(86)-C(84)-C(87)	107.1(12)	C(90)-C(89)-H(89A)	110.3
C(81)-C(84)-C(85)	108.4(10)	O(11)-C(89)-H(89B)	110.5
C(86)-C(84)-C(85)	109.2(13)	C(90)-C(89)-H(89B)	110.4
C(87)-C(84)-C(85)	108.3(12)	H(89A)-C(89)-H(89B)	108.6
C(84)-C(85)-H(85A)	109.4	C(91)-C(90)-C(89)	110.2(18)
C(84)-C(85)-H(85B)	109.4	C(91)-C(90)-H(90A)	109.6
H(85A)-C(85)-H(85B)	109.5	C(89)-C(90)-H(90A)	109.5
C(84)-C(85)-H(85C)	109.6	C(91)-C(90)-H(90B)	109.7
H(85A)-C(85)-H(85C)	109.5	C(89)-C(90)-H(90B)	109.7
H(85B)-C(85)-H(85C)	109.5	H(90A)-C(90)-H(90B)	108.1
C(84)-C(86)-H(86A)	109.4	C(90)-C(91)-C(92)	103.9(17)
C(84)-C(86)-H(86B)	109.4	C(90)-C(91)-H(91A)	110.9
H(86A)-C(86)-H(86B)	109.5	C(92)-C(91)-H(91A)	111.0
C(84)-C(86)-H(86C)	109.5	C(90)-C(91)-H(91B)	111.0
H(86A)-C(86)-H(86C)	109.5	C(92)-C(91)-H(91B)	111.0
H(86B)-C(86)-H(86C)	109.5	H(91A)-C(91)-H(91B)	109.0
C(84)-C(87)-H(87A)	109.4	O(11)-C(92)-C(91)	110.9(14)
C(84)-C(87)-H(87B)	109.5	O(11)-C(92)-H(92A)	109.5
H(87A)-C(87)-H(87B)	109.5	C(91)-C(92)-H(92A)	109.4
C(84)-C(87)-H(87C)	109.5	O(11)-C(92)-H(92B)	109.5
H(87A)-C(87)-H(87C)	109.5	C(91)-C(92)-H(92B)	109.4
H(87B)-C(87)-H(87C)	109.5	H(92A)-C(92)-H(92B)	108.0
C(2)-C(88)-C(83)	117.0(8)	O(12)#2-O(12)-C(96)	0(10)
C(2)-C(88)-H(88A)	108.0	O(12)#2-O(12)-C(93)	0(10)
C(83)-C(88)-H(88A)	108.1	C(96)-O(12)-C(93)	112.5(14)
C(2)-C(88)-H(88B)	108.0	O(12)#2-C(93)-O(12)	0.0(14)
C(83)-C(88)-H(88B)	108.0	O(12)#2-C(93)-C(94)	105.7(14)
H(88A)-C(88)-H(88B)	107.3	O(12)-C(93)-C(94)	105.7(14)
K(1)#1-O(9)-O(19)#1	97.2(3)	C(93)-C(94)-C(95)	104.3(15)
K(1)#1-O(9)-K(1)	92.6(2)	C(96)-C(95)-C(94)	105.1(15)
O(19)#1-O(9)-K(1)	107.9(3)	O(12)#2-C(96)-O(12)	0.0(16)
C(89)-O(11)-C(92)	106.2(13)	O(12)#2-C(96)-C(95)	110.3(16)
C(89)-O(11)-K(1)	126.5(10)	O(12)-C(96)-C(95)	110.3(16)

C(97)-O(13)-C(100)	109.6(17)	C(102)-C(101)-H(10G)	110.6
O(13)-C(97)-C(98)	112(2)	H(10F)-C(101)-H(10G)	108.8
O(13)-C(97)-H(97A)	109.3	C(101)-C(102)-C(103)	109(2)
C(98)-C(97)-H(97A)	109.1	C(101)-C(102)-H(10H)	109.8
O(13)-C(97)-H(97B)	109.3	C(103)-C(102)-H(10H)	109.8
C(98)-C(97)-H(97B)	109.1	C(101)-C(102)-H(10I)	109.9
H(97A)-C(97)-H(97B)	108.0	C(103)-C(102)-H(10I)	109.9
C(97)-C(98)-C(99)	105.0(19)	H(10H)-C(102)-H(10I)	108.3
C(97)-C(98)-H(98A)	110.8	C(102)-C(103)-C(104)	103(2)
C(99)-C(98)-H(98A)	110.4	C(102)-C(103)-H(10J)	111.1
C(97)-C(98)-H(98B)	111.1	C(104)-C(103)-H(10J)	111.1
C(99)-C(98)-H(98B)	110.8	C(102)-C(103)-H(10K)	111.0
H(98A)-C(98)-H(98B)	108.7	C(104)-C(103)-H(10K)	111.1
C(100)-C(99)-C(98)	104.7(19)	H(10J)-C(103)-H(10K)	109.0
C(100)-C(99)-H(99A)	110.7	O(14)-C(104)-C(103)	107(3)
C(98)-C(99)-H(99A)	110.6	O(14)-C(104)-H(10L)	110.3
C(100)-C(99)-H(99B)	110.8	C(103)-C(104)-H(10L)	110.3
C(98)-C(99)-H(99B)	111.0	O(14)-C(104)-H(10M)	110.2
H(99A)-C(99)-H(99B)	108.9	C(103)-C(104)-H(10M)	110.3
O(13)-C(100)-C(99)	107.3(16)	H(10L)-C(104)-H(10M)	108.6
O(13)-C(100)-H(10D)	110.2	O(17)-O(15)-O(10)#1	77.4(4)
C(99)-C(100)-H(10D)	110.3	O(17)-O(15)-O(16)	86.9(4)
O(13)-C(100)-H(10E)	110.4	O(10)#1-O(15)-O(16)	147.5(4)
C(99)-C(100)-H(10E)	110.2	O(13)#3-O(16)-O(15)	109.5(6)
H(10D)-C(100)-H(10E)	108.5	O(13)#3-O(16)-O(18)	162.4(6)
C(104)-O(14)-C(101)	108.3(18)	O(15)-O(16)-O(18)	59.0(3)
O(14)-C(101)-C(102)	105.7(18)	O(15)-O(17)-O(18)	96.2(5)
O(14)-C(101)-H(10F)	110.6	O(12)#2-O(18)-O(17)	109.7(6)
C(102)-C(101)-H(10F)	110.6	O(12)#2-O(18)-O(16)	163.6(6)
O(14)-C(101)-H(10G)	110.6	O(17)-O(18)-O(16)	54.9(4)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1 #2 x,y,z #3 -x,y,-z+1/2

Anisotropic displacement parameters ($Å^2 x\ 10^3$)for [K (4-tert-butylcalix[8]arene-H(THF)$_4$(H_2O)$_7$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
K(1)	73(2)	58(2)	76(2)	11(2)	0(2)	-3(2)
O(1)	32(5)	40(5)	80(5)	13(4)	6(4)	0(4)
C(1)	36(9)	34(8)	40(7)	3(6)	2(6)	6(7)
C(2)	21(9)	42(8)	43(7)	0(6)	6(6)	-2(6)
C(3)	34(8)	48(9)	53(7)	-2(6)	16(6)	-7(7)
C(4)	33(9)	28(7)	41(6)	-10(5)	3(6)	1(6)
C(5)	40(9)	28(8)	43(6)	-7(5)	17(6)	-8(6)
C(6)	37(9)	35(8)	31(6)	2(5)	11(5)	3(7)
C(7)	55(10)	36(8)	53(7)	5(6)	12(7)	6(6)
C(8)	63(13)	64(11)	178(16)	17(10)	-40(11)	13(8)
C(9)	129(16)	83(12)	110(12)	11(9)	48(11)	25(10)
C(10)	139(18)	81(13)	188(17)	70(12)	62(14)	45(11)
C(11)	35(9)	44(8)	40(7)	9(5)	11(6)	1(5)
O(2)	25(5)	44(6)	55(5)	9(4)	2(4)	0(4)
C(12)	32(9)	32(8)	44(7)	6(6)	21(7)	2(6)
C(13)	29(8)	36(8)	49(7)	-1(6)	22(6)	1(6)
C(14)	36(9)	41(9)	62(8)	13(6)	25(7)	10(6)
C(15)	35(9)	40(9)	64(8)	-10(7)	16(7)	-14(6)
C(16)	35(8)	40(9)	47(7)	6(7)	13(6)	0(7)
C(17)	23(8)	40(9)	41(7)	2(6)	13(6)	4(6)
C(18)	40(9)	51(9)	83(9)	-15(7)	26(7)	-10(7)
C(19)	79(13)	55(11)	200(17)	-39(10)	17(11)	17(9)
C(20)	88(14)	73(11)	124(13)	-38(9)	4(10)	-22(9)
C(21)	169(19)	78(12)	161(15)	-33(10)	107(15)	-76(11)
C(22)	32(8)	50(8)	45(7)	5(6)	8(6)	-7(6)
O(3)	29(6)	48(6)	83(6)	-10(4)	10(4)	-11(4)
C(23)	22(9)	38(8)	55(7)	8(6)	9(7)	-1(7)
C(24)	26(9)	42(8)	44(7)	10(6)	14(6)	0(6)
C(25)	37(10)	41(8)	45(7)	4(5)	7(6)	7(7)
C(26)	33(9)	38(8)	45(7)	16(6)	12(6)	5(6)
C(27)	53(11)	24(7)	51(7)	3(6)	19(6)	7(6)
C(28)	29(9)	33(7)	47(7)	8(6)	3(6)	-8(6)
C(29)	39(9)	43(8)	63(8)	-2(6)	23(7)	-7(6)
C(30)	91(16)	72(13)	290(30)	-56(14)	81(16)	-53(11)

C(31)	46(13)	260(20)	147(15)	-75(15)	49(11)	-79(13)
C(32)	44(13)	270(30)	140(15)	122(17)	-33(10)	-44(13)
C(33)	28(8)	52(9)	57(7)	-5(6)	-2(6)	-6(6)
O(4)	45(6)	42(5)	94(6)	-11(5)	28(5)	10(4)
C(34)	21(9)	44(9)	48(7)	-17(6)	8(6)	6(7)
C(35)	44(10)	44(9)	47(7)	-14(6)	14(7)	-5(7)
C(36)	51(10)	44(9)	55(8)	-3(6)	18(7)	-16(7)
C(37)	43(9)	60(9)	44(7)	-9(7)	14(7)	3(8)
C(38)	52(11)	56(9)	37(7)	1(6)	5(7)	25(7)
C(39)	41(10)	47(9)	43(7)	-3(6)	12(6)	6(7)
C(40)	43(12)	106(13)	95(11)	5(9)	34(8)	4(9)
C(41)	150(20)	270(30)	520(50)	250(40)	230(30)	170(20)
C(42)	100(20)	500(50)	120(17)	0(20)	59(14)	110(20)
C(43)	67(19)	280(30)	380(40)	-170(30)	100(20)	-52(19)
C(44)	65(10)	56(9)	48(7)	-12(7)	18(7)	3(7)
O(5)	39(6)	48(6)	91(6)	-9(4)	26(5)	7(4)
C(45)	54(11)	42(9)	60(8)	-3(7)	36(8)	14(8)
C(46)	56(11)	36(9)	61(8)	11(7)	29(8)	19(8)
C(47)	60(10)	60(11)	55(8)	16(7)	17(7)	-6(9)
C(48)	71(13)	45(10)	95(10)	15(8)	35(9)	8(9)
C(49)	67(12)	41(10)	74(9)	-12(6)	29(8)	-9(8)
C(50)	51(10)	31(9)	66(8)	6(7)	28(7)	2(7)
C(51)	103(16)	29(10)	185(18)	-6(11)	66(12)	33(9)
C(52)	190(20)	51(13)	270(30)	41(15)	60(20)	37(13)
C(53)	100(20)	71(14)	480(40)	-11(18)	150(20)	40(12)
C(54)	270(30)	160(20)	210(30)	-70(20)	20(20)	150(20)
C(55)	78(12)	28(7)	59(7)	-12(6)	27(8)	-2(6)
O(6)	40(5)	32(5)	69(5)	4(4)	23(4)	13(4)
C(56)	40(9)	24(8)	52(7)	5(6)	7(6)	-2(6)
C(57)	55(9)	28(8)	51(7)	4(6)	18(6)	7(7)
C(58)	75(10)	29(9)	70(8)	-13(6)	34(8)	-6(7)
C(59)	83(11)	31(9)	65(8)	7(6)	32(8)	0(7)
C(60)	64(10)	49(9)	60(8)	1(7)	30(7)	-8(7)
C(61)	40(8)	44(9)	39(7)	3(6)	12(6)	1(6)
C(62)	171(18)	20(10)	134(14)	-16(8)	79(14)	-30(10)
C(63)	200(30)	32(15)	690(70)	100(20)	90(40)	-50(16)

C(64)	490(50)	120(20)	500(50)	-100(20)	430(50)	-150(30)
C(65)	370(50)	170(30)	200(30)	-10(20)	-40(30)	-190(30)
C(66)	53(9)	34(7)	38(6)	9(5)	13(6)	-2(6)
O(7)	31(5)	30(4)	70(5)	4(3)	19(4)	-10(4)
C(67)	52(10)	20(7)	44(7)	-13(6)	23(7)	-17(6)
C(68)	49(10)	30(7)	35(6)	2(5)	14(6)	4(6)
C(69)	31(9)	48(8)	50(7)	-7(6)	16(7)	-7(7)
C(70)	45(10)	39(8)	43(7)	-5(6)	19(7)	-14(7)
C(71)	29(9)	33(7)	58(7)	2(6)	14(6)	-10(6)
C(72)	41(9)	22(7)	41(7)	-7(5)	8(6)	1(6)
C(73)	45(10)	50(9)	74(9)	10(7)	25(7)	-13(7)
C(74)	70(13)	157(16)	134(14)	-65(12)	39(10)	-63(11)
C(75)	58(13)	114(14)	169(16)	-4(11)	53(11)	-18(10)
C(76)	49(13)	250(20)	197(19)	136(18)	25(12)	-40(13)
C(77)	45(9)	42(8)	54(7)	11(6)	22(6)	0(6)
O(8)	20(6)	48(5)	67(5)	13(4)	2(4)	4(4)
C(78)	29(9)	39(9)	40(7)	13(6)	12(6)	-8(7)
C(79)	26(9)	39(8)	36(6)	17(6)	9(6)	4(7)
C(80)	60(11)	34(8)	49(7)	5(6)	22(7)	-8(7)
C(81)	39(9)	35(8)	36(6)	0(6)	8(6)	-15(7)
C(82)	42(10)	33(8)	53(7)	17(6)	25(7)	10(6)
C(83)	32(9)	34(8)	45(7)	2(6)	19(6)	-9(6)
C(84)	29(9)	44(8)	82(9)	15(6)	8(7)	6(6)
C(85)	50(14)	280(30)	86(12)	26(13)	-15(9)	7(13)
C(86)	54(13)	97(13)	191(17)	43(12)	10(11)	-18(9)
C(87)	60(13)	99(13)	164(15)	-22(11)	8(10)	34(9)
C(88)	32(8)	49(8)	47(7)	-4(6)	18(6)	-4(6)
O(9)	78(7)	63(6)	55(5)	12(4)	21(4)	-8(4)
O(10)	80(8)	89(7)	154(9)	25(6)	56(7)	-5(6)
O(11)	103(10)	122(9)	95(7)	-28(7)	35(7)	-22(7)
C(89)	143(18)	81(12)	68(10)	-17(8)	30(11)	-55(13)
C(90)	150(20)	124(18)	121(15)	5(13)	61(14)	-15(16)
C(91)	130(20)	140(20)	180(20)	-56(17)	31(16)	27(16)
C(92)	190(20)	91(13)	65(10)	-21(10)	57(13)	-46(13)
O(12)	103(12)	135(11)	151(10)	-14(8)	36(9)	-23(9)
C(93)	82(15)	136(16)	139(14)	-35(12)	78(12)	-21(11)

C(94)	101(19)	79(15)	200(20)	43(14)	-23(15)	-17(12)
C(95)	180(20)	86(15)	140(16)	-7(12)	98(17)	-3(16)
C(96)	103(16)	107(16)	118(13)	12(11)	65(12)	39(13)
O(13)	107(13)	142(12)	159(11)	-5(10)	12(9)	46(9)
C(97)	230(30)	180(20)	84(14)	-75(16)	10(15)	-130(20)
C(98)	290(40)	180(30)	150(20)	-130(20)	40(20)	-10(20)
C(99)	180(30)	140(20)	220(30)	-1(19)	90(20)	51(19)
C(100)	110(19)	128(18)	92(12)	-19(11)	-5(11)	4(13)
O(14)	133(13)	149(13)	111(9)	15(8)	39(8)	-2(10)
C(101)	130(20)	170(20)	103(13)	14(14)	54(13)	7(16)
C(102)	140(30)	140(30)	210(30)	20(20)	20(20)	-6(19)
C(103)	250(40)	200(40)	190(30)	-50(20)	110(30)	20(30)
C(104)	200(30)	190(30)	220(30)	80(20)	140(30)	70(30)
O(15)	35(4)	23(4)	11(3)	9(3)	23(3)	-4(3)
O(16)	76(10)	180(12)	196(12)	-40(10)	63(8)	-20(8)
O(17)	182(15)	230(16)	181(13)	-131(12)	-76(10)	92(12)
O(18)	83(10)	148(11)	180(11)	0(9)	-1(8)	-17(7)
O(19)	113(9)	72(7)	104(7)	-25(5)	57(6)	-21(5)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)
for [K (4-tert-butylcalix[8]arene-H$(THF)_4(H_2O)_7$].

	x	y	z	U(eq)
H(1)	589	4015	6506	80
H(3)	1460	2540	6696	53
H(5)	552	1975	7088	43
H(8A)	1812	1934	7264	182
H(8B)	1819	1302	7496	182
H(8C)	1670	1850	7723	182
H(9A)	1046	1134	6411	157
H(9B)	1444	860	6750	157
H(9C)	1446	1463	6477	157
H(10A)	772	1074	7112	201
H(10B)	1021	1328	7629	201

H(10C)	1171	771	7414	201
H(11A)	21	2607	7056	48
H(11B)	43	3280	6931	48
H(2)	-49	3782	6281	66
H(14)	-353	1923	6437	52
H(16)	-1168	2554	5239	49
H(19A)	-436	1047	6052	177
H(19B)	-519	1136	5485	177
H(19C)	-749	649	5671	177
H(20A)	-1146	1526	4954	154
H(20B)	-1479	1716	5168	154
H(20C)	-1372	1043	5152	154
H(21A)	-960	1374	6420	185
H(21B)	-1256	953	6040	185
H(21C)	-1361	1625	6068	185
H(22A)	-1136	3536	4959	52
H(22B)	-806	3971	5267	52
H(3A)	-779	4853	6245	83
H(25)	-1755	3500	5075	51
H(27)	-2046	4699	5922	51
H(30A)	-2343	2999	5400	222
H(30B)	-2361	3151	4859	222
H(30C)	-2750	3136	4997	222
H(31A)	-2519	3770	5912	221
H(31B)	-2912	3875	5476	221
H(31C)	-2631	4410	5697	221
H(32A)	-2893	4108	4645	251
H(32B)	-2490	4133	4540	251
H(32C)	-2603	4640	4845	251
H(33A)	-1097	5260	6526	59
H(33B)	-1459	5046	6676	59
H(4)	-1029	5682	5944	88
H(36)	-2053	5621	6435	60
H(38)	-2152	7083	5631	61
H(41A)	-2802	7050	5539	423
H(41B)	-2601	7381	6039	423

H(41C)	-3017	7088	5935	423
H(42A)	-2405	6161	6813	353
H(42B)	-2781	6564	6698	353
H(42C)	-2363	6854	6805	353
H(43A)	-2719	5634	6072	358
H(43B)	-2901	5964	5566	358
H(43C)	-3084	6053	5989	358
H(44A)	-1308	6882	5295	68
H(44B)	-1700	7250	5164	68
H(5A)	-447	7074	6099	87
H(47)	-1718	8156	5587	71
H(49)	-737	8847	6536	71
H(52A)	-1564	9393	5362	262
H(52B)	-1174	9654	5732	262
H(52C)	-1580	9959	5673	262
H(53A)	-2032	8892	5639	306
H(53B)	-2067	9480	5916	306
H(53C)	-1984	8876	6203	306
H(54A)	-1031	9588	6601	346
H(54B)	-1343	9322	6819	346
H(54C)	-1430	9924	6529	346
H(55A)	-193	7559	6796	65
H(55B)	-178	8239	6912	65
H(6)	211	6765	6215	68
H(58)	131	8898	6498	67
H(60)	810	8278	5756	66
H(63A)	231	9698	6259	479
H(63B)	246	9818	5727	479
H(63C)	534	10157	6179	479
H(64A)	1154	9058	5974	457
H(64B)	1116	9749	6008	457
H(64C)	844	9405	5547	457
H(65A)	792	9459	6877	420
H(65B)	1074	9834	6674	420
H(65C)	1152	9154	6779	420
H(66A)	419	6852	5566	50

H(66B)	702	7287	5418	50
H(69)	1389	7358	5908	51
H(71)	1863	6191	6969	48
H(74A)	2295	7005	7257	178
H(74B)	2139	7636	7055	178
H(74C)	2556	7424	7052	178
H(75A)	2267	6350	6134	165
H(75B)	2388	6233	6704	165
H(75C)	2634	6676	6497	165
H(76A)	1985	7324	5790	251
H(76B)	2371	7610	6149	251
H(76C)	1957	7840	6145	251
H(77A)	1355	5776	7404	55
H(77B)	964	5556	7012	55
H(80)	1967	5276	7474	56
H(82)	1956	3694	6845	48
H(85A)	2835	4335	8206	224
H(85B)	2424	4022	8094	224
H(85C)	2455	4715	8140	224
H(86A)	2998	4907	7557	181
H(86B)	2628	5312	7489	181
H(86C)	2684	4985	7033	181
H(87A)	2946	3829	7489	171
H(87B)	2628	3870	6964	171
H(87C)	2540	3502	7381	171
H(88A)	999	3772	6030	50
H(88B)	1387	3397	6171	50
H(89A)	1611	5297	6127	119
H(89B)	1595	5654	5647	119
H(90A)	2066	5115	5581	152
H(90B)	2058	4728	6034	152
H(91A)	1787	4020	5562	187
H(91B)	1830	4389	5113	187
H(92A)	1220	4642	4879	133
H(92B)	1162	4160	5245	133
H(97A)	662	2727	2061	210

H(97B)	618	2359	2504	210
H(98A)	1111	2203	2013	258
H(98B)	998	1764	2375	258
H(99A)	1530	2017	2991	206
H(99B)	1671	2384	2609	206
H(10D)	1409	2823	3296	143
H(10E)	1539	3188	2907	143
H(10F)	63	2309	5310	154
H(10G)	366	2128	5827	154
H(10H)	74	1445	5047	209
H(10I)	357	1256	5570	209
H(10J)	552	1446	4741	248
H(10K)	807	1122	5230	248
H(10L)	1101	1947	5563	224
H(10M)	986	2162	5012	224

Hydrogen bonds for [K (4-tert-butylcalix[8]arene-H(THF)$_4$(H_2O)$_7$ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(8)	0.82	1.81	2.569(10)	153.6
O(3)-H(3A)...O(4)	0.82	2.92	3.332(10)	113.8
O(5)-H(5A)...O(6)	0.82	1.68	2.476(10)	162.7
O(6)-H(6)...O(7)	0.82	1.81	2.627(9)	173.9
O(6)-H(6)...O(15)#1	0.82	2.75	2.998(8)	99.4

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y+1,-z+1 #2 x,y,z #3 -x,y,-z+1/2

V.2.13. Crystal data and structure refinement for [K_4(4-tert-butylcalix[8]arene-4H)(THF)$_6$(H_2O)$_{12}$] 13

Empirical formula	C136 H180 K4 O26
Formula weight	2387.27
Temperature	243(2) K

Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.286(2) Å	α= 64.93(3)°.
	b = 17.456(4) Å	β= 86.46(3)°.
	c = 18.762(4) Å	γ = 77.18(3)°.
Volume	2973.5(10) $Å^3$	
Z	2	
Density (calculated)	1.157 Mg/m^3	
Absorption coefficient	0.216 mm^{-1}	
F(000)	1109	
Theta range for data collection	1.32 to 21.13°.	
Index ranges	-10<=h<=10, -17<=k<=17, -17<=l<=17	
Reflections collected	11642	
Independent reflections	10419 [R(int) = 0.2374]	
Completeness to theta = 21.13°	93.3 %	
Absorption correction	Spherical	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10419 / 3 / 1244	
Goodness-of-fit on F^2	1.075	
Final R indices [I>2sigma(I)]	R1 = 0.0923, wR2 = 0.2180	
R indices (all data)	R1 = 0.1385, wR2 = 0.2577	
Absolute structure parameter	0.38(14)	
Largest diff. peak and hole	0.478 and -0.539 $e.Å^{-3}$	

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [K_4 (4-tert-butylcalix[8]arene-4H)$(THF)_6(H_2O)_{12}$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
K(1)	6892(3)	6630(3)	3732(2)	52(1)
K(2)	4035(3)	6647(2)	5309(2)	51(1)

K(3)	203(5)	6060(3)	5584(3)	78(2)
K(4)	647(4)	7225(3)	3465(3)	58(1)
O(1)	4850(9)	5962(7)	3400(7)	38(3)
C(1)	4397(15)	5947(11)	2786(11)	39(5)
C(2)	4216(14)	6684(10)	2066(11)	32(4)
C(3)	3852(15)	6686(9)	1401(10)	35(5)
C(4)	3509(15)	6005(9)	1325(10)	28(4)
C(5)	3668(13)	5258(9)	2043(11)	30(4)
C(6)	4021(15)	5264(12)	2792(12)	42(5)
C(7)	3080(16)	5974(13)	607(14)	57(6)
C(8)	2710(50)	6791(17)	-50(20)	240(30)
C(9)	1830(30)	5590(20)	706(15)	200(20)
C(10)	4270(20)	5510(40)	320(20)	330(40)
C(11)	4192(15)	4409(9)	3514(12)	40(5)
O(2)	6298(11)	4620(7)	4292(8)	48(3)
C(12)	6610(19)	3896(10)	4064(12)	52(6)
C(13)	5517(15)	3755(11)	3709(10)	46(6)
C(14)	5863(16)	3135(11)	3455(11)	38(4)
C(15)	7007(18)	2568(11)	3612(12)	47(5)
C(16)	7929(14)	2683(11)	4002(10)	45(5)
C(17)	7817(13)	3399(9)	4200(10)	32(4)
C(18)	7290(20)	1785(15)	3375(15)	73(7)
C(19)	7530(40)	2110(20)	2570(20)	190(20)
C(20)	6103(17)	1335(12)	3463(14)	74(6)
C(21)	8150(20)	961(12)	4091(17)	94(8)
C(22)	8929(19)	3501(11)	4650(12)	56(6)
O(3)	9629(10)	4206(6)	5649(7)	38(3)
C(23)	9453(14)	3347(10)	5984(12)	35(5)
C(24)	9024(17)	3011(12)	5534(11)	45(5)
C(25)	8881(13)	2186(10)	5890(11)	35(5)
C(26)	9135(14)	1634(9)	6747(10)	38(5)
C(27)	9584(17)	2053(15)	7135(13)	60(6)
C(28)	9722(15)	2900(9)	6778(11)	37(5)
C(29)	8913(18)	697(10)	7137(12)	49(5)
C(30)	7643(16)	673(14)	7590(11)	75(7)
C(31)	10157(19)	70(13)	7710(14)	80(7)

C(32)	8890(20)	348(13)	6542(16)	88(8)
C(33)	10160(20)	3265(14)	7309(12)	62(6)
O(4)	8524(11)	4962(7)	6402(7)	47(3)
C(34)	8284(14)	4538(10)	7172(11)	30(4)
C(35)	9094(15)	3743(12)	7640(12)	44(5)
C(36)	8775(16)	3281(11)	8451(11)	45(5)
C(37)	7761(17)	3673(12)	8779(11)	46(5)
C(38)	6899(13)	4457(10)	8320(8)	27(4)
C(39)	7223(18)	4859(9)	7521(11)	45(6)
C(40)	7410(20)	3145(15)	9648(11)	78(8)
C(41)	5990(30)	2950(30)	9669(16)	220(20)
C(42)	7520(20)	3786(18)	10095(13)	92(8)
C(43)	8440(30)	2340(20)	10065(17)	200(20)
C(44)	6360(16)	5774(12)	6970(11)	46(5)
O(5)	6111(9)	7325(7)	5618(7)	39(3)
C(45)	6543(12)	7291(9)	6317(9)	28(4)
C(46)	6727(13)	6534(11)	7006(10)	43(5)
C(47)	7196(15)	6544(13)	7712(11)	45(5)
C(48)	7408(15)	7352(15)	7677(13)	57(6)
C(49)	7350(16)	8031(14)	6972(12)	50(5)
C(50)	6874(12)	8083(8)	6298(10)	21(4)
C(51)	7770(20)	7373(12)	8474(12)	59(6)
C(52)	6920(20)	8076(12)	8555(16)	105(9)
C(53)	9170(20)	7570(20)	8350(20)	149(15)
C(54)	7860(30)	6480(20)	9161(16)	121(10)
C(55)	6826(14)	8891(12)	5515(11)	47(5)
O(6)	4650(11)	8722(8)	4692(8)	53(4)
C(56)	4496(13)	9341(10)	4919(9)	25(4)
C(57)	5436(14)	9427(9)	5379(10)	29(4)
C(58)	5196(15)	10175(10)	5524(10)	37(5)
C(59)	3900(14)	10773(11)	5389(10)	38(5)
C(60)	2874(17)	10567(11)	4998(11)	50(5)
C(61)	3217(16)	9944(11)	4750(11)	39(5)
C(62)	3568(15)	11500(10)	5610(12)	48(5)
C(63)	2870(50)	11220(20)	6440(20)	200(20)
C(64)	2370(30)	12140(20)	5270(30)	190(20)

C(65)	4900(30)	11777(17)	5630(20)	149(15)
C(66)	2096(12)	9766(11)	4400(10)	48(5)
O(7)	11264(10)	9085(9)	3402(8)	51(3)
C(67)	11430(14)	9871(12)	3048(11)	45(5)
C(68)	1855(11)	10271(9)	3530(10)	35(5)
C(69)	2057(18)	11140(13)	3155(13)	55(6)
C(70)	1813(17)	11591(12)	2423(13)	50(5)
C(71)	11393(13)	11267(9)	1897(11)	37(5)
C(72)	11194(13)	10434(12)	2228(11)	43(5)
C(73)	1947(18)	12522(16)	1991(14)	75(7)
C(74)	860(20)	13131(10)	1372(14)	87(9)
C(75)	3300(30)	12592(16)	1570(20)	180(18)
C(76)	2040(40)	12989(19)	2550(16)	142(14)
C(77)	10756(13)	10030(9)	1730(10)	27(4)
O(8)	12382(9)	8315(7)	2600(7)	42(3)
C(78)	12668(16)	8785(10)	1828(10)	35(4)
C(79)	11925(14)	9605(10)	1406(10)	28(4)
C(80)	12187(15)	10029(12)	617(13)	48(5)
C(81)	13223(17)	9683(10)	230(12)	47(5)
C(82)	13914(18)	8854(13)	734(16)	64(7)
C(83)	13728(12)	8348(9)	1532(10)	25(4)
C(84)	13460(18)	10090(12)	-665(14)	62(6)
C(85)	14828(17)	10251(17)	-781(14)	117(11)
C(86)	13590(40)	9466(18)	-995(19)	169(19)
C(87)	12446(19)	10911(11)	-1112(12)	60(5)
C(88)	14681(14)	7534(9)	1970(10)	28(4)
O(9)	5630(30)	9600(40)	2840(30)	330(30)
C(89)	6930(16)	9529(11)	2290(11)	49(5)
C(90)	4870(30)	10130(30)	2140(30)	171(17)
C(91)	5400(50)	10510(30)	1350(30)	220(30)
C(92)	6950(30)	10110(30)	1870(40)	250(30)
O(10)	1980(20)	5210(20)	7935(14)	190(14)
C(93)	2370(30)	4090(20)	9150(20)	180(20)
C(94)	1150(40)	4420(40)	9000(20)	220(30)
C(95)	810(30)	4950(20)	8093(15)	158(15)
C(96)	2920(20)	4780(19)	8550(15)	95(8)

O(11)	8750(20)	5858(17)	2150(14)	132(9)
C(97)	8150(40)	6108(18)	1570(30)	190(20)
C(98)	7870(20)	4711(17)	2064(18)	106(9)
C(99)	7440(30)	5402(15)	1490(20)	147(15)
C(100)	8760(60)	4930(20)	2480(30)	350(50)
O(12)	2630(30)	8100(20)	6463(14)	196(12)
C(101)	2400(40)	7431(17)	6984(12)	169(19)
C(102)	3060(20)	7171(19)	7684(14)	99(9)
C(103)	2920(40)	7980(30)	7740(20)	174(18)
C(104)	2830(60)	8570(20)	6890(20)	240(30)
O(13)	5370(30)	3701(16)	6530(19)	205(14)
C(105)	4030(20)	3430(20)	6587(13)	164(19)
C(106)	5820(40)	3080(20)	7310(40)	270(40)
C(107)	5040(50)	2690(30)	7630(20)	230(30)
C(108)	3970(30)	2910(20)	7408(13)	112(11)
O(14)	8845(17)	8029(13)	1083(12)	97(6)
C(109)	8030(40)	8720(30)	540(30)	210(20)
C(110)	8810(40)	9320(20)	-20(30)	250(30)
C(111)	10170(30)	8800(30)	110(20)	140(14)
C(112)	10086(4)	8114(3)	867(3)	137(13)
O(15)	4509(4)	7836(3)	3814(3)	62(4)
O(16)	6419(4)	5500(3)	5223(3)	42(3)
O(17)	1368(4)	7634(3)	4807(3)	62(4)
O(18)	9599(4)	5638(3)	4227(3)	44(3)
O(19)	8235(4)	7824(3)	2613(3)	71(4)
O(20)	2701(4)	5554(3)	6450(3)	89(5)
O(21)	2799(4)	6024(3)	4393(3)	54(4)
O(22)	8078(4)	7262(3)	4633(3)	55(4)
O(23)	150(4)	7168(3)	6323(3)	83(5)
O(24)	2132(4)	4466(3)	5427(3)	85(5)
O(25)	865(4)	6106(3)	2751(3)	92(6)
O(26)	8710(4)	8829(3)	3529(3)	95(6)

Bond lengths [Å] and angles [°] for [K_4 (4-tert-butylcalix[8]arene-4H)$(THF)_6(H_2O)_{12}$].

