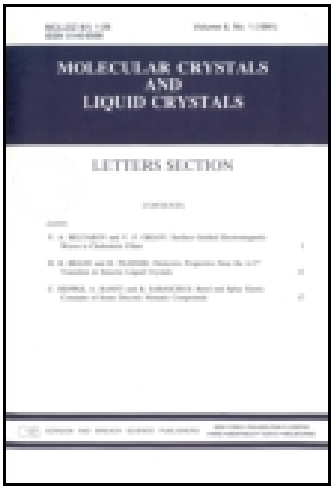

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Ternary Inclusion System of Chair Conformation of 4,6,10,12,16,18,22,24-Octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene: Selective Green Synthesis, Supramolecular Behavior, and Biological Activity

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Ternary Inclusion System of Chair Conformation of 4,6,10,12,16,18,22,24-Octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene: Selective Green Synthesis, Supramolecular Behavior, and Biological Activity

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Calix[4]resorcinarenes form different types of structural conformations. When their methylene carbons are substituted by four phenyl groups, the molecule can adopt both chair and cone conformations depending on the reaction temperature. The solvent-free synthesis of 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene led to the formation of chair conformer (C_{2h}) rather than the cone conformer forming a ternary inclusion complex upon crystallization from wet DMSO. The solid state structure of the ternary inclusion system was determined by single-crystal X-ray diffraction and proved that the host has adopted the chair conformation. The supramolecular interactions in the crystal structure of the solvated compound were carefully investigated. Studies on antimicrobial activities showed that the compound inhibited the growth of Gram-positive bacteria.

Keywords 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene; biological activity; chair-conformation; cone-conformation; DMSO-H_2O solvate; green synthesis; solid-state structure; supramolecular interactions

Introduction

Understanding the self-assembly and the supramolecular interactions of single- and multi-components crystalline solids is of great interest to chemists due to their interesting applications and uses. Generally, a careful examination of molecular arrangement and intermolecular interactions in the crystal structure of a given solid compound is helpful to rationalize the physicochemical properties [1–10].

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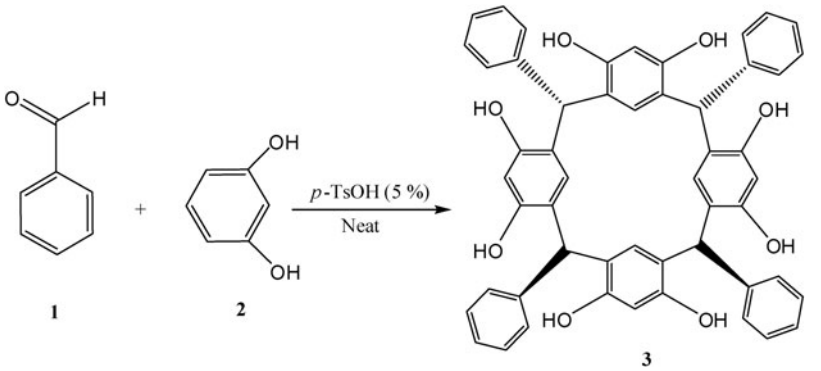
Calixarenes have extensive applications in the biological and pharmaceutical area [11]. Due to their excellent organizing behavior, these classes of synthetic macrocycles have many functionalities [12] and have often been employed as carriers and spatial organizers of various kinds of active substituents [13]. They present well-defined conformational properties and cavities with molecular dimensions that enable them to encapsulate guest drugs [14]. Though most of the calixarene components have been reported for their efficacy against few microorganism species [15–17], some calixarenes have shown interesting activities against cancerous cells [14, 18].

