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Double partial cone conformation for p-cumylcalix[6]arene·2.5 dimethylformamide

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The crystal structure of p-cumylcalix[6]arene·2.5 dimethylformamide has revealed that the compound exists in the double partial cone conformation. The DMF molecules are bound to the calixarene via hydrogen bonds. The compound crystallizes in the triclinic space group $P\bar{1}$ with a = 17.328(3), b = 17.981(3), c = 17.040(3) Å, $\alpha = 116.56(2)$, $\beta = 112.42(1)$, $\gamma = 76.18(2)°$, and $D_c = 1.165\ g\ cm^{-3}$ for Z = 2. Refinement based on 9418 observed reflections led to a final R value of 0.08.

INTRODUCTION

Calix[6]arenes can exist in several conformations, among them eight 'up-down' conformations and others in which one or more of the aryl groups assume an 'out' alignment.[1] In the solid state, up to date, three conformations have been found, one with symmetry planes giving a 'pinched-cone' conformation[2,3] a second with a center of symmetry giving a 'hinged' conformation;[4,5,6,7] recently Atwood *et al.*[8] described the double partial cone conformation of a calix[6]arene sulfonate with three 'up' and three 'down' moities with also a center of symmetry. We describe here the double partial cone conformation without a center of symmetry and the crystal structure of p-cumylcalix[6]arene crystallized from dimethylformamide.

EXPERIMENTAL

Synthesis

The title compound was synthesized according to the modified standard Petrolite Procedure.[9] A mixture of 21.2 g (0.1 mole) of p-cumylphenol, 6.0 g (0.2 mole) of paraformaldehyde and 4 mL (0.05 mole) of 13N KOH in 150 mL of tetraline was refluxed for 4 h in a 250 mL flask equipped with a Dean-Stark water collector. After 20 min all of the solid had gone into solution and after 4 hours a precipitate began to separate. Tetraline was eliminated by evaporation. The cooled reaction mixture was dissolved in 500 mL of CH_2Cl_2 and shaken with 150 mL of 1N HCl. The organic layer was separated and dried over anhydrous Na_2SO_4. Addition of methanol and acetone resulted in the precipitation of a solid which was removed by filtration to give 4.35 g (21%) of paracumylcalix[6]arene.

X-Ray data collection, structure determination and refinement

Single crystals of the title compound were obtained by slow evaporation, at room temperature, of a solution in dimethylformamide (DMF). A single crystal was mounted in a Lindeman capillar and transferred to a CAD4 Nonius Diffractometer. The unit cell was found to be triclinic with cell constants refined from 25 reflections. The intensity data were collected up to $\theta - 73°$ by using ω-2θ scans with graphite monochromatized Cu Kα radiation. Three standard reflections measured every hour showed no significant fluctuation of intensity. 18066 reflections were recorded with $-21 \leqslant h \leqslant 21$, $-22 \leqslant k \leqslant 22$, $0 \leqslant l \leqslant 21$. Lorentz and polarisation corrections and an empirical absorption correction using the programs PSI and EAC of SDP[10] were applied. Cell constants and pertinent details of the experimentation are given in Table 1.

The structure was solved by direct methods (MULTAN)[11] and refined by using SHELX.[12] Several Fourier difference map revealed all the atoms of the macrocycle not found by Multan and three molecules

Table 1 Crystal data and refinement summary

Molecular Formula	$C_{96}H_{96}O_6$, $2.5C_3H_7ON$		
Molecular Weight	1345.8 + 182.7		
Crystal system	Triclinic		
Space group	$P\bar{1}$		
Cell Constants (Å)		(°)	
a	17.328(3)	α	116.56(2)
b	17.981(3)	β	112.42(1)
c	17.040(3)	γ	76.18(2)
Z	2		
V (Å^3)	4375(2)		
Density calc. (g.cm^{-3})	1.165		
Absorption (cm^{-1})	5.39		
Radiation λ Cu Kα (Å)	1.5418		
Temperature (°C)	22		
F(000)	1640		
Number of reflections			
measured	18066		
unique	14089		
used in structure refinement	9418		
Final R	0.08		
Goodness of fit	1.94		

of DMF, two of them with a disorder. The non-H atoms of the macrocycle were refined anisotropically as well as those of one of the three molecules of the solvent. Some hydrogen atoms were found by difference synthesis; the others were allowed to ride on the carbon atoms to which they are bonded. All were refined isotropically.