K(1)-O(16)	2.746(7)	K(1)-O(19)	2.807(6)

K(1)-O(22)	2.828(7)	K(4)-K(1)#2	4.166(5)
K(1)-O(1)	2.833(10)	O(1)-C(1)	1.28(2)
K(1)-O(15)	2.899(7)	O(1)-O(2)	2.438(17)
K(1)-O(18)	2.904(6)	C(1)-C(6)	1.33(2)
K(1)-O(2)	3.397(12)	C(1)-C(2)	1.40(2)
K(1)-K(2)	4.045(4)	C(2)-C(3)	1.32(2)
K(1)-K(4)#1	4.166(5)	C(2)-C(88)#2	1.59(2)
K(1)-K(3)#1	4.658(6)	C(3)-C(4)	1.38(2)
K(2)-O(20)	2.729(6)	C(3)-H(3)	0.9300
K(2)-O(15)	2.786(7)	C(4)-C(5)	1.41(2)
K(2)-O(16)	2.845(6)	C(4)-C(7)	1.47(3)
K(2)-O(5)	2.855(11)	C(5)-C(6)	1.48(3)
K(2)-O(21)	2.867(6)	C(5)-H(5)	0.9300
K(2)-O(17)	2.869(6)	C(6)-C(11)	1.52(3)
K(2)-K(3)	4.236(6)	C(7)-C(8)	1.43(3)
K(2)-K(4)	4.702(6)	C(7)-C(10)	1.52(4)
K(3)-O(22)#2	2.764(7)	C(7)-C(9)	1.54(3)
K(3)-O(4)#2	2.769(10)	C(8)-H(8A)	0.9600
K(3)-O(23)	2.808(8)	C(8)-H(8B)	0.9600
K(3)-O(20)	2.883(6)	C(8)-H(8C)	0.9600
K(3)-O(17)	2.993(6)	C(9)-H(9A)	0.9600
K(3)-O(18)#2	3.066(8)	C(9)-H(9B)	0.9600
K(3)-O(24)	3.153(8)	C(9)-H(9C)	0.9600
K(3)-O(3)#2	3.369(12)	C(10)-H(10A)	0.9600
K(3)-O(21)	3.382(8)	C(10)-H(10B)	0.9600
K(3)-K(4)	3.674(4)	C(10)-H(10C)	0.9600
K(3)-K(1)#2	4.658(6)	C(11)-C(13)	1.52(2)
K(4)-O(25)	2.766(7)	C(11)-H(11A)	0.9700
K(4)-O(21)	2.767(6)	C(11)-H(11B)	0.9700
K(4)-O(19)#2	2.780(6)	O(2)-C(12)	1.46(2)
K(4)-O(8)#2	2.823(12)	O(2)-O(16)	2.796(12)
K(4)-O(18)#2	2.943(6)	C(12)-C(17)	1.32(2)
K(4)-O(17)	3.064(7)	C(12)-C(13)	1.46(3)
K(4)-O(26)#2	3.098(7)	C(13)-C(14)	1.33(3)
K(4)-O(22)#2	3.334(7)	C(14)-C(15)	1.32(2)
K(4)-O(7)#2	3.397(14)	C(14)-H(14)	0.9300

C(15)-C(16)	1.33(3)	C(29)-C(31)	1.59(3)
C(15)-C(18)	1.57(3)	C(30)-H(30A)	0.9600
C(16)-C(17)	1.43(3)	C(30)-H(30B)	0.9600
C(16)-H(16)	0.9300	C(30)-H(30C)	0.9600
C(17)-C(22)	1.55(2)	C(31)-H(31A)	0.9600
C(18)-C(19)	1.41(4)	C(31)-H(31B)	0.9600
C(18)-C(20)	1.56(3)	C(31)-H(31C)	0.9600
C(18)-C(21)	1.61(3)	C(32)-H(32A)	0.9600
C(19)-H(19A)	0.9600	C(32)-H(32B)	0.9600
C(19)-H(19B)	0.9600	C(32)-H(32C)	0.9600
C(19)-H(19C)	0.9600	C(33)-C(35)	1.49(3)
C(20)-H(20A)	0.9600	C(33)-H(33A)	0.9700
C(20)-H(20B)	0.9600	C(33)-H(33B)	0.9700
C(20)-H(20C)	0.9600	O(4)-C(34)	1.35(2)
C(21)-H(21A)	0.9600	O(4)-K(3)#1	2.769(10)
C(21)-H(21B)	0.9600	O(4)-O(16)	2.896(12)
C(21)-H(21C)	0.9600	C(34)-C(39)	1.37(3)
C(22)-C(24)	1.51(3)	C(34)-C(35)	1.39(2)
C(22)-H(22A)	0.9700	C(35)-C(36)	1.45(3)
C(22)-H(22B)	0.9700	C(36)-C(37)	1.38(3)
O(3)-C(23)	1.408(19)	C(36)-H(36)	0.9300
O(3)-O(18)	2.774(11)	C(37)-C(38)	1.40(2)
O(3)-K(3)#1	3.369(12)	C(37)-C(40)	1.56(3)
O(3)-O(24)#3	20.347(15)	C(38)-C(39)	1.42(2)
C(23)-C(24)	1.35(3)	C(38)-H(38)	0.9300
C(23)-C(28)	1.37(3)	C(39)-C(44)	1.58(2)
C(24)-C(25)	1.35(2)	C(40)-C(43)	1.49(3)
C(25)-C(26)	1.49(2)	C(40)-C(41)	1.57(3)
C(25)-H(25)	0.9300	C(40)-C(42)	1.68(4)
C(26)-C(27)	1.39(3)	C(41)-H(41A)	0.9600
C(26)-C(29)	1.55(2)	C(41)-H(41B)	0.9600
C(27)-C(28)	1.38(3)	C(41)-H(41C)	0.9600
C(27)-H(27)	0.9300	C(42)-H(42A)	0.9600
C(28)-C(33)	1.52(3)	C(42)-H(42B)	0.9600
C(29)-C(32)	1.48(3)	C(42)-H(42C)	0.9600
C(29)-C(30)	1.51(3)	C(43)-H(43A)	0.9600

C(43)-H(43B)	0.9600	C(57)-C(58)	1.41(2)
C(43)-H(43C)	0.9600	C(58)-C(59)	1.46(2)
C(44)-C(46)	1.49(3)	C(58)-H(58)	0.9300
C(44)-H(44A)	0.9700	C(59)-C(62)	1.46(3)
C(44)-H(44B)	0.9700	C(59)-C(60)	1.51(2)
O(5)-C(45)	1.38(2)	C(60)-C(61)	1.33(3)
O(5)-O(22)	2.671(13)	C(60)-H(60)	0.9300
C(45)-C(46)	1.39(2)	C(61)-C(66)	1.51(2)
C(45)-C(50)	1.48(2)	C(62)-C(64)	1.44(3)
C(46)-C(47)	1.45(3)	C(62)-C(65)	1.56(3)
C(47)-C(48)	1.45(3)	C(62)-C(63)	1.60(3)
C(47)-H(47)	0.9300	C(63)-H(63A)	0.9600
C(48)-C(49)	1.34(3)	C(63)-H(63B)	0.9600
C(48)-C(51)	1.58(3)	C(63)-H(63C)	0.9600
C(49)-C(50)	1.34(2)	C(64)-H(64A)	0.9600
C(49)-H(49)	0.9300	C(64)-H(64B)	0.9600
C(50)-C(55)	1.54(2)	C(64)-H(64C)	0.9600
C(51)-C(52)	1.40(3)	C(65)-H(65A)	0.9600
C(51)-C(54)	1.53(3)	C(65)-H(65B)	0.9600
C(51)-C(53)	1.54(3)	C(65)-H(65C)	0.9600
C(52)-H(52A)	0.9600	C(66)-C(68)	1.49(2)
C(52)-H(52B)	0.9600	C(66)-H(66A)	0.9700
C(52)-H(52C)	0.9600	C(66)-H(66B)	0.9700
C(53)-H(53A)	0.9600	O(7)-C(67)	1.29(2)
C(53)-H(53B)	0.9600	O(7)-O(8)	2.490(18)
C(53)-H(53C)	0.9600	O(7)-O(26)#4	2.741(11)
C(54)-H(54A)	0.9600	O(7)-O(17)#1	2.763(14)
C(54)-H(54B)	0.9600	O(7)-K(4)#1	3.397(14)
C(54)-H(54C)	0.9600	O(7)-O(22)	4.865(12)
C(55)-C(57)	1.50(2)	C(67)-C(72)	1.43(3)
C(55)-H(55A)	0.9700	C(67)-C(68)#1	1.49(3)
C(55)-H(55B)	0.9700	C(68)-C(69)	1.43(2)
O(6)-C(56)	1.296(19)	C(68)-C(67)#2	1.49(3)
O(6)-H(6)	0.8200	C(69)-C(70)	1.27(3)
C(56)-C(57)	1.41(2)	C(69)-H(69)	0.9300
C(56)-C(61)	1.45(2)	C(70)-C(71)#2	1.45(3)

C(70)-C(73)	1.51(3)	C(85)-H(85A)	0.9600
C(71)-C(72)	1.38(2)	C(85)-H(85B)	0.9600
C(71)-C(70)#1	1.45(3)	C(85)-H(85C)	0.9600
C(71)-H(71)	0.9300	C(86)-H(86A)	0.9600
C(72)-C(77)	1.52(2)	C(86)-H(86B)	0.9600
C(73)-C(74)	1.52(3)	C(86)-H(86C)	0.9600
C(73)-C(75)	1.57(3)	C(87)-H(87A)	0.9600
C(73)-C(76)	1.60(4)	C(87)-H(87B)	0.9600
C(74)-H(74A)	0.9600	C(87)-H(87C)	0.9600
C(74)-H(74B)	0.9600	C(88)-C(2)#1	1.59(2)
C(74)-H(74C)	0.9600	C(88)-H(88A)	0.9700
C(75)-H(75A)	0.9600	C(88)-H(88B)	0.9700
C(75)-H(75B)	0.9600	O(9)-C(90)	1.40(5)
C(75)-H(75C)	0.9600	O(9)-C(89)	1.65(4)
C(76)-H(76A)	0.9600	C(89)-C(92)	0.99(5)
C(76)-H(76B)	0.9600	C(89)-H(89A)	0.9700
C(76)-H(76C)	0.9600	C(89)-H(89B)	0.9700
C(77)-C(79)	1.50(2)	C(90)-C(91)	1.47(5)
C(77)-H(77A)	0.9700	C(90)-H(90A)	0.9700
C(77)-H(77B)	0.9700	C(90)-H(90B)	0.9700
O(8)-C(78)	1.38(2)	C(91)-C(92)	1.76(5)
O(8)-K(4)#1	2.823(12)	C(91)-H(91A)	0.9700
O(8)-O(15)#1	2.977(12)	C(91)-H(91B)	0.9700
C(78)-C(79)	1.37(2)	C(92)-H(92A)	0.9700
C(78)-C(83)	1.41(2)	C(92)-H(92B)	0.9700
C(79)-C(80)	1.39(3)	O(10)-C(95)	1.35(3)
C(80)-C(81)	1.42(3)	O(10)-C(96)	1.39(3)
C(80)-H(80)	0.9300	O(10)-O(20)	2.68(3)
C(81)-C(82)	1.40(3)	O(10)-O(21)	6.22(3)
C(81)-C(84)	1.55(3)	C(93)-C(94)	1.26(5)
C(82)-C(83)	1.41(3)	C(93)-C(96)	1.45(3)
C(82)-H(82)	0.9300	C(93)-H(93A)	0.9700
C(83)-C(88)	1.47(2)	C(93)-H(93B)	0.9700
C(84)-C(86)	1.44(4)	C(94)-C(95)	1.56(4)
C(84)-C(85)	1.48(3)	C(94)-H(94A)	0.9700
C(84)-C(87)	1.51(3)	C(94)-H(94B)	0.9700

C(95)-H(95A)	0.9700	C(105)-H(10N)	0.9700
C(95)-H(95B)	0.9700	C(105)-H(10O)	0.9700
C(96)-H(96A)	0.9700	C(106)-C(107)	1.15(5)
C(96)-H(96B)	0.9700	C(106)-C(108)	1.98(4)
O(11)-C(97)	1.15(5)	C(106)-H(10P)	0.9700
O(11)-C(100)	1.47(4)	C(106)-H(10Q)	0.9700
O(11)-O(25)#1	2.707(17)	C(107)-C(108)	1.12(5)
C(97)-C(99)	1.62(3)	C(107)-H(10R)	0.9700
C(97)-C(100)	2.03(5)	C(107)-H(10S)	0.9700
C(97)-H(97A)	0.9700	C(108)-H(10T)	0.9700
C(97)-H(97B)	0.9700	C(108)-H(10U)	0.9700
C(98)-C(99)	1.23(3)	O(14)-C(112)	1.336(17)
C(98)-C(100)	1.45(5)	O(14)-C(109)	1.34(4)
C(98)-H(98A)	0.9700	O(14)-O(19)	2.79(2)
C(98)-H(98B)	0.9700	C(109)-C(110)	1.48(5)
C(99)-H(99A)	0.9700	C(109)-H(10V)	0.9700
C(99)-H(99B)	0.9700	C(109)-H(10W)	0.9700
C(100)-H(10D)	0.9700	C(110)-C(111)	1.45(4)
C(100)-H(10E)	0.9700	C(110)-H(11C)	0.9700
O(12)-C(101)	1.23(3)	C(110)-H(11D)	0.9700
O(12)-C(104)	1.41(4)	C(111)-C(112)	1.43(4)
C(101)-C(102)	1.36(3)	C(111)-H(11E)	0.9700
C(101)-H(10F)	0.9700	C(111)-H(11F)	0.9700
C(101)-H(10G)	0.9700	C(112)-H(11G)	0.9700
C(102)-C(103)	1.44(4)	C(112)-H(11H)	0.9700
C(102)-H(10H)	0.9700	O(17)-O(23)	2.8944
C(102)-H(10I)	0.9700	O(18)-K(4)#1	2.943(6)
C(103)-C(104)	1.48(5)	O(18)-K(3)#1	3.066(8)
C(103)-H(10J)	0.9700	O(18)-O(25)	9.3254
C(103)-H(10K)	0.9700	O(18)-O(24)#3	17.7940
C(104)-H(10L)	0.9700	O(19)-K(4)#1	2.780(6)
C(104)-H(10M)	0.9700	O(19)-O(26)	3.0503
O(13)-C(106)	1.43(5)	O(21)-O(24)	2.7963
O(13)-C(105)	1.54(3)	O(22)-K(3)#1	2.764(7)
C(105)-C(108)	1.43(3)	O(22)-O(26)	2.8309
C(105)-C(107)	2.02(5)	O(22)-K(4)#1	3.334(7)

O(26)-O(26)#4	0.000(9)	O(26)-K(4)#1	3.098(7)
O(16)-K(1)-O(19)	154.6(2)	O(2)-K(1)-K(4)#1	125.4(2)
O(16)-K(1)-O(22)	79.89(19)	K(2)-K(1)-K(4)#1	132.41(12)
O(19)-K(1)-O(22)	76.60(14)	O(16)-K(1)-K(3)#1	66.74(14)
O(16)-K(1)-O(1)	79.5(3)	O(19)-K(1)-K(3)#1	88.17(14)
O(19)-K(1)-O(1)	125.8(3)	O(22)-K(1)-K(3)#1	33.16(13)
O(22)-K(1)-O(1)	152.8(3)	O(1)-K(1)-K(3)#1	143.5(3)
O(16)-K(1)-O(15)	88.16(19)	O(15)-K(1)-K(3)#1	112.79(18)
O(19)-K(1)-O(15)	98.6(2)	O(18)-K(1)-K(3)#1	39.98(15)
O(22)-K(1)-O(15)	83.70(19)	O(2)-K(1)-K(3)#1	100.8(2)
O(1)-K(1)-O(15)	78.1(3)	K(2)-K(1)-K(3)#1	90.89(10)
O(16)-K(1)-O(18)	80.95(17)	K(4)#1-K(1)-K(3)#1	48.81(8)
O(19)-K(1)-O(18)	82.08(15)	O(20)-K(2)-O(15)	157.5(2)
O(22)-K(1)-O(18)	69.87(14)	O(20)-K(2)-O(16)	102.4(2)
O(1)-K(1)-O(18)	123.6(3)	O(15)-K(2)-O(16)	88.47(19)
O(15)-K(1)-O(18)	152.7(3)	O(20)-K(2)-O(5)	124.2(3)
O(16)-K(1)-O(2)	52.9(2)	O(15)-K(2)-O(5)	77.4(3)
O(19)-K(1)-O(2)	141.9(3)	O(16)-K(2)-O(5)	76.0(2)
O(22)-K(1)-O(2)	128.3(3)	O(20)-K(2)-O(21)	79.77(15)
O(1)-K(1)-O(2)	45.0(3)	O(15)-K(2)-O(21)	81.45(18)
O(15)-K(1)-O(2)	111.2(2)	O(16)-K(2)-O(21)	85.81(19)
O(18)-K(1)-O(2)	81.8(2)	O(5)-K(2)-O(21)	152.3(3)
O(16)-K(1)-K(2)	44.65(13)	O(20)-K(2)-O(17)	81.63(16)
O(19)-K(1)-K(2)	137.30(16)	O(15)-K(2)-O(17)	80.09(17)
O(22)-K(1)-K(2)	79.63(14)	O(16)-K(2)-O(17)	153.5(3)
O(1)-K(1)-K(2)	73.2(3)	O(5)-K(2)-O(17)	123.7(3)
O(15)-K(1)-K(2)	43.53(13)	O(21)-K(2)-O(17)	68.95(13)
O(18)-K(1)-K(2)	121.61(15)	O(20)-K(2)-K(1)	140.73(16)
O(2)-K(1)-K(2)	80.0(2)	O(15)-K(2)-K(1)	45.79(14)
O(16)-K(1)-K(4)#1	114.81(16)	O(16)-K(2)-K(1)	42.70(13)
O(19)-K(1)-K(4)#1	41.53(12)	O(5)-K(2)-K(1)	72.3(3)
O(22)-K(1)-K(4)#1	52.84(13)	O(21)-K(2)-K(1)	80.12(14)
O(1)-K(1)-K(4)#1	154.0(3)	O(17)-K(2)-K(1)	121.15(15)
O(15)-K(1)-K(4)#1	121.91(16)	O(20)-K(2)-K(3)	42.38(13)
O(18)-K(1)-K(4)#1	44.95(12)	O(15)-K(2)-K(3)	115.35(16)

O(16)-K(2)-K(3)	124.10(17)	O(23)-K(3)-O(3)#2	149.2(3)
O(5)-K(2)-K(3)	154.5(3)	O(20)-K(3)-O(3)#2	104.9(3)
O(21)-K(2)-K(3)	52.68(14)	O(17)-K(3)-O(3)#2	150.7(3)
O(17)-K(2)-K(3)	44.89(13)	O(18)#2-K(3)-O(3)#2	50.8(2)
K(1)-K(2)-K(3)	132.75(12)	O(24)-K(3)-O(3)#2	48.6(2)
O(20)-K(2)-K(4)	89.29(14)	O(22)#2-K(3)-O(21)	106.4(2)
O(15)-K(2)-K(4)	68.20(13)	O(4)#2-K(3)-O(21)	136.3(4)
O(16)-K(2)-K(4)	114.44(17)	O(23)-K(3)-O(21)	113.94(19)
O(5)-K(2)-K(4)	143.3(3)	O(20)-K(3)-O(21)	69.39(15)
O(21)-K(2)-K(4)	32.76(12)	O(17)-K(3)-O(21)	60.87(13)
O(17)-K(2)-K(4)	39.05(14)	O(18)#2-K(3)-O(21)	66.82(17)
K(1)-K(2)-K(4)	91.69(10)	O(24)-K(3)-O(21)	50.52(12)
K(3)-K(2)-K(4)	48.19(7)	O(3)#2-K(3)-O(21)	92.0(3)
O(22)#2-K(3)-O(4)#2	90.5(3)	O(22)#2-K(3)-K(4)	60.45(16)
O(22)#2-K(3)-O(23)	87.6(2)	O(4)#2-K(3)-K(4)	131.6(3)
O(4)#2-K(3)-O(23)	106.6(4)	O(23)-K(3)-K(4)	109.53(16)
O(22)#2-K(3)-O(20)	153.5(3)	O(20)-K(3)-K(4)	110.51(17)
O(4)#2-K(3)-O(20)	110.7(3)	O(17)-K(3)-K(4)	53.54(15)
O(23)-K(3)-O(20)	71.66(17)	O(18)#2-K(3)-K(4)	50.82(13)
O(22)#2-K(3)-O(17)	78.39(18)	O(24)-K(3)-K(4)	85.17(16)
O(4)#2-K(3)-O(17)	162.4(4)	O(3)#2-K(3)-K(4)	100.4(2)
O(23)-K(3)-O(17)	59.77(14)	O(21)-K(3)-K(4)	45.95(12)
O(20)-K(3)-O(17)	77.02(15)	O(22)#2-K(3)-K(2)	119.72(18)
O(22)#2-K(3)-O(18)#2	68.31(16)	O(4)#2-K(3)-K(2)	149.6(3)
O(4)#2-K(3)-O(18)#2	83.7(3)	O(23)-K(3)-K(2)	74.15(14)
O(23)-K(3)-O(18)#2	154.1(3)	O(20)-K(3)-K(2)	39.64(12)
O(20)-K(3)-O(18)#2	127.7(3)	O(17)-K(3)-K(2)	42.57(12)
O(17)-K(3)-O(18)#2	104.3(2)	O(18)#2-K(3)-K(2)	108.96(19)
O(22)#2-K(3)-O(24)	130.9(3)	O(24)-K(3)-K(2)	74.88(14)
O(4)#2-K(3)-O(24)	87.8(3)	O(3)#2-K(3)-K(2)	123.4(2)
O(23)-K(3)-O(24)	139.4(2)	O(21)-K(3)-K(2)	42.39(12)
O(20)-K(3)-O(24)	67.72(16)	K(4)-K(3)-K(2)	72.55(11)
O(17)-K(3)-O(24)	109.8(2)	O(22)#2-K(3)-K(1)#2	34.03(14)
O(18)#2-K(3)-O(24)	62.77(17)	O(4)#2-K(3)-K(1)#2	75.5(3)
O(22)#2-K(3)-O(3)#2	101.3(2)	O(23)-K(3)-K(1)#2	120.97(19)
O(4)#2-K(3)-O(3)#2	44.7(4)	O(20)-K(3)-K(1)#2	164.6(2)

O(17)-K(3)-K(1)#2	101.17(17)	O(21)-K(4)-O(7)#2	100.8(3)
O(18)#2-K(3)-K(1)#2	37.48(12)	O(19)#2-K(4)-O(7)#2	101.8(2)
O(24)-K(3)-K(1)#2	99.19(17)	O(8)#2-K(4)-O(7)#2	46.1(3)
O(3)#2-K(3)-K(1)#2	69.1(2)	O(18)#2-K(4)-O(7)#2	152.2(3)
O(21)-K(3)-K(1)#2	96.22(16)	O(17)-K(4)-O(7)#2	50.3(3)
K(4)-K(3)-K(1)#2	58.59(9)	O(26)#2-K(4)-O(7)#2	49.7(2)
K(2)-K(3)-K(1)#2	131.13(16)	O(22)#2-K(4)-O(7)#2	92.6(3)
O(25)-K(4)-O(21)	84.11(18)	O(25)-K(4)-K(3)	109.56(16)
O(25)-K(4)-O(19)#2	78.11(18)	O(21)-K(4)-K(3)	61.46(16)
O(21)-K(4)-O(19)#2	157.3(3)	O(19)#2-K(4)-K(3)	111.61(16)
O(25)-K(4)-O(8)#2	102.8(3)	O(8)#2-K(4)-K(3)	132.8(3)
O(21)-K(4)-O(8)#2	89.6(2)	O(18)#2-K(4)-K(3)	53.85(16)
O(19)#2-K(4)-O(8)#2	107.9(3)	O(17)-K(4)-K(3)	51.78(13)
O(25)-K(4)-O(18)#2	59.88(13)	O(26)#2-K(4)-K(3)	87.83(15)
O(21)-K(4)-O(18)#2	77.08(16)	O(22)#2-K(4)-K(3)	46.14(13)
O(19)#2-K(4)-O(18)#2	81.82(16)	O(7)#2-K(4)-K(3)	100.4(3)
O(8)#2-K(4)-O(18)#2	158.8(3)	O(25)-K(4)-K(1)#2	76.85(14)
O(25)-K(4)-O(17)	150.9(2)	O(21)-K(4)-K(1)#2	120.04(17)
O(21)-K(4)-O(17)	67.45(15)	O(19)#2-K(4)-K(1)#2	42.02(13)
O(19)#2-K(4)-O(17)	127.3(2)	O(8)#2-K(4)-K(1)#2	149.8(3)
O(8)#2-K(4)-O(17)	83.8(3)	O(18)#2-K(4)-K(1)#2	44.18(12)
O(18)#2-K(4)-O(17)	105.6(2)	O(17)-K(4)-K(1)#2	111.41(17)
O(25)-K(4)-O(26)#2	140.3(2)	O(26)#2-K(4)-K(1)#2	74.92(13)
O(21)-K(4)-O(26)#2	134.5(3)	O(22)#2-K(4)-K(1)#2	42.52(12)
O(19)#2-K(4)-O(26)#2	62.24(13)	O(7)#2-K(4)-K(1)#2	124.6(2)
O(8)#2-K(4)-O(26)#2	88.5(3)	K(3)-K(4)-K(1)#2	72.60(11)
O(18)#2-K(4)-O(26)#2	112.6(2)	O(25)-K(4)-K(2)	117.37(16)
O(17)-K(4)-O(26)#2	67.15(15)	O(21)-K(4)-K(2)	34.11(13)
O(25)-K(4)-O(22)#2	116.42(19)	O(19)#2-K(4)-K(2)	163.4(2)
O(21)-K(4)-O(22)#2	107.6(2)	O(8)#2-K(4)-K(2)	75.7(2)
O(19)#2-K(4)-O(22)#2	68.98(13)	O(18)#2-K(4)-K(2)	100.44(16)
O(8)#2-K(4)-O(22)#2	138.1(3)	O(17)-K(4)-K(2)	36.15(11)
O(18)#2-K(4)-O(22)#2	62.69(12)	O(26)#2-K(4)-K(2)	102.24(16)
O(17)-K(4)-O(22)#2	69.25(16)	O(22)#2-K(4)-K(2)	97.27(15)
O(26)#2-K(4)-O(22)#2	52.06(11)	O(7)#2-K(4)-K(2)	68.6(2)
O(25)-K(4)-O(7)#2	147.9(3)	K(3)-K(4)-K(2)	59.26(9)

