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 55535 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1015]

References


Tetramethyl tert-Butylcalix[4]arene Tetraketone

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Abstract

The title molecule, 25,26,27,28-tetraacetylonyx-5,11,17,23-tetra-tert-butylpentacyclo[19.3.1.13.7.11.15.19]-octacosa-1(25),3,5,7(28),9,11,13(27),15,17,19,21,23-dodecaene, has twofold crystallographic symmetry and adopts a distorted cone conformation in the solid state.
The orientation of the four aromatic rings is such that two of the rings are almost parallel to each other and the other two almost normal to one another. This conformation precludes a solvent molecule being enclathrated within the cavity.

**Comment**

The structure determination of the title compound (I) was undertaken to establish the overall molecular conformation in the solid state. The structure of (I).y(CH₃)₂CO has been previously determined in the space group Pbcn using crystals grown from acetone (Arnaud-Neu et al., 1989). Crystals for the present structural determination were obtained by the recrystallization of (I) from methanol/acetonitrile.

The calix[4]arene (I) lies about a twofold axis (Fig. 1) and adopts a distorted cone conformation in the solid state. The conformation of the calix moiety of (I) is defined by the interplanar angles between opposite pairs of aromatic rings [82.3(3)° for rings A and A* (related by the operation y, x, 1-z) and 7.2(2)° for rings B and B*, rings B and B* being tilted so that their tert-butyl groups are pitched slightly away from the calix cavity]. The overall calix conformation for molecule (I) is thus very similar to that observed in the previous structural determination of (I).y(CH₃)₂CO which also has twofold crystallographic symmetry and two sets of opposite rings at interplanar angles of 85° and 9° to one another (Arnaud-Neu et al., 1989).

The O···O intramolecular distances for the cis-adjacent ethereal O atoms are 3.34(1) Å (O1A···O1B) and 2.97(1) Å (O1A···O1B*); the distances between symmetry related ethereal O atoms across the twofold axis are 3.57(1) Å (O1A···O1A*) and 5.17(1) Å (O1B···O1B*). The orientation of the ester groups is presumably determined by crystal packing forces; the shortest intramolecular contacts involving the ester chains are O2A···C7B and O2A···H7BA (3.22 and 2.33 Å, respectively). Intermolecular contacts correspond to normal van der Waals separations. There is no solvent of crystallization present in the lattice and an examination of the structure using PLATON (Spek, 1991) reveals no potential solvent volume.

Final fractional coordinates are listed in Table 1. The molecular dimensions (summarized in Table 2) are unexceptional and serve to establish the conformation. The diagram was prepared using ORTEPII (Johnson, 1976).

**Experimental**

**Crystal data**

C₅₆H₇₂O₈  
Mₑ = 873.17  
Trigonal  
P₃₁2₁  
a = 12.971 (3) Å  
c = 25.740 (12) Å  
V = 3750.3 (19) Å³  
Z = 3  
Dₓ = 1.160 Mg m⁻³

**Data collection**

Enraf–Nonius CAD-4  
ω/2θ scans  
Absorption correction: none  
1400 measured reflections  
1364 independent reflections  
974 observed reflections  
[Iₑ > 2.5σ(Iₑ)]

**Refinement**

Refinement on F  
Final R = 0.048  
Δρₑₑ_max = 0.29 e Å⁻³  
Δρₑₑ_min = −0.18 e Å⁻³

**Mo Kα radiation**

λ = 0.70930 Å  
Cell parameters from 25 reflections  
θ = 5.00–12.00°  
μ = 0.07 mm⁻¹  
T = 293 K  
Block  
0.20 × 0.20 × 0.10 mm  
Colourless

**3 standard reflections**

frequency: 120 min  
intensity variation: < 1%
\[ wR = 0.062 \]
\[ S = 1.59 \]

974 reflections
289 parameters

\[ w = 1/(\sigma^2(F) + 0.0010F^2) \]
(\(\Delta/\sigma\))\text{max} = 0.001


Program(s) used to solve structure: NRCVAX SOLVER. Program(s) used to refine structure: NRCVAX LSQSO. Molecular graphics: NRCVAX. Software used to prepare material for publication: NRCVAX TABLES.

### Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

<table>
<thead>
<tr>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Ueq</th>
</tr>
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<tbody>
<tr>
<td>0.3516 (5)</td>
<td>0.3111 (5)</td>
<td>0.56704 (21)</td>
<td>0.048 (5)</td>
</tr>
<tr>
<td>0.1486 (8)</td>
<td>0.2170 (8)</td>
<td>0.6298 (3)</td>
<td>0.119 (8)</td>
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<tr>
<td>0.4474 (8)</td>
<td>0.3636 (8)</td>
<td>0.6008 (3)</td>
<td>0.040 (7)</td>
</tr>
<tr>
<td>0.5379 (7)</td>
<td>0.3388 (7)</td>
<td>0.6006 (3)</td>
<td>0.040 (7)</td>
</tr>
<tr>
<td>0.6334 (7)</td>
<td>0.4004 (8)</td>
<td>0.6334 (3)</td>
<td>0.042 (7)</td>
</tr>
<tr>
<td>0.6641 (8)</td>
<td>0.4898 (8)</td>
<td>0.6657 (3)</td>
<td>0.046 (7)</td>
</tr>
<tr>
<td>0.5520 (8)</td>
<td>0.5168 (8)</td>
<td>0.6642 (3)</td>
<td>0.047 (8)</td>
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<tr>
<td>0.4529 (7)</td>
<td>0.4546 (7)</td>
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<td>0.040 (7)</td>
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<tr>
<td>0.5462 (7)</td>
<td>0.2613 (7)</td>
<td>0.5583 (3)</td>
<td>0.042 (6)</td>
</tr>
<tr>
<td>0.7542 (7)</td>
<td>0.5508 (8)</td>
<td>0.5694 (3)</td>
<td>0.052 (7)</td>
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<td>0.8194 (10)</td>
<td>0.6917 (9)</td>
<td>0.6761 (4)</td>
<td>0.086 (9)</td>
</tr>
</tbody>
</table>

The structure was solved and refined in \( P3_121 \). No attempt was made to determine any absolute configuration in the light of the paucity of observed data and the low values of the anomalous-scattering components for C and O. Very few reflections were observed beyond \( \theta = 15^\circ \) and data collection was terminated at \( \theta = 20^\circ \).

GF thanks NSERC Canada and MAMcK thanks SERC/Eoals for Grants in Aid of Research.

### References


### Structures of Furo[2,3-b]indolizine Derivatives

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### Abstract

The indolizine and furan rings in the skeleton of 2-benzoyl-9-methylfuro[2,3-b]indolizine (2a) and 8-benzoyl-5,6-dihydro-4H-furo[2′,3′:4,5]pyrrolo[3,2,1-fj]quinoline (2b) are planar [mean deviations 0.014 (3) and 0.005 (3) Å (2b), respectively] and inclined at 5.61° (2a) and 3.25° (2b) to one another. In (2b), The nearly planar cyclohexene ring (fused to the indolizine ring at the 1- and 8-positions) is also nearly coplanar with the indolizine ring [mean deviation 0.140 (3) Å, dihedral angle 5.41°]. The annelation of the cyclohexene ring in (2b)....