Table 2. *Geometric parameters (Å, °)*

C1*A*—C2*A*	1.327 (5)	C1*B*—C2*B*	1.320 (5)
C1*A*—C21*A*	1.483 (4)	C1*B*—C21*B*	1.484 (4)
C2*A*—C3*A*	1.497 (4)	C2*B*—C3*B*	1.503 (4)
C3*A*—C4*A*	1.527 (6)	C3*B*—C4*B*	1.520 (6)
C4*A*—C5*A*	1.529 (5)	C4*B*—C5*B*	1.517 (6)
C5*A*—C6*A*	1.501 (5)	C5*B*—C6*B*	1.510 (5)
C6*A*—O15*A*	1.460 (3)	C6*B*—O15*B*	1.442 (4)
C6*A*—C7*A*	1.516 (5)	C6*B*—C7*B*	1.522 (5)
C7*A*—C8*A*	1.533 (4)	C7*B*—C8*B*	1.527 (5)
C8*A*—C9*A*	1.517 (5)	C8*B*—C9*B*	1.517 (5)
C9*A*—C10*A*	1.531 (5)	C9*B*—C10*B*	1.518 (4)
C10*A*—O11*A*	1.475 (4)	C10*B*—O11*B*	1.474 (3)
C10*A*—C14*A*	1.506 (7)	C10*B*—C14*B*	1.519 (5)
O11*A*—C12*A*	1.322 (4)	O11*B*—C12*B*	1.331 (4)
C12*A*—O13*A*	1.225 (4)	C12*B*—O13*B*	1.224 (4)
C12*A*—C16*A*	1.477 (4)	C12*B*—C16*B*	1.474 (5)
C16*A*—C17*A*	1.411 (4)	C16*B*—C17*B*	1.407 (4)
C16*A*—C21*A*	1.423 (4)	C16*B*—C21*B*	1.416 (4)
C17*A*—O22*A*	1.366 (4)	C17*B*—O22*B*	1.357 (4)
C17*A*—C18*A*	1.379 (5)	C17*B*—C18*B*	1.386 (5)
C18*A*—C19*A*	1.382 (4)	C18*B*—C19*B*	1.375 (5)
C19*A*—O23*A*	1.363 (4)	C19*B*—O23*B*	1.368 (4)
C19*A*—C20*A*	1.390 (4)	C19*B*—C20*B*	1.385 (5)
C20*A*—C21*A*	1.388 (4)	C20*B*—C21*B*	1.385 (5)