K(1)#2-K(4)-K(2)	131.86(15)	H(9A)-C(9)-H(9B)	109.5
C(1)-O(1)-O(2)	115.7(11)	C(7)-C(9)-H(9C)	109.5
C(1)-O(1)-K(1)	135.8(9)	H(9A)-C(9)-H(9C)	109.5
O(2)-O(1)-K(1)	79.9(4)	H(9B)-C(9)-H(9C)	109.5
O(1)-C(1)-C(6)	123.3(17)	C(7)-C(10)-H(10A)	109.5
O(1)-C(1)-C(2)	120.6(13)	C(7)-C(10)-H(10B)	109.5
C(6)-C(1)-C(2)	116.0(16)	H(10A)-C(10)-H(10B)	109.5
C(3)-C(2)-C(1)	123.1(14)	C(7)-C(10)-H(10C)	109.4
C(3)-C(2)-C(88)#2	115.0(15)	H(10A)-C(10)-H(10C)	109.5
C(1)-C(2)-C(88)#2	121.1(14)	H(10B)-C(10)-H(10C)	109.5
C(2)-C(3)-C(4)	125.8(14)	C(13)-C(11)-C(6)	120.7(14)
C(2)-C(3)-H(3)	117.1	C(13)-C(11)-H(11A)	107.1
C(4)-C(3)-H(3)	117.1	C(6)-C(11)-H(11A)	107.1
C(3)-C(4)-C(5)	112.0(15)	C(13)-C(11)-H(11B)	107.2
C(3)-C(4)-C(7)	128.0(15)	C(6)-C(11)-H(11B)	107.2
C(5)-C(4)-C(7)	119.9(14)	H(11A)-C(11)-H(11B)	106.8
C(4)-C(5)-C(6)	122.1(14)	C(12)-O(2)-O(1)	117.7(11)
C(4)-C(5)-H(5)	119.0	C(12)-O(2)-O(16)	156.6(11)
C(6)-C(5)-H(5)	118.9	O(1)-O(2)-O(16)	85.6(5)
C(1)-C(6)-C(5)	120.0(17)	C(12)-O(2)-K(1)	140.2(10)
C(1)-C(6)-C(11)	122.6(17)	O(1)-O(2)-K(1)	55.2(3)
C(5)-C(6)-C(11)	116.3(15)	O(16)-O(2)-K(1)	51.5(2)
C(8)-C(7)-C(4)	115.7(18)	C(17)-C(12)-C(13)	123.3(17)
C(8)-C(7)-C(10)	103(3)	C(17)-C(12)-O(2)	120.1(18)
C(4)-C(7)-C(10)	108.0(16)	C(13)-C(12)-O(2)	116.5(16)
C(8)-C(7)-C(9)	103(3)	C(14)-C(13)-C(12)	114.6(16)
C(4)-C(7)-C(9)	113.8(18)	C(14)-C(13)-C(11)	125.0(15)
C(10)-C(7)-C(9)	113(3)	C(12)-C(13)-C(11)	119.2(17)
C(7)-C(8)-H(8A)	109.5	C(15)-C(14)-C(13)	125.9(16)
C(7)-C(8)-H(8B)	109.4	C(15)-C(14)-H(14)	117.0
H(8A)-C(8)-H(8B)	109.5	C(13)-C(14)-H(14)	117.0
C(7)-C(8)-H(8C)	109.5	C(14)-C(15)-C(16)	115.9(17)
H(8A)-C(8)-H(8C)	109.5	C(14)-C(15)-C(18)	123.2(16)
H(8B)-C(8)-H(8C)	109.5	C(16)-C(15)-C(18)	120.9(16)
C(7)-C(9)-H(9A)	109.4	C(15)-C(16)-C(17)	125.7(14)
C(7)-C(9)-H(9B)	109.5	C(15)-C(16)-H(16)	117.2

C(17)-C(16)-H(16)	117.2	O(18)-O(3)-K(3)#1	58.9(2)
C(12)-C(17)-C(16)	113.2(15)	C(23)-O(3)-O(24)#3	130.9(9)
C(12)-C(17)-C(22)	122.8(17)	O(18)-O(3)-O(24)#3	21.4(2)
C(16)-C(17)-C(22)	123.3(13)	K(3)#1-O(3)-O(24)#3	68.9(2)
C(19)-C(18)-C(20)	104(2)	C(24)-C(23)-C(28)	123.7(16)
C(19)-C(18)-C(15)	107.4(19)	C(24)-C(23)-O(3)	120.7(17)
C(20)-C(18)-C(15)	115.6(18)	C(28)-C(23)-O(3)	115.6(15)
C(19)-C(18)-C(21)	129(3)	C(25)-C(24)-C(23)	117.6(17)
C(20)-C(18)-C(21)	93.0(18)	C(25)-C(24)-C(22)	121.4(16)
C(15)-C(18)-C(21)	107.4(17)	C(23)-C(24)-C(22)	120.2(15)
C(18)-C(19)-H(19A)	109.6	C(24)-C(25)-C(26)	123.8(17)
C(18)-C(19)-H(19B)	109.5	C(24)-C(25)-H(25)	118.1
H(19A)-C(19)-H(19B)	109.5	C(26)-C(25)-H(25)	118.1
C(18)-C(19)-H(19C)	109.4	C(27)-C(26)-C(25)	112.5(15)
H(19A)-C(19)-H(19C)	109.5	C(27)-C(26)-C(29)	125.4(16)
H(19B)-C(19)-H(19C)	109.5	C(25)-C(26)-C(29)	122.1(16)
C(18)-C(20)-H(20A)	109.5	C(28)-C(27)-C(26)	124.1(19)
C(18)-C(20)-H(20B)	109.4	C(28)-C(27)-H(27)	118.0
H(20A)-C(20)-H(20B)	109.5	C(26)-C(27)-H(27)	117.9
C(18)-C(20)-H(20C)	109.5	C(23)-C(28)-C(27)	118.2(17)
H(20A)-C(20)-H(20C)	109.5	C(23)-C(28)-C(33)	125.3(15)
H(20B)-C(20)-H(20C)	109.5	C(27)-C(28)-C(33)	116.6(17)
C(18)-C(21)-H(21A)	109.5	C(32)-C(29)-C(30)	111.7(15)
C(18)-C(21)-H(21B)	109.4	C(32)-C(29)-C(26)	111.0(16)
H(21A)-C(21)-H(21B)	109.5	C(30)-C(29)-C(26)	109.8(16)
C(18)-C(21)-H(21C)	109.5	C(32)-C(29)-C(31)	104.1(17)
H(21A)-C(21)-H(21C)	109.5	C(30)-C(29)-C(31)	110.4(16)
H(21B)-C(21)-H(21C)	109.5	C(26)-C(29)-C(31)	109.7(13)
C(24)-C(22)-C(17)	117.8(15)	C(29)-C(30)-H(30A)	109.5
C(24)-C(22)-H(22A)	107.8	C(29)-C(30)-H(30B)	109.5
C(17)-C(22)-H(22A)	107.8	H(30A)-C(30)-H(30B)	109.5
C(24)-C(22)-H(22B)	107.9	C(29)-C(30)-H(30C)	109.4
C(17)-C(22)-H(22B)	107.9	H(30A)-C(30)-H(30C)	109.5
H(22A)-C(22)-H(22B)	107.2	H(30B)-C(30)-H(30C)	109.5
C(23)-O(3)-O(18)	143.0(11)	C(29)-C(31)-H(31A)	109.5
C(23)-O(3)-K(3)#1	158.1(11)	C(29)-C(31)-H(31B)	109.4

H(31A)-C(31)-H(31B)	109.5	C(38)-C(39)-C(44)	119.3(17)
C(29)-C(31)-H(31C)	109.5	C(43)-C(40)-C(37)	112.9(19)
H(31A)-C(31)-H(31C)	109.5	C(43)-C(40)-C(41)	112(3)
H(31B)-C(31)-H(31C)	109.5	C(37)-C(40)-C(41)	109.8(16)
C(29)-C(32)-H(32A)	109.5	C(43)-C(40)-C(42)	105(2)
C(29)-C(32)-H(32B)	109.5	C(37)-C(40)-C(42)	103.8(18)
H(32A)-C(32)-H(32B)	109.5	C(41)-C(40)-C(42)	114(2)
C(29)-C(32)-H(32C)	109.5	C(40)-C(41)-H(41A)	109.5
H(32A)-C(32)-H(32C)	109.5	C(40)-C(41)-H(41B)	109.6
H(32B)-C(32)-H(32C)	109.5	H(41A)-C(41)-H(41B)	109.5
C(35)-C(33)-C(28)	117.7(15)	C(40)-C(41)-H(41C)	109.3
C(35)-C(33)-H(33A)	107.9	H(41A)-C(41)-H(41C)	109.5
C(28)-C(33)-H(33A)	107.9	H(41B)-C(41)-H(41C)	109.5
C(35)-C(33)-H(33B)	107.9	C(40)-C(42)-H(42A)	109.4
C(28)-C(33)-H(33B)	107.9	C(40)-C(42)-H(42B)	109.5
H(33A)-C(33)-H(33B)	107.2	H(42A)-C(42)-H(42B)	109.5
C(34)-O(4)-K(3)#1	134.4(9)	C(40)-C(42)-H(42C)	109.5
C(34)-O(4)-O(16)	119.8(8)	H(42A)-C(42)-H(42C)	109.5
K(3)#1-O(4)-O(16)	101.1(4)	H(42B)-C(42)-H(42C)	109.5
O(4)-C(34)-C(39)	121.9(14)	C(40)-C(43)-H(43A)	109.5
O(4)-C(34)-C(35)	120.8(16)	C(40)-C(43)-H(43B)	109.4
C(39)-C(34)-C(35)	117.3(17)	H(43A)-C(43)-H(43B)	109.5
C(34)-C(35)-C(36)	119.7(16)	C(40)-C(43)-H(43C)	109.5
C(34)-C(35)-C(33)	122.5(19)	H(43A)-C(43)-H(43C)	109.5
C(36)-C(35)-C(33)	117.0(16)	H(43B)-C(43)-H(43C)	109.5
C(37)-C(36)-C(35)	119.7(15)	C(46)-C(44)-C(39)	115.8(14)
C(37)-C(36)-H(36)	120.2	C(46)-C(44)-H(44A)	108.3
C(35)-C(36)-H(36)	120.1	C(39)-C(44)-H(44A)	108.3
C(36)-C(37)-C(38)	121.6(17)	C(46)-C(44)-H(44B)	108.4
C(36)-C(37)-C(40)	118.3(17)	C(39)-C(44)-H(44B)	108.4
C(38)-C(37)-C(40)	119.1(15)	H(44A)-C(44)-H(44B)	107.4
C(37)-C(38)-C(39)	115.7(15)	C(45)-O(5)-O(22)	113.2(8)
C(37)-C(38)-H(38)	122.2	C(45)-O(5)-K(2)	130.3(9)
C(39)-C(38)-H(38)	122.2	O(22)-O(5)-K(2)	108.9(4)
C(34)-C(39)-C(38)	125.5(14)	O(5)-C(45)-C(46)	121.7(15)
C(34)-C(39)-C(44)	115.2(16)	O(5)-C(45)-C(50)	117.6(13)

C(46)-C(45)-C(50)	120.6(15)	H(54A)-C(54)-H(54B)	109.5
C(45)-C(46)-C(47)	118.6(16)	C(51)-C(54)-H(54C)	109.4
C(45)-C(46)-C(44)	116.8(16)	H(54A)-C(54)-H(54C)	109.5
C(47)-C(46)-C(44)	124.5(16)	H(54B)-C(54)-H(54C)	109.5
C(46)-C(47)-C(48)	118.6(17)	C(57)-C(55)-C(50)	107.8(12)
C(46)-C(47)-H(47)	120.7	C(57)-C(55)-H(55A)	110.1
C(48)-C(47)-H(47)	120.7	C(50)-C(55)-H(55A)	110.1
C(49)-C(48)-C(47)	119.0(19)	C(57)-C(55)-H(55B)	110.2
C(49)-C(48)-C(51)	123.2(18)	C(50)-C(55)-H(55B)	110.2
C(47)-C(48)-C(51)	117.7(18)	H(55A)-C(55)-H(55B)	108.5
C(50)-C(49)-C(48)	125.0(18)	C(56)-O(6)-H(6)	109.5
C(50)-C(49)-H(49)	117.5	O(6)-C(56)-C(57)	124.9(13)
C(48)-C(49)-H(49)	117.5	O(6)-C(56)-C(61)	117.7(13)
C(49)-C(50)-C(45)	117.1(15)	C(57)-C(56)-C(61)	117.1(14)
C(49)-C(50)-C(55)	121.8(15)	C(56)-C(57)-C(58)	119.1(13)
C(45)-C(50)-C(55)	120.7(15)	C(56)-C(57)-C(55)	120.7(14)
C(52)-C(51)-C(54)	117(2)	C(58)-C(57)-C(55)	117.9(15)
C(52)-C(51)-C(53)	107(2)	C(57)-C(58)-C(59)	123.7(15)
C(54)-C(51)-C(53)	108(2)	C(57)-C(58)-H(58)	118.2
C(52)-C(51)-C(48)	109.7(18)	C(59)-C(58)-H(58)	118.2
C(54)-C(51)-C(48)	110.6(18)	C(58)-C(59)-C(62)	125.5(15)
C(53)-C(51)-C(48)	104.3(18)	C(58)-C(59)-C(60)	113.3(16)
C(51)-C(52)-H(52A)	109.4	C(62)-C(59)-C(60)	121.1(14)
C(51)-C(52)-H(52B)	109.5	C(61)-C(60)-C(59)	120.3(15)
H(52A)-C(52)-H(52B)	109.5	C(61)-C(60)-H(60)	119.9
C(51)-C(52)-H(52C)	109.5	C(59)-C(60)-H(60)	119.8
H(52A)-C(52)-H(52C)	109.5	C(60)-C(61)-C(56)	124.7(15)
H(52B)-C(52)-H(52C)	109.5	C(60)-C(61)-C(66)	115.8(16)
C(51)-C(53)-H(53A)	109.5	C(56)-C(61)-C(66)	118.5(15)
C(51)-C(53)-H(53B)	109.5	C(64)-C(62)-C(59)	118(2)
H(53A)-C(53)-H(53B)	109.5	C(64)-C(62)-C(65)	120(2)
C(51)-C(53)-H(53C)	109.5	C(59)-C(62)-C(65)	106.6(13)
H(53A)-C(53)-H(53C)	109.5	C(64)-C(62)-C(63)	88(3)
H(53B)-C(53)-H(53C)	109.5	C(59)-C(62)-C(63)	110.4(18)
C(51)-C(54)-H(54A)	109.5	C(65)-C(62)-C(63)	112(3)
C(51)-C(54)-H(54B)	109.5	C(62)-C(63)-H(63A)	109.5

C(62)-C(63)-H(63B)	109.6	O(17)#1-O(7)-O(22)	47.87(18)
H(63A)-C(63)-H(63B)	109.5	K(4)#1-O(7)-O(22)	43.22(17)
C(62)-C(63)-H(63C)	109.4	O(7)-C(67)-C(72)	127.1(17)
H(63A)-C(63)-H(63C)	109.5	O(7)-C(67)-C(68)#1	118.3(15)
H(63B)-C(63)-H(63C)	109.5	C(72)-C(67)-C(68)#1	114.6(15)
C(62)-C(64)-H(64A)	109.5	C(69)-C(68)-C(67)#2	119.7(15)
C(62)-C(64)-H(64B)	109.5	C(69)-C(68)-C(66)	120.0(16)
H(64A)-C(64)-H(64B)	109.5	C(67)#2-C(68)-C(66)	120.3(14)
C(62)-C(64)-H(64C)	109.4	C(70)-C(69)-C(68)	120.9(18)
H(64A)-C(64)-H(64C)	109.5	C(70)-C(69)-H(69)	119.5
H(64B)-C(64)-H(64C)	109.5	C(68)-C(69)-H(69)	119.5
C(62)-C(65)-H(65A)	109.5	C(69)-C(70)-C(71)#2	123.9(17)
C(62)-C(65)-H(65B)	109.4	C(69)-C(70)-C(73)	124.1(18)
H(65A)-C(65)-H(65B)	109.5	C(71)#2-C(70)-C(73)	112.0(18)
C(62)-C(65)-H(65C)	109.5	C(72)-C(71)-C(70)#1	117.3(16)
H(65A)-C(65)-H(65C)	109.5	C(72)-C(71)-H(71)	121.3
H(65B)-C(65)-H(65C)	109.5	C(70)#1-C(71)-H(71)	121.4
C(68)-C(66)-C(61)	115.4(13)	C(71)-C(72)-C(67)	123.4(18)
C(68)-C(66)-H(66A)	108.4	C(71)-C(72)-C(77)	121.6(16)
C(61)-C(66)-H(66A)	108.3	C(67)-C(72)-C(77)	114.9(16)
C(68)-C(66)-H(66B)	108.4	C(70)-C(73)-C(74)	116.5(17)
C(61)-C(66)-H(66B)	108.5	C(70)-C(73)-C(75)	111.7(18)
H(66A)-C(66)-H(66B)	107.5	C(74)-C(73)-C(75)	106(2)
C(67)-O(7)-O(8)	106.4(11)	C(70)-C(73)-C(76)	114.4(19)
C(67)-O(7)-O(26)#4	118.0(10)	C(74)-C(73)-C(76)	105(2)
O(8)-O(7)-O(26)#4	104.4(5)	C(75)-C(73)-C(76)	101(2)
C(67)-O(7)-O(17)#1	147.3(12)	C(73)-C(74)-H(74A)	109.4
O(8)-O(7)-O(17)#1	96.9(5)	C(73)-C(74)-H(74B)	109.5
O(26)#4-O(7)-O(17)#1	76.5(3)	H(74A)-C(74)-H(74B)	109.5
C(67)-O(7)-K(4)#1	154.0(12)	C(73)-C(74)-H(74C)	109.5
O(8)-O(7)-K(4)#1	54.7(4)	H(74A)-C(74)-H(74C)	109.5
O(26)#4-O(7)-K(4)#1	59.5(3)	H(74B)-C(74)-H(74C)	109.5
O(17)#1-O(7)-K(4)#1	58.6(3)	C(73)-C(75)-H(75A)	109.4
C(67)-O(7)-O(22)	145.5(9)	C(73)-C(75)-H(75B)	109.6
O(8)-O(7)-O(22)	97.7(4)	H(75A)-C(75)-H(75B)	109.5
O(26)#4-O(7)-O(22)	29.70(17)	C(73)-C(75)-H(75C)	109.4

H(75A)-C(75)-H(75C)	109.5	C(78)-C(83)-C(88)	128.1(16)
H(75B)-C(75)-H(75C)	109.5	C(82)-C(83)-C(88)	120.3(14)
C(73)-C(76)-H(76A)	109.5	C(86)-C(84)-C(85)	100(2)
C(73)-C(76)-H(76B)	109.5	C(86)-C(84)-C(87)	113(2)
H(76A)-C(76)-H(76B)	109.5	C(85)-C(84)-C(87)	110.8(17)
C(73)-C(76)-H(76C)	109.5	C(86)-C(84)-C(81)	111.6(18)
H(76A)-C(76)-H(76C)	109.5	C(85)-C(84)-C(81)	108.6(18)
H(76B)-C(76)-H(76C)	109.5	C(87)-C(84)-C(81)	112.9(16)
C(79)-C(77)-C(72)	112.0(11)	C(84)-C(85)-H(85A)	109.4
C(79)-C(77)-H(77A)	109.2	C(84)-C(85)-H(85B)	109.5
C(72)-C(77)-H(77A)	109.2	H(85A)-C(85)-H(85B)	109.5
C(79)-C(77)-H(77B)	109.2	C(84)-C(85)-H(85C)	109.5
C(72)-C(77)-H(77B)	109.2	H(85A)-C(85)-H(85C)	109.5
H(77A)-C(77)-H(77B)	107.9	H(85B)-C(85)-H(85C)	109.5
C(78)-O(8)-O(7)	119.8(10)	C(84)-C(86)-H(86A)	109.4
C(78)-O(8)-K(4)#1	139.5(9)	C(84)-C(86)-H(86B)	109.5
O(7)-O(8)-K(4)#1	79.2(4)	H(86A)-C(86)-H(86B)	109.5
C(78)-O(8)-O(15)#1	116.7(8)	C(84)-C(86)-H(86C)	109.5
O(7)-O(8)-O(15)#1	78.7(4)	H(86A)-C(86)-H(86C)	109.5
K(4)#1-O(8)-O(15)#1	101.4(4)	H(86B)-C(86)-H(86C)	109.5
C(79)-C(78)-O(8)	120.1(16)	C(84)-C(87)-H(87A)	109.5
C(79)-C(78)-C(83)	125.1(16)	C(84)-C(87)-H(87B)	109.5
O(8)-C(78)-C(83)	114.8(13)	H(87A)-C(87)-H(87B)	109.5
C(78)-C(79)-C(80)	118.1(16)	C(84)-C(87)-H(87C)	109.4
C(78)-C(79)-C(77)	123.5(15)	H(87A)-C(87)-H(87C)	109.5
C(80)-C(79)-C(77)	118.1(14)	H(87B)-C(87)-H(87C)	109.5
C(79)-C(80)-C(81)	123.8(16)	C(83)-C(88)-C(2)#1	114.7(12)
C(79)-C(80)-H(80)	118.1	C(83)-C(88)-H(88A)	108.7
C(81)-C(80)-H(80)	118.1	C(2)#1-C(88)-H(88A)	108.6
C(82)-C(81)-C(80)	111.9(19)	C(83)-C(88)-H(88B)	108.5
C(82)-C(81)-C(84)	122(2)	C(2)#1-C(88)-H(88B)	108.6
C(80)-C(81)-C(84)	125.4(15)	H(88A)-C(88)-H(88B)	107.6
C(81)-C(82)-C(83)	129.9(19)	C(90)-O(9)-C(89)	88(3)
C(81)-C(82)-H(82)	115.0	C(92)-C(89)-O(9)	110(4)
C(83)-C(82)-H(82)	115.1	C(92)-C(89)-H(89A)	109.8
C(78)-C(83)-C(82)	111.2(14)	O(9)-C(89)-H(89A)	109.7

C(92)-C(89)-H(89B)	109.7	C(93)-C(94)-H(94B)	108.9
O(9)-C(89)-H(89B)	109.7	C(95)-C(94)-H(94B)	108.9
H(89A)-C(89)-H(89B)	108.2	H(94A)-C(94)-H(94B)	107.8
O(9)-C(90)-C(91)	125(3)	O(10)-C(95)-C(94)	94(2)
O(9)-C(90)-H(90A)	106.2	O(10)-C(95)-H(95A)	113.1
C(91)-C(90)-H(90A)	105.8	C(94)-C(95)-H(95A)	112.9
O(9)-C(90)-H(90B)	105.8	O(10)-C(95)-H(95B)	112.7
C(91)-C(90)-H(90B)	106.1	C(94)-C(95)-H(95B)	112.9
H(90A)-C(90)-H(90B)	106.3	H(95A)-C(95)-H(95B)	110.3
C(90)-C(91)-C(92)	84(3)	O(10)-C(96)-C(93)	106.3(19)
C(90)-C(91)-H(91A)	114.8	O(10)-C(96)-H(96A)	110.4
C(92)-C(91)-H(91A)	114.9	C(93)-C(96)-H(96A)	110.5
C(90)-C(91)-H(91B)	114.5	O(10)-C(96)-H(96B)	110.6
C(92)-C(91)-H(91B)	114.5	C(93)-C(96)-H(96B)	110.5
H(91A)-C(91)-H(91B)	111.8	H(96A)-C(96)-H(96B)	108.7
C(89)-C(92)-C(91)	107(3)	C(97)-O(11)-C(100)	101(2)
C(89)-C(92)-H(92A)	110.3	C(97)-O(11)-O(25)#1	141(2)
C(91)-C(92)-H(92A)	110.4	C(100)-O(11)-O(25)#1	110(2)
C(89)-C(92)-H(92B)	110.2	O(11)-C(97)-C(99)	114(3)
C(91)-C(92)-H(92B)	110.0	O(11)-C(97)-C(100)	45(2)
H(92A)-C(92)-H(92B)	108.5	C(99)-C(97)-C(100)	69.6(18)
C(95)-O(10)-C(96)	114(2)	O(11)-C(97)-H(97A)	108.8
C(95)-O(10)-O(20)	113(2)	C(99)-C(97)-H(97A)	108.8
C(96)-O(10)-O(20)	120.0(18)	C(100)-C(97)-H(97A)	117.9
C(95)-O(10)-O(21)	104.8(17)	O(11)-C(97)-H(97B)	108.7
C(96)-O(10)-O(21)	127.6(17)	C(99)-C(97)-H(97B)	108.7
O(20)-O(10)-O(21)	8.3(3)	C(100)-C(97)-H(97B)	132.5
C(94)-C(93)-C(96)	99(3)	H(97A)-C(97)-H(97B)	107.6
C(94)-C(93)-H(93A)	111.9	C(99)-C(98)-C(100)	104(3)
C(96)-C(93)-H(93A)	111.9	C(99)-C(98)-H(98A)	111.0
C(94)-C(93)-H(93B)	111.9	C(100)-C(98)-H(98A)	111.1
C(96)-C(93)-H(93B)	111.9	C(99)-C(98)-H(98B)	110.9
H(93A)-C(93)-H(93B)	109.6	C(100)-C(98)-H(98B)	110.9
C(93)-C(94)-C(95)	113(3)	H(98A)-C(98)-H(98B)	109.0
C(93)-C(94)-H(94A)	108.9	C(98)-C(99)-C(97)	106(3)
C(95)-C(94)-H(94A)	109.0	C(98)-C(99)-H(99A)	110.6

C(97)-C(99)-H(99A)	110.6	O(12)-C(104)-H(10M)	110.1
C(98)-C(99)-H(99B)	110.5	C(103)-C(104)-H(10M)	110.1
C(97)-C(99)-H(99B)	110.4	H(10L)-C(104)-H(10M)	108.4
H(99A)-C(99)-H(99B)	108.7	C(106)-O(13)-C(105)	94(2)
C(98)-C(100)-O(11)	114(3)	C(108)-C(105)-O(13)	102.7(19)
C(98)-C(100)-C(97)	81(2)	C(108)-C(105)-C(107)	32.5(14)
O(11)-C(100)-C(97)	33.6(14)	O(13)-C(105)-C(107)	72.6(16)
C(98)-C(100)-H(10D)	108.8	C(108)-C(105)-H(10N)	111.3
O(11)-C(100)-H(10D)	108.8	O(13)-C(105)-H(10N)	111.3
C(97)-C(100)-H(10D)	116.7	C(107)-C(105)-H(10N)	134.4
C(98)-C(100)-H(10E)	108.9	C(108)-C(105)-H(10O)	111.1
O(11)-C(100)-H(10E)	108.9	O(13)-C(105)-H(10O)	111.2
C(97)-C(100)-H(10E)	128.7	C(107)-C(105)-H(10O)	111.1
H(10D)-C(100)-H(10E)	107.7	H(10N)-C(105)-H(10O)	109.1
C(101)-O(12)-C(104)	103(3)	C(107)-C(106)-O(13)	112(3)
O(12)-C(101)-C(102)	114(3)	C(107)-C(106)-C(108)	29(3)
O(12)-C(101)-H(10F)	108.8	O(13)-C(106)-C(108)	83.8(16)
C(102)-C(101)-H(10F)	108.7	C(107)-C(106)-H(10P)	109.3
O(12)-C(101)-H(10G)	108.7	O(13)-C(106)-H(10P)	108.8
C(102)-C(101)-H(10G)	108.8	C(108)-C(106)-H(10P)	128.4
H(10F)-C(101)-H(10G)	107.7	C(107)-C(106)-H(10Q)	109.3
C(101)-C(102)-C(103)	101(3)	O(13)-C(106)-H(10Q)	109.5
C(101)-C(102)-H(10H)	111.6	C(108)-C(106)-H(10Q)	114.3
C(103)-C(102)-H(10H)	111.5	H(10P)-C(106)-H(10Q)	107.9
C(101)-C(102)-H(10I)	111.6	C(108)-C(107)-C(106)	121(4)
C(103)-C(102)-H(10I)	111.5	C(108)-C(107)-C(105)	43(2)
H(10H)-C(102)-H(10I)	109.4	C(106)-C(107)-C(105)	81(3)
C(102)-C(103)-C(104)	99(2)	C(108)-C(107)-H(10R)	106.6
C(102)-C(103)-H(10J)	111.9	C(106)-C(107)-H(10R)	107.1
C(104)-C(103)-H(10J)	111.9	C(105)-C(107)-H(10R)	106.8
C(102)-C(103)-H(10K)	111.9	C(108)-C(107)-H(10S)	107.5
C(104)-C(103)-H(10K)	111.8	C(106)-C(107)-H(10S)	107.0
H(10J)-C(103)-H(10K)	109.6	C(105)-C(107)-H(10S)	140.8
O(12)-C(104)-C(103)	108(3)	H(10R)-C(107)-H(10S)	106.8
O(12)-C(104)-H(10L)	110.0	C(107)-C(108)-C(105)	105(3)
C(103)-C(104)-H(10L)	110.1	C(107)-C(108)-C(106)	30(2)

C(105)-C(108)-C(106)	78(2)	K(1)-O(16)-O(2)	75.6(3)
C(107)-C(108)-H(10T)	110.3	K(1)-O(16)-K(2)	92.65(19)
C(105)-C(108)-H(10T)	110.7	O(2)-O(16)-K(2)	116.9(3)
C(106)-C(108)-H(10T)	109.3	K(1)-O(16)-O(4)	116.0(3)
C(107)-C(108)-H(10U)	111.3	O(2)-O(16)-O(4)	122.9(4)
C(105)-C(108)-H(10U)	111.0	K(2)-O(16)-O(4)	118.0(3)
C(106)-C(108)-H(10U)	134.1	K(2)-O(17)-O(23)	99.44(14)
H(10T)-C(108)-H(10U)	108.9	K(2)-O(17)-K(3)	92.53(19)
C(112)-O(14)-C(109)	106(2)	O(23)-O(17)-K(3)	56.94(17)
C(112)-O(14)-O(19)	116.4(9)	K(2)-O(17)-K(4)	104.8(2)
C(109)-O(14)-O(19)	112(3)	O(23)-O(17)-K(4)	126.34(12)
O(14)-C(109)-C(110)	110(3)	K(3)-O(17)-K(4)	74.67(17)
O(14)-C(109)-H(10V)	109.6	O(3)-O(18)-K(1)	110.8(3)
C(110)-C(109)-H(10V)	109.7	O(3)-O(18)-K(4)#1	142.4(3)
O(14)-C(109)-H(10W)	109.7	K(1)-O(18)-K(4)#1	90.87(17)
C(110)-C(109)-H(10W)	109.6	O(3)-O(18)-K(3)#1	70.3(3)
H(10V)-C(109)-H(10W)	108.1	K(1)-O(18)-K(3)#1	102.5(2)
C(111)-C(110)-C(109)	104(3)	K(4)#1-O(18)-K(3)#1	75.34(16)
C(111)-C(110)-H(11C)	110.9	O(3)-O(18)-O(25)	95.2(2)
C(109)-C(110)-H(11C)	110.9	K(1)-O(18)-O(25)	28.98(12)
C(111)-C(110)-H(11D)	110.8	K(4)#1-O(18)-O(25)	115.76(12)
C(109)-C(110)-H(11D)	110.9	K(3)#1-O(18)-O(25)	121.39(13)
H(11C)-C(110)-H(11D)	108.9	O(3)-O(18)-O(24)#3	155.3(2)
C(112)-C(111)-C(110)	101(2)	K(1)-O(18)-O(24)#3	45.77(13)
C(112)-C(111)-H(11E)	111.6	K(4)#1-O(18)-O(24)#3	57.86(13)
C(110)-C(111)-H(11E)	111.5	K(3)#1-O(18)-O(24)#3	117.22(13)
C(112)-C(111)-H(11F)	111.6	O(25)-O(18)-O(24)#3	60.4
C(110)-C(111)-H(11F)	111.6	K(4)#1-O(19)-O(14)	106.6(4)
H(11E)-C(111)-H(11F)	109.4	K(4)#1-O(19)-K(1)	96.45(19)
O(14)-C(112)-C(111)	114.7(15)	O(14)-O(19)-K(1)	128.4(4)
O(14)-C(112)-H(11G)	108.7	K(4)#1-O(19)-O(26)	64.01(15)
C(111)-C(112)-H(11G)	108.6	O(14)-O(19)-O(26)	131.3(3)
O(14)-C(112)-H(11H)	108.6	K(1)-O(19)-O(26)	100.28(14)
C(111)-C(112)-H(11H)	108.6	O(10)-O(20)-K(2)	138.6(7)
H(11G)-C(112)-H(11H)	107.6	O(10)-O(20)-K(3)	103.9(5)
K(2)-O(15)-K(1)	90.68(18)	K(2)-O(20)-K(3)	98.0(2)