Our research group has been interested in designing and preparing different types of host molecules, and investigating their ability to form host–guest complexes with different guests' functionalities [19–32]. Herein, we describe the selective synthesis, structural conformation, supramolecularity, and bioactivity of the chair conformation of 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene host **3** (Scheme 1) and its DMSO-H_2O ternary inclusion system, as part of an ongoing project aiming to design, synthesize, and investigate new calix [4] arenes compounds [33, 34]. The current biological study is performed as a baseline for investigation, in our group, for planned calixarene derivatives synthesis and a structure–activity relationship deduction.



Scheme 1. Synthetic route for 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenyl-calix[4]arene host **3**.

Experimental Details

Single-crystal X-ray Data Collection and Structure Determination

X-ray quality crystals of compound **3** were obtained by dissolving 30 mg of **3** in 1 mL of dimethylsulfoxide. Slow evaporation of the resulted solution led to X-ray quality crystals which were examined under microscope to select a crystal for single-crystal X-ray crystallography. Single-crystal X-ray diffraction data were collected on a Bruker SMART APEX 1000 diffractometer equipped with a CCD detector and MoKα sealed tube at 223(2) K. SMART [35] was used for collecting frame data, indexing reflection, and determination of lattice parameters. SAINT [35] was used for integration of intensity of reflections and scaling. SADABS [36] was used for adsorption correction and SHELXTL [37] was used for space group, structure determination, and least-square refinements on F^2. All hydrogen atoms were placed in calculated positions for the purpose of structure factor calculations. All nonhydrogen atoms were refined anisotropically [37], and all hydrogen atoms were included in calculated positions with isotropic thermal motion linked to that of the bonded

Table 1. Crystal data and structure refinement for (**3**)·$(DMSO)_8$·$(H_2O)_3$ lattice inclusion compound

Empirical formula	C_{68} H_{94} O_{19} S_8	
Formula weight	1471.91 g/mol	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/*c*	
Unit cell dimensions	$a = 23.2700(10)$ Å	$\alpha = 90^\circ$
	$b = 16.1224(6)$ Å	$\beta = 101.9960(10)^\circ$
	$c = 20.9495(8)$ Å	$\gamma = 90^\circ$
Volume	7687.9(5) $Å^3$	
Z	4	
Density (calculated)	1.272 Mg/m^3	
Absorption coefficient	0.297 mm^{-1}	
$F(000)$	3128	
Crystal size	$0.34 \times 0.16 \times 0.14$ mm^3	
Theta range for data collection	1.55°–25.00°	
Index ranges	$-25 \le h \le 27$, $-19 \le k \le 19$, $-23 \le l \le 24$	
Reflections collected	43810	
Independent reflections	13542 [R(int) = 0.0658]	
Completeness to theta = 25.00°	100.0%	
Absorption correction	Sadabs (Sheldrick 2001)	
Max. and min. transmission	0.9596 and 0.9057	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints/parameters	13542/25/914	
Goodness-of-fit on F^2	1.030	
Final R indices [$I > 2\,\sigma(I)$]	$R1 = 0.0825$, $wR2 = 0.1875$	
R indices (all data)	$R1 = 0.1355$, $wR2 = 0.2131$	
Largest diff. peak and hole	0.878 and −0.607 e^3	

atom. The numerical details, data processing, and refinement of the X-ray structure of (**3**).$(DMSO)_8$.$(H_2O)_3$ lattice inclusion compound are listed in Table 1.

Materials and Physical Measurements

All solvents were used as analytical reagent grade and used as purchased. Melting point was measured on a Stuart scientific melting point apparatus in open capillary tubes. The infrared spectrum was recorded over the range 4000–500 cm^{-1}, on a Maltson 5000 FTIR spectrophotometer. The ^{1}H-NMR and ^{13}C-NMR experiments were conducted on a Bruker 500 MHz at the University of Jordan. Chemical shifts were referenced to *TMS* as the internal standard and deuterated dimethylsulfoxide (DMSO-d_6) as the solvent. X-ray single crystal

structure determination was carried out at Department of Chemistry, National University of Singapore.

