

Table 2. Geometric parameters (Å, °)

(I)			
O(1)—N(1)	1.401 (3)	O(2)—N(2)	1.410 (3)
N(1)—C(1)	1.275 (3)	N(2)—C(2)	1.281 (2)
C(1)—C(2)	1.489 (3)	C(1)—C(6)	1.505 (3)
C(2)—C(3)	1.503 (3)	C(3)—C(4)	1.517 (3)
C(4)—C(5)	1.508 (4)	C(5)—C(6)	1.526 (4)
(II)			
N(1)—O(1)	1.412 (3)	N(1)—C(1)	1.274 (4)
N(2)—O(2)	1.417 (3)	N(2)—C(2)	1.276 (4)
C(1)—C(2)	1.478 (4)	C(1)—C(7)	1.492 (5)
C(2)—C(3)	1.503 (5)	C(3)—C(4)	1.527 (6)
C(4)—C(5)	1.506 (10)	C(5)—C(6)	1.522 (9)
C(6)—C(7)	1.522 (5)		
(III)			
C(1)—C(2)	1.492 (7)	C(1)—C(8)	1.485 (7)
C(1)—N(1)	1.293 (7)	C(2)—C(3)	1.495 (4)
C(2)—N(2)	1.291 (3)	C(3)—C(4)	1.540 (5)
C(4)—C(5)	1.520 (4)	C(5)—C(6)	1.522 (4)
C(6)—C(7)	1.532 (7)	C(7)—C(8)	1.519 (6)
N(1)—O(1)	1.413 (4)	O(1)—H(w1)	0.850 (33)
N(2)—O(2)	1.409 (2)	O(2)—H(w2)	0.878 (43)
(I)			
O(1)—N(1)—C(1)	111.8 (2)	O(2)—N(2)—C(2)	111.4 (2)
N(1)—C(1)—C(2)	116.8 (2)	N(1)—C(1)—C(6)	123.2 (2)
C(2)—C(1)—C(6)	119.8 (2)	N(2)—C(2)—C(1)	116.3 (2)
N(2)—C(2)—C(3)	125.1 (2)	C(1)—C(2)—C(3)	118.6 (2)
C(2)—C(3)—C(4)	113.0 (2)	C(3)—C(4)—C(5)	109.6 (2)
C(4)—C(5)—C(6)	109.8 (2)	C(1)—C(6)—C(5)	113.6 (2)
(II)			
O(1)—N(1)—C(1)	113.0 (2)	O(2)—N(2)—C(2)	112.6 (2)
N(1)—C(1)—C(2)	115.0 (2)	N(1)—C(1)—C(7)	124.6 (3)
C(2)—C(1)—C(7)	120.4 (3)	N(2)—C(2)—C(1)	124.6 (3)
N(2)—C(2)—C(3)	125.7 (3)	C(1)—C(2)—C(3)	118.5 (3)
C(2)—C(3)—C(4)	111.8 (3)	C(3)—C(4)—C(5)	113.2 (5)
C(4)—C(5)—C(6)	115.7 (5)	C(5)—C(6)—C(7)	115.0 (4)
C(1)—C(7)—C(6)	116.2 (3)		
(III)			
C(2)—C(1)—C(8)	120.2 (4)	C(2)—C(1)—N(1)	115.2 (4)
C(8)—C(1)—N(1)	124.7 (5)	C(1)—C(2)—C(3)	119.5 (3)
C(1)—C(2)—N(2)	116.3 (3)	C(3)—C(2)—N(2)	124.3 (2)
C(2)—C(3)—C(4)	114.4 (3)	C(3)—C(4)—C(5)	116.9 (3)
C(4)—C(5)—C(6)	115.7 (3)	C(5)—C(6)—C(7)	114.5 (3)
C(6)—C(7)—C(8)	114.7 (4)	C(1)—C(8)—C(7)	113.6 (4)
C(1)—N(1)—O(1)	111.2 (4)	N(1)—O(1)—H(w1)	97.7 (22)
C(2)—N(2)—O(2)	111.8 (2)	N(2)—O(2)—H(w2)	98.5 (25)