C21*A*—C1*A*—C2*A*—C3*A*	172.9 (3)
C1*A*—C2*A*—C3*A*—C4*A*	131.1 (4)
C2*A*—C3*A*—C4*A*—C5*A*	−64.6 (4)
C3*A*—C4*A*—C5*A*—C6*A*	−71.4 (4)
C4*A*—C5*A*—C6*A*—C7*A*	164.1 (3)
C5*A*—C6*A*—C7*A*—C8*A*	−67.3 (5)
C6*A*—C7*A*—C8*A*—C9*A*	−73.8 (5)
C7*A*—C8*A*—C9*A*—C10*A*	175.4 (3)
C8*A*—C9*A*—C10*A*—O11*A*	−71.9 (4)
C9*A*—C10*A*—O11*A*—C12*A*	−160.7 (3)
C10*A*—O11*A*—C12*A*—C16*A*	−177.6 (3)
O11*A*—C12*A*—C16*A*—C21*A*	−21.7 (4)
C12*A*—C16*A*—C21*A*—C1*A*	−14.0 (5)
C2*A*—C1*A*—C21*A*—C16*A*	155.8 (3)
C21*B*—C1*B*—C2*B*—C3*B*	174.5 (3)
C1*B*—C2*B*—C3*B*—C4*B*	122.0 (4)
C2*B*—C3*B*—C4*B*—C5*B*	−64.6 (4)
C3*B*—C4*B*—C5*B*—C6*B*	−71.1 (4)
C4*B*—C5*B*—C6*B*—C7*B*	177.0 (3)
C5*B*—C6*B*—C7*B*—C8*B*	−66.0 (4)
C6*B*—C7*B*—C8*B*—C9*B*	−72.9 (4)
C7*B*—C8*B*—C9*B*—C10*B*	163.9 (3)
C8*B*—C9*B*—C10*B*—O11*B*	−75.2 (4)
C9*B*—C10*B*—O11*B*—C12*B*	−158.3 (3)
C10*B*—O11*B*—C12*B*—C16*B*	−171.7 (3)
O11*B*—C12*B*—C16*B*—C21*B*	−23.6 (5)
C12*B*—C16*B*—C21*B*—C1*B*	−13.9 (5)
C2*B*—C1*B*—C21*B*—C16*B*	156.7 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O22*A*—H23*A*···O13*A*	0.94 (7)	1.69 (6)	2.592 (3)	159 (6)
O23*A*—H24*A*···O*W*3	0.86 (5)	1.77 (5)	2.632 (4)	171 (5)
O15*A*—H20*A*···O*W*2^{i}	0.94 (6)	1.79 (6)	2.698 (4)	162 (5)
O22*B*—H23*B*···O13*B*	0.89 (6)	1.79 (6)	2.580 (4)	147 (5)
O23*B*—H24*B*···O*W*1^{ii}	0.84 (4)	1.94 (4)	2.767 (3)	165 (3)
O15*B*—H20*B*···O15*A*^{iii}	0.86 (5)	2.03 (6)	2.886 (4)	172 (4)
O*W*1—H1O*W*···O23*A*^{iv}	1.02 (6)	1.90 (6)	2.840 (4)	151 (4)
O*W*1—H2O*W*···O15*B*^{v}	0.77 (7)	1.99 (7)	2.754 (4)	171 (7)
O*W*2—H3O*W*···O*W*1	0.90 (7)	1.96 (7)	2.842 (4)	168 (6)
O*W*2—H4O*W*···O22*A*	1.06 (7)	1.96 (7)	2.968 (4)	157 (5)
O*W*3—H5O*W*···O23*B*^{vi}	0.88 (7)	2.03 (6)	2.810 (4)	148 (5)
O*W*3—H6O*W*···O15*A*^{vii}	0.99 (10)	1.82 (10)	2.794 (4)	167 (9)

Symmetry codes: (i) $x-1, y-1, z-1$; (ii) $x, y, z-1$; (iii) $1+x, 2+y, 1+z$; (iv) $x, 1+y, z$; (v) $x-1, y-1, z$; (vi) $x, y-1, 1+z$; (vii) $1+x, 1+y, 1+z$.

Data collection: *P*4 (*XSCANS*; Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1204). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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17β-Estradiol 3-Benzoate†

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Abstract

In the title compound, 17β-hydroxyestra-1,3,5(10)-trien-3-yl benzoate, $C_{25}H_{28}O_3$, the *B*, *C* and *D* rings adopt envelope, chair and envelope conformations, respectively. Both phenyl rings are planar. The structure

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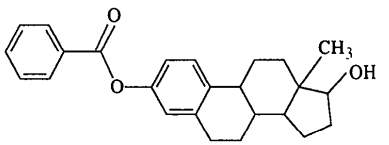
is internally stabilized by C—H···O hydrogen-bond interactions and is also externally stabilized by one O—H···O and one C—H···O hydrogen bond.

Comment

17β-Estradiol 3-benzoate is a synthetic estrogen which has important effects on utero development, menstrual cycle and ovulation. It is responsible for secondary sexual features in women (Muray & Gilman, 1975) and is an important activator of the adenylate cyclase enzyme in the hypothalamus (Zubin & Defagot, 1994). Recently, it has been recognized as an estrogen and progesterone receptor expression regulator (Young, Nag & Crews, 1995). Furthermore, it is involved in glucose–plasma level reduction (Jaccoby, Arnon, Snapir & Robinson, 1995), in transport across the blood–brain barrier (Bishop & Simpinks, 1995) and thymus parenchyma (Martin, Casares, Alonso, Nieuwenhuis, Vicente & Zapata, 1995). Interestingly, 17β-estradiol 3-benzoate is associated with increased risk of mammary cancer development (Yamanouchi, Ishii, Susuki, Onada, Wakabayashi & Inano, 1995). In order to obtain detailed information of its molecular conformation, we determined the structure of the title compound, (I), using X-ray techniques.