K(4)-O(21)-O(24)	112.87(13)	K(3)#1-O(22)-K(4)#1	73.41(17)
K(4)-O(21)-K(2)	113.1(2)	K(1)-O(22)-K(4)#1	84.64(18)
O(24)-O(21)-K(2)	107.71(13)	O(26)-O(22)-K(4)#1	59.67(11)
K(4)-O(21)-K(3)	72.59(16)	O(5)-O(22)-O(7)	133.0(3)
O(24)-O(21)-K(3)	60.50(12)	K(3)#1-O(22)-O(7)	86.2(2)
K(2)-O(21)-K(3)	84.93(18)	K(1)-O(22)-O(7)	118.9(2)
K(4)-O(21)-O(10)	111.2(3)	O(26)-O(22)-O(7)	28.67(16)
O(24)-O(21)-O(10)	59.5(3)	K(4)#1-O(22)-O(7)	44.23(19)
K(2)-O(21)-O(10)	53.3(3)	K(3)-O(23)-O(17)	63.30(14)
K(3)-O(21)-O(10)	42.7(2)	O(21)-O(24)-K(3)	68.98(12)
O(5)-O(22)-K(3)#1	104.8(3)	K(4)-O(25)-O(18)	75.22(12)
O(5)-O(22)-K(1)	99.0(3)	O(26)#4-O(26)-O(22)	0(10)
K(3)#1-O(22)-K(1)	112.8(2)	O(26)#4-O(26)-O(19)	0(10)
O(5)-O(22)-O(26)	119.1(2)	O(22)-O(26)-O(19)	72.7
K(3)#1-O(22)-O(26)	114.86(13)	O(26)#4-O(26)-K(4)#1	0(10)
K(1)-O(22)-O(26)	105.30(13)	O(22)-O(26)-K(4)#1	68.27(12)
O(5)-O(22)-K(4)#1	176.4(3)	O(19)-O(26)-K(4)#1	53.74(12)

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 x-1,y,z #3 x,y+1,z-1 #4 x,y,z

Anisotropic displacement parameters ($Å^2$x 10^3)for [K_4 (4-tert-butylcalix[8]arene)-4H$(THF)_6(H_2O)_{12}$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
K(1)	45(2)	64(3)	49(3)	-17(2)	7(2)	-32(2)
K(2)	39(2)	57(3)	55(3)	-19(2)	-11(2)	-16(2)
K(3)	87(3)	85(3)	57(4)	-4(3)	-12(3)	-59(3)
K(4)	55(2)	59(3)	60(3)	-17(2)	-1(2)	-27(2)
O(1)	42(6)	48(7)	23(8)	0(6)	3(5)	-42(5)
C(1)	53(10)	40(10)	36(13)	-13(9)	-6(8)	-39(8)
C(2)	44(9)	28(9)	45(14)	-41(10)	2(8)	2(7)
C(3)	49(9)	15(8)	15(11)	18(7)	2(7)	-8(7)
C(4)	56(9)	12(8)	16(11)	-4(7)	-7(7)	-10(6)
C(5)	26(8)	17(8)	47(13)	-17(8)	-3(7)	3(6)

C(6)	36(9)	58(12)	42(13)	-37(10)	3(8)	1(8)
C(7)	37(9)	68(13)	77(18)	-39(12)	-1(9)	-17(8)
C(8)	560(90)	57(16)	110(30)	-21(17)	-170(40)	-30(30)
C(9)	310(40)	300(40)	54(18)	-30(20)	-30(20)	-280(40)
C(10)	76(16)	890(120)	200(40)	-430(70)	13(18)	-30(30)
C(11)	45(10)	9(7)	64(14)	-4(8)	-9(8)	-23(7)
O(2)	67(7)	53(7)	46(9)	-36(7)	-13(6)	-24(6)
C(12)	76(13)	17(9)	53(14)	-3(8)	33(9)	-21(9)
C(13)	43(9)	47(11)	28(11)	22(9)	-19(8)	-46(8)
C(14)	39(10)	54(11)	52(13)	-47(10)	15(8)	-22(8)
C(15)	55(12)	33(10)	57(15)	-14(9)	-6(10)	-27(9)
C(16)	25(9)	63(12)	41(12)	-33(9)	23(8)	20(8)
C(17)	13(7)	18(7)	36(10)	5(7)	5(6)	20(6)
C(18)	73(14)	89(16)	74(19)	-46(14)	-16(12)	-22(12)
C(19)	380(50)	180(30)	190(40)	-180(30)	240(40)	-240(30)
C(20)	46(10)	60(11)	105(17)	-25(11)	12(9)	-17(8)
C(21)	101(16)	44(10)	150(20)	-48(12)	-36(14)	-7(10)
C(22)	95(13)	39(10)	57(15)	-23(10)	-27(10)	-43(9)
O(3)	39(6)	22(6)	32(8)	11(5)	3(5)	-10(5)
C(23)	29(8)	21(9)	53(15)	-21(9)	15(8)	4(7)
C(24)	74(12)	58(12)	25(13)	-31(10)	21(9)	-36(9)
C(25)	19(8)	37(10)	43(13)	-7(9)	-8(7)	-10(7)
C(26)	28(8)	14(8)	33(11)	21(7)	-27(7)	17(6)
C(27)	47(11)	107(18)	33(14)	-28(13)	1(9)	-34(10)
C(28)	43(9)	10(8)	36(13)	15(8)	-17(8)	-8(7)
C(29)	89(13)	16(8)	42(13)	4(8)	-18(10)	-41(8)
C(30)	47(10)	106(16)	46(12)	4(11)	-9(8)	-40(10)
C(31)	70(12)	69(13)	78(18)	-21(12)	4(11)	10(10)
C(32)	126(17)	32(10)	110(20)	-15(12)	33(14)	-57(11)
C(33)	65(13)	89(15)	29(14)	-8(11)	5(10)	-46(11)
O(4)	74(8)	53(7)	13(8)	9(6)	6(5)	-58(6)
C(34)	22(8)	36(9)	37(13)	-17(9)	-3(7)	-11(7)
C(35)	27(8)	60(12)	57(15)	-28(11)	-7(8)	-24(8)
C(36)	46(10)	34(10)	29(13)	5(9)	-10(8)	6(8)
C(37)	47(10)	66(12)	24(12)	-14(10)	20(8)	-24(9)
C(38)	12(7)	47(10)	4(9)	10(7)	3(6)	-14(6)

C(39)	75(13)	12(8)	39(14)	9(8)	-32(10)	-22(8)
C(40)	64(12)	102(16)	0(10)	29(10)	-11(8)	10(11)
C(41)	170(30)	430(60)	46(17)	0(20)	54(16)	-240(30)
C(42)	93(14)	150(20)	18(14)	-20(14)	0(10)	-18(14)
C(43)	110(20)	200(30)	70(20)	100(20)	12(15)	34(19)
C(44)	33(8)	79(13)	30(12)	-25(10)	14(7)	-16(8)
O(5)	38(6)	41(7)	36(9)	-18(6)	-11(5)	-1(5)
C(45)	13(7)	34(9)	19(11)	-6(8)	0(6)	17(6)
C(46)	15(7)	63(12)	29(12)	7(9)	-2(7)	-22(7)
C(47)	27(8)	83(13)	34(12)	-26(10)	-4(7)	-27(8)
C(48)	26(9)	106(17)	57(17)	-43(14)	1(8)	-34(9)
C(49)	40(10)	100(15)	21(12)	-22(11)	10(8)	-45(9)
C(50)	16(7)	18(7)	30(12)	-6(7)	13(6)	-17(5)
C(51)	117(17)	36(10)	29(14)	-13(9)	-6(10)	-23(10)
C(52)	160(20)	59(11)	130(20)	-67(13)	-10(16)	-21(13)
C(53)	63(14)	290(40)	150(30)	-160(30)	-7(14)	-16(17)
C(54)	200(30)	160(30)	42(18)	-43(17)	3(15)	-110(20)
C(55)	20(8)	86(13)	34(12)	-26(10)	8(7)	-9(8)
O(6)	38(6)	46(8)	56(10)	-4(7)	-10(6)	-8(5)
C(56)	30(8)	41(10)	4(9)	-8(7)	-7(6)	-11(7)
C(57)	38(9)	20(8)	34(11)	-20(7)	17(7)	-1(6)
C(58)	31(8)	34(9)	27(11)	14(8)	-3(7)	-21(7)
C(59)	19(8)	63(11)	22(11)	-18(9)	2(7)	13(7)
C(60)	49(10)	25(9)	55(13)	13(9)	-24(8)	-22(7)
C(61)	53(11)	33(10)	31(11)	-2(8)	-3(7)	-34(8)
C(62)	36(9)	32(9)	75(16)	-27(10)	33(9)	-7(8)
C(63)	330(50)	160(30)	110(30)	-70(20)	160(30)	-90(30)
C(64)	110(20)	170(30)	340(60)	-180(40)	-80(30)	80(20)
C(65)	140(20)	110(19)	310(40)	-190(30)	40(20)	-47(16)
C(66)	0(6)	67(11)	45(12)	1(9)	-3(6)	3(6)
O(7)	35(6)	76(9)	46(9)	-20(7)	4(5)	-33(6)
C(67)	24(8)	66(12)	29(12)	2(10)	-7(7)	-26(8)
C(68)	0(6)	34(9)	45(12)	10(8)	-25(6)	-3(6)
C(69)	70(13)	70(13)	35(16)	-14(11)	-14(10)	-42(10)
C(70)	70(11)	62(12)	44(15)	-26(11)	14(9)	-59(9)
C(71)	23(8)	13(8)	51(13)	11(8)	-11(7)	-7(6)

C(72)	6(7)	71(13)	47(14)	-25(11)	0(7)	1(7)
C(73)	40(10)	105(18)	60(17)	-19(13)	3(9)	-10(10)
C(74)	118(16)	4(8)	108(19)	31(9)	-52(13)	-46(9)
C(75)	180(30)	58(15)	260(40)	0(20)	110(30)	-94(18)
C(76)	270(40)	110(20)	60(20)	-26(15)	-80(20)	-80(20)
C(77)	20(7)	22(8)	39(12)	-11(7)	-12(7)	-4(6)
O(8)	24(5)	45(7)	55(10)	-21(7)	-7(5)	-2(4)
C(78)	49(10)	28(9)	19(11)	6(8)	-2(8)	-26(8)
C(79)	33(8)	35(9)	23(11)	-9(8)	9(7)	-33(7)
C(80)	18(8)	58(11)	69(17)	-25(11)	-3(8)	-16(8)
C(81)	43(10)	25(9)	55(15)	8(9)	-20(9)	-15(8)
C(82)	46(11)	53(13)	110(20)	-51(14)	12(11)	-14(9)
C(83)	8(7)	27(8)	38(12)	-11(8)	5(6)	-8(6)
C(84)	55(11)	49(11)	86(19)	-29(12)	28(10)	-24(9)
C(85)	35(10)	150(20)	75(17)	30(14)	-17(9)	-6(11)
C(86)	280(40)	84(19)	80(30)	-14(17)	60(20)	40(20)
C(87)	95(14)	54(11)	45(14)	-29(10)	9(10)	-27(10)
C(88)	40(8)	18(8)	25(10)	-2(7)	-5(6)	-19(6)
O(9)	140(20)	630(80)	260(40)	-190(50)	40(20)	-200(40)
C(89)	28(8)	42(9)	47(11)	18(8)	-18(7)	-20(7)
C(90)	53(15)	260(50)	170(40)	-60(30)	50(20)	-60(20)
C(91)	250(50)	220(40)	130(40)	-90(30)	-100(30)	120(40)
C(92)	43(16)	190(40)	570(110)	-210(60)	20(30)	-40(20)
O(10)	93(14)	270(30)	93(17)	30(17)	10(12)	-38(16)
C(93)	97(19)	160(30)	170(30)	70(20)	-38(18)	-110(20)
C(94)	170(40)	340(60)	90(30)	0(30)	60(30)	-150(40)
C(95)	200(30)	230(30)	70(19)	-20(20)	-3(18)	-180(30)
C(96)	49(11)	170(20)	73(17)	-37(16)	-26(11)	-48(13)
O(11)	158(15)	190(20)	117(18)	-86(16)	18(12)	-137(15)
C(97)	170(30)	55(17)	370(70)	-70(30)	-30(40)	-73(19)
C(98)	112(16)	86(18)	110(20)	-26(17)	-11(15)	-38(13)
C(99)	170(20)	56(14)	180(30)	20(17)	-90(20)	-80(15)
C(100)	470(80)	80(20)	410(90)	60(30)	-270(70)	-160(40)
O(12)	300(30)	260(40)	65(17)	-70(20)	20(16)	-130(30)
C(101)	350(40)	120(20)	9(12)	77(13)	-90(19)	-180(30)
C(102)	87(14)	160(20)	48(15)	-53(15)	-11(10)	13(14)

C(103)	280(40)	250(40)	110(30)	-120(30)	90(30)	-210(40)
C(104)	580(80)	140(30)	130(40)	-100(30)	180(40)	-250(40)
O(13)	210(30)	135(16)	180(30)	31(16)	70(20)	-72(18)
C(105)	61(13)	280(40)	45(14)	69(19)	-34(10)	-110(20)
C(106)	170(30)	100(20)	520(90)	-100(40)	-280(50)	30(20)
C(107)	280(60)	280(50)	100(30)	30(30)	10(30)	-210(50)
C(108)	86(16)	170(30)	44(15)	14(14)	-17(11)	-75(17)
O(14)	89(12)	130(14)	96(15)	-47(12)	-3(10)	-70(11)
C(109)	120(30)	260(50)	140(40)	20(40)	10(20)	-30(30)
C(110)	190(40)	160(30)	210(40)	30(30)	150(40)	60(30)
C(111)	110(20)	210(30)	120(30)	-60(20)	2(17)	-110(20)
C(112)	68(16)	210(30)	110(30)	-70(20)	-14(15)	28(18)
O(15)	46(7)	74(9)	51(10)	-10(7)	-8(6)	-16(6)
O(16)	58(7)	36(6)	42(9)	-24(6)	-6(6)	-15(5)
O(17)	83(9)	45(7)	70(11)	-25(7)	7(7)	-38(6)
O(18)	21(5)	67(8)	44(9)	-22(6)	-4(5)	-8(5)
O(19)	87(9)	80(9)	47(10)	-14(7)	32(7)	-52(7)
O(20)	52(8)	104(11)	84(12)	-18(9)	-13(7)	-5(7)
O(21)	47(7)	45(7)	69(11)	-22(7)	4(6)	-12(6)
O(22)	55(7)	82(9)	49(10)	-42(8)	4(6)	-26(6)
O(23)	50(7)	140(14)	88(13)	-77(11)	27(7)	-20(8)
O(24)	61(8)	79(10)	95(12)	-5(9)	-31(8)	-34(7)
O(25)	63(8)	137(15)	63(12)	-20(10)	-1(7)	-41(9)
O(26)	60(8)	83(11)	107(13)	3(9)	22(7)	-44(7)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3)
for $[K_4$ (4-tert-butylcalix[8]arene)-4H$(THF)_6(H_2O)_{12}]$.

	x	y	z	U(eq)
H(3)	3826	7191	944	42
H(5)	3547	4744	2046	35
H(8A)	2444	6704	-491	366
H(8B)	3462	7064	-195	366
H(8C)	1986	7154	77	366

H(9A)	1983	5028	1143	299
H(9B)	1664	5531	235	299
H(9C)	1078	5964	800	299
H(10A)	4558	4924	708	497
H(10B)	4991	5805	224	497
H(10C)	4025	5499	-164	497
H(11A)	3980	4542	3966	48
H(11B)	3519	4119	3470	48
H(14)	5240	3100	3138	46
H(16)	8715	2261	4160	55
H(19A)	7700	1650	2402	291
H(19B)	8299	2370	2469	291
H(19C)	6771	2544	2277	291
H(20A)	6377	859	3319	110
H(20B)	5367	1744	3125	110
H(20C)	5835	1126	4000	110
H(21A)	8330	469	3966	141
H(21B)	7651	834	4563	141
H(21C)	8973	1089	4170	141
H(22A)	9778	3327	4446	67
H(22B)	8811	4112	4524	67
H(25)	8609	1946	5582	42
H(27)	9807	1743	7669	72
H(30A)	7511	91	7832	112
H(30B)	6900	1043	7236	112
H(30C)	7711	871	7989	112
H(31A)	10042	-516	7924	120
H(31B)	10218	237	8131	120
H(31C)	10960	112	7420	120
H(32A)	8810	-244	6801	133
H(32B)	9706	380	6261	133
H(32C)	8147	684	6178	133
H(33A)	10691	2788	7746	74
H(33B)	10738	3653	7013	74
H(36)	9252	2723	8750	54
H(38)	6161	4698	8527	33

H(41A)	5347	3485	9429	332
H(41B)	5771	2642	10206	332
H(41C)	5989	2603	9386	332
H(42A)	6849	4310	9873	138
H(42B)	8387	3924	10019	138
H(42C)	7391	3489	10648	138
H(43A)	9277	2486	10082	293
H(43B)	8525	1976	9791	293
H(43C)	8168	2038	10593	293
H(44A)	6428	5837	6431	56
H(44B)	5433	5784	7107	56
H(47)	7358	6042	8179	54
H(49)	7665	8501	6948	60
H(52A)	6943	8599	8094	158
H(52B)	7199	8126	9009	158
H(52C)	6021	7985	8619	158
H(53A)	9801	7086	8338	224
H(53B)	9407	7684	8782	224
H(53C)	9168	8074	7867	224
H(54A)	8474	6054	9044	181
H(54B)	6995	6343	9242	181
H(54C)	8166	6492	9630	181
H(55A)	7451	9221	5543	56
H(55B)	7067	8721	5087	56
H(6)	5154	8290	5005	79
H(58)	5902	10293	5717	44
H(60)	1996	10877	4931	59
H(63A)	2690	11696	6591	296
H(63B)	3458	10734	6835	296
H(63C)	2052	11062	6405	296
H(64A)	2264	12577	5466	292
H(64B)	1618	11876	5399	292
H(64C)	2435	12410	4704	292
H(65A)	4737	12238	5798	224
H(65B)	5260	11971	5115	224
H(65C)	5530	11291	5994	224

H(66A)	2289	9153	4518	57
H(66B)	1281	9887	4657	57
H(69)	2370	11377	3454	67
H(71)	11265	11609	1359	44
H(74A)	1079	13691	1110	131
H(74B)	23	13184	1620	131
H(74C)	798	12903	994	131
H(75A)	3359	13189	1303	270
H(75B)	3354	12352	1187	270
H(75C)	4029	12277	1947	270
H(76A)	2201	13557	2239	212
H(76B)	2762	12655	2932	212
H(76C)	1220	13036	2815	212
H(77A)	10252	9602	2050	32
H(77B)	10174	10476	1298	32
H(80)	11654	10571	324	57
H(82)	14601	8599	506	77
H(85A)	14968	10540	-1334	175
H(85B)	15465	9710	-557	175
H(85C)	14939	10609	-526	175
H(86A)	13766	9732	-1544	254
H(86B)	12769	9268	-934	254
H(86C)	14304	8983	-726	254
H(87A)	12687	11165	-1652	91
H(87B)	12424	11311	-882	91
H(87C)	11582	10780	-1086	91
H(88A)	14843	7494	2490	34
H(88B)	15521	7551	1705	34
H(89A)	7745	9306	2614	59
H(89B)	6876	9130	2062	59
H(90A)	4320	10607	2221	205
H(90B)	4259	9790	2099	205
H(91A)	5145	11133	1079	262
H(91B)	5285	10235	1005	262
H(92A)	7666	10062	1515	295
H(92B)	7091	10494	2087	295

H(93A)	2630	3554	9081	215
H(93B)	2632	3982	9676	215
H(94A)	864	4797	9261	262
H(94B)	654	3963	9202	262
H(95A)	686	4599	7832	190
H(95B)	41	5434	7981	190
H(96A)	3094	5171	8760	114
H(96B)	3755	4543	8370	114
H(97A)	8751	6288	1136	233
H(97B)	7474	6614	1515	233
H(98A)	8337	4273	1895	127
H(98B)	7143	4502	2392	127
H(99A)	6481	5575	1502	177
H(99B)	7672	5346	1005	177
H(10D)	9662	4613	2468	418
H(10E)	8518	4735	3026	418
H(10F)	2638	6972	6807	203
H(10G)	1446	7517	7068	203
H(10H)	3987	6904	7672	119
H(10I)	2641	6770	8117	119
H(10J)	3688	7998	7999	209
H(10K)	2115	8112	8001	209
H(10L)	2090	9063	6779	291
H(10M)	3644	8782	6733	291
H(10N)	3296	3934	6405	196
H(10O)	4022	3102	6282	196
H(10P)	6637	2685	7285	321
H(10Q)	6031	3376	7611	321
H(10R)	5353	2110	7654	278
H(10S)	5039	2627	8169	278
H(10T)	3446	3248	7667	135
H(10U)	3581	2422	7497	135
H(10V)	7417	9018	805	255
H(10W)	7509	8529	262	255
H(11C)	8499	9510	-554	298
H(11D)	8760	9819	92	298

H(11E)	10409	8591	-290	169
H(11F)	10809	9133	124	169
H(11G)	10582	7574	862	164
H(11H)	10514	8209	1260	164

Hydrogen bonds for [K_4 (4-tert-butylcalix[8]arene)-4H$(THF)_6(H_2O)_{12}$] [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(6)-H(6)...O(5)	0.82	1.70	2.516(16)	173.2

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 x-1,y,z #3 x,y+1,z-1 #4 x,y,z

V.2.14. [Rb_2((4-tert-butyl calix[8]arene-H))$_2(THF)_8(H_2O)_{14}$] 14

Besides the large number of water molecules, It was not possible to anisotropize the two 4-tert-butylcalix[8]arene because data were not enough for so many atoms. The tertio-butyl groups are really distorted and some tertio- butyl were not fully determined.

The Cc group is rather uncommon but when the structure **14** is solved in a higher symmetry group (C2/c), the symmetry properties do not match the water aggregates present in the structure. Therefore it seems wise to use this group more than another.

Empirical formula	C208 H314 O46 Rb2	
Formula weight	3721.64	
Temperature	240(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 35.745(7) Å	α= 90°.
	b = 22.459(5) Å	β= 109.53(3)°.
	c = 28.450(6) Å	γ = 90°.

Volume	21525(7) $Å^3$
Z	4
Density (calculated)	1.031 Mg/m^3
Absorption coefficient	0.517 mm^{-1}
F(000)	6780
Theta range for data collection	1.19 to 19.88°.
Index ranges	-34<=h<=34, -13<=k<=20, -27<=l<=26
Reflections collected	18188
Independent reflections	12170 [R(int) = 0.2383]
Completeness to theta = 19.88°	81.7 %
Absorption correction	Spherical
Refinement method	Full-matrix-block least-squares on F^2
Data / restraints / parameters	12170 / 1 / 1276
Goodness-of-fit on F^2	1.150
Final R indices [I>2sigma(I)]	R1 = 0.1449, wR2 = 0.2990
R indices (all data)	R1 = 0.3162, wR2 = 0.3750
Absolute structure parameter	0.00
Largest diff. peak and hole	2.316 and -0.609 $e.Å^{-3}$

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Rb_2((4-tert-butylcalix[8]arene-H))$_2$$(THF)_8$$(H_2O)_{14}$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Rb(1)	504(1)	5060(3)	5529(2)	72
Rb(2)	-496(2)	5553(3)	4471(2)	68
O(1)	-696(6)	3314(12)	6089(9)	46
C(1)	-862(8)	2732(18)	6104(13)	37
C(2)	-1285(8)	2674(16)	5709(12)	28
C(3)	-1477(8)	2193(17)	5762(12)	30
C(4)	-1297(12)	1640(30)	5995(18)	81
C(5)	-854(8)	1793(17)	6367(12)	33

C(6)	-580(20)	2260(40)	6430(30)	151
C(7)	-1501(7)	1106(14)	6106(11)	10
C(8)	-1450(14)	1010(30)	6580(20)	107
C(9)	-1898(13)	1210(30)	6090(20)	99
C(10)	-1280(30)	400(50)	5820(40)	234
C(11)	-243(9)	2318(19)	6697(13)	42
O(2)	46(6)	3298(12)	6187(8)	44
C(12)	183(8)	2745(19)	6117(13)	33
C(13)	40(9)	2193(19)	6399(13)	36
C(14)	214(7)	1745(16)	6384(11)	19
C(15)	473(10)	1590(20)	6069(15)	58
C(16)	599(11)	2160(20)	5957(15)	57
C(17)	452(8)	2676(18)	5886(12)	26
C(18)	746(11)	980(30)	6197(17)	70
C(19)	548(18)	560(40)	6410(30)	156
C(20)	770(30)	910(60)	5660(40)	231
C(21)	1060(30)	990(50)	5830(40)	212
C(22)	615(10)	3140(20)	5636(15)	66
O(3)	503(7)	4138(15)	6260(10)	75
C(23)	921(11)	3940(20)	6382(15)	61
C(24)	985(8)	3491(16)	6084(12)	24
C(25)	1335(9)	3335(18)	6114(14)	42
C(26)	1643(9)	3521(19)	6440(14)	44
C(27)	1611(9)	3951(17)	6783(13)	36
C(28)	1276(9)	4215(19)	6806(14)	42
C(29)	2077(15)	2690(30)	6160(20)	115
C(30)	2137(11)	3260(20)	6524(17)	68
C(31)	2279(10)	2940(20)	6970(16)	63
C(32)	2345(9)	3827(18)	6360(13)	40
C(33)	1233(11)	4630(20)	7076(15)	59
O(4)	830(4)	5630(9)	6521(7)	16
C(34)	1204(12)	5770(20)	6689(18)	79
C(35)	1438(7)	5262(15)	7059(12)	24
C(36)	1884(9)	5340(19)	7346(14)	53
C(37)	2050(8)	5804(17)	7210(13)	34
C(38)	1825(7)	6309(14)	6895(11)	16

C(39)	1426(8)	6306(17)	6667(12)	29
C(40)	2523(9)	5960(20)	7509(15)	49
C(41)	2586(13)	6050(30)	8020(20)	99
C(42)	2696(11)	5330(20)	7248(16)	75
C(43)	2688(11)	6410(20)	7339(16)	66
C(44)	1200(10)	6710(20)	6311(15)	65
O(5)	460(7)	6638(12)	6439(9)	51
C(45)	639(8)	7137(16)	6597(11)	22
C(46)	1031(7)	7192(15)	6550(11)	19
C(47)	1188(7)	7765(15)	6758(11)	17
C(48)	1052(8)	8162(18)	6935(12)	34
C(49)	694(7)	8088(16)	6931(11)	23
C(50)	491(7)	7558(16)	6849(11)	21
C(51)	1264(9)	8765(18)	7125(13)	39
C(52)	1302(9)	9214(19)	6570(14)	50
C(53)	1014(11)	9210(20)	7309(16)	71
C(54)	1691(14)	8640(30)	7500(20)	109
C(55)	43(9)	7460(20)	6884(15)	65
O(6)	-270(8)	6524(16)	6173(11)	81
C(56)	-401(11)	7100(20)	5963(16)	64
C(57)	-283(6)	7570(13)	6350(10)	1
C(58)	-438(10)	8160(20)	6192(15)	58
C(59)	-773(11)	8110(20)	5784(17)	66
C(60)	-935(11)	7690(20)	5517(16)	58
C(61)	-785(9)	7155(19)	5578(14)	42
C(62)	-936(8)	8784(17)	5660(12)	31
C(63)	-1089(15)	9070(30)	6160(20)	122
C(64)	-1289(13)	8820(30)	5070(20)	105
C(65)	-629(11)	9380(20)	5801(16)	72
C(66)	-972(7)	6603(14)	5315(11)	11
O(7)	-798(6)	5637(14)	5957(10)	67
C(67)	-1178(8)	5889(16)	5808(12)	21
C(68)	-1268(8)	6210(18)	5450(13)	33
C(69)	-1658(9)	6520(20)	5281(14)	51
C(70)	-1953(8)	6211(17)	5489(13)	30
C(71)	-1854(10)	5840(20)	5812(16)	56

C(72)	-1445(9)	5680(20)	5976(14)	50
C(73)	-2421(9)	6409(19)	5272(14)	43
C(74)	-2451(10)	7170(20)	5133(15)	56
C(75)	-2606(11)	6150(20)	4769(17)	73
C(76)	-2634(13)	6150(30)	5566(19)	92
C(77)	-1320(9)	5150(20)	6386(14)	52
O(8)	-1074(7)	4318(15)	5887(11)	68
C(78)	-1418(11)	4200(20)	6024(16)	64
C(79)	-1621(9)	4544(18)	6216(13)	38
C(80)	-1945(8)	4417(19)	6322(14)	41
C(81)	-2190(7)	3971(14)	6062(10)	11
C(82)	-2002(9)	3610(20)	5821(14)	53
C(83)	-1648(10)	3650(20)	5702(16)	59
C(84)	-2599(6)	3833(13)	6107(10)	1
C(85)	-2809(9)	3300(20)	5804(14)	52
C(86)	-2854(13)	4350(30)	5910(20)	99
C(87)	-2568(16)	4100(30)	6530(20)	127
C(88)	-1501(14)	3240(30)	5450(20)	66
O(9)	-826(8)	4952(19)	3615(12)	106
C(89)	-1238(7)	4947(14)	3243(10)	11
C(90)	-1444(10)	5430(20)	2988(15)	56
C(91)	-1842(9)	5326(19)	2716(14)	51
C(92)	-2054(9)	4812(19)	2779(14)	41
C(93)	-1853(13)	4420(30)	3044(19)	96
C(94)	-1407(9)	4460(20)	3367(14)	46
C(95)	-2484(9)	4700(20)	2491(15)	50
C(96)	-2670(15)	4130(30)	2740(20)	120
C(97)	-2567(11)	4680(20)	1934(17)	72
C(98)	-2706(12)	5390(30)	2533(17)	85
C(99)	-1196(10)	3930(20)	3672(14)	48
O(10)	-421(7)	4115(15)	3541(11)	73
C(100)	-639(15)	3500(30)	3380(20)	104
C(101)	-996(13)	3400(30)	3486(19)	81
C(102)	-1218(11)	2950(20)	3323(16)	62
C(103)	-1040(11)	2350(20)	3121(17)	68
C(104)	-652(10)	2550(20)	3012(16)	61

