organic compounds

(2R*,4R*,7S*,10R*,12R*)-3,11,13,15-Tetraoxapentacyclo[5.5.3.0^{1,7}.0^{2,4}.-0^{10,12}]pentadeca-5,8-dien-14-one

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The title compound, $C_{11}H_8O_5$, features a 'skipped' diene, an *anti*-bis(epoxide) and a cyclic carbonate, all embedded in a densely functionalized [4.4.3]propellane scaffold. The crystal packing of this diepoxide is effected primarily by C—H···O hydrogen bonds, which link the molecules into tapes along the b axis. Inter-tape connectivity is brought about by centrosymmetrically disposed pairs of C···O contacts [3.183 (4) Å] between the C$^{\delta+}$=O$^{\delta-}$ dipoles of neighbouring carbonate moieties.

Related literature

For our report on the crystal structure of (1*s*,6*s*)-11,13-dioxatricyclo[4.4.3.0^{1,6}]trideca-2,4,7,9-tetraen-12-one, the synthetic precursor of the title compound, see: Mehta & Sen (2011). For salient references related to the chemistry of molecules, featuring two abutting 1,3-cyclohexadiene (CHD) units embedded in a rigid 11,13-dioxa[4.4.3]propellane framework, see: Ashkenazi *et al.* (1978); Paquette *et al.* (1990). For references representing our own previous studies on the modes of self-assembly in oxygenated CHDs, see: Mehta & Sen (2010) and citations therein. For a reference related to *VEGA ZZ* 3.0.0 the program used to generate the MEP surface diagram of the title compound, see: Pedretti *et al.* (2004). For a discussion on graph-set analysis of hydrogen bonds, see: Bernstein *et al.* (1995); Etter *et al.* (1990). For a discussion on ring-puckering parameters, see: Cremer & Pople (1975). For details of the Cambridge Structural Database, see: Allen (2002); Bruno *et al.* (2002).
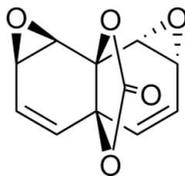
Experimental

Crystal data

$C_{11}H_8O_5$	V = 914.3 (6) Å^3
M_r = 220.17	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 7.170 (3) Å	μ = 0.13 mm^{-1}
b = 7.809 (3) Å	T = 291 K
c = 16.357 (6) Å	0.23 × 0.17 × 0.13 mm
β = 93.301 (6)°	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	6504 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	1691 independent reflections
	1319 reflections with $I > 2\sigma(I)$
T_{min} = 0.971, T_{max} = 0.984	R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.072	145 parameters
$wR(F^2)$ = 0.162	H-atom parameters constrained
S = 1.18	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
1691 reflections	$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4^i	0.98	2.53	3.509 (5)	174
C3—H3···O5^{ii}	0.98	2.55	3.313 (4)	134
C10—H10···O5^i	0.98	2.49	3.379 (4)	151

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, y + 1, z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2009).

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supporting information

(2*R**,4*R**,7*S**,10*R**,12*R**)-3,11,13,15-Tetraoxapentacyclo-[5.5.3.0^{1,7}.0^{2,4}.0^{10,12}]pentadeca-5,8-dien-14-one

Goverdhan Mehta, Saikat Sen and C. S. Ananda Kumar

S1. Comment

In a recent communication, we had introduced the C—H⋯O hydrogen bonded self-assembly of the crystalline tetraene **1** – the synthetic precursor of the title compound **2** (Fig. 1) – as a foil to highlight the rather singular crystal packing (lacking in any obvious hydrogen bonds) observed in two structurally related [4.4.3]propellanes (Mehta & Sen, 2011). During the course of this study, we recognized that the tricyclic carbonate **1** features a novel structural attribute – namely, two abutting 1,3-cyclohexadiene (CHD) units embedded in a rigid 11,13-dioxa[4.4.3]propellane framework (Ashkenazi *et al.*, 1978; Paquette *et al.*, 1990). In view of our on-going activity in delineating the patterns of self-assembly in oxygenated CHDs (Mehta & Sen, 2010), it was of considerable interest to investigate the diversely oxy-functionalized CHD moieties that might be accessed from the tetraene **1**.

m-Chloroperbenzoic acid (*m*CPBA) mediated epoxidation of **1** was carried out and though sluggish, led to the formation of the title compound **2** in presence of excess peracid (Fig. 1). Routine characterization of **2** by NMR spectroscopy revealed it to be the product of an unsymmetrical *bis*-epoxidation of the C_{2v}-symmetric **1**. This was decidedly an uncanny result that was quite unpredictable and could not be readily reconciled with stereo-electronic preferences. In order to settle its stereo-structure by single-crystal XRD analysis, the diepoxide **2** was crystallized by slow evaporation of its saturated solution in 1:2 EtOAc-hexanes.