(I)

Bond distances and angles are quite similar to those found in the related compound estradiol 3-*p*-bromobenzoate (Tsukuda, Sato, Shiro & Koyama, 1968). The molecule consists of two phenyl rings (*A* and *E*), two six-membered rings (*B* and *C*) and one five-membered ring (*D*). The *B*/*C* and *C*/*D* ring junctions are *trans*. According to the torsion angles (Table 2) and the puckering parameter values (φ_2, θ_2 and Q), the six-membered rings (*B* and *C*) occur in envelope and chair conformations, respectively, while ring *D* (φ_2 and Q) adopts an envelope conformation (Cremer & Pople, 1975). The two phenyl rings (*A* and *E*) are planar within 0.003 (3) and 0.008 (3) Å, respectively, and the dihedral angle between them is 62.4 (1)°.

The hydroxy group located at C17 forms an intermolecular hydrogen bond with a symmetry-related carbonyl O2 atom at a distance of 2.966 (3) Å; H···O2^{i} 2.28 (6) Å and O1—H···O2^{i} 134 (5)° [symmetry code: (i) $-x$, $y+\frac{1}{2}$, $-z-\frac{3}{2}$]. There is a short intramolecular C—H···O hydrogen bond [C19···O2 2.882 (4), H19···O2 2.54 (4) Å and C19—H19···O2 102 (3)°] which stabilizes the molecule internally. In addition, there is one intermolecular C1—H···O1 hydrogen-bond interaction which helps stabilize the molecules in the crystal; C1···O1^{ii} 3.162 (4), H1···O1^{ii} 2.33 (3) Å and C1—H1···O1^{ii} 144 (3)° [symmetry code: (ii) $-x+1$, $y-\frac{1}{2}$, $-z-\frac{3}{2}$] (Desiraju, 1991).

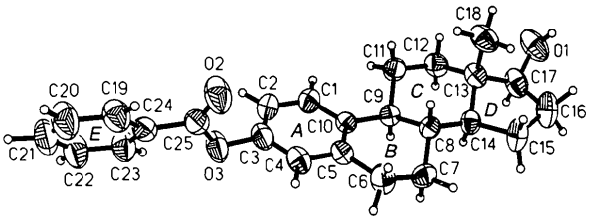


Fig. 1. The molecular structure of the title compound shown with the atom-labeling scheme and 50% probability displacement ellipsoids.

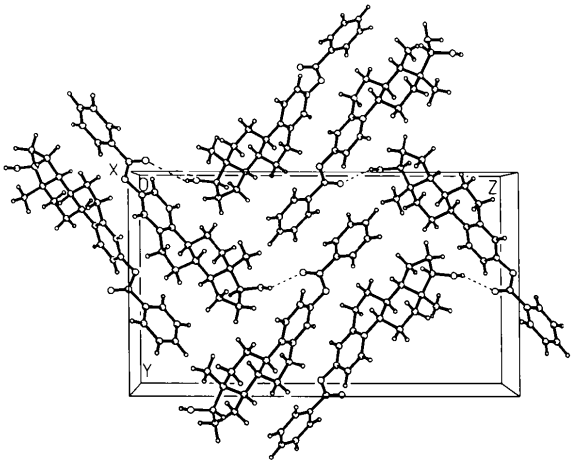


Fig. 2. A perspective drawing of the packing arrangement along the *a* axis, with dashed lines indicating the O—H···O hydrogen bonding.

Experimental

The title compound was purchased from the Sigma Chemical Company and crystallized from EtOH/MeOH (1:1) solution by slow evaporation of the solvent at room temperature.