C(105)	-412(10)	3030(20)	3285(15)	52
C(106)	-1282(10)	1840(20)	2966(15)	51
C(107)	-1343(10)	1590(20)	3344(16)	61
C(108)	-1715(12)	1940(20)	2599(17)	78
C(109)	-1118(13)	1440(30)	2560(19)	94
C(110)	-63(7)	3177(15)	3197(11)	15
O(11)	283(6)	4113(12)	3913(9)	45
C(111)	463(8)	3527(16)	3931(12)	25
C(112)	195(18)	3130(40)	3500(30)	130
C(113)	460(9)	2520(20)	3740(14)	50
C(114)	797(8)	2376(16)	4202(12)	24
C(115)	966(10)	2900(20)	4504(15)	57
C(116)	787(8)	3554(17)	4368(13)	35
C(117)	1015(14)	1720(30)	4270(20)	96
C(118)	1279(8)	1754(17)	4797(13)	37
C(119)	1203(11)	1750(30)	3971(18)	75
C(120)	647(11)	1360(20)	4413(16)	71
C(121)	1037(14)	4020(30)	4800(20)	114
O(12)	784(5)	4967(11)	3886(8)	33
C(122)	1180(12)	4630(30)	4151(19)	81
C(123)	1282(10)	4250(20)	4534(16)	56
C(124)	1703(8)	4101(18)	4697(13)	39
C(125)	1980(11)	4450(20)	4575(17)	64
C(126)	1867(9)	4832(18)	4201(13)	37
C(127)	1478(8)	5073(19)	3949(13)	36
C(128)	2419(9)	4197(18)	4816(14)	39
C(129)	2615(11)	4180(20)	4365(16)	64
C(130)	2526(18)	4280(40)	5310(30)	139
C(131)	2689(17)	4740(40)	5180(20)	124
C(132)	1394(8)	5486(18)	3569(12)	35
O(13)	1063(6)	6381(12)	4029(9)	47
C(133)	1445(8)	6555(17)	4084(12)	24
C(134)	1573(9)	6020(18)	3799(13)	36
C(135)	1960(13)	6110(30)	3756(19)	95
C(136)	2126(14)	6650(30)	3990(20)	96
C(137)	2011(10)	7080(20)	4261(15)	59

C(138)	1655(11)	6920(20)	4253(15)	55
C(139)	2626(19)	7570(40)	3860(30)	164
C(140)	2820(19)	5890(40)	3980(30)	157
C(141)	2510(11)	6930(20)	3304(17)	68
C(142)	2910(20)	6800(50)	4390(40)	217
C(143)	1490(12)	7420(20)	4578(18)	50
O(14)	693(7)	7404(14)	4070(11)	61
C(144)	871(9)	7900(18)	3978(13)	36
C(145)	1236(12)	7850(20)	4192(17)	71
C(146)	1476(14)	8510(30)	4190(20)	95
C(147)	1277(11)	8920(20)	3883(16)	57
C(148)	917(13)	8900(30)	3611(19)	91
C(149)	681(6)	8338(13)	3668(10)	4
C(150)	1440(20)	9840(40)	4060(30)	166
C(151)	1497(15)	9940(30)	4450(20)	111
C(152)	2059(14)	9330(30)	4250(20)	101
C(153)	1298(17)	9980(40)	3520(30)	146
C(154)	242(10)	8260(20)	3412(16)	68
O(15)	-20(6)	7337(12)	3885(9)	40
C(155)	-173(11)	7850(20)	3866(16)	56
C(156)	-43(9)	8340(20)	3637(14)	40
C(157)	-160(11)	8950(20)	3719(15)	70
C(158)	-483(10)	9040(20)	3808(14)	49
C(159)	-612(9)	8610(19)	4134(14)	44
C(160)	-451(9)	7900(20)	4032(14)	43
C(161)	-632(9)	9660(20)	3927(14)	43
C(162)	-354(12)	10150(30)	4007(17)	82
C(163)	-1001(16)	9720(30)	4010(20)	127
C(164)	-926(16)	9800(30)	3340(20)	130
C(165)	-576(9)	7446(19)	4296(14)	49
O(16)	-574(5)	6484(11)	3613(8)	32
C(166)	-962(9)	6710(19)	3633(14)	44
C(167)	-936(12)	7170(30)	4000(18)	79
C(168)	-1345(9)	7400(20)	3917(15)	53
C(169)	-1669(10)	7160(20)	3604(15)	52
C(170)	-1643(9)	6622(18)	3299(13)	38

C(171)	-1274(7)	6484(15)	3314(11)	17
C(172)	-2061(10)	7390(20)	3537(15)	56
C(173)	-2106(10)	7710(20)	3975(15)	52
C(174)	-2280(17)	7570(40)	2920(20)	135
C(175)	-2426(16)	6910(30)	3420(20)	137
C(176)	-1272(8)	5955(15)	2902(12)	25
O(17)	1270(9)	5465(15)	5473(13)	75
C(177)	1352(10)	5810(20)	5194(16)	55
C(178)	1838(15)	5860(30)	5390(30)	168
C(179)	1945(9)	5450(20)	5790(20)	92
C(180)	1688(14)	5190(30)	5781(16)	105
O(18)	-1287(6)	5314(15)	4483(14)	100
C(181)	-1612(11)	5614(17)	4190(20)	102
C(182)	-2070(30)	5200(40)	4200(30)	210
C(183)	-1760(40)	4700(40)	4790(50)	234
C(184)	-1390(30)	4660(20)	4780(30)	149
O(19)	706(15)	3030(30)	7339(19)	179
C(185)	1070(19)	2400(20)	7440(20)	132
C(186)	1510(20)	2490(30)	7760(30)	166
C(187)	1430(15)	3220(30)	8030(30)	131
C(188)	945(9)	3270(20)	7721(19)	70
O(20)	-1079(12)	7322(18)	2264(17)	122
C(189)	-866(12)	7840(30)	2630(20)	125
C(190)	-1210(30)	8260(40)	2700(30)	212
C(191)	-1512(17)	7850(30)	2200(20)	117
C(192)	-1387(12)	7370(30)	2110(20)	130
O(21)	-648(7)	2688(16)	4648(11)	90
C(193)	-921(12)	2370(20)	4766(19)	80
C(194)	-712(16)	2030(40)	4950(30)	1361
C(195)	-490(20)	1820(40)	4750(30)	203
C(196)	-320(9)	2310(30)	4583(14)	68
O(22)	849(15)	8340(30)	5325(19)	281
C(197)	520(30)	8900(30)	5020(30)	200
C(198)	262(15)	8840(30)	5300(30)	117
C(299)	70(30)	8170(40)	5440(50)	284
C(200)	413(17)	8160(30)	5580(30)	149

O(23)	-1093(7)	6549(19)	7036(14)	100
C(201)	-1456(14)	6650(20)	7040(16)	60
C(202)	-1530(30)	7100(40)	6870(80)	470
C(203)	-1232(11)	7347(17)	6695(14)	43
C(204)	-922(17)	6900(20)	6891(17)	83
O(24)	1028(13)	4245(18)	2776(19)	81
C(205)	950(30)	3650(80)	3050(30)	836
C(206)	1630(40)	2670(80)	3860(100)	716
C(207)	1632(14)	3350(30)	3280(30)	155
C(208)	1504(15)	3970(30)	2860(20)	147
O(25)	-6(7)	4392(14)	4716(10)	52
O(26)	38(8)	6103(16)	5352(18)	126
O(27)	-395(8)	4998(16)	5568(12)	80
O(28)	371(7)	5767(12)	4429(13)	73
O(29)	-459(9)	3703(15)	5148(13)	75
O(30)	404(9)	6884(18)	4811(15)	97
O(31)	-494(6)	6404(16)	2315(11)	80
O(32)	-153(5)	4534(11)	6618(7)	6
O(33)	-444(12)	5730(30)	7384(15)	188
O(34)	-337(13)	4020(30)	7607(18)	186
O(35)	526(10)	4988(16)	2496(16)	109
O(36)	21(4)	5397(9)	1661(7)	23
O(37)	171(5)	6167(10)	3355(7)	3
O(38)	0	460(70)	2500	479

Bond lengths [Å] and angles [°] for $[Rb_2((4\text{-tert-butylcalix[8]arene-H}))_2(THF)_8(H_2O)_{14}]$.

Rb(1)-O(26)	2.82(3)	Rb(1)-Rb(2)	3.986(5)
Rb(1)-O(25)	2.85(3)	Rb(2)-O(9)	2.69(4)
Rb(1)-O(3)	2.94(3)	Rb(2)-O(26)	2.87(5)
Rb(1)-O(17)	2.94(3)	Rb(2)-O(18)	2.89(2)
Rb(1)-O(4)	2.96(2)	Rb(2)-O(25)	3.09(3)
Rb(1)-O(27)	3.26(3)	Rb(2)-O(16)	3.15(2)
Rb(1)-O(28)	3.40(4)	Rb(2)-O(28)	3.18(2)
Rb(1)-C(23)	3.46(5)	Rb(2)-O(27)	3.27(3)

Rb(2)-C(166)	3.54(4)	C(20)-C(21)	1.00(11)
O(1)-C(1)	1.44(4)	C(22)-C(24)	1.70(5)
O(1)-H(1)	0.8200	C(22)-H(22A)	0.9700
C(1)-C(6)	1.55(8)	C(22)-H(22B)	0.9700
C(1)-C(2)	1.56(5)	O(3)-C(23)	1.48(4)
C(2)-C(3)	1.32(5)	C(23)-C(24)	1.39(5)
C(2)-C(88)	1.53(6)	C(23)-C(28)	1.55(5)
C(3)-C(4)	1.44(6)	C(24)-C(25)	1.28(4)
C(3)-H(3)	0.9300	C(25)-C(26)	1.25(5)
C(4)-C(7)	1.50(6)	C(25)-H(25)	0.9300
C(4)-C(5)	1.62(6)	C(26)-C(27)	1.40(5)
C(5)-C(6)	1.41(8)	C(26)-C(30)	1.80(5)
C(5)-H(5)	0.9300	C(27)-C(28)	1.36(4)
C(6)-C(11)	1.19(7)	C(27)-H(27)	0.9300
C(7)-C(8)	1.32(5)	C(28)-C(33)	1.25(5)
C(7)-C(9)	1.43(4)	C(29)-C(30)	1.63(7)
C(7)-C(10)	2.06(11)	C(30)-C(31)	1.40(6)
C(8)-C(9)	1.79(7)	C(30)-C(32)	1.62(6)
C(11)-C(13)	1.55(4)	C(33)-C(35)	1.60(5)
C(11)-H(11A)	0.9700	C(33)-H(33A)	0.9700
C(11)-H(11B)	0.9700	C(33)-H(33B)	0.9700
O(2)-C(12)	1.37(4)	O(4)-C(34)	1.30(4)
O(2)-H(2)	0.8200	C(34)-C(39)	1.45(6)
C(12)-C(17)	1.34(3)	C(34)-C(35)	1.59(6)
C(12)-C(13)	1.65(5)	C(35)-C(36)	1.54(4)
C(13)-C(14)	1.19(5)	C(36)-C(37)	1.32(5)
C(14)-C(15)	1.53(4)	C(36)-H(36)	0.9300
C(14)-H(14)	0.9300	C(37)-C(38)	1.50(4)
C(15)-C(16)	1.44(6)	C(37)-C(40)	1.65(5)
C(15)-C(18)	1.64(7)	C(38)-C(39)	1.36(4)
C(16)-C(17)	1.26(5)	C(38)-H(38)	0.9300
C(16)-H(16)	0.9300	C(39)-C(44)	1.39(5)
C(17)-C(22)	1.48(5)	C(40)-C(41)	1.41(6)
C(18)-C(19)	1.43(8)	C(40)-C(43)	1.35(6)
C(18)-C(20)	1.56(10)	C(40)-C(42)	1.80(6)
C(18)-C(21)	1.79(9)	C(44)-C(46)	1.51(5)

C(44)-H(44A)	0.9700	C(66)-H(66A)	0.9700
C(44)-H(44B)	0.9700	C(66)-H(66B)	0.9700
O(5)-C(45)	1.29(4)	O(7)-C(67)	1.40(4)
O(5)-H(5A)	0.8200	O(7)-H(7)	0.8200
C(45)-C(50)	1.39(4)	C(67)-C(68)	1.20(4)
C(45)-C(46)	1.46(3)	C(67)-C(72)	1.29(4)
C(46)-C(47)	1.45(4)	C(68)-C(69)	1.49(5)
C(47)-C(48)	1.20(4)	C(69)-C(70)	1.54(4)
C(47)-H(47)	0.9300	C(69)-H(69)	0.9300
C(48)-C(49)	1.29(3)	C(70)-C(71)	1.21(5)
C(48)-C(51)	1.56(5)	C(70)-C(73)	1.64(5)
C(49)-C(50)	1.37(5)	C(71)-C(72)	1.43(5)
C(49)-H(49)	0.9300	C(71)-H(71)	0.9300
C(50)-C(55)	1.65(4)	C(72)-C(77)	1.61(5)
C(51)-C(53)	1.55(5)	C(73)-C(76)	1.43(5)
C(51)-C(54)	1.56(6)	C(73)-C(75)	1.48(5)
C(51)-C(52)	1.92(5)	C(73)-C(74)	1.75(6)
C(55)-C(57)	1.60(5)	C(77)-C(79)	1.70(5)
C(55)-H(55A)	0.9700	C(77)-H(77A)	0.9700
C(55)-H(55B)	0.9700	C(77)-H(77B)	0.9700
O(6)-C(56)	1.43(5)	O(8)-C(78)	1.43(4)
O(6)-H(6)	0.8200	O(8)-H(8)	0.8200
C(56)-C(61)	1.45(5)	C(78)-C(79)	1.30(5)
C(56)-C(57)	1.49(5)	C(78)-C(83)	1.58(6)
C(57)-C(58)	1.44(5)	C(79)-C(80)	1.32(3)
C(58)-C(59)	1.37(5)	C(80)-C(81)	1.37(5)
C(58)-H(58)	0.9300	C(80)-H(80)	0.9300
C(59)-C(60)	1.22(6)	C(81)-C(82)	1.37(4)
C(59)-C(62)	1.63(6)	C(81)-C(84)	1.54(3)
C(60)-C(61)	1.31(5)	C(82)-C(83)	1.42(4)
C(60)-H(60)	0.9300	C(82)-H(82)	0.9300
C(61)-C(66)	1.49(5)	C(83)-C(88)	1.37(6)
C(62)-C(65)	1.69(6)	C(84)-C(87)	1.31(6)
C(62)-C(64)	1.74(6)	C(84)-C(86)	1.47(6)
C(62)-C(63)	1.79(6)	C(84)-C(85)	1.52(5)
C(66)-C(68)	1.52(4)	C(86)-C(87)	1.81(8)

C(88)-H(88A)	0.9700	C(110)-H(11C)	0.9700
C(88)-H(88B)	0.9700	C(110)-H(11D)	0.9700
O(9)-C(89)	1.50(4)	O(11)-C(111)	1.46(4)
C(89)-C(90)	1.37(5)	O(11)-H(11)	0.8200
C(89)-C(94)	1.35(5)	C(111)-C(116)	1.39(4)
C(90)-C(176)	1.39(5)	C(111)-C(112)	1.55(7)
C(90)-C(91)	1.39(5)	C(112)-C(113)	1.69(8)
C(91)-C(92)	1.43(5)	C(113)-C(114)	1.49(5)
C(91)-H(91)	0.9300	C(113)-H(113)	0.9300
C(92)-C(93)	1.22(6)	C(114)-C(115)	1.47(5)
C(92)-C(95)	1.50(5)	C(114)-C(117)	1.65(7)
C(93)-C(94)	1.55(6)	C(115)-C(116)	1.59(5)
C(93)-H(93)	0.9300	C(115)-H(115)	0.9300
C(94)-C(99)	1.52(5)	C(116)-C(121)	1.64(7)
C(95)-C(97)	1.51(5)	C(117)-C(119)	1.25(5)
C(95)-C(96)	1.70(7)	C(117)-C(118)	1.48(6)
C(95)-C(98)	1.76(7)	C(117)-C(120)	1.71(6)
C(99)-C(101)	1.56(6)	C(121)-C(123)	1.44(5)
C(99)-H(99A)	0.9700	C(121)-H(12A)	0.9700
C(99)-H(99B)	0.9700	C(121)-H(12B)	0.9700
O(10)-C(100)	1.58(7)	O(12)-C(122)	1.57(5)
O(10)-H(10)	0.8200	O(12)-H(12)	0.8200
C(100)-C(101)	1.42(5)	C(122)-C(123)	1.33(6)
C(100)-C(105)	1.40(7)	C(122)-C(127)	1.70(6)
C(101)-C(102)	1.28(7)	C(123)-C(124)	1.46(5)
C(102)-C(103)	1.67(7)	C(124)-C(125)	1.39(5)
C(102)-H(102)	0.9300	C(124)-H(124)	0.9300
C(103)-C(106)	1.41(6)	C(125)-C(126)	1.32(5)
C(103)-C(104)	1.58(5)	C(125)-C(128)	1.59(5)
C(104)-C(105)	1.45(6)	C(126)-C(127)	1.44(5)
C(104)-H(104)	0.9300	C(126)-H(126)	0.9300
C(105)-C(110)	1.39(4)	C(127)-C(132)	1.38(5)
C(106)-C(107)	1.29(5)	C(128)-C(130)	1.34(7)
C(106)-C(108)	1.57(6)	C(128)-C(129)	1.65(5)
C(106)-C(109)	1.72(6)	C(128)-C(131)	1.68(8)
C(110)-C(112)	1.04(7)	C(130)-C(131)	1.28(9)

C(132)-C(134)	1.41(5)	C(155)-C(156)	1.42(6)
C(132)-H(13A)	0.9700	C(156)-C(157)	1.49(6)
C(132)-H(13B)	0.9700	C(157)-C(158)	1.27(4)
O(13)-C(133)	1.38(3)	C(157)-H(157)	0.9300
O(13)-H(13)	0.8200	C(158)-C(159)	1.51(5)
C(133)-C(138)	1.11(5)	C(158)-C(161)	1.57(6)
C(133)-C(134)	1.60(5)	C(159)-C(160)	1.74(6)
C(134)-C(135)	1.44(4)	C(159)-H(159)	0.9300
C(135)-C(136)	1.41(7)	C(160)-C(165)	1.43(5)
C(135)-H(135)	0.9300	C(161)-C(163)	1.43(5)
C(136)-C(137)	1.39(7)	C(161)-C(162)	1.45(6)
C(137)-C(138)	1.31(4)	C(161)-C(164)	1.67(7)
C(137)-H(137)	0.9300	C(165)-C(167)	1.42(6)
C(138)-C(143)	1.68(6)	C(165)-H(16A)	0.9700
C(143)-C(145)	1.52(6)	C(165)-H(16B)	0.9700
C(143)-H(14A)	0.9700	O(16)-C(166)	1.49(3)
C(143)-H(14B)	0.9700	C(166)-C(171)	1.28(4)
O(14)-C(144)	1.35(4)	C(166)-C(167)	1.46(6)
O(14)-H(14C)	0.8200	C(167)-C(168)	1.49(5)
C(144)-C(145)	1.25(5)	C(168)-C(169)	1.31(5)
C(144)-C(149)	1.34(4)	C(168)-H(168)	0.9300
C(145)-C(146)	1.72(7)	C(169)-C(172)	1.44(5)
C(146)-C(147)	1.30(6)	C(169)-C(170)	1.51(5)
C(146)-H(146)	0.9300	C(170)-C(171)	1.34(3)
C(147)-C(148)	1.26(5)	C(171)-C(176)	1.67(4)
C(148)-C(149)	1.56(6)	C(172)-C(173)	1.49(5)
C(148)-H(148)	0.9300	C(172)-C(175)	1.63(8)
C(149)-C(154)	1.50(4)	C(172)-C(174)	1.72(7)
C(150)-C(151)	1.08(8)	C(176)-H(17A)	0.9700
C(150)-C(153)	1.48(9)	C(176)-H(17B)	0.9700
C(154)-C(156)	1.38(4)	O(17)-C(177)	1.22(4)
C(154)-H(15A)	0.9700	O(17)-C(180)	1.58(6)
C(154)-H(15B)	0.9700	C(177)-C(178)	1.64(6)
O(15)-C(155)	1.28(5)	C(178)-C(179)	1.40(7)
O(15)-H(15)	0.8200	C(179)-C(180)	1.08(6)
C(155)-C(160)	1.24(4)	O(18)-C(181)	1.36(6)

O(18)-C(184)	1.80(5)	O(23)-C(202)	1.92(9)
C(181)-C(182)	1.90(5)	O(23)-O(33)#1	2.87(5)
C(182)-C(183)	2.04(15)	C(201)-C(202)	1.11(13)
C(183)-C(184)	1.31(10)	C(202)-C(203)	1.43(13)
O(19)-C(188)	1.26(6)	C(203)-C(204)	1.45(6)
O(19)-C(185)	1.89(8)	O(24)-C(205)	1.61(16)
O(19)-O(34)#1	4.62(7)	O(24)-C(208)	1.75(8)
C(185)-C(186)	1.56(8)	C(206)-C(207)	2.3(3)
C(186)-C(187)	1.89(10)	C(207)-C(208)	1.79(10)
C(187)-C(188)	1.67(6)	O(25)-O(29)#1	2.80(4)
O(20)-C(192)	1.04(5)	O(26)-O(30)#1	2.92(5)
O(20)-C(189)	1.58(8)	O(27)-O(29)#1	3.13(5)
O(20)-C(191)	1.90(6)	O(27)-O(32)#1	3.01(3)
O(20)-O(31)#1	2.91(5)	O(28)-O(30)#1	2.72(5)
C(189)-C(190)	1.62(9)	O(28)-O(37)	3.03(4)
C(190)-C(191)	1.75(10)	O(28)-O(36)#2	3.06(4)
C(191)-C(192)	1.21(8)	O(29)-O(29)#1	0.00(10)
O(21)-C(193)	1.33(5)	O(30)-O(30)#1	0.00000(12)
O(21)-C(196)	1.50(6)	O(31)-O(31)#1	0.00(9)
O(21)-C(194)	1.75(6)	O(31)-O(37)#1	3.16(3)
O(21)-O(29)#1	2.65(5)	O(31)-O(35)#2	3.23(5)
C(193)-C(194)	1.07(5)	O(32)-O(32)#1	0.00(4)
C(194)-C(195)	1.22(11)	O(32)-O(34)#3	2.58(6)
C(195)-C(196)	1.42(9)	O(32)-O(34)	3.30(5)
O(22)-C(197)	1.74(10)	O(33)-O(33)#1	0.00(18)
O(22)-C(200)	1.97(8)	O(33)-O(33)#3	3.02(8)
C(197)-C(198)	1.42(7)	O(34)-O(34)#1	0.00(17)
C(198)-C(200)	1.73(7)	O(34)-O(34)#3	2.68(8)
C(198)-C(299)	1.76(9)	O(35)-O(36)#1	2.62(5)
C(299)-C(200)	1.17(9)	O(36)-O(36)#1	0.00(6)
O(23)-C(204)	1.16(5)	O(36)-O(37)#2	1.85(3)
O(23)-C(201)	1.32(4)	O(37)-O(37)#1	0.00(6)

O(26)-Rb(1)-O(25)	96.5(10)	O(26)-Rb(1)-O(17)	103.8(9)
O(26)-Rb(1)-O(3)	124.8(8)	O(25)-Rb(1)-O(17)	118.4(8)
O(25)-Rb(1)-O(3)	92.9(6)	O(3)-Rb(1)-O(17)	118.6(7)

O(26)-Rb(1)-O(4)	80.3(10)	O(9)-Rb(2)-O(16)	74.5(9)
O(25)-Rb(1)-O(4)	160.6(5)	O(26)-Rb(2)-O(16)	104.5(8)
O(3)-Rb(1)-O(4)	73.9(7)	O(18)-Rb(2)-O(16)	107.4(5)
O(17)-Rb(1)-O(4)	80.9(6)	O(25)-Rb(2)-O(16)	129.1(5)
O(26)-Rb(1)-O(27)	60.7(9)	O(9)-Rb(2)-O(28)	100.3(6)
O(25)-Rb(1)-O(27)	68.5(9)	O(26)-Rb(2)-O(28)	66.2(8)
O(3)-Rb(1)-O(27)	73.2(5)	O(18)-Rb(2)-O(28)	177.5(10)
O(17)-Rb(1)-O(27)	164.3(9)	O(25)-Rb(2)-O(28)	69.4(7)
O(4)-Rb(1)-O(27)	93.6(7)	O(16)-Rb(2)-O(28)	72.8(6)
O(26)-Rb(1)-O(28)	63.6(10)	O(9)-Rb(2)-O(27)	123.3(8)
O(25)-Rb(1)-O(28)	69.1(8)	O(26)-Rb(2)-O(27)	60.1(11)
O(3)-Rb(1)-O(28)	161.7(7)	O(18)-Rb(2)-O(27)	73.4(8)
O(17)-Rb(1)-O(28)	69.6(8)	O(25)-Rb(2)-O(27)	65.7(8)
O(4)-Rb(1)-O(28)	124.4(5)	O(16)-Rb(2)-O(27)	160.9(7)
O(27)-Rb(1)-O(28)	102.5(6)	O(28)-Rb(2)-O(27)	107.2(8)
O(26)-Rb(1)-C(23)	144.8(9)	O(9)-Rb(2)-C(166)	78.1(11)
O(25)-Rb(1)-C(23)	101.3(8)	O(26)-Rb(2)-C(166)	107.3(7)
O(3)-Rb(1)-C(23)	25.1(6)	O(18)-Rb(2)-C(166)	84.5(5)
O(17)-Rb(1)-C(23)	94.1(7)	O(25)-Rb(2)-C(166)	151.0(6)
O(4)-Rb(1)-C(23)	72.9(9)	O(16)-Rb(2)-C(166)	24.9(5)
O(27)-Rb(1)-C(23)	98.3(6)	O(28)-Rb(2)-C(166)	96.2(6)
O(28)-Rb(1)-C(23)	151.5(6)	O(27)-Rb(2)-C(166)	143.1(6)
O(26)-Rb(1)-Rb(2)	46.1(10)	O(9)-Rb(2)-Rb(1)	123.8(8)
O(25)-Rb(1)-Rb(2)	50.5(5)	O(26)-Rb(2)-Rb(1)	45.1(6)
O(3)-Rb(1)-Rb(2)	121.2(5)	O(18)-Rb(2)-Rb(1)	125.0(6)
O(17)-Rb(1)-Rb(2)	119.4(7)	O(25)-Rb(2)-Rb(1)	45.3(5)
O(4)-Rb(1)-Rb(2)	124.6(4)	O(16)-Rb(2)-Rb(1)	126.4(3)
O(27)-Rb(1)-Rb(2)	52.5(5)	O(28)-Rb(2)-Rb(1)	55.3(6)
O(28)-Rb(1)-Rb(2)	50.3(4)	O(27)-Rb(2)-Rb(1)	52.2(5)
C(23)-Rb(1)-Rb(2)	142.9(7)	C(166)-Rb(2)-Rb(1)	144.4(6)
O(9)-Rb(2)-O(26)	165.6(6)	C(1)-O(1)-H(1)	109.5
O(9)-Rb(2)-O(18)	77.5(8)	O(1)-C(1)-C(6)	116(4)
O(26)-Rb(2)-O(18)	115.9(10)	O(1)-C(1)-C(2)	111(3)
O(9)-Rb(2)-O(25)	79.9(8)	C(6)-C(1)-C(2)	131(4)
O(26)-Rb(2)-O(25)	90.2(7)	C(3)-C(2)-C(88)	122(2)
O(18)-Rb(2)-O(25)	108.9(8)	C(3)-C(2)-C(1)	114(3)

C(88)-C(2)-C(1)	119(3)	C(15)-C(14)-H(14)	115.0
C(2)-C(3)-C(4)	126(3)	C(16)-C(15)-C(14)	102(3)
C(2)-C(3)-H(3)	117.2	C(16)-C(15)-C(18)	126(3)
C(4)-C(3)-H(3)	117.2	C(14)-C(15)-C(18)	120(3)
C(3)-C(4)-C(7)	127(3)	C(17)-C(16)-C(15)	136(3)
C(3)-C(4)-C(5)	108(4)	C(17)-C(16)-H(16)	112.2
C(7)-C(4)-C(5)	118(4)	C(15)-C(16)-H(16)	112.2
C(6)-C(5)-C(4)	137(5)	C(16)-C(17)-C(12)	111(3)
C(6)-C(5)-H(5)	111.7	C(16)-C(17)-C(22)	120(3)
C(4)-C(5)-H(5)	111.7	C(12)-C(17)-C(22)	128(3)
C(11)-C(6)-C(5)	132(7)	C(19)-C(18)-C(20)	122(7)
C(11)-C(6)-C(1)	129(7)	C(19)-C(18)-C(15)	108(4)
C(5)-C(6)-C(1)	98(5)	C(20)-C(18)-C(15)	96(5)
C(8)-C(7)-C(9)	81(3)	C(19)-C(18)-C(21)	139(6)
C(8)-C(7)-C(4)	116(4)	C(20)-C(18)-C(21)	34(4)
C(9)-C(7)-C(4)	114(4)	C(15)-C(18)-C(21)	108(4)
C(8)-C(7)-C(10)	110(4)	C(21)-C(20)-C(18)	85(10)
C(9)-C(7)-C(10)	129(4)	C(20)-C(21)-C(18)	61(8)
C(4)-C(7)-C(10)	105(3)	C(17)-C(22)-C(24)	107(3)
C(7)-C(8)-C(9)	52(3)	C(17)-C(22)-H(22A)	110.2
C(7)-C(9)-C(8)	47(2)	C(24)-C(22)-H(22A)	110.2
C(6)-C(11)-C(13)	109(5)	C(17)-C(22)-H(22B)	110.2
C(6)-C(11)-H(11A)	109.8	C(24)-C(22)-H(22B)	110.2
C(13)-C(11)-H(11A)	109.8	H(22A)-C(22)-H(22B)	108.5
C(6)-C(11)-H(11B)	109.8	C(23)-O(3)-Rb(1)	98(2)
C(13)-C(11)-H(11B)	109.8	C(24)-C(23)-O(3)	116(3)
H(11A)-C(11)-H(11B)	108.2	C(24)-C(23)-C(28)	120(3)
C(12)-O(2)-H(2)	109.5	O(3)-C(23)-C(28)	124(3)
C(17)-C(12)-O(2)	122(3)	C(24)-C(23)-Rb(1)	103(2)
C(17)-C(12)-C(13)	123(3)	O(3)-C(23)-Rb(1)	57(2)
O(2)-C(12)-C(13)	115(2)	C(28)-C(23)-Rb(1)	108(3)
C(14)-C(13)-C(11)	128(3)	C(25)-C(24)-C(23)	121(3)
C(14)-C(13)-C(12)	112(3)	C(25)-C(24)-C(22)	115(3)
C(11)-C(13)-C(12)	120(3)	C(23)-C(24)-C(22)	124(3)
C(13)-C(14)-C(15)	130(3)	C(26)-C(25)-C(24)	124(4)
C(13)-C(14)-H(14)	115.0	C(26)-C(25)-H(25)	118.0