Some constraints were applied to the two other solvent molecules, due to the disorder and the refinement was isotropic.

The final R with unit weights was 0.08 calculated on 9418 F(hkl) with 1153 variable parameters. The final difference electron density map showed a highest residual peak of ca. 0.4 e Å^{-3}. Scattering factors for non hydrogen atoms were taken from the International Tables for X-Ray Crystallography;[13] for H atoms from the scattering tables for Stewart *et al.*[14] The final values of the positional parameters are given in Table 2.

RESULTS AND DISCUSSION

Conformation of the calix[6]arene molecule

Some characteristic covalent bond lengths, bond angles and torsion angles calculated from PARST[15] are given in Table 3, with the numbering scheme given in Fig 1.

There are no extraordinary features about the values of bonds in the different rings. In the phenol rings internal angles at the OH groups are larger than 120° (121.1(5)→122.4(6)) and at the para position they have values from 116.2(5) to 118.5(5); it is known that this is related to the withdrawing or releasing properties of the substituents.[16] Figure 2 shows that the macrocycle adopts a structure that may be described as a double partial cone, that is to say three moieites 'up' and the three others 'down'.

Angles at the methylene groups were calculated and reported in Table 3: it is seen that the values at C(2) and C(20) are larger than the others respectively 114.8(3) and 115.7(6). The inclinations of the phenolic rings were calculated with respect to reference planes of three oxygen atoms: thus the dihedral angles between planes 1, 3, 5 and O(10) O(41) O(42) are respectively 143.3(3), 98.3(4) and 133.7(4)°; in the same manner the angles between planes 7, 9, 11 and O(37) O(38) O(39) have values 139.3(2), 107.9(3) and 128.3(3)°.

Bonding pattern with dimethylformamide molecules.

As illustrated in Fig 2 two dimethylformamide molecules are bound to the calix[6]arene through hydrogen bonds. On one part, the conformation of the macrocycle seems to be stabilized by intramolecular hydrogen bonds as is usually found with calixarenes. On other part each half cone is bound by intermolecular hydrogen bond with the solvent molecules.

Table 4 shows the characteristic values for H bonds. The geometry of the hydrogen bonds is not the same for each half of the calix[6]arene. One half contains intramolecular H-bonds between O(39)...O(38)...O(37), and an intermolecular one betwee O(37) and O(400) of one DMF molecule. The positions of the hydrogen atoms are in good agreement with the dissymetries of the angles at C–C–O at the phenolic rings as accordance with Hirsheld.[17]

In the second half, on the molecule, the central O(41) forms an intermolecular H-bond with the solvent molecule. The characteristic values of these bonds are not very good due to the disorder of the DMF molecule with bonds in two positions for the oxygen atom. Both O(40) and O(42) seems to share their hydrogen with O(41), again in good agreement with the theory of Hirshfeld.[17]

PACKING

Pairs of calixarenes lie over centers of symmetry. Two unique DMF molecules are situated between these pairs in such a way that the guest is inside half a cone and bonded to the neighbouring calixarene by hydrogen bonds as described previously. This situation is repeated along the 'b' axis. In the 'a' direction, the third DMF disordered is found between each pair of calixarenes around the center of symmetry at 0, 0, 1/2.

