DOI: 10.6060/mhc190440o

# Cooperation Effect of Classical O-H…N and Non-Classical C-H…N Hydrogen Bonding at the Formation of Supramolecular Tubes Based on Novel 1,2,4-Triazolyl Derivative of Calix[4]arene in Crystalline Phase

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Synthesis of new 1,2,4-triazolyl containing macrocyclic ligand on the calix[4]arene platform (Schiff base) was performed by condensation of the corresponding upper rim tetraaldehyde derivative of calix[4]arene in 1,3-alternate conformations with 4-amino-4H-1,2,4-triazole. The structure of obtained compound was established by the physical methods (<sup>1</sup>H, <sup>13</sup>C NMR spectroscopies, MALDI mass spectrometry) including single crystal X-ray diffraction. It was found that the cooperation of classical and non-classical hydrogen bonding leads to formation of 1D zigzag supramolecular tubes in the crystalline phase.

Keywords: Hydrogen bonding, Schiff base, calix[4]arene, imine, triazole, supramolecular chemistry.

# Кооперативный эффект классического О-H…N и неклассического С-H…N водородного связывания при образовании супрамолекулярных трубок на основе нового 1,2,4-триазолильного производного каликс[4]арена в кристаллической фазе

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Осуществлён синтез нового 1,2,4-триазолилсодержащего макроциклического лиганда на платформе каликс[4] арена (основания Шиффа) конденсацией соответствующего тетраальдегида каликс[4]арена в конформации 1,3-альтернат с 4-амино-4H-1,2,4-триазолом. Структура соединения была установлена физическими методами (<sup>1</sup>H, <sup>13</sup>C ЯМР спектроскопия, MALDI масс-спектрометрия, рентгеноструктурный анализ). На основе данных рентгеноструктурного анализа было показано кооперативное влияние классического и неклассического водородного связывания на образование супрамолекулярных зигзагообразных трубок в кристаллической фазе.

Ключевые слова: Водородная связь, основание Шиффа, каликс[4]арен, имин, триазол, супрамолекулярная химия.

## Introduction

Calix[4]arenes<sup>[1]</sup> are macrocyclic compounds composed of four phenolic moieties bridged by the methylene groups and attracts a growing interest in organic supramolecular chemistry as receptors towards different substrates of organic and inorganic nature leading to creation on of new sensors, molecular transport and molecular devices.<sup>[2]</sup> In particular, owing to ability to adopt different conformations (cone, partial cone, 1,3-alternate and 1,2-alternate) of molecular platform with unlimited possibilities of functionalization of upper and low rim they can be considered as efficient polydentate ligands for binding the metal cations thus forming the discrete coordination complexes like in case of *cone* conformation when coordination groups are pointed only from one side of macrocyclic platform or may lead to formation of extended periodic hybrid metalorganic structures in crystal phase – coordination polymers (or networks) in case of cation binding from the both sides of macrocyclic platform which is typical for the calix[4] arenes derivatives adopted in the 1,3-alternate conformation. Many functional derivatives of calix[4]arene platform bearing hydroxyl, carboxylic, pyridyl, phosphonate, sulfonate groups as binding sites were synthesized and the creation of different coordination compounds in crystal phase was achieved. It was shown that some of them can exhibit specific magnetic (single molecular magnets, spin crossover etc.), luminescent and catalytic properties.<sup>[3]</sup> In this regard, the synthesis of new calix[4] arene based ligands able to form the functional crystal materials, still appeals considerable attention.

Among the most used ligands involved in coordination polymers construction 1,2,4-triazolyl based compound are of particular interest due to the presence of two outward orientated N donor atoms in the molecular structure which may perform the linear coordination pattern while interaction with the different transition metal cations. It was shown that coordination compounds obtained using the ligands with 1,2,4-triazolyl binding sites are very promising for development of spin crossover materials (SCO).<sup>[4]</sup> Thus, the fusion of calix[4]arene macrocyclic platform with 1,2,4-triazolyl coordination sites may contribute to formation of new extended metal-organic structures upon the combining with transition metal cations such as Fe<sup>II</sup>, Co<sup>II</sup>, Mn<sup>II</sup>, and help to create new functional materials exhibiting the SCO properties, in particular.