Preparation of the 4,6,10,12,16,18,22,24-octahydroxy-2,8,14, 20-tetraphenyl-resorcin[4]arene 3

Synthesis of the chair conformation of compound **3** was selectively achieved by using solvent-free procedure that was published by Scott et al. [38]. Benzaldehyde and resorcinol (1:1) were mixed together in the presence of catalytic amount of *p*-toluenesulfonic acid (ca. 5%). The mixture was removed into small mortar and ground manually until it produced a gummy material which solidified after 2 hr. The crude mixture was reground again, washed with water, filtered, and dried to give compound **3**. IR (KBr), cm^{-1}: 3393, 1614, 1508, 1429, 1280, 1207, 1080, 931, 700, 551; ^{1}H-NMR (500 MHz, DMSO), δ (ppm): 5.64 (s, 4H), 6.33 (s, 4H), 6.74–6.76 (m, 8H), 6.92–7.00 (m, 12 H), 8.58 (bs, 8H); 13C-NMR (125 MHz, DMSO), δ (ppm): 60.71, 101.96, 102.42, 106.15, 120.24, 124.48, 127.10, 128.52–133.24, 145.68, 152.50, 158.40.

In vitro Antimicrobial Activity

The antibacterial activity of compound **3** was determined by measuring the inhibition zones in agar diffusion test according to the National Committee for Clinical Laboratory Standards (NCCLS) [39] using three different concentrations (100, 300, and 500 μg /disk) of the test sample. In addition, the minimum inhibitory concentration (MIC) was calculated by serial dilution assay using microtiter plates with 1 mg/mL of the test sample and 100 μg/mL of the positive control (Chloramphenicol) as starting concentrations. It was performed as follows [40]: test organisms were incubated overnight (16 hr) prior to the assay. Test samples (100 μL) were diluted serially in 96 well plates and each microbial strain suspension (100 μL of 2×10^6 cell/mL) was added in each well. All prepared cultures were incubated for 24–48 hr. The minimum inhibitory concentration is defined as the lowest concentration of the substance that inhibited the growth (visible turbidity) of the test microorganisms. The type of inhibition, bacteriostatic or bactericidal, was determined by platting the preparations in which there were no visible growths on agar plates with incubation for 24 hr. The test microorganisms used in this study were *Bacillus subtilis* (ATCC 6633), *Staphylococcus aureus* (ATCC 43300), *Escherichia coli* (ATCC 25922), *Enterobacter aerogenes* (ATCC 13048), and *Micrococcus luteus* (ATCC 10240).

Results and Discussion

Calix[4]resorcinarenes have been prepared in different methods and conditions, both in the solid state (neat) [38, 41] and in solution [42–45]. Mattay et al. [42] studied the acid-catalyzed condensation reaction of 2-hydroxyresorcinol with aldehydes in aqueous media under reflux conditions and at room temperature. The product obtained under room temperature exhibited the chair-conformation, while the product obtained under reflux conditions exhibited the cone-conformation.

In the present work, the chair conformation of 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene host **3** was selectively prepared in high yield and purity by direct reaction of resorcinol and benzaldehyde in the presence of catalytic amount of solid *p*-toluenesulfonic at room temperature under solvent-free conditions [38]. FTIR, ^{1}H-NMR, ^{13}C-NMR, MS of the prepared compound were in full agreement with what have been previously reported by Cram et al. [43].

