Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# *p*-Cumylcalix[4]arene–*para*-xylene (1/1)

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.004 Å
H-atom completeness 95%
*R* factor = 0.055
*wR* factor = 0.155
Data-to-parameter ratio = 18.2

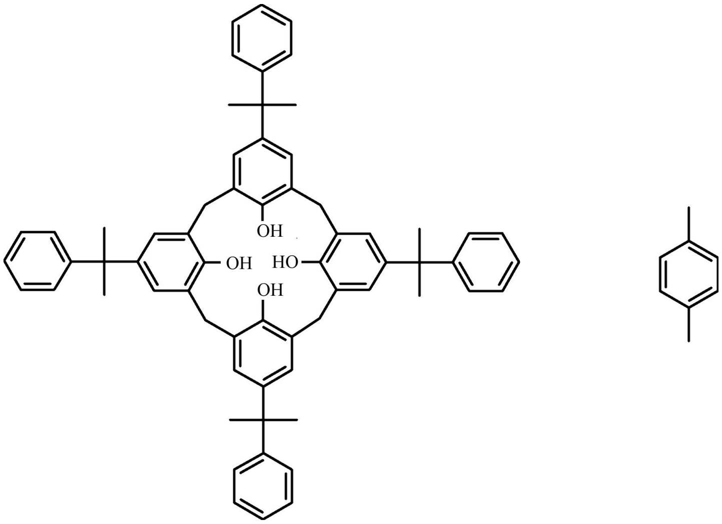
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 April 2005
Accepted 23 May 2005
Online 10 June 2005

In the complex between *p*-cumylcalix[4]arene and *para*-xylene [systematic name: 4-(2-phenylisopropyl)calix[4]arene–1,4-dimethylbenzene (1/1)], $C_{64}H_{64}O_4{\cdot}C_8H_{10}$, the macrocycle has a cone conformation, which is stabilized by intramolecular hydrogen bonds. The guest *para*-xylene molecule is situated inside the cavity, giving an *endo*-calix complex. The host and guest molecules both have twofold rotation symmetry.

## Comment

Many calixarenes form complexes with neutral molecules in the solid state. We describe here the structure of *p*-cumylcalix[4]arene with *p*-xylene as guest. The macrocycle and the guest each have a twofold symmetry axis. The calixarene is in a cone conformation; the dihedral angles between the planes of the phenol rings and the mean plane through the methylene C atoms are 121.50 (14) and126.01 (15)°, similar to those found for *p*-tertbutyl or *p*-isopropyl calix[4]arenes (Andreetti *et al.*, 1979; Perrin *et al.*, 1992). The cone conformation is stabilized by strong hydrogen bonds between the OH groups [O$\cdots$O = 2.684 (4) and 2.622 (4) Å]. The dihedral angles between the phenol ring and the benzene ring of the cumyl group have values of 81.88 (15) and 85.03 (15)°. The cumyl groups are oriented out of the cavity. The *p*-xylene guest lies along the [010] direction. Some $CH_3\cdots\pi$ interactions are observed between one of the $CH_3$ groups of the xylene and the phenol rings. The complex is an *endo*-calix complex.



(I)

## Experimental

The synthesis of *p*-cumylcalix[4]arene was accomplished following the Zinke procedure (Zinke *et al.*, 1948) and an adapted method, as described previously (Gutsche & Iqbal, 1990; Makha & Raston, 2001). A mixture of *p*-cumylphenol (20.1 g, 0.095 mol), formaldehyde solution (13 ml, 37%) and 10 *N* NaOH (0.19 g, 0.0049 mol) was stirred and heated at 383–393 K for 2 h in a 250 ml flask equipped with a Dean–Stark water collector. Warm diphenyl ether (165 ml) was added and the contents were heated for 2 h at 393 K, before ramping the temperature to 533 K over a period of 0.5 h. Refluxing at 533 K was maintained for 3 h, forming a dark-amber solution, and the mixture was then allowed to cool to room temperature. Diphenyl ether was evaporated, and the viscous material obtained was washed and dried *in vacuo*, giving an amber oil. Upon addition of acetone (150 ml), *p*-cumylcalix[4]arene was obtained as a beige powder (yield 12%). Crystals of the complex were obtained from a saturated solution in *p*-xylene at room temperature.

### *Crystal data*

$C_{64}H_{64}O_4{\cdot}C_8H_{10}$
$M_r$ = 1003.31
Monoclinic, $P2/n$
$a$ = 14.66 (3) Å
$b$ = 12.62 (3) Å
$c$ = 15.14 (3) Å
$\beta$ = 96.62 (3)°
$V$ = 2782 (10) Å$^3$
$Z$ = 2
$D_x$ = 1.198 Mg m$^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 6550 reflections
**$\theta$ =? –**
**?** °
$\mu$ = 0.07 mm$^{-1}$
$T$ = 293 (2) K
Plate, colorless
0.80 × 0.50 × 0.20 mm

### *Data collection*

Nonius KappaCCD diffractometer
$\varphi$ or $\omega$ scans
Absorption correction: none
12312 measured reflections
6372 independent reflections
4031 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.038
$\theta_{\rm max}$ = 27.5°
$h = -18 \rightarrow 19$
$k = -16 \rightarrow 16$
$l = -19 \rightarrow 19$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.155
$S$ = 1.02
6372 reflections
351 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0771P)^2 + 0.6028P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.45 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −0.19 e Å$^{-3}$

H atoms were constrained using a riding model [C—H = 0.93–0.97 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C)].

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

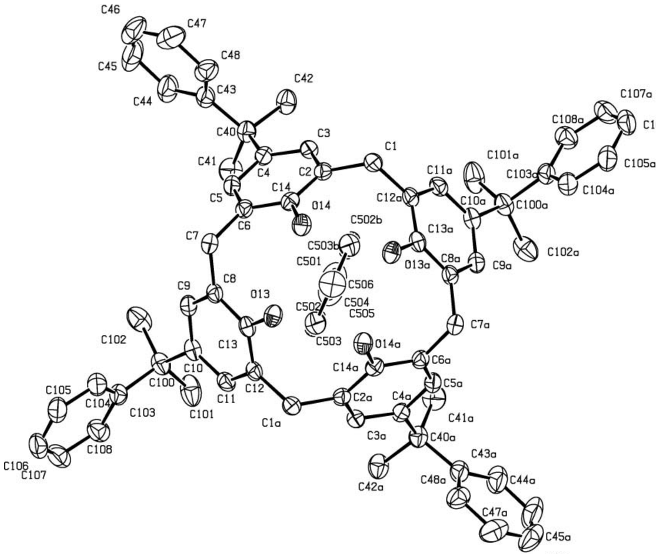


**Figure 1**
A view of (I), showing the atomic numbering scheme and displacement ellipsoids at the 50% probability level. H atoms have been omitted for clarity. Atoms labeled with the suffixes *a* and *b* are at the symmetry position $\frac{1}{2} - x, y, \frac{1}{2} - z$..

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