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cis-3,3′-Dimethyldibenzo-18-crown-6

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Key indicators

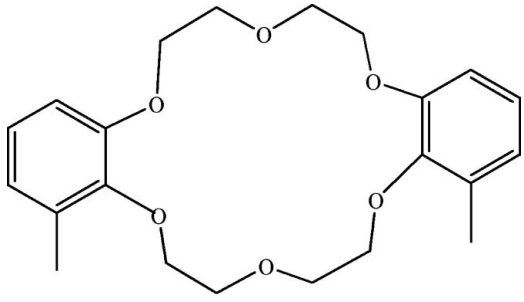
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.062
wR factor = 0.210
Data-to-parameter ratio = 14.7

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

In the title molecule (systematic name: 10,26-dimethyl-2,5,8,15,18,21-hexaoxatricyclo[20.4.0.0^{9,14}]tetracosa-9,11,13,-22,24,26-hexaene), $C_{22}H_{28}O_6$, the two benzene rings make a dihedral angle of 63.9 (2)°, indicating a boat-like molecular conformation. The crystal packing is mainly stabilized by van der Waals forces.

Comment

In continuation of our ongoing programme directed to the development of crown ether chemistry (Niu *et al.*, 2005), we present here the synthesis and crystal structure of the title compound, (I) (Fig. 1).



(I)

In (I), the average Csp^2—O bond length is 1.372 (4) Å (see also Table 1). The interatomic distances O1···O2 [2.664 (4) Å] and O4···O5 [2.641 (4) Å] are somewhat shorter than those reported earlier (Yang *et al.*, 1988). The other intramolecular O···O distances lie in the range 2.941 (4)–3.592 (4) Å. The two benzene rings make a dihedral angle of 63.9 (2)°, indicating a boat-like conformation of the molecule. The crystal packing is mainly stabilized by van der Waals forces.

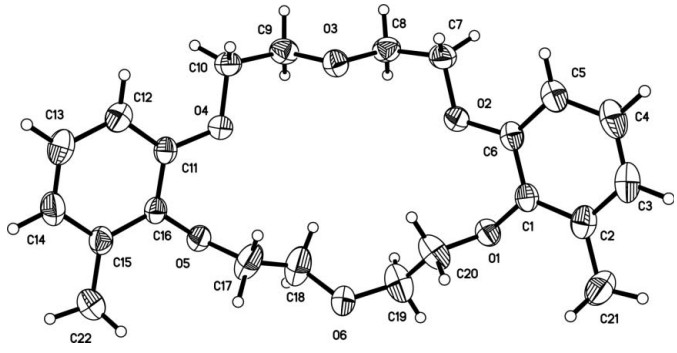


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The reaction was carried out under a nitrogen atmosphere. 3-Methylcatechol (19.84 g, 0.16 mol), sodium hydroxide (6.4 g, 0.16 mol) and dichloroethyl ether (12 g, 0.084 mol) were in turn added to a solution of isopentyl alcohol (80 ml) in a four-mouth flask and the mixture was stirred for 2.5 h. After cooling to room temperature, the mixture was filtered. The solid was recrystallized from *n*-heptane to obtain crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_{22}H_{28}O_6$
M_r = 388.44
Monoclinic, $P2_1/n$
a = 9.354 (3) Å
b = 7.828 (3) Å
c = 29.122 (11) Å
β = 95.386 (5)°
V = 2123.0 (13) Å^3
Z = 4
D_x = 1.215 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colourless
0.40 × 0.32 × 0.28 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.976
10489 measured reflections
3714 independent reflections
1897 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.210
S = 1.00
3714 reflections
253 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0806P)^2 + 1.8496P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.375 (4)	O3—C8	1.415 (5)
O1—C20	1.435 (5)	O3—C9	1.416 (5)
O2—C6	1.369 (4)	O4—C11	1.365 (4)
O2—C7	1.424 (4)	O4—C10	1.426 (4)

All H atoms were placed geometrically (C—H = 0.93–0.97 Å) and treated as riding on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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