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Cyclooctane-1,2,5,6-tetrayl tetraacetate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.055
wR factor = 0.148
Data-to-parameter ratio = 19.1

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

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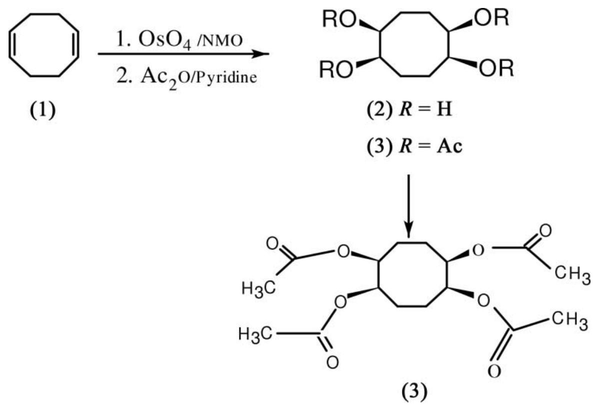
In the title compound, $C_{16}H_{24}O_8$, the cyclooctane ring has a non-planar structure. Weak intermolecular C—H···O hydrogen bonding occurs in the crystal structure.

Comment

Development of an enantioselective catalytic version of the dihydroxylation reaction based on cinchona alkaloids facilitated preparation of highly oxygenated molecules (Kolb *et al.*, 1994). The osmium-catalysed bis-dihydroxylation of 1,5-cyclooctadiene, (1), has utilized cyclooctane-1,2,5,6-tetrol, (2), as a scaffold for a supramolecule (Takata *et al.*, 1997; Kawazoe *et al.*, 2001). The title compound, (3), is a derivative of (2). Here we report the crystal structure of (3) in order to confirm its formation and understand its reactivity.



The molecule of (3) (Fig. 1) consists of a cyclooctyl ring carrying four substituents; bond distances are normal. The cyclooctane system has a non-planar structure; puckering coordinates (Cremer & Pople, 1975) are as follows: φ_2 = −161.68 (7)°, φ_3 = 167.2 (2)°, θ_2 = 0.219 (3)°, θ_3 = 0.012 (3)°, θ_4 = 0.808 (3)° and Q_T = 0.837 (3) Å.

Weak intermolecular C—H···O hydrogen bonding occurs in the crystal structure (Table 1).

Experimental

Compound (3) was prepared according to literature methods (Kawazoe *et al.*, 2001; VanRheenen *et al.*, 1976). A 100 ml three-necked round-bottomed flask equipped with a magnetic stirrer and a nitrogen inlet was charged with 2.63 g (19.44 mmol) of *N*-methylmorpholine *N*-oxide (NMO), 8 ml of water and 6 ml of acetone. To this solution were added *ca* 62 mg of OsO_4 (0.248 mmol) and 1.0 g (9.25 mmol) of 1,5-cyclooctadiene, (1). The resulting mixture was stirred vigorously under nitrogen at room temperature. During the overnight stirring, the reaction mixture became homogeneous. After 14 h, the reaction was complete. Sodium hydrosulfite (1.0 g) and 4.0 g

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of Florisil slurried in 8 ml of water were added, the slurry was stirred for 10 min. and the mixture was filtered through a pad of 0.5 g of Celite in a 50 ml sintered-glass funnel. The Celite cake was washed with acetone (4 × 40 ml). The mixture was evaporated and the residue was chromatographed over silica gel (eluent: water/ethanol/ethyl acetate 1:2:5) to afford tetrol (2) (97%) (Kawazoe *et al.*, 2001). Tetrol (2) was acetylated as described in the literature (Powell *et al.*, 1972). Recrystallization of compound (3) from dichloromethane/hexane (1:3) gave colourless crystals of (3) (m.p. 365–367 K).

Crystal data

$C_{16}H_{24}O_8$
$M_r = 344.35$
Monoclinic, $P2_1/c$
$a = 11.8989$ (12) Å
$b = 7.7568$ (8) Å
$c = 20.080$ (2) Å
$\beta = 93.567$ (2)°
$V = 1849.7$ (3) Å^3
$Z = 4$
$D_x = 1.237$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.32 × 0.26 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
9906 measured reflections
4150 independent reflections
1635 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.066$
$\theta_{\rm max} = 28.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.148$
$S = 0.84$
4150 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.22$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C4—H4A⋯O6$^{\rm i}$	0.97	2.58	3.510 (3)	160

Symmetry code: (i) $-x+1, -y+2, -z$.

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined to fit the electron density; $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. Other H atoms were placed in calculated positions, with C—H = 0.97 (methylene) or 0.98 Å (methine), and refined in riding mode with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

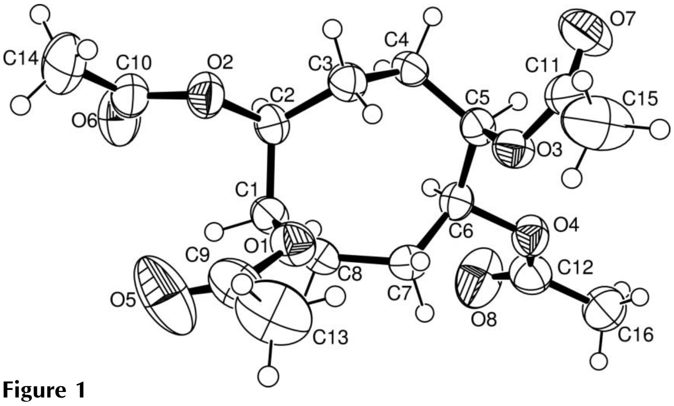

Figure 1
The molecular structure of (3), shown with 70% probability displacement ellipsoids (arbitrary spheres for H atoms).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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