Table 2 Positional parameters

Atom	x	y	z	B (Å²)	Atom	x	y	z	B (Å²)
O(42)	0.5378(3)	0.1780(3)	0.4275(4)	4.7(2)	O(39)	0.2181(2)	0.3623(2)	0.3686(2)	4.3(1)
C(42)	0.6061(3)	0.1793(3)	0.4058(3)	3.3(2)	C(39)	0.1923(3)	0.3440(3)	0.4248(3)	3.3(2)
C(3)	0.5866(3)	0.2058(3)	0.3332(3)	3.2(1)	C(21)	0.2458(3)	0.3650(3)	0.5156(3)	3.1(1)
C(4)	0.6521(3)	0.2067(3)	0.3067(3)	3.4(2)	C(22)	0.2207(3)	0.3514(3)	0.5760(3)	3.5(2)
C(5)	0.7337(3)	0.1822(3)	0.3452(3)	3.3(2)	C(23)	0.1464(3)	0.3172(3)	0.5490(3)	3.7(2)
C(6)	0.7501(3)	0.1572(3)	0.4171(3)	3.6(2)	C(24)	0.0961(3)	0.2959(3)	0.4573(3)	3.7(2)
C(7)	0.6866(3)	0.1562(3)	0.4480(3)	3.3(2)	C(25)	0.1183(3)	0.3080(3)	0.3935(3)	3.3(1)
C(50)	0.8031(3)	0.1831(3)	0.3109(3)	3.7(2)	C(230)	0.1229(3)	0.3042(2)	0.6201(3)	4.2(2)
C(51)	0.8862(3)	0.1384(4)	0.3496(5)	5.0(2)	C(231)	0.0458(5)	0.2568(5)	0.5776(5)	6.8(3)
C(52)	0.7758(4)	0.1396(5)	0.2042(4)	5.7(2)	C(232)	0.1965(5)	0.2537(5)	0.6636(6)	7.0(3)
C(53)	0.8206(3)	0.2742(3)	0.3440(4)	4.0(2)	C(233)	0.1084(3)	0.3907(2)	0.6932(3)	5.5(2)
C(54)	0.8238(4)	0.3307(4)	0.4330(4)	4.9(2)	C(234)	0.0336(3)	0.4374(2)	0.6676(3)	7.3(3)
C(55)	0.8441(4)	0.4123(4)	0.4664(6)	6.4(3)	C(235)	0.0132(3)	0.5142(2)	0.7322(3)	11.5(6)
C(56)	0.8616(4)	0.4375(5)	0.4123(7)	7.1(4)	C(236)	0.0678(3)	0.5444(2)	0.8225(3)	13.8(8)
C(57)	0.8580(4)	0.3842(5)	0.3235(7)	6.9(4)	C(237)	0.1426(3)	0.4978(2)	0.8481(3)	13.5(6)
C(58)	0.8375(4)	0.3033(4)	0.2904(5)	5.3(2)	C(238)	0.1629(3)	0.4209(2)	0.7835(3)	8.4(4)
O(41)	0.5713(4)	0.1632(4)	0.5970(5)	4.3(2)	O(38)	0.1559(2)	0.2720(2)	0.1827(2)	3.9(1)
C(41)	0.6384(3)	0.2138(3)	0.6505(3)	3.5(2)	C(38)	0.1412(3)	0.1972(3)	0.1770(3)	3.3(1)
C(9)	0.7047(3)	0.2001(3)	0.6178(3)	3.6(2)	C(27)	0.0953(3)	0.1994(3)	0.2294(3)	3.4(2)
C(10)	0.7662(3)	0.2554(4)	0.6680(4)	4.3(2)	C(28)	0.0806(3)	0.1249(3)	0.2248(3)	3.7(2)
C(11)	0.7632(3)	0.3255(3)	0.7499(4)	4.3(2)	C(29)	0.1103(3)	0.0476(3)	0.1688(3)	3.6(2)
C(12)	0.6958(3)	0.3353(3)	0.7786(4)	4.1(2)	C(30)	0.1552(3)	0.0476(3)	0.1164(3)	3.6(2)
C(13)	0.6331(3)	0.2814(3)	0.7322(3)	3.4(2)	C(31)	0.1708(3)	0.1216(3)	0.1188(3)	3.3(1)
C(110)	0.8310(4)	0.3866(4)	0.7982(4)	5.4(2)	C(290)	0.0913(4)	−0.0328(3)	0.1667(4)	4.6(2)
C(111)	0.8320(5)	0.4193(5)	0.7288(7)	8.1(4)	C(291)	−0.0030(5)	−0.0341(5)	0.1397(6)	7.6(3)
C(112)	0.8101(6)	0.4651(6)	0.8791(7)	10.2(5)	C(292)	0.1320(7)	−0.0329(5)	0.2646(6)	8.4(4)
C(113)	0.9154(3)	0.3466(4)	0.8377(4)	4.8(2)	C(293)	0.1263(3)	−0.1103(3)	0.0978(3)	3.7(2)
C(114)	0.9257(5)	0.3065(5)	0.8941(5)	6.6(3)	C(294)	0.2035(4)	−0.1507(4)	0.1244(4)	5.1(2)
C(115)	1.0025(6)	0.2754(6)	0.9346(7)	8.9(4)	C(295)	0.2339(4)	−0.2200(4)	0.0604(5)	5.6(3)
C(116)	1.0711(6)	0.2832(7)	0.9241(9)	12.0(6)	C(296)	0.1874(4)	−0.2489(4)	−0.0306(5)	5.3(2)