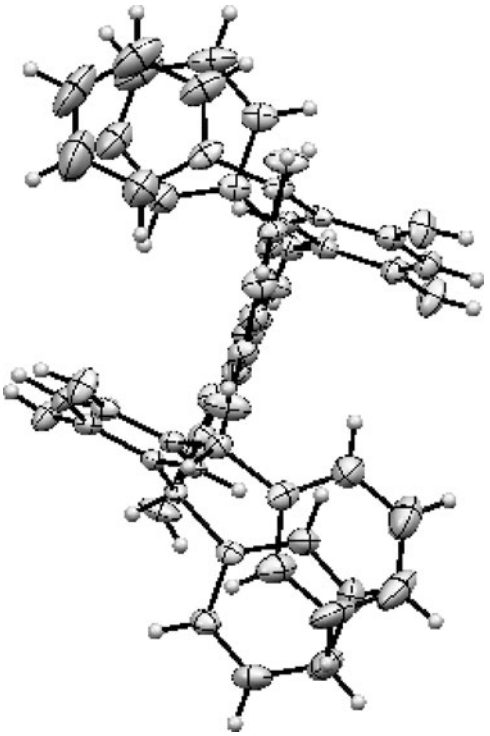


Figure 1. ORTEP plot of chair conformation of compound **3** showing 50% probability ellipsoids.

Crystallization of **3** from dimethylsulfoxide led to inclusion crystals of (**3**).$(DMSO)_8$.$(H_2O)_3$ in a monoclinic system with P2(1)/*c* space group. The asymmetric unit contains one molecule of compound **3**, eight DMSO molecules and three water molecules. Five of the eight DMSO molecules showed disorder of the positions of S, this is quite usual for DMSO. The crystal structure consists of extensive network of hydrogen bonds. Hydrogen atoms of the compound **3** and the DMSO molecules were put at calculated positions. The hydrogen atoms of the water molecules were located from different maps and refined with restrains in bond lengths and thermal parameters. Compound **3** adopted the chair-conformation with approximate C_{2h} symmetry. Morales-Morales et al. have reported on the formation of the cone-conformation of **3** with approximate C_{2v} symmetry [46]. Like its chair-conformation, the cone-conformation of **3** formed a ternary inclusion complex with the same space group but with different formula; (**3**) $(DMSO)_9 \cdot (H_2O)$. The molecular structure of **3** including thermal displacement ellipses with 50% probability is illustrated in Fig. 1. Hydrogen bonding was observed between various phenolic OH groups of the host molecule **3** and the oxygen atoms of the DMSO and water molecules. Intramolecular hydrogen bonding does not exist since the shortest distance between hydroxyl groups is 3.82 Å (Fig. 2). However, all hydrogen donor–acceptor components (solvent and water) are involved in very interesting set of intermolecular hydrogen bonding with different motifs as presented in Table 2. The completely hydrogen-bonded, solvated structure of molecule **3** is shown in Fig. 3. Packing of compound **3** as ternary inclusion system ((**3**).$(DMSO)_8$.(H_2O)) is presented in Fig. 4.

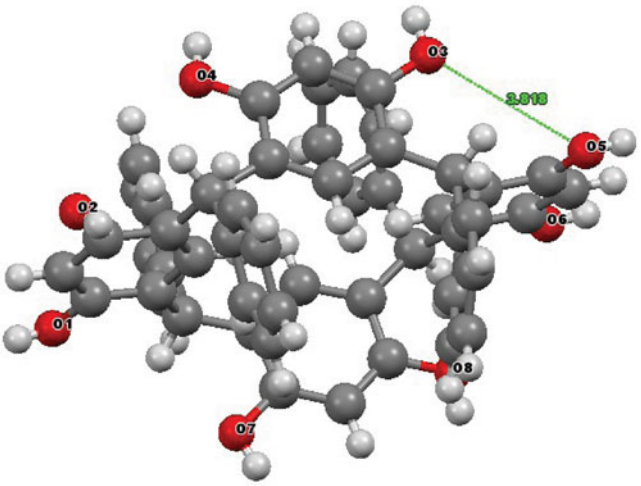


Figure 2. Shortest distance between hydroxyl groups of one molecule of compound **3**.