Crystal data

$C_{25}H_{28}O_3$	Cu $K\alpha$ radiation
M_r = 376.47	λ = 1.54178 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 15–45°
a = 6.410 (3) Å	μ = 0.623 mm^{-1}
b = 13.340 (4) Å	T = 293 (2) K
c = 23.851 (5) Å	Rectangular
V = 2039.5 (12) Å^3	0.28 × 0.25 × 0.15 mm
Z = 4	Colorless
D_x = 1.226 Mg m^{-3}	
D_m = 1.225 Mg m^{-3}	
D_m measured by flotation in benzene/chloroform	

Data collection

$P4$ diffractometer	θ_{max} = 62.47°
$\theta/2\theta$ scans	h = 0 → 7
Absorption correction: none	k = 0 → 15
	l = 0 → 27

1888 measured reflections
1888 independent reflections
1785 observed reflections
$[I > 2\sigma(I)]$

3 standard reflections
frequency: 100 min
intensity decay: 2.0%

Refinement

Refinement on F^2
$R(F) = 0.0348$
$wR(F^2) = 0.0958$
$S = 1.039$
1888 reflections
365 parameters
All H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0576P)^2 + 0.2782P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.008$
$\Delta\rho_{max} = 0.118$ e Å^{-3}
$\Delta\rho_{min} = -0.094$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C1	0.2346 (5)	−0.3346 (2)	−0.5991 (1)	0.0579 (6)
C2	0.1615 (5)	−0.4069 (2)	−0.5629 (1)	0.0647 (7)
C3	−0.0255 (4)	−0.3909 (2)	−0.5360 (1)	0.0564 (6)
C4	−0.1390 (4)	−0.3059 (2)	−0.5443 (1)	0.0551 (6)
C5	−0.0662 (4)	−0.2324 (2)	−0.5815 (1)	0.0465 (5)
C6	−0.1976 (5)	−0.1401 (2)	−0.5907 (1)	0.0666 (8)
C7	−0.0867 (4)	−0.0568 (2)	−0.6217 (1)	0.0563 (6)
C8	0.0383 (4)	−0.0976 (2)	−0.6708 (1)	0.0416 (5)
C9	0.2103 (3)	−0.1671 (2)	−0.6484 (1)	0.0402 (5)
C10	0.1239 (3)	−0.2467 (2)	−0.6091 (1)	0.0420 (5)
C11	0.3443 (4)	−0.2104 (2)	−0.6963 (1)	0.0540 (6)
C12	0.4338 (4)	−0.1283 (2)	−0.7343 (1)	0.0554 (6)
C13	0.2635 (4)	−0.0588 (2)	−0.7563 (1)	0.0496 (5)
C14	0.1408 (4)	−0.0168 (2)	−0.7063 (1)	0.0460 (5)
C15	0.0107 (5)	0.0683 (2)	−0.7319 (1)	0.0707 (8)
C16	0.1522 (6)	0.1091 (3)	−0.7797 (2)	0.0830 (10)
C17	0.3419 (5)	0.0396 (2)	−0.7819 (1)	0.0626 (7)
C18	0.1257 (6)	−0.1143 (3)	−0.7989 (1)	0.0695 (8)
C19	−0.3752 (5)	−0.6860 (2)	−0.4658 (1)	0.0727 (8)
C20	−0.3742 (7)	−0.7629 (3)	−0.4263 (1)	0.0909 (12)
C21	−0.2178 (8)	−0.7701 (3)	−0.3879 (1)	0.0913 (11)
C22	−0.0633 (7)	−0.6994 (3)	−0.3865 (1)	0.0829 (9)
C23	−0.0612 (5)	−0.6224 (2)	−0.4253 (1)	0.0664 (7)
C24	−0.2163 (4)	−0.6159 (2)	−0.4652 (1)	0.0529 (6)
C25	−0.2116 (4)	−0.5371 (2)	−0.5093 (1)	0.0540 (6)
O1	0.4391 (4)	0.0315 (2)	−0.8353 (1)	0.0861 (7)
O2	−0.3017 (4)	−0.5410 (2)	−0.5531 (1)	0.0884 (7)
O3	−0.0857 (4)	−0.4615 (1)	−0.4948 (1)	0.0709 (6)