C(117)	1.0641(5)	0.3213(9)	0.869(1)	15.3(9)	C(297)	0.1123(4)	−0.2093(4)	−0.0588(4)	5.5(2)
C(118)	0.9868(5)	0.3534(7)	0.8278(7)	10.3(5)	C(298)	0.0805(3)	−0.1399(4)	0.0044(4)	4.6(2)
O(40)	0.4340(2)	0.2753(2)	0.5859(2)	4.4(1)	O(37)	0.3182(2)	0.2494(2)	0.1913(3)	4.0(1)
C(40)	0.4389(3)	0.3519(3)	0.6595(3)	3.2(1)	C(37)	0.3622(3)	0.1706(3)	0.1704(3)	3.1(2)
C(15)	0.4960(3)	0.3646(3)	0.7468(3)	3.0(1)	C(33)	0.3135(3)	0.1042(3)	0.1031(3)	3.1(1)
C(16)	0.4940(3)	0.4444(3)	0.8165(3)	3.2(2)	C(34)	0.3547(3)	0.0242(3)	0.0808(3)	3.5(2)
C(17)	0.4370(3)	0.5094(3)	0.8005(3)	3.2(1)	C(35)	0.4384(3)	0.0088(3)	0.1205(3)	3.4(2)
C(18)	0.3822(3)	0.4945(3)	0.7122(3)	3.3(2)	C(36)	0.4838(3)	0.0769(3)	0.1866(3)	3.4(2)
C(19)	0.3838(3)	0.4155(3)	0.6402(3)	3.1(1)	C(1)	0.4464(3)	0.1584(3)	0.2127(3)	2.9(1)
C(170)	0.4326(3)	0.5936(3)	0.8830(3)	3.7(2)	C(350)	0.4812(3)	−0.0811(3)	0.0925(4)	4.3(2)
C(171)	0.3864(4)	0.5795(4)	0.9357(5)	5.6(2)	C(351)	0.4332(6)	−0.1353(4)	0.1036(7)	7.5(4)
C(172)	0.3804(5)	0.6620(4)	0.8499(5)	6.1(2)	C(352)	0.4702(5)	−0.1161(4)	−0.0125(6)	6.8(3)
C(173)	0.5197(3)	0.6218(3)	0.9409(3)	3.8(2)	C(353)	0.5739(4)	−0.0824(3)	0.1492(3)	5.0(2)
C(174)	0.5495(4)	0.6462(4)	1.0368(4)	4.9(2)	C(354)	0.6349(4)	−0.0872(3)	0.1118(3)	7.0(3)
C(175)	0.6271(5)	0.6770(5)	1.0873(5)	7.0(3)	C(355)	.7192(4)	−0.0858(3)	0.1650(3)	10.5(5)
C(176)	0.6770(5)	0.6845(5)	1.0471(6)	7.3(3)	C(356)	0.7427(4)	−0.0795(3)	0.2556(3)	12.5(5)
C(177)	0.6484(5)	0.6600(5)	0.9523(6)	7.0(3)	C(357)	0.6818(4)	−0.0746(3)	0.2929(3)	10.4(4)
C(178)	0.5722(4)	0.6290(4)	0.9012(4)	5.3(2)	C(358)	0.5974(4)	−0.0760(3)	0.2398(3)	8.0(3)
C(2)	0.4979(3)	0.2306(3)	0.2869(4)	3.4(2)	C(20)	0.3257(3)	0.4021(3)	0.5440(3)	3.5(2)
C(8)	0.7097(4)	0.1286(3)	0.5270(4)	4.0(2)	C(26)	0.0624(3)	0.2814(3)	0.2941(3)	3.7(2)
C(14)	0.5602(3)	0.2970(3)	0.7668(4)	3.6(2)	C(32)	0.2205(3)	0.1165(3)	0.0601(3)	3.5(1)
O(400)	0.3821(3)	0.3992(2)	0.2953(3)	6.1(2)	O(500)	0.5800(7)	0.0495(7)	0.6587(8)	10.8(4)
C(400)	0.3614(4)	0.4522(4)	0.3624(4)	4.8(2)	C(500)	0.6093(9)	−0.016(1)	0.673(1)	7.7(4)
N(400)	0.3908(3)	0.5275(3)	0.4099(3)	4.4(2)	N(500)	0.6557(4)	−0.0912(4)	0.6058(4)	8.5(2)
C(401)	0.3652(8)	0.5876(5)	0.4886(7)	9.0(5)	C(501)	0.650(1)	−0.083(1)	0.527(1)	9.1(5)
C(402)	0.4500(6)	0.5514(6)	0.3878(7)	8.2(4)	C(502)	0.6715(4)	−0.1554(4)	0.6417(5)	8.6(2)
					O(550)	0.6186(7)	0.0114(7)	0.5733(8)	12.0
					C(550)	0.621(1)	−0.037(1)	0.618(2)	12.0
					C(551)	0.7152(8)	−0.1315(8)	0.5411(9)	12.0
					O(600)	0.0045(7)	−0.0788(7)	0.3924(8)	12.0
					C(600)	0.002(1)	−0.021(1)	0.470(1)	12.0
					N(600)	0.0688(5)	0.0237(5)	0.5103(5)	12.0
					C(601)	0.130(1)	0.034(1)	0.495(1)	12.0