Table 2. Hydrogen bond lengths (Å) and angles (°) for (**3**)·$(DMSO)_8$·$(H_2O)_3$

D-H . . . A	*d*(D-H)	*d*(H . . . A)	*d*(D . . . A)	<(DHA)
O(1)-H(1) . . . O(2S)	0.83	1.90	2.709(4)	165.4
O(1)-H(1) . . . S(2)	0.83	2.95	3.630(3)	140.6
O(3)-H(3) . . . O(3W)#1	0.83	1.85	2.649(5)	161.3
O(4)-H(4) . . . O(3S)#2	0.83	1.90	2.724(4)	172.7
O(4)-H(4) . . . S(3)#2	0.83	2.80	3.548(3)	151.4
O(5)-H(5) . . . O(8SA)#1	0.83	1.74	2.567(11)	172.3
O(5)-H(5) . . . O(8S)#1	0.83	1.95	2.723(7)	155.0
O(5)-H(5) . . . S(8A)#1	0.83	2.80	3.561(5)	153.4
O(6)-H(6) . . . O(1S)#1	0.83	1.85	2.672(4)	171.7
O(6)-H(6) . . . S(1)#1	0.83	2.81	3.501(3)	141.6
O(7)-H(7) . . . O(1W)	0.83	1.90	2.715(5)	168.6
O(8)-H(8) . . . O(7S)#1	0.83	1.85	2.669(5)	169.6
O(1W)-H(1WA) . . . O(2S)#3	0.899(10)	1.959(14)	2.853(5)	173(6)
O(1W)-H(1W) . . . O(6S)	0.897(10)	1.834(18)	2.720(6)	169(7)
O(1W)-H(1W) . . . S(6)	0.897(10)	2.86(4)	3.639(5)	146(5)
O(2W)-H(2W) . . . O(3S)	0.901(10)	1.874(14)	2.773(6)	175(8)
O(2W)-H(2WA) . . . O(1S)#3	0.900(10)	1.95(2)	2.828(6)	165(8)
O(2W)-H(2WA) . . . S(1)#3	0.900(10)	2.98(5)	3.730(5)	142(7)
O(3W)-H(3W) . . . O(4S)	0.895(10)	1.84(2)	2.705(6)	162(6)
O(3W)-H(3W) . . . S(4)	0.895(10)	2.85(3)	3.690(4)	156(5)
O(3W)-H(3W) . . . S(4A)	0.895(10)	2.99(3)	3.840(19)	159(5)
O(3W)-H(3WA) . . . O(2W)#4	0.897(10)	1.840(15)	2.730(7)	171(6)

Notes. Symmetry transformations used to generate equivalent atoms:
#1: x, $y+1$, z; #2: $x-1$, y, z; #3: $-x+1$, $y+1/2$, $-z+1/2$; #4: $x-1$, $y-1$, z.

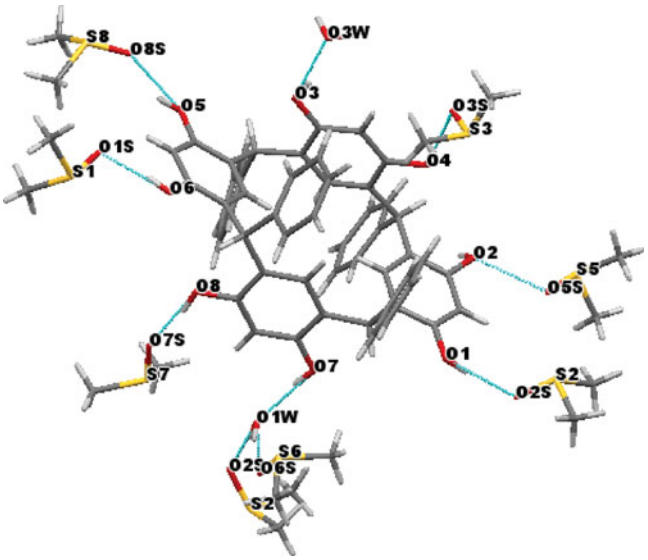


Figure 3. Intermolecular hydrogen bonding present between **3**, water, and DMSO.