Table 2. *Selected geometric parameters (Å, °)*

C1—C2	1.378 (4)	C13—C17	1.533 (3)
C1—C10	1.391 (3)	C13—C14	1.536 (3)
C2—C3	1.377 (4)	C13—C18	1.536 (3)
C3—C4	1.361 (4)	C14—C15	1.535 (3)
C3—O3	1.414 (3)	C15—C16	1.556 (4)
C4—C5	1.401 (3)	C16—C17	1.530 (5)
C5—C10	1.398 (3)	C17—O1	1.422 (3)
C5—C6	1.509 (4)	C19—C24	1.384 (4)
C6—C7	1.511 (4)	C19—C20	1.392 (4)
C7—C8	1.519 (3)	C20—C21	1.361 (5)
C8—C14	1.521 (3)	C21—C22	1.367 (5)
C8—C9	1.535 (3)	C22—C23	1.382 (4)
C9—C10	1.522 (3)	C23—C24	1.379 (4)
C9—C11	1.541 (3)	C24—C25	1.487 (3)
C11—C12	1.533 (3)	C25—O2	1.195 (3)
C12—C13	1.525 (4)	C25—O3	1.337 (3)
C2—C1—C10	121.6 (3)	C17—C13—C14	99.5 (2)
C3—C2—C1	118.7 (3)	C12—C13—C18	110.3 (2)
C4—C3—C2	121.7 (2)	C17—C13—C18	109.7 (2)
C4—C3—O3	120.7 (2)	C14—C13—C18	113.3 (2)
C2—C3—O3	117.2 (2)	C8—C14—C15	120.8 (2)
C3—C4—C5	119.8 (2)	C8—C14—C13	113.3 (2)
C10—C5—C4	119.5 (2)	C15—C14—C13	103.9 (2)
C10—C5—C6	121.9 (2)	C14—C15—C16	103.5 (2)
C4—C5—C6	118.6 (2)	C17—C16—C15	106.1 (2)
C5—C6—C7	114.2 (2)	O1—C17—C16	115.2 (2)
C6—C7—C8	111.2 (2)	O1—C17—C13	115.8 (2)
C7—C8—C14	113.8 (2)	C16—C17—C13	104.2 (2)
C7—C8—C9	109.1 (2)	C24—C19—C20	119.2 (3)
C14—C8—C9	108.1 (2)	C21—C20—C19	120.7 (3)
C10—C9—C8	112.0 (2)	C20—C21—C22	120.1 (3)
C10—C9—C11	113.5 (2)	C21—C22—C23	120.2 (3)
C8—C9—C11	111.7 (2)	C24—C23—C22	120.1 (3)
C1—C10—C5	118.6 (2)	C23—C24—C19	119.6 (2)
C1—C10—C9	120.5 (2)	C23—C24—C25	121.2 (2)
C5—C10—C9	120.9 (2)	C19—C24—C25	119.1 (2)
C12—C11—C9	112.3 (2)	O2—C25—O3	123.4 (2)
C13—C12—C11	111.7 (2)	O2—C25—C24	125.3 (2)
C12—C13—C17	115.0 (2)	O3—C25—C24	111.3 (2)
C12—C13—C14	108.7 (2)	C25—O3—C3	119.2 (2)
C10—C5—C6—C7	−12.4 (4)	C11—C12—C13—C14	54.6 (3)
C5—C6—C7—C8	43.0 (3)	C9—C8—C14—C13	59.6 (2)
C6—C7—C8—C9	−63.5 (3)	C12—C13—C14—C8	−59.6 (2)
C7—C8—C9—C10	52.0 (2)	C17—C13—C14—C15	47.0 (2)
C14—C8—C9—C11	−55.3 (2)	C13—C14—C15—C16	−32.4 (3)
C6—C5—C10—C9	2.1 (3)	C14—C15—C16—C17	5.0 (3)
C8—C9—C10—C5	−22.4 (3)	C15—C16—C17—C13	24.1 (3)
C8—C9—C11—C12	53.8 (3)	C14—C13—C17—C16	−43.3 (3)
C9—C11—C12—C13	−53.4 (3)		

Data collection: *P4* diffractometer software. Cell refinement: *XSCANS* (Siemens, 1991). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1186). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5,17-Diethoxycarbonyl-25,26,27,28-tetrahydroxycalix[4]arene