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: (4/3)*[a²*B(1,1)+b²*B(2,2)+c²*B(3,3)+ab(cos gamma)*B(1,2)+ac(cos beta)*B(1,3)+bc(cos alpha)*B(2,3)].

Table 3 Selected bond distances (Å), bond angles (°) and torsion angles (°) with

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
O42	C42	1.376(9)	O40	C40	1.382(5)	O37	C37	1.398(5)
O41	C41	1.402(8)	O39	C39	1.379(8)	O38	C38	1.383(7)
C1	C2	1.518(6)	C13	C14	1.507(9)	C25	C26	1.510(6)
C2	C3	1.500(6)	C14	C15	1.506(7)	C26	C27	1.529(6)
C7	C8	1.520(9)	C19	C20	1.500(6)	C31	C32	1.512(9)
C8	C9	1.524(6)	C20	C21	1.496(8)	C32	C33	1.503(6)
C5	C50	1.531(9)	C17	C170	1.548(6)	C29	C290	1.540(9)
C50	C51	1.532(7)	C170	C171	1.54(1)	C290	C291	1.52(1)
C50	C52	1.546(8)	C170	C172	1.533(9)	C290	C292	1.54(1)
C50	C53	1.541(8)	C170	C173	1.514(7)	C290	C293	1.537(7)
C11	C110	1.528(8)	C23	C230	1.539(9)	C35	C350	1.549(6)
C110	C111	1.55(2)	C230	C231	1.512(9)	C350	C351	1.53(1)
C112	C112	1.56(1)	C230	C232	1.522(9)	C350	C352	1.56(1)
C110	C113	1.513(8)	C230	C233	1.534(5)	C350	C353	1.528(7)

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
O42	C42	C3	114.4(4)	O39	C39	C21	115.2(4)
O42	C42	C7	124.5(6)	O39	C39	C25	122.4(4)
O41	C41	C9	119.6(5)	O38	C38	C27	118.4(4)
O41	C41	C13	118.6(5)	O38	C38	C31	120.5(5)
O40	C40	C15	122.5(4)	O37	C37	C1	123.0(4)
O40	C40	C19	115.3(4)	O37	C37	C33	115.0(4)
C32	C42	C7	121.1(6)	C21	C39	C25	122.4(6)
C9	C41	C13	121.6(4)	C27	C38	C31	121.1(5)
C15	C40	C19	122.2(4)	C33	C37	C1	122.1(4)
C4	C5	C6	116.8(6)	C22	C23	C24	117.7(6)
C10	C11	C12	116.2(4)	C28	C29	C30	117.5(6)
C16	C17	C18	118.5(4)	C28	C29	C30	117.5(6)
C5	C50	C51	112.9(6)	C23	C230	C231	113.1(5)
C5	C50	C52	109.6(4)	C23	C230	C232	107.4(6)
C5	C50	C53	108.9(4)	C23	C230	C233	107.4(4)
C51	C50	C52	108.(4)	C231	C230	C232	108.2(5)
C51	C50	C53	106.5(4)	C231	C230	C233	110.0(4)
C52	C50	C53	110.9(6)	C232	C230	C233	110.7(4)
C11	C110	C111	108.6(5)	C29	C290	C291	109.3(5)
C11	C110	C112	111.2(6)	C29	C290	C292	108.7(5)
C11	C110	C113	111.2(6)	C29	C290	C293	110.7(6)
C111	C110	C112	105.4(7)	C291	C290	C292	108.4(8)
C111	C110	C113	112.4(6)	C291	C290	C293	110.0(4)
C112	C110	C113	107.8(5)	C292	C290	C293	109.6(5)
C17	C170	C171	106.1(5)	C35	C350	C351	108.2(6)
C17	C170	C172	111.3(4)	C35	C350	C352	107.8(5)
C17	C170	C173	110.2(4)	C35	C350	C353	111.2(3)
C171	C170	C172	107.2(5)	C351	C350	C352	106.5(6)
C171	C170	C173	114.0(5)	C351	C350	C353	112.3(6)
C172	C170	C173	107.9(5)	C352	C350	C353	110.7(6)
C1	C2	C3	114.8(3)	C19	C20	C21	115.7(6)
C7	C8	C9	113.4(5)	C25	C26	C27	113.1(4)
C13	C14	C15	113.6(6)	C31	C32	C33	113.3(5)