C(24)-C(25)-H(25)	118.0	C(39)-C(38)-H(38)	118.3
C(25)-C(26)-C(27)	119(3)	C(37)-C(38)-H(38)	118.3
C(25)-C(26)-C(30)	125(3)	C(38)-C(39)-C(44)	127(3)
C(27)-C(26)-C(30)	116(3)	C(38)-C(39)-C(34)	119(4)
C(28)-C(27)-C(26)	128(3)	C(44)-C(39)-C(34)	112(3)
C(28)-C(27)-H(27)	116.1	C(41)-C(40)-C(43)	108(4)
C(26)-C(27)-H(27)	116.1	C(41)-C(40)-C(37)	110(3)
C(33)-C(28)-C(27)	130(4)	C(43)-C(40)-C(37)	118(4)
C(33)-C(28)-C(23)	122(3)	C(41)-C(40)-C(42)	126(4)
C(27)-C(28)-C(23)	107(3)	C(43)-C(40)-C(42)	101(3)
C(31)-C(30)-C(29)	96(4)	C(37)-C(40)-C(42)	93(3)
C(31)-C(30)-C(32)	127(4)	C(39)-C(44)-C(46)	111(3)
C(29)-C(30)-C(32)	114(3)	C(39)-C(44)-H(44A)	109.4
C(31)-C(30)-C(26)	109(3)	C(46)-C(44)-H(44A)	109.4
C(29)-C(30)-C(26)	105(3)	C(39)-C(44)-H(44B)	109.4
C(32)-C(30)-C(26)	104(3)	C(46)-C(44)-H(44B)	109.4
C(28)-C(33)-C(35)	119(3)	H(44A)-C(44)-H(44B)	108.0
C(28)-C(33)-H(33A)	107.5	C(45)-O(5)-H(5A)	109.5
C(35)-C(33)-H(33A)	107.5	O(5)-C(45)-C(50)	123(2)
C(28)-C(33)-H(33B)	107.5	O(5)-C(45)-C(46)	115(3)
C(35)-C(33)-H(33B)	107.5	C(50)-C(45)-C(46)	122(3)
H(33A)-C(33)-H(33B)	107.0	C(45)-C(46)-C(47)	107(2)
C(34)-O(4)-Rb(1)	119(2)	C(45)-C(46)-C(44)	121(3)
O(4)-C(34)-C(39)	134(4)	C(47)-C(46)-C(44)	132(3)
O(4)-C(34)-C(35)	108(3)	C(48)-C(47)-C(46)	132(3)
C(39)-C(34)-C(35)	117(3)	C(48)-C(47)-H(47)	113.8
C(36)-C(35)-C(34)	119(3)	C(46)-C(47)-H(47)	113.8
C(36)-C(35)-C(33)	119(3)	C(47)-C(48)-C(49)	116(4)
C(34)-C(35)-C(33)	121(3)	C(47)-C(48)-C(51)	125(3)
C(37)-C(36)-C(35)	115(4)	C(49)-C(48)-C(51)	119(3)
C(37)-C(36)-H(36)	122.5	C(48)-C(49)-C(50)	126(3)
C(35)-C(36)-H(36)	122.5	C(48)-C(49)-H(49)	117.2
C(36)-C(37)-C(38)	125(3)	C(50)-C(49)-H(49)	117.2
C(36)-C(37)-C(40)	120(3)	C(49)-C(50)-C(45)	114(2)
C(38)-C(37)-C(40)	114(3)	C(49)-C(50)-C(55)	125(3)
C(39)-C(38)-C(37)	123(3)	C(45)-C(50)-C(55)	119(3)

C(53)-C(51)-C(54)	115(4)	C(64)-C(62)-C(63)	116(3)
C(53)-C(51)-C(48)	115(3)	C(61)-C(66)-C(68)	126(2)
C(54)-C(51)-C(48)	109(4)	C(61)-C(66)-H(66A)	105.8
C(53)-C(51)-C(52)	98(3)	C(68)-C(66)-H(66A)	105.8
C(54)-C(51)-C(52)	109(3)	C(61)-C(66)-H(66B)	105.8
C(48)-C(51)-C(52)	109(3)	C(68)-C(66)-H(66B)	105.8
C(57)-C(55)-C(50)	109(2)	H(66A)-C(66)-H(66B)	106.2
C(57)-C(55)-H(55A)	109.8	C(67)-O(7)-H(7)	109.5
C(50)-C(55)-H(55A)	109.8	C(68)-C(67)-C(72)	121(3)
C(57)-C(55)-H(55B)	109.8	C(68)-C(67)-O(7)	117(3)
C(50)-C(55)-H(55B)	109.8	C(72)-C(67)-O(7)	120(3)
H(55A)-C(55)-H(55B)	108.2	C(67)-C(68)-C(69)	121(3)
C(56)-O(6)-H(6)	109.5	C(67)-C(68)-C(66)	123(3)
O(6)-C(56)-C(61)	120(4)	C(69)-C(68)-C(66)	108(3)
O(6)-C(56)-C(57)	111(3)	C(68)-C(69)-C(70)	111(4)
C(61)-C(56)-C(57)	117(4)	C(68)-C(69)-H(69)	124.6
C(58)-C(57)-C(56)	116(3)	C(70)-C(69)-H(69)	124.6
C(58)-C(57)-C(55)	122(3)	C(71)-C(70)-C(69)	123(3)
C(56)-C(57)-C(55)	122(3)	C(71)-C(70)-C(73)	119(3)
C(59)-C(58)-C(57)	109(4)	C(69)-C(70)-C(73)	119(3)
C(59)-C(58)-H(58)	125.5	C(70)-C(71)-C(72)	116(3)
C(57)-C(58)-H(58)	125.5	C(70)-C(71)-H(71)	122.1
C(60)-C(59)-C(58)	134(5)	C(72)-C(71)-H(71)	122.1
C(60)-C(59)-C(62)	121(4)	C(67)-C(72)-C(71)	126(4)
C(58)-C(59)-C(62)	105(4)	C(67)-C(72)-C(77)	119(3)
C(59)-C(60)-C(61)	122(5)	C(71)-C(72)-C(77)	115(3)
C(59)-C(60)-H(60)	118.9	C(76)-C(73)-C(75)	105(3)
C(61)-C(60)-H(60)	118.9	C(76)-C(73)-C(70)	110(4)
C(60)-C(61)-C(56)	115(4)	C(75)-C(73)-C(70)	109(3)
C(60)-C(61)-C(66)	127(4)	C(76)-C(73)-C(74)	122(3)
C(56)-C(61)-C(66)	117(4)	C(75)-C(73)-C(74)	101(3)
C(59)-C(62)-C(65)	122(3)	C(70)-C(73)-C(74)	109(3)
C(59)-C(62)-C(64)	110(3)	C(72)-C(77)-C(79)	113(3)
C(65)-C(62)-C(64)	113(3)	C(72)-C(77)-H(77A)	109.1
C(59)-C(62)-C(63)	110(3)	C(79)-C(77)-H(77A)	109.1
C(65)-C(62)-C(63)	83(3)	C(72)-C(77)-H(77B)	109.1

C(79)-C(77)-H(77B)	109.1	C(90)-C(89)-C(94)	125(3)
H(77A)-C(77)-H(77B)	107.8	C(90)-C(89)-O(9)	127(3)
C(78)-O(8)-H(8)	109.5	C(94)-C(89)-O(9)	104(3)
C(79)-C(78)-O(8)	130(4)	C(89)-C(90)-C(176)	125(3)
C(79)-C(78)-C(83)	117(3)	C(89)-C(90)-C(91)	116(4)
O(8)-C(78)-C(83)	108(3)	C(176)-C(90)-C(91)	118(4)
C(78)-C(79)-C(80)	128(4)	C(90)-C(91)-C(92)	123(4)
C(78)-C(79)-C(77)	102(3)	C(90)-C(91)-H(91)	118.3
C(80)-C(79)-C(77)	129(3)	C(92)-C(91)-H(91)	118.3
C(79)-C(80)-C(81)	119(3)	C(93)-C(92)-C(91)	115(4)
C(79)-C(80)-H(80)	120.5	C(93)-C(92)-C(95)	120(4)
C(81)-C(80)-H(80)	120.5	C(91)-C(92)-C(95)	124(4)
C(80)-C(81)-C(82)	112(3)	C(92)-C(93)-C(94)	127(5)
C(80)-C(81)-C(84)	124(2)	C(92)-C(93)-H(93)	116.4
C(82)-C(81)-C(84)	124(3)	C(94)-C(93)-H(93)	116.4
C(81)-C(82)-C(83)	135(4)	C(89)-C(94)-C(99)	127(3)
C(81)-C(82)-H(82)	112.6	C(89)-C(94)-C(93)	110(4)
C(83)-C(82)-H(82)	112.6	C(99)-C(94)-C(93)	121(4)
C(88)-C(83)-C(82)	127(4)	C(97)-C(95)-C(92)	113(3)
C(88)-C(83)-C(78)	127(3)	C(97)-C(95)-C(96)	117(4)
C(82)-C(83)-C(78)	104(3)	C(92)-C(95)-C(96)	112(4)
C(87)-C(84)-C(86)	81(4)	C(97)-C(95)-C(98)	100(3)
C(87)-C(84)-C(81)	101(3)	C(92)-C(95)-C(98)	103(3)
C(86)-C(84)-C(81)	107(3)	C(96)-C(95)-C(98)	112(3)
C(87)-C(84)-C(85)	139(4)	C(94)-C(99)-C(101)	127(3)
C(86)-C(84)-C(85)	107(3)	C(94)-C(99)-H(99A)	105.6
C(81)-C(84)-C(85)	114(2)	C(101)-C(99)-H(99A)	105.6
C(84)-C(86)-C(87)	46(3)	C(94)-C(99)-H(99B)	105.6
C(84)-C(87)-C(86)	53(3)	C(101)-C(99)-H(99B)	105.6
C(83)-C(88)-C(2)	122(4)	H(99A)-C(99)-H(99B)	106.1
C(83)-C(88)-H(88A)	106.7	C(100)-O(10)-H(10)	109.5
C(2)-C(88)-H(88A)	106.7	C(101)-C(100)-C(105)	123(5)
C(83)-C(88)-H(88B)	106.7	C(101)-C(100)-O(10)	118(4)
C(2)-C(88)-H(88B)	106.7	C(105)-C(100)-O(10)	116(4)
H(88A)-C(88)-H(88B)	106.6	C(102)-C(101)-C(100)	123(5)
C(89)-O(9)-Rb(2)	132(2)	C(102)-C(101)-C(99)	115(4)

C(100)-C(101)-C(99)	120(5)	C(113)-C(114)-C(117)	120(3)
C(101)-C(102)-C(103)	120(4)	C(115)-C(114)-C(117)	125(3)
C(101)-C(102)-H(102)	120.0	C(114)-C(115)-C(116)	123(3)
C(103)-C(102)-H(102)	120.0	C(114)-C(115)-H(115)	118.5
C(106)-C(103)-C(104)	131(4)	C(116)-C(115)-H(115)	118.5
C(106)-C(103)-C(102)	120(3)	C(111)-C(116)-C(115)	109(3)
C(104)-C(103)-C(102)	108(4)	C(111)-C(116)-C(121)	142(4)
C(105)-C(104)-C(103)	120(3)	C(115)-C(116)-C(121)	109(3)
C(105)-C(104)-H(104)	119.9	C(119)-C(117)-C(118)	112(4)
C(103)-C(104)-H(104)	119.9	C(119)-C(117)-C(114)	103(4)
C(110)-C(105)-C(104)	119(3)	C(118)-C(117)-C(114)	101(4)
C(110)-C(105)-C(100)	119(4)	C(119)-C(117)-C(120)	145(5)
C(104)-C(105)-C(100)	113(4)	C(118)-C(117)-C(120)	93(3)
C(107)-C(106)-C(103)	110(4)	C(114)-C(117)-C(120)	95(3)
C(107)-C(106)-C(108)	102(3)	C(123)-C(121)-C(116)	96(3)
C(103)-C(106)-C(108)	117(4)	C(123)-C(121)-H(12A)	112.6
C(107)-C(106)-C(109)	121(4)	C(116)-C(121)-H(12A)	112.6
C(103)-C(106)-C(109)	109(3)	C(123)-C(121)-H(12B)	112.6
C(108)-C(106)-C(109)	96(3)	C(116)-C(121)-H(12B)	112.6
C(112)-C(110)-C(105)	115(4)	H(12A)-C(121)-H(12B)	110.1
C(112)-C(110)-H(11C)	108.4	C(122)-O(12)-H(12)	109.5
C(105)-C(110)-H(11C)	108.4	C(123)-C(122)-O(12)	131(3)
C(112)-C(110)-H(11D)	108.4	C(123)-C(122)-C(127)	129(4)
C(105)-C(110)-H(11D)	108.4	O(12)-C(122)-C(127)	97(3)
H(11C)-C(110)-H(11D)	107.5	C(122)-C(123)-C(121)	128(4)
C(111)-O(11)-H(11)	109.5	C(122)-C(123)-C(124)	112(3)
C(116)-C(111)-O(11)	103(3)	C(121)-C(123)-C(124)	120(4)
C(116)-C(111)-C(112)	147(4)	C(125)-C(124)-C(123)	122(4)
O(11)-C(111)-C(112)	110(3)	C(125)-C(124)-H(124)	118.8
C(110)-C(112)-C(111)	138(7)	C(123)-C(124)-H(124)	118.8
C(110)-C(112)-C(113)	129(7)	C(126)-C(125)-C(124)	121(3)
C(111)-C(112)-C(113)	92(4)	C(126)-C(125)-C(128)	125(3)
C(114)-C(113)-C(112)	135(4)	C(124)-C(125)-C(128)	112(4)
C(114)-C(113)-H(113)	112.4	C(125)-C(126)-C(127)	129(3)
C(112)-C(113)-H(113)	112.4	C(125)-C(126)-H(126)	115.5
C(113)-C(114)-C(115)	113(3)	C(127)-C(126)-H(126)	115.5

C(132)-C(127)-C(126)	124(3)	C(138)-C(143)-H(14A)	110.7
C(132)-C(127)-C(122)	132(3)	C(145)-C(143)-H(14B)	110.7
C(126)-C(127)-C(122)	103(3)	C(138)-C(143)-H(14B)	110.7
C(130)-C(128)-C(125)	107(4)	H(14A)-C(143)-H(14B)	108.8
C(130)-C(128)-C(129)	140(4)	C(144)-O(14)-H(14C)	109.5
C(125)-C(128)-C(129)	107(3)	C(145)-C(144)-O(14)	109(4)
C(130)-C(128)-C(131)	49(4)	C(145)-C(144)-C(149)	127(4)
C(125)-C(128)-C(131)	107(4)	O(14)-C(144)-C(149)	124(3)
C(129)-C(128)-C(131)	101(3)	C(144)-C(145)-C(143)	133(4)
C(131)-C(130)-C(128)	80(5)	C(144)-C(145)-C(146)	112(4)
C(130)-C(131)-C(128)	52(4)	C(143)-C(145)-C(146)	112(4)
C(134)-C(132)-C(127)	105(3)	C(147)-C(146)-C(145)	116(4)
C(134)-C(132)-H(13A)	110.7	C(147)-C(146)-H(146)	121.8
C(127)-C(132)-H(13A)	110.7	C(145)-C(146)-H(146)	121.8
C(134)-C(132)-H(13B)	110.7	C(148)-C(147)-C(146)	128(5)
C(127)-C(132)-H(13B)	110.7	C(147)-C(148)-C(149)	116(5)
H(13A)-C(132)-H(13B)	108.8	C(147)-C(148)-H(148)	122.1
C(133)-O(13)-H(13)	109.5	C(149)-C(148)-H(148)	122.1
C(138)-C(133)-O(13)	140(4)	C(144)-C(149)-C(154)	117(3)
C(138)-C(133)-C(134)	121(3)	C(144)-C(149)-C(148)	120(3)
O(13)-C(133)-C(134)	100(3)	C(154)-C(149)-C(148)	124(3)
C(132)-C(134)-C(135)	112(3)	C(151)-C(150)-C(153)	155(10)
C(132)-C(134)-C(133)	135(2)	C(156)-C(154)-C(149)	125(3)
C(135)-C(134)-C(133)	112(3)	C(156)-C(154)-H(15A)	106.1
C(136)-C(135)-C(134)	111(4)	C(149)-C(154)-H(15A)	106.1
C(136)-C(135)-H(135)	124.7	C(156)-C(154)-H(15B)	106.1
C(134)-C(135)-H(135)	124.7	C(149)-C(154)-H(15B)	106.1
C(135)-C(136)-C(137)	135(4)	H(15A)-C(154)-H(15B)	106.3
C(138)-C(137)-C(136)	105(5)	C(155)-O(15)-H(15)	109.5
C(138)-C(137)-H(137)	127.3	C(160)-C(155)-O(15)	117(4)
C(136)-C(137)-H(137)	127.3	C(160)-C(155)-C(156)	122(4)
C(133)-C(138)-C(137)	136(4)	O(15)-C(155)-C(156)	121(3)
C(133)-C(138)-C(143)	115(3)	C(154)-C(156)-C(155)	122(4)
C(137)-C(138)-C(143)	109(4)	C(154)-C(156)-C(157)	118(3)
C(145)-C(143)-C(138)	105(3)	C(155)-C(156)-C(157)	119(3)
C(145)-C(143)-H(14A)	110.7	C(158)-C(157)-C(156)	119(4)

C(158)-C(157)-H(157)	120.4	C(168)-C(169)-C(172)	123(4)
C(156)-C(157)-H(157)	120.4	C(168)-C(169)-C(170)	120(3)
C(157)-C(158)-C(159)	121(4)	C(172)-C(169)-C(170)	117(3)
C(157)-C(158)-C(161)	125(4)	C(171)-C(170)-C(169)	115(3)
C(159)-C(158)-C(161)	104(3)	C(166)-C(171)-C(170)	123(3)
C(158)-C(159)-C(160)	107(3)	C(166)-C(171)-C(176)	125(2)
C(158)-C(159)-H(159)	126.6	C(170)-C(171)-C(176)	112(2)
C(160)-C(159)-H(159)	126.6	C(169)-C(172)-C(173)	116(3)
C(155)-C(160)-C(165)	124(4)	C(169)-C(172)-C(175)	118(4)
C(155)-C(160)-C(159)	120(3)	C(173)-C(172)-C(175)	100(3)
C(165)-C(160)-C(159)	113(3)	C(169)-C(172)-C(174)	108(3)
C(163)-C(161)-C(162)	121(4)	C(173)-C(172)-C(174)	127(4)
C(163)-C(161)-C(158)	122(4)	C(175)-C(172)-C(174)	83(4)
C(162)-C(161)-C(158)	116(3)	C(90)-C(176)-C(171)	112(2)
C(163)-C(161)-C(164)	81(3)	C(90)-C(176)-H(17A)	109.4
C(162)-C(161)-C(164)	101(4)	C(171)-C(176)-H(17A)	109.3
C(158)-C(161)-C(164)	96(3)	C(90)-C(176)-H(17B)	109.0
C(167)-C(165)-C(160)	112(4)	C(171)-C(176)-H(17B)	109.0
C(167)-C(165)-H(16A)	109.1	H(17A)-C(176)-H(17B)	107.8
C(160)-C(165)-H(16A)	109.1	C(177)-O(17)-C(180)	103(3)
C(167)-C(165)-H(16B)	109.1	C(177)-O(17)-Rb(1)	132(3)
C(160)-C(165)-H(16B)	109.1	C(180)-O(17)-Rb(1)	125(3)
H(16A)-C(165)-H(16B)	107.8	O(17)-C(177)-C(178)	106(4)
C(166)-O(16)-Rb(2)	92.2(17)	C(179)-C(178)-C(177)	102(3)
C(171)-C(166)-C(167)	128(3)	C(180)-C(179)-C(178)	110(4)
C(171)-C(166)-O(16)	116(3)	C(179)-C(180)-O(17)	116(5)
C(167)-C(166)-O(16)	116(3)	C(181)-O(18)-C(184)	115(4)
C(171)-C(166)-Rb(2)	107(3)	C(181)-O(18)-Rb(2)	122.4(19)
C(167)-C(166)-Rb(2)	98(3)	C(184)-O(18)-Rb(2)	122(3)
O(16)-C(166)-Rb(2)	62.9(16)	O(18)-C(181)-C(182)	108(4)
C(165)-C(167)-C(166)	124(3)	C(181)-C(182)-C(183)	92(5)
C(165)-C(167)-C(168)	127(4)	C(184)-C(183)-C(182)	107(7)
C(166)-C(167)-C(168)	108(4)	C(183)-C(184)-O(18)	109(7)
C(169)-C(168)-C(167)	125(4)	C(188)-O(19)-C(185)	87(4)
C(169)-C(168)-H(168)	117.7	C(188)-O(19)-O(34)#1	90(3)
C(167)-C(168)-H(168)	117.7	C(185)-O(19)-O(34)#1	153(3)

C(186)-C(185)-O(19)	121(4)	C(198)-C(200)-O(22)	79(3)
C(185)-C(186)-C(187)	95(3)	C(204)-O(23)-C(201)	121(5)
C(188)-C(187)-C(186)	97(4)	C(204)-O(23)-C(202)	87(6)
O(19)-C(188)-C(187)	136(5)	C(201)-O(23)-C(202)	34(4)
C(192)-O(20)-C(189)	114(5)	C(204)-O(23)-O(33)#1	96(4)
C(192)-O(20)-C(191)	35(5)	C(201)-O(23)-O(33)#1	143(4)
C(189)-O(20)-C(191)	79(3)	C(202)-O(23)-O(33)#1	174(7)
C(192)-O(20)-O(31)#1	138(5)	C(202)-C(201)-O(23)	104(6)
C(189)-O(20)-O(31)#1	108(3)	C(201)-C(202)-C(203)	114(7)
C(191)-O(20)-O(31)#1	173(3)	C(201)-C(202)-O(23)	42(4)
O(20)-C(189)-C(190)	107(4)	C(203)-C(202)-O(23)	73(4)
C(189)-C(190)-C(191)	83(4)	C(204)-C(203)-C(202)	99(6)
C(192)-C(191)-C(190)	117(6)	O(23)-C(204)-C(203)	101(5)
C(192)-C(191)-O(20)	30(2)	C(205)-O(24)-C(208)	87(6)
C(190)-C(191)-O(20)	89(4)	C(208)-C(207)-C(206)	162(5)
O(20)-C(192)-C(191)	115(7)	O(24)-C(208)-C(207)	113(3)
C(193)-O(21)-C(196)	114(4)	O(29)#1-O(25)-Rb(1)	104.0(12)
C(193)-O(21)-C(194)	38(2)	O(29)#1-O(25)-Rb(2)	101.4(9)
C(196)-O(21)-C(194)	80(3)	Rb(1)-O(25)-Rb(2)	84.2(8)
C(193)-O(21)-O(29)#1	113(3)	Rb(1)-O(26)-Rb(2)	88.8(12)
C(196)-O(21)-O(29)#1	118(2)	Rb(1)-O(26)-O(30)#1	104.5(11)
C(194)-O(21)-O(29)#1	121(4)	Rb(2)-O(26)-O(30)#1	94.7(15)
C(194)-C(193)-O(21)	93(4)	O(29)#1-O(27)-O(32)#1	91.0(11)
C(193)-C(194)-C(195)	120(6)	O(29)#1-O(27)-Rb(1)	88.5(10)
C(193)-C(194)-O(21)	49(3)	O(32)#1-O(27)-Rb(1)	95.1(8)
C(195)-C(194)-O(21)	84(4)	O(29)#1-O(27)-Rb(2)	91.1(11)
C(194)-C(195)-C(196)	105(7)	O(32)#1-O(27)-Rb(2)	170.1(12)
C(195)-C(196)-O(21)	88(4)	Rb(1)-O(27)-Rb(2)	75.3(6)
C(197)-O(22)-C(200)	81(3)	O(30)#1-O(28)-O(37)	95.4(14)
C(198)-C(197)-O(22)	96(4)	O(30)#1-O(28)-O(36)#2	126.8(15)
C(197)-C(198)-C(200)	100(4)	O(37)-O(28)-O(36)#2	35.5(7)
C(197)-C(198)-C(299)	126(5)	O(30)#1-O(28)-Rb(2)	92.1(8)
C(200)-C(198)-C(299)	39(3)	O(37)-O(28)-Rb(2)	100.2(9)
C(200)-C(299)-C(198)	69(5)	O(36)#2-O(28)-Rb(2)	82.8(7)
C(299)-C(200)-C(198)	72(4)	O(30)#1-O(28)-Rb(1)	95.1(13)
C(299)-C(200)-O(22)	139(7)	O(37)-O(28)-Rb(1)	168.4(11)

O(36)#2-O(28)-Rb(1)	133.0(10)	O(34)#3-O(32)-O(34)	52.4(16)
Rb(2)-O(28)-Rb(1)	74.5(6)	O(33)#1-O(33)-O(33)#3	0(10)
O(31)#1-O(31)-O(37)#1	0(10)	O(34)#1-O(34)-O(34)#3	0(10)
O(31)#1-O(31)-O(35)#2	0(10)	O(34)#1-O(34)-O(32)	0(10)
O(37)#1-O(31)-O(35)#2	74.7(11)	O(34)#3-O(34)-O(32)	49.8(15)
O(32)#1-O(32)-O(34)#3	0(10)	O(36)#1-O(36)-O(37)#2	0(10)
O(32)#1-O(32)-O(34)	0(10)	O(37)#1-O(37)-O(28)	0(10)

Symmetry transformations used to generate equivalent atoms:

#1 x,y,z #2 -x,y,-z+1/2 #3 -x,y,-z+3/2

Anisotropic displacement parameters ($Å^2 x\ 10^3$)for $[Rb_2((4\text{-tert-butylcalix[8]arene-H}))_2(THF)_8(H_2O)_{14}]$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Rb(1)	47	48	104	-2	2	-3
Rb(2)	71	28	95	-3	13	8
O(17)	114	15	94	-31	31	-7
C(177)	50	41	29	47	-47	5
C(178)	81	176	262	114	77	-67
C(179)	13	48	212	4	32	28
C(180)	91	173	27	47	-11	47
O(18)	21	98	186	124	43	46
C(181)	86	10	295	-81	179	-67
C(182)	347	157	172	-131	146	-244
C(183)	398	0	386	-67	241	-85
C(184)	333	0	204	-38	209	-85
O(19)	216	169	141	-91	43	-47
C(185)	220	52	180	-93	142	28
C(186)	188	43	144	-21	-105	84
C(187)	110	0	211	64	-42	10
C(188)	38	45	84	-24	-37	20
O(20)	148	68	175	-98	87	-90
C(189)	49	133	183	82	27	-21

C(190)	293	247	194	-36	212	-35
C(191)	150	32	120	-41	-19	63
C(192)	23	143	136	-51	-93	-6
O(21)	41	104	101	-33	-6	-80
C(193)	78	46	122	-64	43	4
C(194)	729	2000	2000	2000	1313	1348
C(195)	222	24	196	-16	-150	-20
C(196)	11	146	36	-3	-8	-26
O(22)	249	420	178	-120	76	223
C(197)	490	0	198	-15	231	49
C(198)	137	0	235	36	91	29
C(299)	231	66	569	237	152	138
C(200)	137	82	242	129	84	147
O(23)	33	77	152	-17	-21	-1
C(201)	129	26	62	-75	79	-79
C(202)	174	0	1225	-54	219	-8
C(203)	89	2	52	31	45	3
C(204)	176	23	27	34	5	-25
O(24)	129	0	180	-43	140	-48
C(205)	349	1949	249	-306	151	-916
C(206)	393	351	1338	-776	201	-210
C(207)	88	41	254	-98	-52	37
C(208)	95	187	178	79	70	-89
O(25)	56	28	40	-17	-26	8
O(26)	57	47	286	20	75	33
O(27)	103	41	64	68	-12	36
O(28)	30	23	164	-25	31	19
O(29)	134	5	90	50	40	51
O(30)	83	59	143	-69	32	-39
O(31)	45	88	83	-48	-11	22
O(32)	0	17	0	-6	-3	17
O(33)	171	220	79	-55	-85	112
O(35)	131	3	215	2	87	-12
O(36)	3	5	47	23	-9	33
O(37)	0	0	0	-7	-12	9
O(38)	174	884	512	0	292	0

Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3)
for $[Rb_2((4\text{-tert-butylcalix[8]arene-H}))_2(THF)_8(H_2O)_{14}]$.

	x	y	z	U(eq)
H(1)	-609	3329	5856	69
H(3)	-1753	2207	5639	36
H(5)	-756	1491	6600	39
H(11A)	-188	2044	6975	50
H(11B)	-202	2720	6829	50
H(2)	-18	3294	6438	66
H(14)	188	1445	6597	23
H(16)	852	2151	5931	68
H(22A)	409	3416	5461	79
H(22B)	718	2951	5396	79
H(25)	1364	3068	5879	50
H(27)	1847	4069	7023	43
H(33A)	1329	4495	7418	71
H(33B)	950	4703	6991	71
H(36)	2026	5080	7597	64
H(38)	1967	6640	6855	19
H(44A)	984	6494	6068	78
H(44B)	1364	6883	6137	78
H(5A)	395	6625	6134	77
H(47)	1442	7840	6754	21
H(49)	562	8422	6988	27
H(55A)	19	7053	6993	78
H(55B)	1	7729	7126	78
H(6)	-166	6346	5997	122
H(58)	-328	8512	6344	69
H(60)	-1174	7761	5262	70
H(66A)	-754	6344	5318	13
H(66B)	-1102	6717	4970	13