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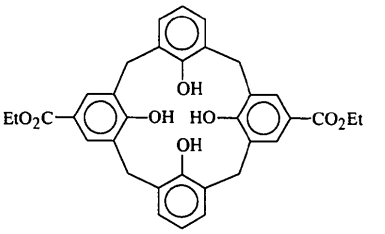
Abstract

The title compound, diethyl 25,26,27,28-tetrahydroxypentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25),3,5,7(18),9,11,13(27),15,17,19(26),21,23-dodecaene-5,17-dicarboxylate, $C_{34}H_{32}O_8$, a tetrahydroxy calix[4]arene, adopts an open-cone conformation in the solid state; this conformation is dictated by the presence of intramolecular O—H···O hydrogen bonding of the phenolic OH groups [O···O 2.673 (2)–2.708 (2) Å]. The planes of the aromatic rings are inclined at 120.8 (1), 133.1 (1), 118.0 (1) and 128.5 (1)° to the plane of the methylene C atoms which link them. Self-inclusion occurs when the molecules assemble in pairs about inversion centres.

Comment

The use of calixarenes as building blocks for the construction of more sophisticated host molecules and larger molecular assemblies requires calixarene derivatives with various functional groups (Böhmer, 1995). For their synthesis, two principally different strategies are available: the more-or-less selective modification of the easily accessible *tert*-butylcalixarenes (van Loon, Verboom & Reinhoudt, 1992) or the direct synthesis of special calixarenes by fragment condensation (Böhmer, Marschollek & Zetta, 1987; Böhmer, Merkel & Kunz, 1987), and of course both strategies may be combined. The diester title compound (I) was synthesized as part of a series of such synthetic studies.



(I)

A view of (I) with our crystallographic numbering scheme is given in Fig. 1. Molecular dimensions are normal [*e.g.* mean aromatic C—C 1.388 (8) Å, mean C_{ar}—O_{phenol} 1.378 (7) Å]. The molecule has an 'open-cone' conformation dictated by the intramolecular O—H···O hydrogen bonding of the phenolic OH groups with O···O 2.673 (2)–2.708 (2) Å. The phenolic H atoms are disordered equally over two sites to give two sets of intramolecular eight-membered (O—H···)$_4$ rings. The open-cone conformation is quantitatively described by the interplanar angles between the planes of the aromatic rings (*A–D* as indicated by the atomic labelling) and the plane of the methylene C atoms (C7*A*,C7*B*,C7*C*,C7*D*) which link them; these values are 120.8 (1), 133.1 (1), 118.0 (1) and 128.5 (1)° for aromatic rings *A* to *D*, respectively. An alternative way of describing calixarene conformations involving the values of torsion angles about the methylene CH_2 atoms was introduced by Ugozzoli & Andreetti (1992); these values are shown in Table 2.

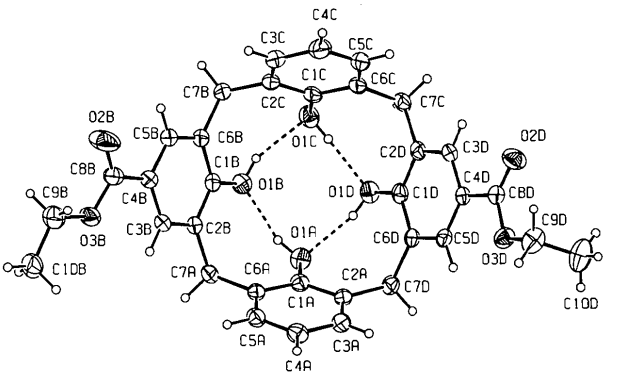


Fig. 1. A view of (I) with our numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Only one of the two orientations of the disordered phenolic H atoms is shown.

Pairs of molecules pack about inversion centres (Fig. 2) such that the ethyl C atoms C9*D* and C10*D* of one molecule lie in the cup of the inversion-related molecule to generate loosely held dimers. The shortest intermolecular contact (calix cup···ethyl-C atom) is 3.577 (3) Å between calix ring atom C5*D* and ethyl C atom C9*D* (at equivalent position 1 $-x$, 1 $-y$, $-z$).