Table 3. Hydrogen-bond parameters (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
(I)				
O(1)—H(10)...O(2) <sup>i</sup>	0.82	2.10	2.85	153
O(2)—H(20)...N(1) <sup>ii</sup>	0.91	1.85	2.76	174
(II)				
O(1)—H(1)...N(2) <sup>iii</sup>	1.08	1.77	2.80	158
O(2)—H(2)...N(1) <sup>iv</sup>	1.00	1.81	2.76	159
(III)				
O(1)—H(w1)...O(2) <sup>j</sup>	0.85	2.18	2.81	131
O(2)—H(w2)...N(1) <sup>y</sup>	0.88	1.85	2.72	176
Symmetry codes: (i) $x, y, -1+z$ ; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$ ; (iii) $-\frac{1}{2}+x, \frac{1}{2}-y, z$ ; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, z$ ; (v) $\frac{3}{2}-x, -y, \frac{1}{2}+z$ .				

The starting alicyclic  $\alpha$ -dioximes were synthesized by the method reported (Peshkova, Savostina & Ivanova, 1977). The single crystals suitable for X-ray analysis were obtained by recrystallization of water-methanol-ethyl acetate (I, III) and benzene-2-propanol (II) mixtures.

The program package *SHELXTL* (Sheldrick, 1984) was used throughout.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55658 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: VS0102]

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Structure of *p*-Isopropylcalix[4]arene-Toluene (1/1) Complex†

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## Abstract

In the 5,11,17,23-tetraisopropyl-25,26,27,28-tetrahydroxycalix[4]arene-toluene (1/1) complex, a fourfold axis runs through the center of the calixarene (host) molecule and the toluene (guest) molecule adopts

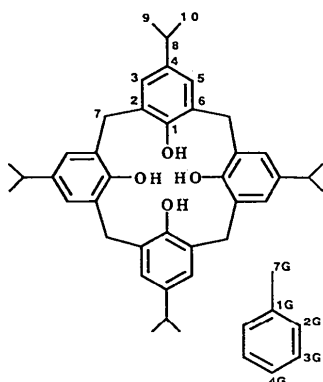
† 5,11,17,23-Tetraisopropylpentacyclo[19.3.1.1<sup>3,7</sup>.1<sup>9,13</sup>.1<sup>15,19</sup>]-octacosane-1(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecacene-25,26,27,28-tetraol-toluene (1/1).

four statistically equal orientations around the fourfold axis. Several weak van der Waals interactions are observed between the host and the guest molecules. There is a cyclic network of hydrogen bonds between the four hydroxy groups [ $O1-O1'' = 2.665(4) \text{ \AA}$ ], which stabilizes the cone conformation.

### Comment

Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1. Distances, angles and selected torsion angles are given in Table 2.

Calixarenes are cyclic phenol-methylene oligomers which include a selected molecule in the hydrophobic cavity. Various inclusion complexes have been studied and their crystal structures determined. We synthesized *p*-isopropylcalix[4]arene and carried out an X-ray structure determination of the 1/1 complex with toluene as a guest.



A crystallographic fourfold axis runs through the center of the inclusion complex ( $x = 0.25, y = 0.25$  and  $x = 0.75, y = 0.75$ ); C1G, C4G and C7G of the guest molecule are on this axis.

The methyl group of the toluene adopts a quite different position from that in the *p*-*tert*-butylcalix[4]arene-toluene complex (Andreotti, 1979). In the case of *p*-*tert*-butylcalix[4]arene, the toluene methyl group is inside the cone cavity of the host molecule but in the title complex it is outside the cone.

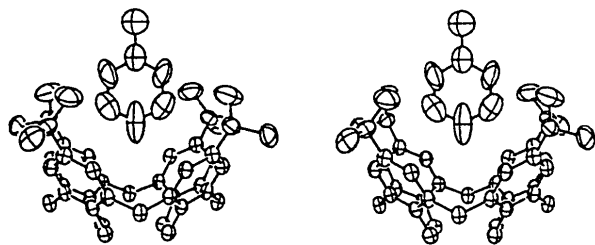


Fig. 1. ORTEPII (Johnson, 1976) drawing of the inclusion complex.