H(7)	-816	5276	5912	101
H(69)	-1716	6852	5076	62
H(71)	-2033	5666	5945	67
H(77A)	-1331	5302	6701	62
H(77B)	-1049	5033	6436	62
H(8)	-1142	4480	5613	102
H(80)	-2006	4627	6568	49
H(82)	-2139	3258	5711	64
H(88A)	-1320	3448	5322	80
H(88B)	-1723	3109	5169	80
H(91)	-1976	5608	2481	61
H(93)	-1981	4065	3054	115
H(99A)	-1389	3744	3800	58
H(99B)	-991	4093	3958	58
H(10)	-229	4132	3444	110
H(102)	-1480	2954	3317	74
H(104)	-577	2345	2771	73
H(11C)	-41	2922	2932	18
H(11D)	-82	3584	3079	18
H(11)	283	4203	4192	67
H(113)	376	2188	3534	60
H(115)	1188	2854	4787	68
H(12A)	870	4321	4873	137
H(12B)	1190	3817	5109	137
H(12)	630	4915	4040	50
H(124)	1790	3762	4891	46
H(126)	2067	4964	4085	44
H(13A)	1504	5362	3316	43
H(13B)	1110	5536	3415	43
H(13)	1035	6366	4303	70
H(135)	2083	5853	3598	114
H(137)	2155	7415	4417	71
H(14A)	1335	7228	4755	60
H(14B)	1711	7629	4817	60
H(14C)	486	7495	4116	92
H(146)	1734	8580	4404	114

H(148)	799	9209	3391	110
H(15A)	169	8526	3127	82
H(15B)	207	7859	3278	82
H(15)	16	7267	3620	60
H(157)	0	9275	3705	84
H(159)	-750	8706	4348	53
H(16A)	-370	7146	4402	58
H(16B)	-612	7615	4592	58
H(168)	-1373	7727	4100	63
H(17A)	-1416	6096	2568	30
H(17B)	-1001	5876	2920	30

Hydrogen bonds for $[Rb_2((4\text{-tert-butylcalix[8]arene-H}))_2(THF)_8(H_2O)_{14}]$ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(2)	0.82	2.21	2.57(3)	106.8
O(1)-H(1)...O(29)#1	0.82	2.40	3.19(3)	160.3
O(2)-H(2)...O(32)#1	0.82	2.90	3.21(2)	104.8
O(5)-H(5A)...O(26)	0.82	2.46	3.19(5)	148.5
O(6)-H(6)...O(26)	0.82	2.26	3.05(4)	161.8
O(7)-H(7)...O(8)	0.82	2.33	3.11(5)	158.0
O(7)-H(7)...O(27)	0.82	2.14	2.53(3)	109.1
O(8)-H(8)...O(27)	0.82	2.95	3.24(3)	103.4
O(10)-H(10)...O(11)	0.82	1.87	2.38(3)	119.3
O(10)-H(10)...O(36)#2	0.82	2.98	3.35(3)	109.9
O(11)-H(11)...O(25)	0.82	2.12	2.87(3)	152.9
O(12)-H(12)...O(28)	0.82	2.53	3.05(2)	122.3
O(12)-H(12)...O(36)#2	0.82	2.73	2.939(19)	96.5
O(13)-H(13)...O(14)	0.82	2.61	2.67(4)	85.3
O(13)-H(13)...O(28)	0.82	2.85	3.34(3)	120.6
O(14)-H(14C)...O(15)	0.82	1.74	2.43(3)	140.2
O(14)-H(14C)...O(30)#1	0.82	2.50	2.89(4)	109.9
O(15)-H(15)...O(37)	0.82	2.69	3.22(2)	123.0

Symmetry transformations used to generate equivalent atoms:

#1 x,y,z #2 -x,y,-z+1/2 #3 -x,y,-z+3/2

V.2.15. [Cs(4-tert-butylcalix[8]arene-H)(THF)$_5$(H$_2$O)$_6$] 15

Empirical formula	C108 H163 Cs O19	
Formula weight	1898.35	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 35.968(7) Å	α= 90°.
	b = 22.437(5) Å	β= 109.72(3)°.
	c = 28.549(6) Å	γ = 90°.
Volume	21688(8) Å^3	
Z	8	
Density (calculated)	1.139 Mg/m^3	
Absorption coefficient	0.403 mm^{-1}	
F(000)	7896	
Theta range for data collection	1.20 to 24.76°.	
Index ranges	-41<=h<=41, -26<=k<=26, -33<=l<=30	
Reflections collected	48218	
Independent reflections	17628 [R(int) = 0.1805]	
Completeness to theta = 24.76°	94.9 %	
Absorption correction	Spherical	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	17628 / 0 / 1140	
Goodness-of-fit on F^2	2.169	
Final R indices [I>2sigma(I)]	R1 = 0.1893, wR2 = 0.4194	
R indices (all data)	R1 = 0.2498, wR2 = 0.4525	

Largest diff. peak and hole 7.731 and -0.930 e.Å^{-3}

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for [Cs(4-tert-butylcalix[8]arene-H)$(THF)_5(H_2O)_6$]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cs(1)	527(1)	308(1)	5534(1)	70(1)
O(1)	27(3)	2041(4)	6236(5)	63(3)
C(1)	172(4)	2599(6)	6205(6)	50(4)
C(2)	50(3)	3074(6)	6416(5)	49(4)
C(3)	208(5)	3623(7)	6385(7)	69(5)
C(4)	485(5)	3728(7)	6145(7)	64(4)
C(5)	592(5)	3221(7)	5932(6)	60(4)
C(6)	444(4)	2663(6)	5959(5)	49(4)
C(7)	675(7)	4332(8)	6146(9)	93(7)
C(8)	375(19)	4845(14)	6030(30)	380(50)
C(9)	831(15)	4557(15)	6664(16)	240(20)
C(10)	972(17)	4336(15)	5910(30)	390(60)
C(11)	588(4)	2157(6)	5738(6)	51(4)
O(2)	555(2)	1217(4)	6449(4)	54(3)
C(12)	931(3)	1408(5)	6431(5)	41(3)
C(13)	948(3)	1853(6)	6101(5)	41(3)
C(14)	1316(4)	2028(6)	6124(5)	47(3)
C(15)	1660(4)	1800(6)	6442(5)	43(3)
C(16)	1631(4)	1359(6)	6772(5)	47(3)
C(17)	1261(3)	1141(5)	6769(5)	36(3)
C(18)	2078(4)	2014(7)	6482(6)	51(4)
C(19)	2056(6)	2419(15)	6016(11)	151(14)
C(20)	2336(5)	1499(10)	6449(10)	104(8)
C(21)	2275(6)	2295(12)	6992(9)	120(9)
C(22)	1254(4)	675(6)	7140(5)	41(3)
O(3)	830(3)	-273(4)	6565(4)	53(3)
C(23)	1229(3)	-351(6)	6772(5)	38(3)
C(24)	1438(3)	104(5)	7079(5)	35(3)

C(25)	1840(4)	7(7)	7310(5)	50(4)
C(26)	2044(4)	-499(6)	7234(5)	43(3)
C(27)	1815(4)	-924(6)	6910(5)	44(3)
C(28)	1403(3)	-871(6)	6682(5)	41(3)
C(29)	2489(4)	-563(7)	7496(6)	57(4)
C(30)	2652(5)	-1142(9)	7347(10)	99(8)
C(31)	2695(5)	10(11)	7424(10)	110(9)
C(32)	2575(6)	-667(18)	8031(8)	172(16)
C(33)	1183(4)	-1357(6)	6327(5)	45(3)
O(4)	442(2)	-1233(4)	6473(4)	55(3)
C(34)	639(3)	-1762(6)	6609(5)	41(3)
C(35)	1013(3)	-1844(6)	6558(5)	39(3)
C(36)	1202(4)	-2380(6)	6688(5)	45(3)
C(37)	1056(4)	-2844(6)	6901(5)	44(3)
C(38)	676(3)	-2761(5)	6944(5)	36(3)
C(39)	476(3)	-2235(6)	6804(5)	37(3)
C(40)	1279(4)	-3435(6)	7062(6)	53(4)
C(41)	1701(5)	-3306(8)	7366(9)	98(8)
C(42)	1092(6)	-3863(8)	7308(9)	93(7)
C(43)	1305(6)	-3758(9)	6600(8)	90(6)
C(44)	63(3)	-2163(6)	6844(5)	41(3)
O(5)	-274(3)	-1187(4)	6196(4)	59(3)
C(45)	-438(3)	-1740(6)	6065(5)	44(3)
C(46)	-277(3)	-2232(6)	6366(5)	42(3)
C(47)	-451(4)	-2785(6)	6212(5)	46(3)
C(48)	-776(4)	-2865(6)	5784(6)	50(4)
C(49)	-931(4)	-2361(6)	5512(5)	46(3)
C(50)	-772(3)	-1795(6)	5654(5)	43(3)
C(51)	-954(4)	-3498(7)	5675(7)	66(5)
C(52)	-656(6)	-3948(9)	5677(11)	118(10)
C(53)	-1290(6)	-3495(10)	5152(8)	100(7)
C(54)	-1137(8)	-3678(10)	6061(11)	125(10)
C(55)	-971(4)	-1241(7)	5352(5)	54(4)
O(6)	-794(3)	-364(5)	6135(4)	69(3)
C(56)	-1180(3)	-531(6)	5911(5)	45(4)
C(57)	-1274(3)	-961(6)	5533(5)	40(3)

C(58)	-1668(4)	-1151(6)	5321(5)	46(3)
C(59)	-1970(4)	-928(6)	5466(5)	45(3)
C(60)	-1868(4)	-483(6)	5844(6)	46(3)
C(61)	-1461(4)	-278(6)	6068(5)	48(3)
C(62)	-2398(4)	-1116(7)	5247(5)	49(4)
C(63)	-2445(6)	-1784(11)	5154(12)	124(10)
C(64)	-2590(6)	-795(13)	4794(11)	161(16)
C(65)	-2620(6)	-1035(16)	5615(12)	173(16)
C(66)	-1382(4)	174(6)	6465(6)	58(4)
O(7)	-1045(3)	1038(4)	6006(4)	59(3)
C(67)	-1423(4)	1155(6)	6012(5)	46(3)
C(68)	-1593(4)	757(6)	6251(5)	48(4)
C(69)	-1956(4)	907(8)	6278(6)	61(4)
C(70)	-2175(4)	1398(8)	6061(6)	55(4)
C(71)	-2005(4)	1767(7)	5794(5)	53(4)
C(72)	-1632(4)	1662(7)	5772(5)	51(4)
C(73)	-2584(5)	1525(10)	6097(7)	76(5)
C(74)	-2540(9)	1680(30)	6581(18)	330(50)
C(75)	-2759(9)	2087(18)	5870(18)	210(20)
C(76)	-2858(7)	1037(17)	5910(20)	250(30)
C(77)	-1461(4)	2096(7)	5489(6)	55(4)
O(8)	-669(3)	2029(5)	6141(5)	66(3)
C(78)	-852(4)	2576(6)	6112(6)	46(3)
C(79)	-1251(4)	2613(7)	5801(5)	49(3)
C(80)	-1447(4)	3155(7)	5784(7)	65(5)
C(81)	-1268(5)	3643(7)	6060(7)	67(5)
C(82)	-873(5)	3570(7)	6356(7)	63(4)
C(83)	-664(4)	3061(7)	6388(5)	51(3)
C(84)	-1483(6)	4231(9)	6040(12)	109(9)
C(85)	-1931(7)	4130(12)	5915(17)	189(19)
C(86)	-1447(12)	4562(14)	5511(19)	240(20)
C(87)	-1299(13)	4603(16)	6469(14)	240(30)
C(88)	-231(4)	3021(7)	6705(6)	59(4)
O(9)	1326(4)	-138(8)	5515(6)	103(5)
C(89)	1681(6)	119(10)	5805(8)	92(6)
C(90)	2000(8)	-267(16)	5811(10)	130(10)

C(91)	1852(10)	-594(16)	5356(15)	178(17)
C(92)	1419(8)	-574(10)	5223(9)	103(7)
O(10)	1072(6)	-1323(16)	7958(9)	183(12)
C(93)	1442(10)	1326(13)	2908(10)	125(10)
C(94)	1674(10)	1812(15)	3204(17)	171(17)
C(95)	1510(30)	2150(20)	3426(19)	340(60)
C(96)	1095(13)	2000(30)	3214(17)	230(30)
O(11)	-684(7)	2608(16)	4716(10)	187(11)
C(97)	820(20)	6870(20)	5100(30)	270(40)
C(98)	670(20)	6420(20)	5126(18)	240(30)
C(99)	328(12)	6520(30)	5269(18)	210(20)
C(100)	368(10)	7160(20)	5479(14)	169(16)
O(12)	-1015(6)	2117(13)	7337(8)	168(10)
C(101)	1405(7)	7838(13)	2914(9)	118(10)
C(102)	1527(13)	7283(16)	2800(20)	220(20)
C(103)	1212(16)	6960(30)	2420(20)	300(40)
C(104)	864(15)	7410(30)	2384(12)	280(40)
O(13)	0	4760(30)	7500	480(80)
C(105)	265(17)	5190(30)	7840(30)	380(60)
C(106)	148(16)	5680(30)	7790(20)	300(40)
O(14)	-406(5)	387(6)	5570(6)	100(4)
O(15)	84(4)	-964(6)	5347(5)	83(4)
O(16)	-416(2)	1225(3)	7315(2)	15(2)
O(17)	-436(5)	1582(7)	5233(7)	123(6)
O(18)	-25(4)	417(8)	6696(3)	138(8)
O(20)	434(1)	421(2)	2564(2)	3(1)

Bond lengths [Å] and angles [°] for [Cs(4-tert-butylcalix[8]arene-H)$(THF)_5(H_2O)_6$].

Cs(1)-O(9)	3.061(15)	Cs(1)-C(12)	3.501(13)
Cs(1)-O(3)	3.064(10)	Cs(1)-C(23)	3.877(12)
Cs(1)-O(15)#1	3.098(12)	Cs(1)-C(13)	3.909(13)
Cs(1)-O(15)	3.224(13)	Cs(1)-Cs(1)#1	4.217(3)
Cs(1)-O(2)	3.289(11)	O(1)-C(1)	1.370(17)
Cs(1)-O(14)	3.397(15)	O(1)-O(8)	2.426(13)
Cs(1)-O(14)#1	3.410(16)	O(1)-O(2)	2.570(13)

O(1)-O(17)#2	2.97(2)	C(26)-C(29)	1.527(19)
C(1)-C(6)	1.39(2)	C(27)-C(28)	1.408(17)
C(1)-C(2)	1.37(2)	C(28)-C(33)	1.517(18)
C(2)-C(3)	1.37(2)	C(29)-C(32)	1.47(3)
C(2)-C(88)	1.51(2)	C(29)-C(31)	1.53(2)
C(3)-C(4)	1.41(3)	C(29)-C(30)	1.54(2)
C(4)-C(5)	1.40(2)	C(33)-C(35)	1.509(18)
C(4)-C(7)	1.52(2)	O(4)-C(34)	1.371(15)
C(5)-C(6)	1.37(2)	O(4)-O(5)	2.426(12)
C(6)-C(11)	1.47(2)	O(4)-O(15)	3.090(16)
C(7)-C(9)	1.48(4)	C(34)-C(35)	1.412(18)
C(7)-C(10)	1.44(5)	C(34)-C(39)	1.416(18)
C(7)-C(8)	1.53(4)	C(35)-C(36)	1.370(18)
C(11)-C(13)	1.520(17)	C(36)-C(37)	1.39(2)
O(2)-C(12)	1.435(15)	C(37)-C(38)	1.424(19)
O(2)-O(18)#2	3.009(12)	C(37)-C(40)	1.538(18)
O(2)-O(3)	3.471(13)	C(38)-C(39)	1.370(16)
C(12)-C(17)	1.389(17)	C(39)-C(44)	1.537(16)
C(12)-C(13)	1.391(19)	C(40)-C(42)	1.48(2)
C(13)-C(14)	1.360(18)	C(40)-C(41)	1.50(2)
C(14)-C(15)	1.363(19)	C(40)-C(43)	1.53(3)
C(15)-C(16)	1.395(19)	C(44)-C(46)	1.500(18)
C(15)-C(18)	1.543(18)	O(5)-C(45)	1.371(15)
C(16)-C(17)	1.414(17)	O(5)-O(6)	2.592(15)
C(17)-C(22)	1.494(19)	O(5)-O(15)	3.144(18)
C(18)-C(20)	1.51(2)	C(45)-C(50)	1.372(17)
C(18)-C(21)	1.52(3)	C(45)-C(46)	1.402(18)
C(18)-C(19)	1.59(3)	C(46)-C(47)	1.392(18)
C(22)-C(24)	1.479(17)	C(47)-C(48)	1.39(2)
O(3)-C(23)	1.368(14)	C(48)-C(49)	1.379(19)
O(3)-O(4)	2.530(13)	C(48)-C(51)	1.55(2)
C(23)-C(28)	1.387(19)	C(49)-C(50)	1.395(19)
C(23)-C(24)	1.391(18)	C(50)-C(55)	1.543(18)
C(24)-C(25)	1.390(18)	C(51)-C(52)	1.47(3)
C(25)-C(26)	1.41(2)	C(51)-C(54)	1.52(3)
C(26)-C(27)	1.390(18)	C(51)-C(53)	1.57(3)

C(55)-C(57)	1.50(2)	C(80)-C(81)	1.37(2)
O(6)-C(56)	1.371(15)	C(81)-C(82)	1.40(2)
O(6)-O(14)	2.984(19)	C(81)-C(84)	1.52(2)
O(6)-O(18)#2	3.210(11)	C(82)-C(83)	1.35(2)
O(6)-O(7)	3.260(15)	C(83)-C(88)	1.52(2)
C(56)-C(61)	1.36(2)	C(84)-C(87)	1.44(4)
C(56)-C(57)	1.40(2)	C(84)-C(85)	1.55(3)
C(57)-C(58)	1.404(17)	C(84)-C(86)	1.73(5)
C(58)-C(59)	1.38(2)	O(9)-C(89)	1.39(2)
C(59)-C(60)	1.423(19)	O(9)-C(92)	1.40(3)
C(59)-C(62)	1.512(17)	C(89)-C(90)	1.43(3)
C(60)-C(61)	1.461(18)	C(90)-C(91)	1.43(4)
C(61)-C(66)	1.47(2)	C(91)-C(92)	1.47(4)
C(62)-C(64)	1.44(2)	O(10)-C(93)#3	1.38(3)
C(62)-C(65)	1.53(3)	O(10)-C(96)#3	1.68(6)
C(62)-C(63)	1.52(3)	O(10)-O(20)	15.29(3)
C(66)-C(68)	1.53(2)	C(93)-O(10)#4	1.38(3)
O(7)-C(67)	1.391(17)	C(93)-C(94)	1.46(4)
O(7)-O(14)	3.299(18)	C(94)-C(95)	1.25(6)
C(67)-C(68)	1.39(2)	C(95)-C(96)	1.46(9)
C(67)-C(72)	1.407(19)	C(96)-O(10)#4	1.68(6)
C(68)-C(69)	1.37(2)	O(11)-C(97)#5	1.44(5)
C(69)-C(70)	1.38(2)	O(11)-C(100)#5	1.52(4)
C(70)-C(71)	1.40(2)	O(11)-O(17)#2	2.72(4)
C(70)-C(73)	1.53(2)	C(97)-C(98)	1.17(6)
C(71)-C(72)	1.38(2)	C(97)-O(11)#5	1.44(5)
C(72)-C(77)	1.52(2)	C(98)-C(99)	1.42(6)
C(73)-C(74)	1.38(4)	C(99)-C(100)	1.54(6)
C(73)-C(76)	1.45(3)	C(100)-O(11)#5	1.52(4)
C(73)-C(75)	1.46(4)	O(12)-C(104)#5	1.33(5)
C(77)-C(79)	1.50(2)	O(12)-C(101)#5	1.35(3)
O(8)-C(78)	1.381(16)	O(12)-O(16)#2	2.96(2)
O(8)-O(17)#2	3.15(2)	C(101)-C(102)	1.39(4)
C(78)-C(83)	1.380(19)	C(101)-O(12)#5	1.35(3)
C(78)-C(79)	1.414(19)	C(102)-C(103)	1.47(5)
C(79)-C(80)	1.40(2)	C(103)-C(104)	1.58(8)

C(104)-O(12)#5	1.33(5)	O(15)-Cs(1)#1	3.098(12)
O(13)-C(105)	1.48(6)	O(16)-O(16)#2	0.00(2)
O(13)-C(105)#6	1.48(6)	O(16)-O(18)#6	3.279(10)
C(105)-C(106)	1.15(6)	O(17)-O(17)#2	0.00(4)
C(106)-C(106)#6	1.63(10)	O(17)-O(15)#1	2.77(2)
O(14)-O(17)#2	2.84(2)	O(18)-O(18)#2	0.00(2)
O(14)-O(18)#2	3.041(18)	O(18)-O(20)#7	11.746(10)
O(14)-Cs(1)#1	3.410(16)	O(20)-O(20)#7	3.021(10)

O(9)-Cs(1)-O(3)	81.2(4)	O(14)#1-Cs(1)-C(12)	153.0(3)
O(9)-Cs(1)-O(15)#1	123.7(4)	O(9)-Cs(1)-C(23)	64.2(4)
O(3)-Cs(1)-O(15)#1	155.1(3)	O(3)-Cs(1)-C(23)	18.4(3)
O(9)-Cs(1)-O(15)	97.0(4)	O(15)#1-Cs(1)-C(23)	170.8(3)
O(3)-Cs(1)-O(15)	77.1(3)	O(15)-Cs(1)-C(23)	86.9(3)
O(15)#1-Cs(1)-O(15)	96.4(3)	O(2)-Cs(1)-C(23)	71.2(3)
O(9)-Cs(1)-O(2)	116.1(3)	O(14)-Cs(1)-C(23)	109.8(3)
O(3)-Cs(1)-O(2)	66.1(3)	O(14)#1-Cs(1)-C(23)	120.5(3)
O(15)#1-Cs(1)-O(2)	100.0(3)	C(12)-Cs(1)-C(23)	67.7(3)
O(15)-Cs(1)-O(2)	124.1(3)	O(9)-Cs(1)-C(13)	93.6(3)
O(9)-Cs(1)-O(14)	163.9(4)	O(3)-Cs(1)-C(13)	91.0(3)
O(3)-Cs(1)-O(14)	91.6(3)	O(15)#1-Cs(1)-C(13)	89.1(3)
O(15)#1-Cs(1)-O(14)	64.0(4)	O(15)-Cs(1)-C(13)	162.7(3)
O(15)-Cs(1)-O(14)	67.3(3)	O(2)-Cs(1)-C(13)	38.5(3)
O(2)-Cs(1)-O(14)	73.1(3)	O(14)-Cs(1)-C(13)	101.0(3)
O(9)-Cs(1)-O(14)#1	70.2(4)	O(14)#1-Cs(1)-C(13)	134.3(3)
O(3)-Cs(1)-O(14)#1	125.9(3)	C(12)-Cs(1)-C(13)	20.7(3)
O(15)#1-Cs(1)-O(14)#1	68.5(4)	C(23)-Cs(1)-C(13)	85.4(3)
O(15)-Cs(1)-O(14)#1	62.6(3)	O(9)-Cs(1)-Cs(1)#1	120.0(2)
O(2)-Cs(1)-O(14)#1	167.9(3)	O(3)-Cs(1)-Cs(1)#1	119.75(18)
O(14)-Cs(1)-O(14)#1	103.4(3)	O(15)#1-Cs(1)-Cs(1)#1	49.5(2)
O(9)-Cs(1)-C(12)	94.8(3)	O(15)-Cs(1)-Cs(1)#1	46.9(2)
O(3)-Cs(1)-C(12)	70.8(3)	O(2)-Cs(1)-Cs(1)#1	123.82(16)
O(15)#1-Cs(1)-C(12)	105.4(3)	O(14)-Cs(1)-Cs(1)#1	51.9(3)
O(15)-Cs(1)-C(12)	143.6(3)	O(14)#1-Cs(1)-Cs(1)#1	51.6(3)
O(2)-Cs(1)-C(12)	24.1(3)	C(12)-Cs(1)-Cs(1)#1	144.1(2)
O(14)-Cs(1)-C(12)	96.4(3)	C(23)-Cs(1)-Cs(1)#1	133.16(19)

C(13)-Cs(1)-Cs(1)#1	135.93(17)	O(18)#2-O(2)-O(3)	66.4(5)
C(1)-O(1)-O(8)	113.8(8)	Cs(1)-O(2)-O(3)	53.8(2)
C(1)-O(1)-O(2)	113.9(8)	C(17)-C(12)-C(13)	123.8(11)
O(8)-O(1)-O(2)	132.1(5)	C(17)-C(12)-O(2)	116.3(11)
C(1)-O(1)-O(17)#2	110.9(10)	C(13)-C(12)-O(2)	119.8(11)
O(8)-O(1)-O(17)#2	70.6(5)	C(17)-C(12)-Cs(1)	104.4(8)
O(2)-O(1)-O(17)#2	96.2(6)	C(13)-C(12)-Cs(1)	96.4(8)
O(1)-C(1)-C(6)	118.3(13)	O(2)-C(12)-Cs(1)	69.6(6)
O(1)-C(1)-C(2)	120.0(14)	C(14)-C(13)-C(12)	116.1(11)
C(6)-C(1)-C(2)	121.8(14)	C(14)-C(13)-C(11)	119.8(13)
C(3)-C(2)-C(1)	118.0(16)	C(12)-C(13)-C(11)	124.0(12)
C(3)-C(2)-C(88)	118.3(15)	C(14)-C(13)-Cs(1)	120.7(9)
C(1)-C(2)-C(88)	123.5(14)	C(12)-C(13)-Cs(1)	62.9(7)
C(2)-C(3)-C(4)	124.0(17)	C(11)-C(13)-Cs(1)	89.6(7)
C(5)-C(4)-C(3)	114.7(15)	C(13)-C(14)-C(15)	125.0(13)
C(5)-C(4)-C(7)	122.4(17)	C(14)-C(15)-C(16)	117.2(13)
C(3)-C(4)-C(7)	122.8(17)	C(14)-C(15)-C(18)	125.1(12)
C(6)-C(5)-C(4)	123.1(17)	C(16)-C(15)-C(18)	117.6(12)
C(5)-C(6)-C(1)	118.3(15)	C(15)-C(16)-C(17)	121.8(12)
C(5)-C(6)-C(11)	118.7(15)	C(12)-C(17)-C(16)	116.0(12)
C(1)-C(6)-C(11)	123.0(13)	C(12)-C(17)-C(22)	125.3(11)
C(9)-C(7)-C(10)	112(4)	C(16)-C(17)-C(22)	118.7(11)
C(9)-C(7)-C(4)	109(2)	C(20)-C(18)-C(15)	111.3(13)
C(10)-C(7)-C(4)	114(2)	C(20)-C(18)-C(21)	106.1(17)
C(9)-C(7)-C(8)	88(4)	C(15)-C(18)-C(21)	109.2(13)
C(10)-C(7)-C(8)	118(3)	C(20)-C(18)-C(19)	104.0(19)
C(4)-C(7)-C(8)	113(2)	C(15)-C(18)-C(19)	110.1(12)
C(6)-C(11)-C(13)	113.3(12)	C(21)-C(18)-C(19)	116.0(18)
C(12)-O(2)-O(1)	114.0(7)	C(24)-C(22)-C(17)	114.0(11)
C(12)-O(2)-O(18)#2	157.1(8)	C(23)-O(3)-O(4)	113.4(8)
O(1)-O(2)-O(18)#2	88.2(5)	C(23)-O(3)-Cs(1)	116.7(8)
C(12)-O(2)-Cs(1)	86.2(7)	O(4)-O(3)-Cs(1)	104.9(4)
O(1)-O(2)-Cs(1)	115.8(4)	C(23)-O(3)-O(2)	112.9(8)
O(18)#2-O(2)-Cs(1)	88.8(3)	O(4)-O(3)-O(2)	132.9(4)
C(12)-O(2)-O(3)	92.8(7)	Cs(1)-O(3)-O(2)	60.0(2)
O(1)-O(2)-O(3)	151.5(4)	O(3)-C(23)-C(28)	120.1(11)

O(3)-C(23)-C(24)	116.4(12)	C(36)-C(37)-C(38)	116.9(11)
C(28)-C(23)-C(24)	123.5(10)	C(36)-C(37)-C(40)	122.6(13)
O(3)-C(23)-Cs(1)	44.9(6)	C(38)-C(37)-C(40)	120.5(12)
C(28)-C(23)-Cs(1)	110.3(8)	C(39)-C(38)-C(37)	120.9(12)
C(24)-C(23)-Cs(1)	110.0(8)	C(38)-C(39)-C(34)	120.8(11)
C(25)-C(24)-C(23)	116.1(12)	C(38)-C(39)-C(44)	119.8(11)
C(25)-C(24)-C(22)	121.0(12)	C(34)-C(39)-C(44)	119.4(11)
C(23)-C(24)-C(22)	122.8(11)	C(42)-C(40)-C(41)	112.6(16)
C(24)-C(25)-C(26)	124.4(13)	C(42)-C(40)-C(37)	115.1(13)
C(27)-C(26)-C(25)	115.6(11)	C(41)-C(40)-C(37)	109.4(12)
C(27)-C(26)-C(29)	123.4(12)	C(42)-C(40)-C(43)	105.7(16)
C(25)-C(26)-C(29)	120.9(12)	C(41)-C(40)-C(43)	104.2(16)
C(26)-C(27)-C(28)	123.1(13)	C(37)-C(40)-C(43)	109.1(13)
C(23)-C(28)-C(27)	117.1(11)	C(46)-C(44)-C(39)	115.6(11)
C(23)-C(28)-C(33)	124.2(11)	C(45)-O(5)-O(4)	111.4(8)
C(27)-C(28)-C(33)	118.5(12)	C(45)-O(5)-O(6)	113.0(8)
C(32)-C(29)-C(31)	109(2)	O(4)-O(5)-O(6)	135.6(5)
C(32)-C(29)-C(26)	110.0(15)	C(45)-O(5)-O(15)	100.6(9)
C(31)-C(29)-C(26)	109.2(13)	O(4)-O(5)-O(15)	65.9(4)
C(32)-C(29)-C(30)	100.8(19)	O(6)-O(5)-O(15)	108.0(5)
C(31)-C(29)-C(30)	115.1(17)	O(5)-C(45)-C(50)	119.3(12)
C(26)-C(29)-C(30)	112.1(12)	O(5)-C(45)-C(46)	119.5(11)
C(35)-C(33)-C(28)	115.1(11)	C(50)-C(45)-C(46)	121.1(12)
C(34)-O(4)-O(5)	121.4(8)	C(47)-C(46)-C(45)	117.3(12)
C(34)-O(4)-O(3)	119.5(7)	C(47)-C(46)-C(44)	121.5(12)
O(5)-O(4)-O(3)	119.0(5)	C(45)-C(46)-C(44)	121.3(12)
C(34)-O(4)-O(15)	117.2(9)	C(48)-C(47)-C(46)	123.3(13)
O(5)-O(4)-O(15)	68.3(4)	C(47)-C(48)-C(49)	116.7(13)
O(3)-O(4)-O(15)	87.9(4)	C(47)-C(48)-C(51)	117.7(13)
O(4)-C(34)-C(35)	120.3(12)	C(49)-C(48)-C(51)	125.4(13)
O(4)-C(34)-C(39)	121.1(12)	C(48)-C(49)-C(50)	122.3(12)
C(35)-C(34)-C(39)	118.6(12)	C(45)-C(50)-C(49)	119.1(12)
C(36)-C(35)-C(34)	119.3(12)	C(45)-C(50)-C(55)	120.7(13)
C(36)-C(35)-C(33)	121.1(12)	C(49)-C(50)-C(55)	120.2(11)
C(34)-C(35)-C(33)	119.5(12)	C(52)-C(51)-C(54)	108(2)
C(35)-C(36)-C(37)	123.3(13)	C(52)-C(51)-C(48)	112.2(13)