Herein we proposed the synthesis of new macrocyclic polydentate ligand adopted *1,3-alternate* conformation and bearing four 1,2,4-triazolyl coordinating sites connected to the macrocyclic platform through imine (Schiff base) function. Some aspects of its crystal structure focused on the presence of the heterocyclic moieties were described as well.

### Experimental

and used without further purification. Calix[4]arene 1 was pre-

pared according to the standard procedure.<sup>[5]</sup> <sup>1</sup>H and <sup>13</sup>C NMR

All reagents were purchased from commercial sources

NMR spectrometer. Mass spectra (MS (ES+)) were obtained on a MALDI-TOF Dynamo Finnigan mass spectrometer using 1,8,9-trihydroxyanthracene or *p*-nitroaniline as the matrix.

The X-ray diffraction data for the single crystal of 4 as a trihydrate,  $C_{52}H_{56}N_{16}O_4\cdot 3H_2O$ , were collected in an  $\omega$  and  $\phi$ -scan mode on a Bruker Kappa Apex II CCD diffractometer equipped with an Oxford Cryostream LT device using graphite monochromated MoK $\alpha$  (0.71073 Å) radiation at 173(2) K. Data collection: images were indexed and integrated using the APEX3 data reduction package (v2015.9-0, Bruker AXS). Final cell parameters were determined by global refinement of reflections from the complete data set. Data were corrected for systematic errors and absorption by means of SADABS-2014/5 based on the Laue symmetry using equivalent reflections. The structure was solved by the direct methods using SHELXT-2018/2<sup>[6]</sup> and refined by the full-matrix least-squares on F<sup>2</sup> using SHELXL-2018/3.<sup>[7]</sup> Molecule 4 and one of the water molecules in the crystal are on a special position on the axe 2. Non-hydrogen atoms were refined anisotropically. The positions of the hydrogen atoms of methyl groups were found using rotating group refinement with idealized tetrahedral angles. The other hydrogen atoms were inserted at the calculated positions and refined as riding atoms. The disorder of O1B-pivot propyl group was resolved using free variables and reasonable restraints on geometry and anisotropic displacement parameters. CCDC 1906183 contains the supplementary crystallographic information that can be obtained free of charge via https://www.ccdc.cam. ac.uk/structures/.

#### Synthesis

Compound 6. Tetra-O-propoxy tetraaldehyde derivative of calix[4]arene 3 (0.1 g, 0.14 mmol) and 4-amino-4H-1,2,4triazole (0.176 g, 2 mmol) and 0.1 g of molecular sieves (5 Å) were mixed in dry pyridine (40 ml) and stirred under refluxing conditions during 80 hours. The completing of reaction was monitored by MALDI TOF spectrometry. After cooling the reaction mixture was filtrated and the solvent was evaporated to dryness. White precipitation was formed after adding of methanol (50 ml) to residue. The powder was filtrated and washed by methanol (50 ml) and water (50 ml) affording the white solid (0.078 g, 57 %yield). M.p. > 320 °C. Anal. Calcd. for  $C_{52}H_{62}N_{16}O_{7}$ : C 61.04, H 6.11, N 21.90 %. Found: C 61.15, H 6.20, N 21.56 %. MS (ES+) m/z 969.48 [M]<sup>+</sup>, calculated 969.13; 992.52 [M+Na]<sup>+</sup>, calculated 992.12. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ<sub>H</sub> ppm: 0.63 (12H, s, -CH<sub>3</sub>), 1.28 (8H, m, -CH<sub>2</sub>-), 3.55 (8H, t, O-CH<sub>2</sub>-), 3.94 (8H, s, Ar-CH<sub>2</sub>-Ar), 7.61 (8H, s, Ar-H), 8.94 (4H, s, -N=CH-Ar), 7.61 (8H, s, trz-H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ<sub>c</sub> ppm: 9.68, 22.26, 36.88, 71.77, 125.84, 130.09, 134.16, 138.75, 157.49, 160.18.