This methyl group has interactions with the isopropyl groups of adjacent host molecules [ $C10-C7G = 3.98(2) \text{ \AA}$ ].

### Experimental

#### Crystal data

$C_{40}H_{48}O_4 \cdot C_7H_8$   
 $M_r = 684.96$   
 Tetragonal  
 $P4/n$   
 $a = 12.790(1) \text{ \AA}$   
 $c = 12.493(2) \text{ \AA}$   
 $V = 2043.6(4) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 1.11 \text{ Mg m}^{-3}$

Cu  $K\alpha$  radiation  
 $\lambda = 1.5418 \text{ \AA}$   
 Cell parameters from 20 reflections  
 $\theta = 20-30.5^\circ$   
 $\mu = 0.505 \text{ mm}^{-1}$   
 $T = 288 \text{ K}$   
 $0.50 \times 0.50 \times 0.40 \text{ mm}$   
 Colorless

#### Data collection

Rigaku AFC-5 automated diffractometer  
 $\omega-2\theta$  scans  
 Absorption correction: none  
 1915 measured reflections  
 1665 independent reflections  
 1004 observed reflections ( $3\sigma$  cut-off)

$R_{int} = 0.0589$   
 $\theta_{max} = 63^\circ$   
 $h = -14 \rightarrow 0$   
 $k = 0 \rightarrow 14$   
 $l = 0 \rightarrow 14$   
 3 standard reflections monitored every 100 reflections  
 intensity variation:  $<5.7\%$

#### Refinement

Refinement on  $F$   
 Final  $R = 0.070$   
 $wR = 0.077$   
 $S = 1.042$   
 1003 reflections  
 155 parameters  
 Parameters of selected H atoms refined

$w = 1/[\sigma^2(F_o) - 0.00535|F_o| + 0.00128|F_o|^2]$   
 $(\Delta/\sigma)_{max} = 0.93$   
 $\Delta\rho_{max} = 0.33 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.26 \text{ e \AA}^{-3}$   
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )

The origin is at  $\bar{1}$  on  $n$ , at  $\frac{1}{4}, -\frac{1}{4}, 0$  from  $\bar{4}$ .  $U_{eq} = (U_{11} + U_{22} + U_{33})/3$ .

	$x$	$y$	$z$	$U_{eq}$
O1	0.1844 (2)	0.1181 (2)	-0.0488 (2)	0.0731 (10)
C1	0.1569 (3)	0.0668 (3)	0.0445 (3)	0.0606 (12)
C2	0.0584 (3)	0.0836 (3)	0.0894 (3)	0.0626 (13)
C3	0.0318 (3)	0.0257 (3)	0.1797 (3)	0.0697 (14)
C4	0.0993 (3)	-0.0463 (3)	0.2250 (4)	0.0733 (15)
C5	0.1975 (3)	-0.0585 (3)	0.1799 (4)	0.0703 (15)
C6	0.2279 (3)	-0.0033 (3)	0.0897 (3)	0.0638 (13)
C7	-0.0176 (3)	0.1628 (3)	0.0428 (4)	0.0664 (14)
C8	0.0649 (4)	-0.1116 (4)	0.3210 (4)	0.0973 (20)
C9	0.1292 (9)	-0.0913 (8)	0.4177 (6)	0.194 (5)
C10	0.0581 (6)	-0.2294 (5)	0.2915 (6)	0.139 (3)
C1G*	0.25	0.25	0.5451 (14)	0.167 (8)
C2G†	0.1682 (15)	0.2028 (28)	0.4889 (14)	0.239 (12)
C3G†	0.1656 (24)	0.2070 (41)	0.3765 (14)	0.253 (17)
C4G*	0.25	0.25	0.3205 (14)	0.261 (20)
C7G*	0.25	0.25	0.6678 (14)	0.221 (9)
HO1†	0.161 (13)	0.192 (4)	-0.048 (7)	0.14 (4)
HO2†	0.255 (6)	0.149 (14)	-0.044 (7)	0.17 (5)

\*Occupancy 0.25.

†Occupancy 0.50.