C(54)-C(51)-C(48)	110.5(15)	C(67)-O(7)-O(6)	114.5(8)
C(52)-C(51)-C(53)	109.3(17)	C(67)-O(7)-O(14)	153.7(9)
C(54)-C(51)-C(53)	108.0(17)	O(6)-O(7)-O(14)	54.1(4)
C(48)-C(51)-C(53)	108.7(15)	O(7)-C(67)-C(68)	118.8(12)
C(57)-C(55)-C(50)	113.6(11)	O(7)-C(67)-C(72)	120.6(13)
C(56)-O(6)-O(5)	115.6(9)	C(68)-C(67)-C(72)	120.6(13)
C(56)-O(6)-O(14)	119.2(9)	C(69)-C(68)-C(67)	117.1(14)
O(5)-O(6)-O(14)	88.9(5)	C(69)-C(68)-C(66)	122.6(15)
C(56)-O(6)-O(18)#2	161.6(10)	C(67)-C(68)-C(66)	120.3(14)
O(5)-O(6)-O(18)#2	82.8(5)	C(68)-C(69)-C(70)	125.7(16)
O(14)-O(6)-O(18)#2	58.7(4)	C(71)-C(70)-C(69)	115.4(14)
C(56)-O(6)-O(7)	90.7(8)	C(71)-C(70)-C(73)	122.2(15)
O(5)-O(6)-O(7)	149.5(5)	C(69)-C(70)-C(73)	122.4(16)
O(14)-O(6)-O(7)	63.6(4)	C(70)-C(71)-C(72)	122.3(14)
O(18)#2-O(6)-O(7)	71.8(4)	C(71)-C(72)-C(67)	118.8(14)
C(61)-C(56)-O(6)	118.8(13)	C(71)-C(72)-C(77)	119.1(14)
C(61)-C(56)-C(57)	121.9(11)	C(67)-C(72)-C(77)	122.2(13)
O(6)-C(56)-C(57)	119.3(14)	C(74)-C(73)-C(76)	113(4)
C(56)-C(57)-C(58)	119.1(13)	C(74)-C(73)-C(75)	96(3)
C(56)-C(57)-C(55)	122.8(11)	C(76)-C(73)-C(75)	111(3)
C(58)-C(57)-C(55)	118.1(13)	C(74)-C(73)-C(70)	109.0(18)
C(59)-C(58)-C(57)	122.8(13)	C(76)-C(73)-C(70)	112.8(19)
C(58)-C(59)-C(60)	116.9(12)	C(75)-C(73)-C(70)	114.2(19)
C(58)-C(59)-C(62)	124.6(13)	C(79)-C(77)-C(72)	113.0(13)
C(60)-C(59)-C(62)	118.4(13)	C(78)-O(8)-O(1)	116.8(8)
C(59)-C(60)-C(61)	121.2(14)	C(78)-O(8)-O(17)#2	119.2(10)
C(56)-C(61)-C(60)	118.0(13)	O(1)-O(8)-O(17)#2	62.8(5)
C(56)-C(61)-C(66)	124.7(12)	O(8)-C(78)-C(83)	122.4(12)
C(60)-C(61)-C(66)	117.3(14)	O(8)-C(78)-C(79)	116.9(12)
C(64)-C(62)-C(65)	111(2)	C(83)-C(78)-C(79)	120.7(13)
C(64)-C(62)-C(63)	110(2)	C(78)-C(79)-C(80)	117.8(14)
C(65)-C(62)-C(63)	100.6(19)	C(78)-C(79)-C(77)	121.9(13)
C(64)-C(62)-C(59)	110.2(13)	C(80)-C(79)-C(77)	120.3(13)
C(65)-C(62)-C(59)	112.1(13)	C(81)-C(80)-C(79)	122.7(14)
C(63)-C(62)-C(59)	112.7(14)	C(80)-C(81)-C(82)	116.0(14)
C(61)-C(66)-C(68)	109.8(11)	C(80)-C(81)-C(84)	122.4(16)

C(82)-C(81)-C(84)	121.6(17)	C(104)#5-O(12)-O(16)#2	114(3)
C(83)-C(82)-C(81)	124.6(15)	C(101)#5-O(12)-O(16)#2	132(2)
C(82)-C(83)-C(78)	118.4(13)	C(102)-C(101)-O(12)#5	107(3)
C(82)-C(83)-C(88)	121.7(14)	C(101)-C(102)-C(103)	113(4)
C(78)-C(83)-C(88)	119.9(13)	C(102)-C(103)-C(104)	98(4)
C(87)-C(84)-C(81)	113(2)	O(12)#5-C(104)-C(103)	108(3)
C(87)-C(84)-C(85)	115(3)	C(105)-O(13)-C(105)#6	97(5)
C(81)-C(84)-C(85)	111.1(18)	C(106)-C(105)-O(13)	114(5)
C(87)-C(84)-C(86)	110(3)	C(105)-C(106)-C(106)#6	103(5)
C(81)-C(84)-C(86)	103(2)	O(17)#2-O(14)-O(6)	138.3(7)
C(85)-C(84)-C(86)	104(3)	O(17)#2-O(14)-O(18)#2	106.8(7)
C(2)-C(88)-C(83)	114.4(13)	O(6)-O(14)-O(18)#2	64.4(4)
C(89)-O(9)-C(92)	106.7(17)	O(17)#2-O(14)-O(7)	76.0(5)
C(89)-O(9)-Cs(1)	122.2(13)	O(6)-O(14)-O(7)	62.3(4)
C(92)-O(9)-Cs(1)	131.0(13)	O(18)#2-O(14)-O(7)	73.4(5)
O(9)-C(89)-C(90)	109(2)	O(17)#2-O(14)-Cs(1)	87.9(5)
C(89)-C(90)-C(91)	104(2)	O(6)-O(14)-Cs(1)	129.3(5)
C(92)-C(91)-C(90)	105(3)	O(18)#2-O(14)-Cs(1)	86.3(5)
C(91)-C(92)-O(9)	108(2)	O(7)-O(14)-Cs(1)	148.8(5)
C(93)#3-O(10)-C(96)#3	98(2)	O(17)#2-O(14)-Cs(1)#1	98.1(6)
C(93)#3-O(10)-O(20)	73.8(17)	O(6)-O(14)-Cs(1)#1	107.7(5)
C(96)#3-O(10)-O(20)	129.8(16)	O(18)#2-O(14)-Cs(1)#1	149.1(6)
O(10)#4-C(93)-C(94)	109(3)	O(7)-O(14)-Cs(1)#1	131.5(5)
C(95)-C(94)-C(93)	118(5)	Cs(1)-O(14)-Cs(1)#1	76.6(3)
C(94)-C(95)-C(96)	104(5)	Cs(1)#1-O(15)-O(4)	150.4(5)
C(95)-C(96)-O(10)#4	106(3)	Cs(1)#1-O(15)-O(5)	109.8(4)
C(97)#5-O(11)-C(100)#5	104(4)	O(4)-O(15)-O(5)	45.8(3)
C(97)#5-O(11)-O(17)#2	125(3)	Cs(1)#1-O(15)-Cs(1)	83.6(3)
C(100)#5-O(11)-O(17)#2	110(2)	O(4)-O(15)-Cs(1)	89.5(4)
C(98)-C(97)-O(11)#5	117(6)	O(5)-O(15)-Cs(1)	108.8(4)
C(97)-C(98)-C(99)	110(6)	O(16)#2-O(16)-O(18)#6	0(10)
C(98)-C(99)-C(100)	107(4)	O(17)#2-O(17)-O(15)#1	0(10)
O(11)#5-C(100)-C(99)	99(4)	O(18)#2-O(18)-O(20)#7	0(10)
C(104)#5-O(12)-C(101)#5	114(4)	O(20)#7-O(20)-O(10)	85.4(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x,-y,-z+1 #2 x,y,z #3 x,-y,z+1/2 #4 x,-y,z-1/2

#5 -x,-y+1,-z+1 #6 -x,y,-z+3/2 #7 -x,y,-z+1/2

Anisotropic displacement parameters ($Å^2 x\ 10^3$)for [Cs(4-tert-butylcalix[8]arene-H)$(THF)_5(H_2O)_6$]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cs(1)	73(1)	62(1)	67(1)	1(1)	14(1)	-4(1)
O(1)	40(5)	35(5)	118(9)	-4(6)	34(6)	-1(4)
C(1)	31(6)	40(8)	74(10)	-8(7)	11(7)	3(6)
C(2)	28(6)	50(9)	57(8)	3(7)	-1(6)	-4(5)
C(3)	81(11)	37(9)	82(11)	5(8)	18(10)	-11(8)
C(4)	63(10)	50(10)	82(11)	2(9)	31(9)	-8(8)
C(5)	70(9)	47(9)	64(9)	1(8)	25(8)	-6(7)
C(6)	40(7)	47(9)	56(8)	6(7)	10(7)	-7(6)
C(7)	108(16)	42(10)	137(19)	-9(11)	55(15)	-22(10)
C(8)	420(80)	70(20)	840(160)	170(50)	440(100)	100(30)
C(9)	330(60)	100(30)	250(40)	-80(30)	60(40)	-130(30)
C(10)	400(80)	80(20)	900(170)	-130(50)	490(110)	-130(40)
C(11)	45(7)	42(8)	59(8)	-11(7)	8(6)	0(6)
O(2)	36(5)	37(5)	95(7)	0(5)	30(5)	-11(4)
C(12)	33(6)	29(7)	66(8)	-1(6)	25(6)	-9(5)
C(13)	27(6)	39(7)	51(7)	-2(6)	7(6)	-1(5)
C(14)	47(8)	48(8)	53(8)	0(7)	27(7)	-9(6)
C(15)	45(7)	39(7)	47(7)	3(6)	17(6)	-6(6)
C(16)	43(7)	41(8)	51(8)	-3(6)	7(6)	-14(6)
C(17)	23(6)	29(7)	60(8)	-9(6)	20(6)	-5(5)
C(18)	37(7)	57(9)	69(9)	6(7)	29(7)	-7(6)
C(19)	63(12)	220(30)	180(20)	120(20)	51(15)	-24(15)
C(20)	51(10)	92(16)	170(20)	-8(15)	45(13)	-13(10)
C(21)	82(14)	140(20)	125(18)	-44(17)	24(13)	-83(15)
C(22)	33(6)	41(8)	58(8)	5(6)	28(6)	-3(5)
O(3)	37(5)	42(5)	78(6)	1(5)	16(5)	3(4)
C(23)	25(5)	39(7)	53(7)	14(6)	18(5)	0(5)
C(24)	32(6)	34(7)	45(7)	8(5)	22(6)	3(5)

C(25)	41(7)	48(8)	58(8)	5(7)	10(7)	-14(6)
C(26)	39(7)	30(7)	63(8)	0(6)	21(6)	-4(5)
C(27)	34(6)	44(8)	56(8)	2(7)	19(6)	6(6)
C(28)	26(6)	43(8)	58(8)	-7(6)	20(6)	-13(5)
C(29)	48(8)	45(8)	70(10)	-6(7)	10(7)	-7(6)
C(30)	39(8)	87(14)	150(20)	-8(14)	8(11)	13(8)
C(31)	35(9)	124(18)	150(20)	41(16)	11(11)	-19(10)
C(32)	54(12)	370(50)	84(15)	100(20)	7(11)	64(19)
C(33)	39(7)	48(8)	58(8)	1(7)	29(7)	-10(6)
O(4)	30(4)	45(6)	90(7)	6(5)	22(5)	1(4)
C(34)	33(6)	34(7)	46(7)	-3(6)	-1(6)	-9(5)
C(35)	25(6)	42(7)	51(7)	-6(6)	13(6)	-3(5)
C(36)	34(6)	37(8)	63(8)	-9(6)	14(6)	1(5)
C(37)	50(8)	33(7)	49(7)	-3(6)	16(6)	11(6)
C(38)	24(5)	33(7)	49(7)	7(6)	11(5)	-2(5)
C(39)	18(5)	41(7)	48(7)	0(6)	7(5)	3(5)
C(40)	44(7)	40(8)	70(9)	-1(7)	13(7)	5(6)
C(41)	57(10)	51(10)	160(20)	-15(12)	2(12)	2(8)
C(42)	91(13)	60(12)	146(19)	29(12)	61(14)	19(10)
C(43)	87(13)	70(13)	110(16)	-13(11)	31(12)	26(10)
C(44)	28(6)	52(8)	47(7)	6(6)	17(6)	16(5)
O(5)	41(5)	51(6)	75(7)	10(5)	5(5)	-6(4)
C(45)	26(6)	40(7)	61(8)	-4(6)	7(6)	-15(5)
C(46)	27(6)	32(7)	66(9)	2(6)	16(6)	4(5)
C(47)	29(6)	47(8)	65(9)	6(7)	18(6)	4(5)
C(48)	40(7)	39(8)	73(10)	2(7)	23(7)	3(6)
C(49)	25(6)	55(9)	56(8)	-3(7)	10(6)	-6(6)
C(50)	23(6)	47(8)	57(8)	10(7)	12(6)	8(5)
C(51)	41(8)	56(10)	105(13)	-23(9)	28(9)	-17(7)
C(52)	78(13)	61(13)	190(30)	-38(15)	17(16)	-10(10)
C(53)	95(14)	78(14)	102(15)	-12(12)	-2(12)	-20(11)
C(54)	150(20)	81(16)	190(30)	-29(17)	120(20)	-65(16)
C(55)	39(7)	72(11)	55(8)	25(8)	21(7)	8(7)
O(6)	39(5)	51(6)	99(8)	3(6)	0(5)	-14(4)
C(56)	26(6)	37(7)	61(8)	9(7)	1(6)	-6(5)
C(57)	24(6)	40(7)	53(7)	13(6)	10(6)	2(5)

C(58)	38(7)	43(8)	53(8)	0(6)	13(6)	-5(6)
C(59)	40(7)	36(7)	58(8)	9(6)	14(6)	-10(6)
C(60)	38(7)	37(8)	69(9)	-7(7)	25(7)	-11(5)
C(61)	44(7)	40(7)	53(7)	10(7)	7(6)	-10(6)
C(62)	30(7)	62(9)	53(8)	-7(7)	11(6)	-12(6)
C(63)	78(13)	96(18)	190(30)	-32(19)	37(17)	-22(13)
C(64)	58(12)	180(30)	170(30)	100(20)	-49(15)	-60(15)
C(65)	66(13)	270(40)	200(30)	-130(30)	72(18)	-76(19)
C(66)	56(8)	48(9)	56(8)	0(7)	2(7)	-10(6)
O(7)	42(5)	47(6)	95(8)	-1(6)	31(5)	1(4)
C(67)	46(8)	41(8)	50(8)	-1(6)	15(7)	-4(6)
C(68)	40(8)	50(9)	44(7)	-9(7)	0(6)	-5(6)
C(69)	48(9)	66(11)	64(9)	-9(8)	13(8)	-17(8)
C(70)	39(7)	66(11)	57(9)	-8(8)	14(7)	-5(7)
C(71)	36(7)	66(10)	47(8)	-5(7)	2(6)	7(7)
C(72)	51(8)	52(9)	46(7)	5(7)	13(7)	-11(7)
C(73)	50(9)	106(16)	79(12)	-11(11)	33(9)	-9(9)
C(74)	80(20)	640(120)	280(50)	-270(70)	70(30)	40(40)
C(75)	110(20)	240(40)	310(60)	120(40)	120(30)	100(30)
C(76)	64(15)	190(40)	500(80)	-130(40)	110(30)	-46(19)
C(77)	54(8)	47(9)	65(9)	4(7)	20(7)	-1(7)
O(8)	39(5)	49(6)	115(9)	-2(6)	30(6)	8(4)
C(78)	40(7)	34(7)	72(9)	-1(7)	29(7)	-3(6)
C(79)	47(8)	49(9)	58(8)	3(7)	27(7)	3(6)
C(80)	46(8)	54(10)	93(12)	32(9)	22(8)	23(7)
C(81)	70(10)	28(8)	100(13)	4(8)	23(10)	12(7)
C(82)	66(10)	36(8)	89(11)	0(8)	30(9)	2(7)
C(83)	47(7)	51(9)	59(8)	4(7)	26(7)	5(6)
C(84)	90(13)	47(11)	200(30)	5(15)	53(16)	34(10)
C(85)	83(16)	100(20)	400(60)	-50(30)	100(30)	31(15)
C(86)	200(40)	90(20)	390(70)	110(30)	60(40)	50(20)
C(87)	270(50)	160(30)	190(30)	-80(30)	-40(30)	140(30)
C(88)	55(9)	55(9)	60(9)	-5(7)	9(8)	15(7)
O(9)	77(8)	135(14)	105(10)	-51(10)	42(8)	-36(8)
C(89)	90(14)	95(16)	90(14)	-30(12)	30(12)	-34(12)
C(90)	89(16)	200(30)	105(18)	30(20)	31(15)	-1(19)

C(91)	140(30)	160(30)	180(30)	-60(30)	-10(20)	60(20)
C(92)	140(20)	82(15)	94(15)	-27(13)	41(15)	-10(14)
O(10)	97(13)	320(40)	130(17)	-70(20)	42(13)	26(19)
C(93)	190(30)	100(20)	111(19)	1(16)	90(20)	0(20)
C(94)	130(20)	90(20)	240(40)	40(30)	0(30)	14(19)
C(95)	750(170)	140(40)	160(40)	20(30)	180(70)	190(70)
C(96)	210(40)	380(80)	170(40)	150(40)	160(30)	220(50)
O(11)	138(17)	250(30)	170(20)	50(20)	56(16)	-40(20)
C(97)	410(100)	130(40)	390(80)	130(50)	290(80)	100(50)
C(98)	450(100)	140(40)	180(40)	-40(30)	190(50)	-20(60)
C(99)	150(30)	260(60)	160(40)	140(40)	-20(30)	-40(40)
C(100)	130(20)	200(40)	160(30)	50(30)	20(20)	-60(30)
O(12)	96(13)	280(30)	118(14)	50(17)	20(12)	32(16)
C(101)	93(16)	130(20)	100(17)	-31(16)	-4(14)	39(15)
C(102)	210(40)	120(30)	390(70)	-110(40)	190(50)	-30(30)
C(103)	210(50)	370(90)	290(70)	-220(60)	60(50)	-120(50)
C(104)	300(60)	370(80)	82(19)	-10(30)	-40(30)	-270(60)
O(13)	900(200)	180(60)	260(70)	0	100(100)	0
C(105)	200(50)	200(70)	520(130)	-10(70)	-150(70)	110(50)
C(106)	210(60)	320(80)	330(80)	-220(70)	30(50)	-10(50)
O(14)	116(11)	78(10)	121(11)	4(8)	61(9)	-3(8)
O(15)	90(9)	74(8)	85(8)	14(7)	31(7)	-6(7)
O(16)	0(3)	33(4)	15(3)	62(3)	6(3)	50(3)
O(17)	139(14)	98(12)	144(14)	-25(11)	63(12)	-45(10)
O(18)	129(10)	270(19)	23(4)	-42(7)	35(5)	-197(13)
H	156(13)	57(13)	11(12)	113(10)	-109(9)	276(12)
O(20)	0(2)	0(3)	0(2)	4(2)	-11(2)	-5(2)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [Cs(4-tert-butylcalix[8]arene-H)$(THF)_5(H_2O)_6$].

	x	y	z	U(eq)
H(3)	127	3945	6532	82
H(5)	771	3266	5764	72

H(8A)	510	5216	6039	577
H(8B)	243	4850	6270	577
H(8C)	184	4789	5703	577
H(9A)	954	4937	6668	353
H(9B)	1022	4280	6865	353
H(9C)	618	4598	6793	353
H(10A)	1087	4726	5940	587
H(10B)	853	4234	5567	587
H(10C)	1173	4050	6072	587
H(11A)	653	2298	5454	61
H(11B)	377	1866	5619	61
H(14)	1334	2326	5906	56
H(16)	1861	1204	7000	56
H(19A)	2317	2549	6044	227
H(19B)	1893	2760	6007	227
H(19C)	1946	2192	5715	227
H(20A)	2367	1231	6722	156
H(20B)	2591	1646	6464	156
H(20C)	2216	1292	6141	156
H(21A)	2276	2015	7246	180
H(21B)	2133	2646	7022	180
H(21C)	2542	2401	7028	180
H(22A)	1390	828	7471	49
H(22B)	982	601	7112	49
H(25)	1985	297	7529	61
H(27)	1939	-1259	6840	53
H(30A)	2615	-1132	6998	148
H(30B)	2514	-1479	7416	148
H(30C)	2928	-1177	7534	148
H(31A)	2634	84	7075	165
H(31B)	2975	-33	7579	165
H(31C)	2604	338	7573	165
H(32A)	2854	-713	8192	258
H(32B)	2442	-1022	8077	258
H(32C)	2483	-334	8173	258
H(33A)	970	-1175	6060	55

H(33B)	1363	-1537	6179	55
H(36)	1439	-2437	6632	54
H(38)	563	-3068	7068	43
H(41A)	1812	-3040	7185	147
H(41B)	1848	-3671	7432	147
H(41C)	1712	-3124	7674	147
H(42A)	825	-3939	7096	140
H(42B)	1090	-3698	7618	140
H(42C)	1238	-4229	7370	140
H(43A)	1044	-3851	6381	134
H(43B)	1454	-4119	6700	134
H(43C)	1434	-3505	6431	134
H(44A)	33	-2454	7080	49
H(44B)	46	-1771	6979	49
H(47)	-344	-3117	6406	56
H(49)	-1150	-2399	5224	56
H(52A)	-778	-4334	5609	178
H(52B)	-452	-3953	5997	178
H(52C)	-543	-3851	5426	178
H(53A)	-1486	-3205	5151	151
H(53B)	-1409	-3883	5085	151
H(53C)	-1179	-3396	4899	151
H(54A)	-1328	-3384	6073	188
H(54B)	-935	-3706	6381	188
H(54C)	-1265	-4057	5973	188
H(55A)	-769	-948	5367	65
H(55B)	-1095	-1355	5006	65
H(58)	-1727	-1439	5072	55
H(60)	-2064	-320	5949	55
H(63A)	-2332	-1993	5463	187
H(63B)	-2312	-1900	4928	187
H(63C)	-2721	-1880	5012	187
H(64A)	-2560	-374	4858	242
H(64B)	-2866	-895	4673	242
H(64C)	-2473	-900	4550	242
H(65A)	-2612	-623	5708	259

H(65B)	-2498	-1273	5905	259
H(65C)	-2890	-1157	5461	259
H(66A)	-1100	244	6608	69
H(66B)	-1475	31	6727	69
H(69)	-2062	654	6458	73
H(71)	-2147	2094	5625	63
H(74A)	-2373	2029	6673	496
H(74B)	-2422	1361	6801	496
H(74C)	-2794	1774	6604	496
H(75A)	-2791	2083	5523	315
H(75B)	-2589	2411	6030	315
H(75C)	-3012	2137	5909	315
H(76A)	-2879	947	5578	373
H(76B)	-3113	1148	5926	373
H(76C)	-2764	692	6119	373
H(77A)	-1278	1885	5366	66
H(77B)	-1674	2247	5203	66
H(80)	-1709	3187	5578	78
H(82)	-744	3895	6543	75
H(85A)	-2061	4507	5901	283
H(85B)	-1976	3887	6167	283
H(85C)	-2036	3934	5598	283
H(86A)	-1575	4943	5463	356
H(86B)	-1572	4313	5228	356
H(86C)	-1174	4614	5547	356
H(87A)	-1444	4969	6437	353
H(87B)	-1032	4687	6493	353
H(87C)	-1300	4398	6765	353
H(88A)	-172	3335	6953	71
H(88B)	-186	2643	6879	71
H(89A)	1682	180	6141	110
H(89B)	1713	504	5668	110
H(90A)	2068	-533	6095	156
H(90B)	2232	-39	5823	156
H(91A)	1934	-411	5099	213
H(91B)	1947	-1002	5403	213

H(92A)	1321	-959	5282	124
H(92B)	1298	-477	4873	124
H(93A)	1416	1378	2562	150
H(93B)	1574	949	3022	150
H(94A)	1906	1640	3451	205
H(94B)	1767	2056	2986	205
H(95A)	1558	2564	3367	414
H(95B)	1612	2074	3782	414
H(96A)	958	2290	2964	273
H(96B)	975	1994	3472	273
H(97A)	785	6940	4749	324
H(97B)	1100	6829	5273	324
H(98A)	850	6166	5370	284
H(98B)	586	6218	4807	284
H(99A)	318	6237	5519	253
H(99B)	88	6486	4983	253
H(10D)	124	7382	5348	202
H(10E)	456	7161	5840	202
H(10F)	1547	8153	2813	142
H(10G)	1457	7873	3269	142
H(10H)	1617	7046	3104	262
H(10I)	1749	7336	2686	262
H(10J)	1166	6568	2537	358
H(10K)	1264	6923	2112	358
H(10L)	736	7527	2040	330
H(10M)	669	7218	2500	330
H(10N)	305	5071	8182	452
H(10O)	519	5183	7795	452
H(10P)	361	5960	7839	362
H(10Q)	6	5768	8015	362
H	-610(5)	1274(6)	7225(4)	109(9)

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VII. Appendices

VII.1. Abbreviations

Solvent designations

Br-2DB18C6: Dibromodibenzo-18-crown-6
Br-4DB18C6: Tetrabromodibenzo-18-crown-6
DMSO: Dimethylsulfoxide
$CDCl_3$: Deuterated chloroform
EtOH: Ethanol
EtO^-: Ethanolate
THF: Tetrahydrofurane

IR designations

ν: Stretching vibration
s: strong
m: medium
w: weak

1H-NMR designations

s: singlet
d: doublet
t: triplet
m: multiplet

VII.2. Summary

This thesis reports the new crystal structures of crown ethers and calixarenes as well as the ionic conduction properties of crown ether channels.

Chapter I gives general knowledge about ion channels. It deals also with the state of the art on crown ethers and calix[n]arenes.

Chapter II present the different compounds obtained with crown ethers and calixarenes.
In a first part, it shows a study of ionic conduction through crown ether channel structures **1** and **2**. It describes as well all the attempts done to obtain the isostructural structure of **1** with tribromide ions. It presents a pH-dependency of the DB18C6 to form crystals.
Finally this chapter ends on calix[n]arene channel structures (with n=6 and 8) and a comparison between 4-tert-butylcalix[8]arene structures which evolve from a single molecule to a dimer and finally channel compounds thanks to water aggregates. Those aggregates are dependent of the alkali metal present. Potassium ions are related on water cubes and caesium ions influence on the octahedral organization of the water molecules. Rubidium ions tolerate both: On average, the structure with rubidium ions is an alternation of cubes and octahedron separated by a heterooctahedron of water molecules and rubidium ions.

Chapter III summarizes related work and describes a possible developments of this project: the possible applications of crown ethers, and the derivatisation of the upper rim of calixarenes.

VIII. Curriculum Vitae

PERSONAL INFORMATIONS

Nationality: French

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EDUCATION

1996 – 1998: University Diploma in Technology at the "Institut Universitaire de Technologie", Montpellier, Department of Chemistry, Sète, France (Environmental Chemistry)

1998 – 2000: BSc in Chemistry at the UFR (Unité de formation et de recherche) of Science Montpellier II, France

2000 – 2001: Master in Chemistry at the UFR of Science Montpellier II, France

2001 – 2002: DEA Matériaux at the UFR of Science Montpellier II, France

2002 – 2003: PhD in Chemistry at the University of Karlsruhe, Germany

2003 – 2006: PhD in Chemistry at the University of Basel, Switzerland

PROFESSIONAL EXPERIENCES

1998: Professional training on alumine granulation in Salindres, France

2001: Professional training on anodic thin metallic films for Li-ion micro-batteries at the UFR of Science Montpellier II, France

2002: Professional training on the synthesis of tin and germanium particles to be used as electrode materials in Li-ion batteries

2002-2006: Ph-D diploma on ionic channels with crown ether and calix[n]arenas (with n= 6, 8)

2006: Referee for Analytica Chimica Acta

TEACHING

03/2004-07/2004: Inorganic Chemistry Tutorial IV for Chemistry Students, University of Basel, Switzerland.

03/2005-07/2005: Inorganic Chemistry Tutorial IV for Chemistry Students, University of Basel, Switzerland.

10/2005-07/2006: Analytical Chemistry Tutorial I for Pharmacy Students, University of Basel, Switzerland.

CONFERENCES AND PRESENTATIONS

- Poster at the 3rd International Conference on the Chemistry of the Alkali and Alkaline Earth Metals, ALKCHEM, in Würzburg (**2003**, Germany)

Crystallographic study of calixarenes of alkali and alkaline earth metals

- Poster at the 36th International Conference on Coordination Chemistry in Merida (**2004**, Mexico)

Towards ionic channels

- Poster at the fall meetings of the Swiss Chemical Society in Zürich (**2004**, Switzerland)

Towards ionic channels

- Poster at the 8th International Conference on Calixarenes in Prague (**2005**, Czech Republic)

Towards ionic channels

- Oral presentation at the 37th International Conference on Coordination Chemistry in Capetown (**2006**, South Africa)
Ionic channels and ion conduction.

SCIENTIFIC PUBLICATIONS

R. D. Bergougnant, A. Y. Robin, K. M. Fromm, "Hooked-on" Calix[8]arenes: A $(H_2O)_{10}$ Cluster with an Unprecedented Structure. Crystal Growth & Design (**2005**), 5(5), 1691-1694

K. M. Fromm, E. D. Gueneau, A. Y. Robin, W. Maudez, J. Sague, R. Bergougnant, Recent advances in the chemistry of "clusters" and coordination polymers of alkali, alkaline earth metal and group 11 compounds. Z. Anorg. Allg. Chem. (**2005**), 631(10), 1725-1740

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OTHERS

Languages: French, English, German
Computer skills: Schakal, Shelx, Mercury, Structure resolution programs (IPDS STOE)
Single crystal X-ray measurements on STOE IPDS II diffractometer
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REFERENCES

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