#### Crystallization Conditions

Compound 4 (0.005 g, 5 mmol) was dissolved in DMF (1 ml) in crystallization tube. The following slow liquid-liquid diffusion of MeOH (1 ml) into DMF solution of 4 afforded monocrystals suitable for X-Ray diffraction in several days.

## **Results and Discussion**

The synthesis of targeted triazolyl compound was achieved in several steps using conventional calix[4] arene 1 as a starting material (Scheme 1). On the first stage the interaction of calix[4]arene with PrI in presence of CsOH led to formation of tetra-O-alkylated compound 2 adopted in *1,3-alternate* conformation in quantitative yield. On the next step the formylation of 1 was performed using the Duff reaction which was carried out in acetic Supramolecular Tubes Based on Novel 1,2,4-Triazolyl Derivative of Calix[4]arene



Scheme 1.

acid media in presence of HMTA and afforded to desired tetra aldehyde compound **3**. Finally, the treating of **3** with 20-fold excess of 4-amino-4*H*-1,2,4-triazole in dry pyridine under refluxing conditions during 80 hours in presence of molecular sieves (5 Å) led to formation of targeted Schiff base **4** containing four triazolyl moieties. Molecular sieves were used on the last stage in order to increase the speed of reaction. The structures of intermediate compounds **2** and **3** were confirmed by <sup>1</sup>H NMR spectroscopy which data were in accordance with earlier compounds. The novel triazolyl derivative **4** were also characterized in solution using a combination of <sup>1</sup>H, <sup>13</sup>C NMR spectroscopies and MALDI mass-spectrometry (see Experimental part).

As expected, the number of signals and the proton splitting in observed <sup>1</sup>H NMR spectrum of 4 demonstrate the symmetrical structure of obtained compound which is in accordance with structure of calixarene derivative in 1,3-alternate conformations (Figure 1). The appear-

ance of singlet peak belonging to imine -HC=N- proton instead of aldehyde proton is observed in the range of  $\delta = 8.94$  ppm. When considering the signals of aromatic protons of calix[4]arene platform they demonstrate the singlet peak found in the region of  $\delta = 7.61$  ppm. The aromatic part of <sup>1</sup>H NMR spectrum of obtained triazolyl compound contains also the singlet peak corresponding to CH aromatic protons of triazolyl moieties at  $\delta = 9.04$  ppm. The signals corresponding to protons of methylene bridges connecting the aromatic rings of macrocyclic platform demonstrate also the singlet peak at 3.94 ppm proving 1,3-alternate conformation of calixarene while the cone conformation presents two doublet peaks corresponding to AB proton system in <sup>1</sup>H NMR spectrum. Concerning the proton signals referred to lower rim substituents of 4, two triplet peaks belonging to propyl groups appears at  $\delta = 3.55$  and 0.63 ppm and one multiplet is observed in the range of  $\delta = 1.28$  ppm.



Figure 1. <sup>1</sup>H NMR spectra of compounds 4 (400 MHz, 25 °C, DMSO-*d6*).

## Single Crystal X-Ray Study

X-Ray diffraction study revealed that compound 4 crystallizes in C2/c space group and presents hydrate with 3 molecules of water (Table 1). As expected and confirmed by <sup>1</sup>H NMR spectra in solution, in crystal calixarene adopts *1,3-alternate* conformation and can be described as cylinder (Figure 2). The  $C_2$ -axis of the second order passes through C1 and C3 atoms belonging to methylene bridges.



Figure 2. Crystal structure of 4 (hydrogen atoms and minor disordered components are omitted for clarity).