Table 2. Bond lengths (Å), angles (°) and selected torsion angles (°) with *e.s.d.*'s in parentheses

C1—O1	1.383 (5)	C5—C6	1.385 (6)
C1—C2	1.396 (6)	C6—C7 <sup>i</sup>	1.527 (6)
C1—C6	1.395 (6)	C8—C9	1.484 (13)
C2—C3	1.392 (6)	C8—C10	1.554 (10)
C2—C7	1.520 (6)	C1G—C2G	1.40 (4)
C3—C4	1.383 (6)	C1G—C7G	1.53 (2)
C4—C5	1.385 (6)	C2G—C3G	1.41 (6)
C4—C8	1.526 (7)	C3G—C4G	1.40 (6)
O1—C1—C2	119.7 (3)	C1—C6—C7 <sup>ii</sup>	121.2 (4)
O1—C1—C6	118.7 (4)	C5—C6—C7 <sup>ii</sup>	120.5 (4)
C2—C1—C6	121.6 (4)	C2—C7—C6 <sup>iii</sup>	112.7 (4)
C1—C2—C3	117.7 (4)	C4—C8—C9	112.6 (6)
C1—C2—C7	121.7 (4)	C4—C8—C10	111.1 (5)
C3—C2—C7	120.6 (4)	C9—C8—C10	113.2 (6)
C2—C3—C4	122.2 (4)	C2G—C1G—C7G	120 (2)
C3—C4—C5	118.3 (4)	C1G—C2G—C3G	120 (3)
C3—C4—C8	120.4 (4)	C2G—C3G—C4G	120 (4)
C5—C4—C8	121.3 (4)	C2G—C1G—C2G <sup>i</sup>	120 (2)
C4—C5—C6	121.9 (4)	C3G—C4G—C3G <sup>i</sup>	120 (3)
C1—C6—C5	118.3 (4)		
C1—C2—C7—C6 <sup>iii</sup>	−88.8 (5)	C3—C4—C8—C9	−117.5 (6)
C1—C6—C7 <sup>ii</sup> —C2 <sup>ii</sup>	88.4 (5)	C3—C4—C8—C10	114.3 (5)

Symmetry codes: (i)  $\frac{1}{2} - x, \frac{1}{2} - y, z$ ; (ii)  $\frac{1}{2} - y, x, z$ ; (iii)  $y, \frac{1}{2} - x, z$ .

Programs used: *SHELXS86* (Sheldrick, 1985); *SHELX76* (Sheldrick, 1976); *ORTEPII* (Johnson, 1976); *The Universal Crystallographic Computing System—Osaka* (1979).

The occupancy of the guest molecule was refined as variable; the final refined occupancy showed the molecular ratio of host to guest as 1:1. The H atom of the OH group has two orientations.

There are two circular frameworks including four O—H...O hydrogen bonds as clockwise and anticlockwise rotations. The positive peaks of these H atoms were indistinct on the difference Fourier maps.

There are not enough strong interactions to hold the guest molecule fixed rigidly although several weak interactions are observed. This causes the high *R* value and the high temperature factors of the guest molecule.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55703 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1013]

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**Structure of MoAs<sub>2</sub>O<sub>7</sub>: corrigendum and addendum.** By E. HUMS, *Siemens AG, Bereich Energieerzeugung (KWU), Hammerbacherstrasse 12 und 14, D - 8520 Erlangen, Germany* and H. BURZLAFF and W. ROTHAMMEL, *Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen-Nürnberg, Bismarckstrasse 10, D-8520 Erlangen, Germany*

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### Abstract

Data collection and refinement for the title compound, molybdenum diarsenate, were repeated; the results are presented.

The crystal structure of the title compound was solved and published by the authors (Hums, Burzlaff & Rothammel, 1991). At that time the refinement did not allow the application of anisotropic displacement parameters for all

atoms, since one  $\beta(2,2)$  parameter ran negative. Moreover, unusually high residua could be recognized in the difference Fourier map. Therefore the data collection was repeated using more stable conditions for the measuring temperature and extending the observed region of the reciprocal space such that averaging for symmetrically equivalent reflections was possible. The sample and the instrument were not changed. The new data allowed a complete refinement as usual; the *Experimental* contains the crystal data and the experimental procedure relating to the new data collection. Table 1 contains the structural