Atom 1	Atom 2	Atom 3	Atom 4	Angle	Atom 1	Atom 2	Atom 3	Atom 4	Angle
C1	C2	C3	C42	−81.4(6)	C19	C20	C21	C39	−173.8(5)
C7	C8	C9	C41	103.4(6)	C25	C26	C27	C38	−97.8(6)
C13	C14	C15	C40	76.2(6)	C31	C32	C33	C37	83.2(6)

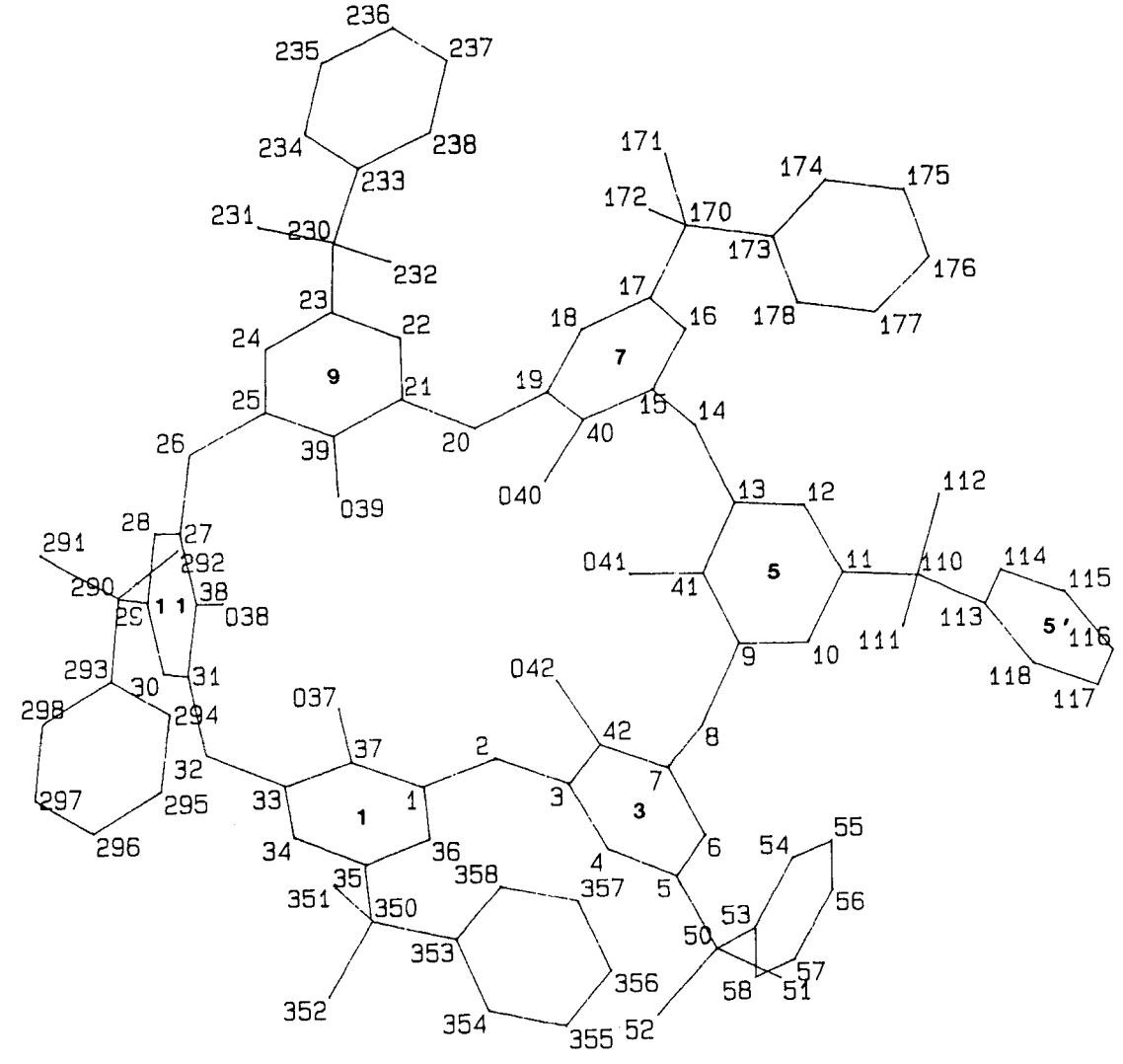

Figure 1 Numbering scheme.

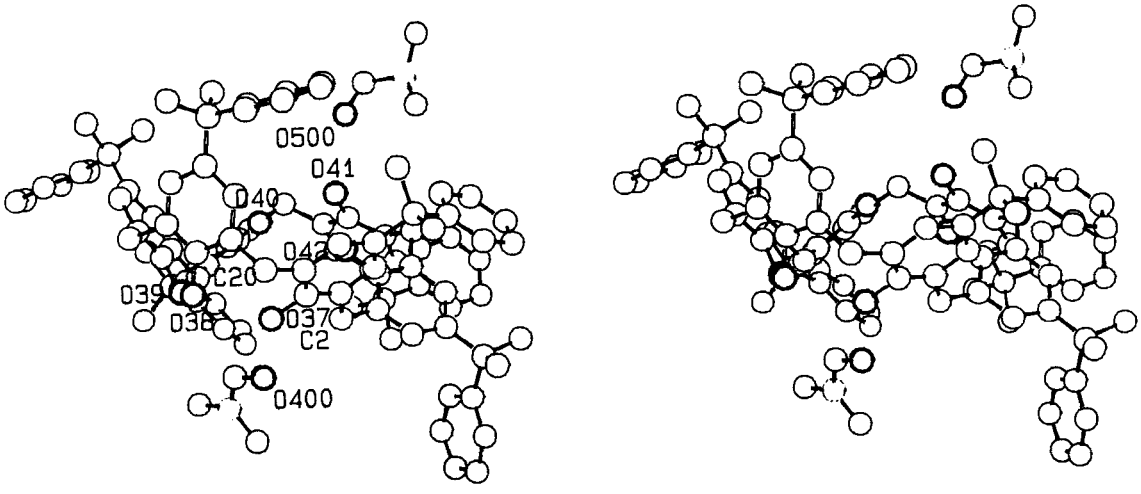

Figure 2 Structure of the p-cumylcalix[6]arene:dimethylformamide complex as a double partial cone conformation.

Table 4 Geometry of hydrogen bonds

Distances	Distances	Distances	Angles
O(42)...H(42)	O(42)...O(41)	H(42)...O(41)	O(42)–H(42)...O(41)
0.60(7)	2.846(7)	2.03(7)	164(9)
O(40)...H(40)	O(40)...O(41)	H(40)...O(41)	O(40)–H(40)...O(41)
0.87(4)	2.730(7)	1.90(4)	164(4)
O(39)...H(39)	O(39)...O(38)	H(39)...O(38)	O(39)–H(39)...O(38)
0.76(5)	2.735(4)	2.03(5)	152(7)
O(38)...H(38)	O(38)...O(37)	H(38)...O(37)	O(38)–H(38)...O(37)
0.84(6)	2.707(5)	1.89(6)	164(6)
O(37)...H(37)	O(37)...O(400)	H(37)...O(400)	O(37)–H(37)...O(400)
0.69(6)	2.69(5)	2.12(6)	140(7)
O(41)...H(41)	O(41)...O(500)	H(41)...O(500)	O(41)–H(41)...O(500)
0.61(8)	2.63(2)	2.26(9)	147(10)
O(41)...H(417)	O(41)...O(550)	H(41)...O(550)	O(41)–H(41)...O(550)
0.61(8)	2.54(1)	2.14(6)	153(10)

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