The imine fragments connecting triazolyl moieties with calix[4]arene platform adopts trans planar configuration (torsion angles between C3A-C7A и N1A-N2A atoms do not exceed 3.5°), and are co-planar to triazolyl and calix[4] arene aryl units (dihedral angles are about 7°) which caused by  $\pi$ -conjugation of presenting aromatic systems through -N=C- bond. The bond distances of such conjugated aromatic fragments are in accordance with reported before.<sup>[8]</sup> Comparing to described earlier Schiff bases derived from calix[4]arene and displaying similar but inversed imine junction between calix[4]arene and coordinating sites (such as pyridyl, furanyl, phenolic, pyrrolyl, thienyl)<sup>[9]</sup> on the upper rim of macrocycle one may establish that obtained compound 4 possesses of enhanced rigidity of molecular platform which may be useful in construction of coordination polymers behaving as tetrahedral shaped molecular building block. The opposite aryl moieties of 4 form considerably small dihedral angle (21.13°) that leads to a distance of about 7.5 Å between N atoms from distal triazolyl substituents. Other metrics corresponding to macrocyclic calix[4]arene platform (bond distances and angles) are in accordance with those reported for the parent compounds.<sup>[10]</sup> Oxypropyl substituents are found to be adopted in trans-planar zigzag conformation.

In crystal, the molecules of **4** are arranged into the chains resulting from the H-bonding between the nitro-

Table 1. Crystal data and structure refinement for 4.

Chemical formula	$C_{52}H_{56}N_{16}O_4 \cdot 3(H_2O)$		
Empirical formula	$C_{52}H_{62}N_{16}O_{7}$		
Formula weight	1023.17		
Temperature	173(2) K		
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	C2/c		
Unit cell dimensions	$a = 14.4846(12) \text{ Å}  \alpha = 90^{\circ}$ b = 16.3788(14)  Å $\beta = 103.985(4)^{\circ}$ $c = 23.2915(19) \text{ Å}  \gamma = 90^{\circ}$		
Volume	5361.9(8) Å <sup>3</sup>		
Ζ	4 (molecule <b>4</b> and one of the water molecules in the special position on the axe 2)		
Density (calculated)	1.267 g/cm <sup>3</sup>		
Absorption coefficient	$0.088 \text{ mm}^{-1}$		
<i>F</i> (000)	2168		
Crystal size	$0.547 \times 0.320 \times 0.277 \ mm^3$		
$\theta$ range for data collection	3.021 to 25.648°		
Index ranges	$\begin{array}{l} -17 <= h <= 17, \\ -19 <= k <= 19, \\ -28 <= l <= 20 \end{array}$		
Reflections collected	23401		
Independent reflections	5049		
Observed reflections [I>2sigma(I)]	3330		
R <sub>int</sub>	0.0554		
R <sub>o</sub>	0.0564		
Completeness to $\theta = 25.242^{\circ}$	99.3 %		
Absorption correction	Semi-empirical from equivalents		
Max. and min. transmission	0.7204 and 0.6402		
Data / restraints / parameters	5049 / 52 / 369		
Goodness-of-fit on $F^2$	1.036		
Final <i>R</i> indices [I>2sigma(I)]	$R_1 = 0.0547, wR^2 = 0.1387$		
R indices (all data)	$R_1 = 0.0932, wR^2 = 0.1590$		
Extinction coefficient	n/a		
Largest diff. peak and hole	0.216 and -0.533 e. Å <sup>-3</sup>		

gen atoms of triazolyl rings with two water molecules acting as bridges and forming the four centered square-like connection node (Figure 3a). The chains formed by classical OH...N hydrogen bonding are linked together by nonclassical C-H...O and C-H...N hydrogen bond interactions: C atom of triazolyl rings and one of the imino carbon atoms display short contacts with nitrogen atoms belonging to triazolyl units (Table 2) leading to 2D layer formation along x0z plane. It should be noted that non-classical hydrogen bonding observed in crystal promotes inclusion phenomenon of distal propyl substituents into the calixarenes cavities (Figure 3b, 4). The inclusion event may also



Figure 3. Portion of crystal structure of 4 showing: (a) 1D H-bonded chain resulting from interaction of 4 with water molecules, (b) antiparallel arrangement of H-bonded chains along x0z plane upon the non-classical CH...N bonding (hydrogen atoms and minor disordered components are omitted for clarity).

be confirmed by the fact that positions of propyl C atoms pointed between two triazolyl moieties are well-refined with 100 % occupancy whereas two remaining propyl groups of **4** are found to be disordered. The disorder was modelled and refined including alternative positions with relative occupancies for C11B-C12B and C13B-C14B pairs equal to 0.746(6): 0.254(6).