In Vitro Antimicrobial Activity

The antibacterial activity of 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenylresorcin[4]arene **3** is presented in Table 3. It was active only against two Gram-positive bacteria, namely *S. aureus* and *M. luteus* at concentrations $\geq$ 300 μg/disk. Gram-negative

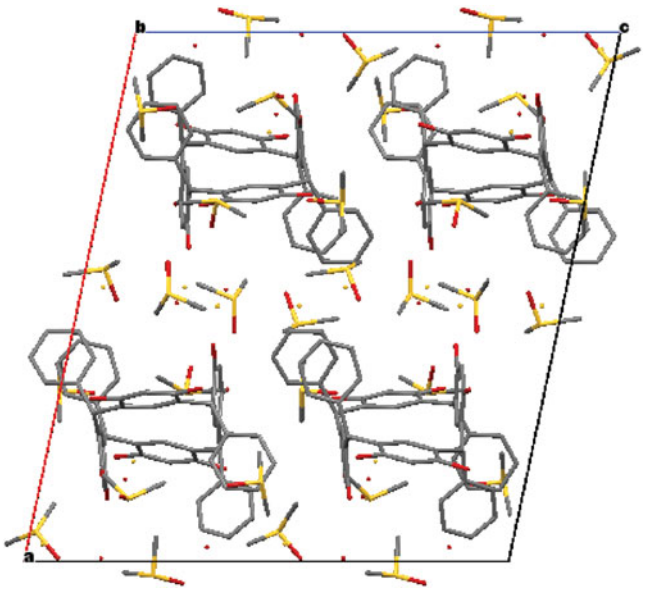


Figure 4. Crystal structure packing of (**3**)·$(DMSO)_8$·(H_2O). All hydrogen atoms were omitted for clarity.

Table 3. Inhibition zone (mm) caused by chair conformation of compound **3** in agar diffusion test

	Inhibition zone (mm)	
Organisms	300 μg/disk	500 μg/disk
Staphylococcus aureus	10	11
Micrococcus luteus	10	13

bacteria were resistant to all tested concentrations. The MIC value of compound **3** against the susceptible bacteria in serial dilution assay was 250 μg/mL and 125 μg/mL for *M. luteus* and *S. aureus* with biostatic effect, respectively. Chloramphenicol was 100–200 fold more active than the tested compound in microtiter plate. Similar results were obtained previously in our group [40] as well as by Abosadiya and his co-researcher [17]. It was found that Gram-positive bacteria were more susceptible than Gram-negative test strains at concentrations comparable to the used ones in this work or even higher. In these previous works the tested Calix[4]resorcinarenes bore para- or ortho- and meta-substituted phenyl groups at their methylene carbons and the MIC values were larger than those in this study, indicating the effect of substitution and type of substituent on the bioactivity of these calixarenes.

Conclusions

Green Chemistry attracted many chemists so that practical methods and reaction conditions for the preparation of compounds with low environment-damage cost have been developed. In this paper, a solvent-free route was used to successfully and selectively prepare 4,6,10,12,16,18,22,24-octahydroxy-2,8,14,20-tetraphenyl-resorcin[4]arene **3** with a chair conformation rather than its boat conformation. The crystal structure of the obtained chair conformation was determined, analyzed and compared to the boat conformation which was reported previously. Compound **3** showed a moderate antimicrobial activity against Gram-positive bacteria. It caused formation of inhibition zones 10–13 mm and has MIC values 125–250 μg/mL.

Acknowledgments

The authors would like to thank Mutah University, the University of Jordan, and the National University of Singapore for conducting the experiments needed to complete this work.

Supplementary Material

Crystallographic data (cif) for (**3**)·$(DMSO)_8$·$(H_2O)_3$ have been deposited in the Cambridge Structural Data Centre (CCDC) with reference number (917787). This data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html [or from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223–336033; Email: deposit@ccdc.cam.ac.uk].

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