The 3D crystal packing of 4 is completed by antiparallel stacking of the layers along y axis (Figure 5). One of three molecules of water presenting in the crystal is lying in the interstices without any specific interactions with other crystal components.

On another hand, in terms of supramolecular selforganization of compound 4, in crystal one may observe the 1D zigzag supramolecular chain formation along x0yplane which composing of calixarene molecules displaying the shortest intermolecular contacts of 3.349(4) Å and 3.303(4) Å between C9B...N4A and C9A...N4B atoms belonging to triazolyl moieties (Figure 6). In this respect, the crystal packing can be described as assemble

D-HA	d (D-H), Å	D(DA), Å	D(HA), Å	Angle D-HA, °
O1-H21N4A (intra)	0.87	2.926(4)	2.07	168
O1-H11N3A' (-1/2-x, 3/2-y, -z)	0.87	3.254(4)	2.43	160
O1-H11N4A' (-1/2-x, 3/2-y, -z)	0.87	3.048(4)	2.20	166
C8A-H8AN1B (-1/2+x, 3/2-y, -1/2+z)	0.95	3.571(3)	2.62	176
C9B-H9BO1" (1/2–x, –1/2+y, 1/2–z)	0.95	3.429(4)	2.51	164
C7B-H7BO1" (1/2–x, –1/2+y, 1/2–z)	0.95	3.382(4)	2.45	166
C8B-H8BN1A (1/2+x, 3/2-y, 1/2+z)	0.95	3.549(3)	2.60	175

Table 2. Hydrogen bonds parameters in crystal of 4.



**Figure 4**. Portion of crystal structure of **4** demonstrating the inclusion phenomenon of propyl substituents into the calixarene cavity upon the H-bonding involving C atom of triazolyl moieties and N atom of imine groups of calixarenes (water molecules, hydrogen atoms and minor disordered components are omitted for clarity).



Figure 5. Fragment of crystal packing of 4 showing the antiparallel stacking of H-bonded layers along the y axis.



**Figure 6**. Fragment of crystal structure of 4 and schematic representation showing: (a) formation of 1D zigzag supramolecular tubes based on calix[4]arene molecules displaying the short intermolecular contacts between triazolyl moieties; (b) their arrangement along the  $x\partial y$  plane (water molecules, hydrogen atoms and minor disordered components are omitted for clarity).

of interlaced supramolecular tubes. Although the observed interaction between triazolyl moieties cannot be attributed to hydrogen bonding because of too small D-H-A angles ( $\angle$ C9A-H9A-N4B = 121.7°;  $\angle$ C9B-H9B-N4A = 123.9°), it allows to assume that, to the best of our knowledge, this is the first example when the triazolyl containing compounds demonstrate the self-assembly affording to supramolecular tubular type architecture in the crystal phase.

# Conclusion

It was shown the facile and useful synthetic pathway for preparation of upper rim Schiff base on calix[4]arene platform using tetraaldehyde derivatives of calix[4]arene in 1,3-alternate conformations and amino triazolyl compound as starting materials. The detailed X-ray analysis of crystal structure of obtained compound 4 revealed the formation of interlaced 1D zigzag supramolecular tubes mediated by the classical OH ... N interactions involving water solvate molecules and triazolyl fragments of 4 in cooperation with non-classical CH...N hydrogen bonding. In addition, the obtained compound can be considered as attractive molecular building blocks for synthesis of new coordination polymers, especially with the aim of creation of perspective SCO materials. The coordination ability of 4 towards transition metal cations are currently under investigation.

Acknowledgments. This work was financially supported by Russian Science Foundation (project N 17-73-20117). The diffraction and spectral data were registered on the equipment of Assigned Spectral-Analytical Center of FRC Kazan Scientific Center of RAS.

The authors would like to thank Dr. Robert R. Fayzullin from Arbuzov Institute of Organic and Physical Chemistry, FRC Kazan Scientific Center, Russian Academy of Science for performing X-ray diffraction analysis and crystal structure determination.

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Received 05.04.2019 Accepted 19.04.2019