The crystal structure of **2** was solved and refined in the centrosymmetric monoclinic space group $P2_1/c$ ($Z = 4$). Interestingly, the two epoxy functionalities in **2** were found to have a 1,4-*anti* relationship to one another (Fig. 2). As suggested by their conformational analysis [Ring C1–C6: $q_2 = 0.225$ (3) Å, $q_3 = 0.086$ (3) Å, $\varphi_2 = 322.5$ (9)°, $Q_T = 0.241$ (3) Å, $\theta_2 = 69.2$ (7)°; Ring C1/C6–C10: $q_2 = 0.201$ (4) Å, $q_3 = -0.080$ (4) Å, $\varphi_2 = 200.6$ (10)°, $Q_T = 0.215$ (3) Å, $\theta_2 = 111.8$ (11)°], the two cyclohexene rings in **2** adopt a puckered form, somewhat intermediate between a pure twist-boat (*TB*) and a pure half-chair (*HC*) conformation (Cremer & Pople, 1975). Puckering parameters of the central five-membered ring [$q_2 = 0.238$ (3) Å, $\varphi_2 = 51.9$ (7)°] are close to the values expected for an ideal C_2-symmetric 'twist′ (*T*) conformation (Cremer & Pople, 1975).

Molecular packing in **2** was brought about primarily *via* the agency of C—H⋯O hydrogen bonds which linked the molecules into zigzag tapes essentially along the *b* axis (Fig. 3). Within each of these tapes, a pair of hydrogen bonds (C2—H2⋯O4 and C10—H10⋯O5), defining an $R^2_2(11)$ motif (Etter *et al.*, 1990; Bernstein *et al.*, 1995) connected the diepoxide molecules related by the 2_1 symmetry, while another H-bond (C3—H3⋯O5) consolidated the architecture by linking the molecules, translated along the *b* axis. While no inter-tape hydrogen bonds were observed, a closer analysis of the crystal packing in **2** revealed C⋯O short contacts [C11⋯O5, $d = 3.183$ (4) Å, symmetry code: $-x + 1$, $-y$, $-z + 1$] between the molecular tapes along the longest *c* axis. These C⋯O contacts involved centrosymmetrically disposed pairs of neighbouring $C^{\delta+}$═$O^{\delta-}$ dipoles (Fig. 3) and described an interesting cyclic dipolar interaction motif, much akin to an

$R^2_2(4)$ O—H⋯O hydrogen bonding loop. In fact, a Cambridge Structural Database search [CSD version 5.33 (November 2011); Allen, 2002; Bruno *et al.*, 2002] with such a C⋯O interaction quadrilateral involving the carbonyl group of carbonate moieties (Fig. 4), generated only 10 hits (CSD codes: ARUJEC, CHPCBO, JOKSUX, MHIQXI, QENJUP, SECPUL, SEGCAI, VECQUP, WIZQOL and XEXVEB). Even within this coterie, the observed C⋯O interaction motif was, in most cases, adventitious/supportive in nature and resulted merely on account of centrosymmetrically related pairs of hydrogen bonds forcing the carbonyls to approach closer to one another. It is worth mentioning at this point that the centrosymmetric C⋯O interaction motif in **2**, though unsupported by hydrogen bonds, might owe its existence to a congenial synergy between the shape and charge distribution in the molecule. As illustrated in Figure 5, each of the two carbonyl groups, involved in the C⋯O short contacts, fits in a complementary lock-and-key fashion within the single accessible 'groove′ (defined by the central five-membered ring and the cyclohexene, bearing the *endo* epoxy moiety) that the diepoxide **2** presents. Coincidentally, this groove also bears that 'face′ of the carbonyl functionality in **2** which features a well defined $C^{\delta+}=O^{\delta-}$ dipolar charge separation (Fig. 6).

To summarize, we have provided herein the first report of an attempted oxyfunctionalization of the tetraene **1** and the complete structural elucidation of the diepoxide **2**, obtained in the endeavour. The supramolecular structure of **2**, as obtained from the analysis of its single-crystal XRD data, was found to be quite noteworthy, particularly because the carbonyl group of the carbonate moiety in **2** not only functioned as the donor in two C—H⋯O hydrogen bonds, but participated in a scarcely encountered cyclic dipolar interaction motif as well.

S2. Experimental

As delineated in Figure 1, the title compound was obtained by *m*CPBA mediated epoxidation of the tetraene **1**. Thus, **1** (0.188 g, 1.00 mmol) was dissolved in dichloromethane (8 ml) and solid *m*-chloroperbenzoic acid (70% purity, 0.740 g, 3.00 mmol) was added portion wise to the stirred solution, cooled on an ice-bath. Thereafter, the reaction mixture was allowed to stir while gradually warming to room temperature on its own. The progress of the reaction was monitored by thin layer chromatography. Even after allowing the reaction to proceed for three days at room temperature, the TLC profile showed no apparent change beyond the disappearance of the starting material and the formation of **2** as the predominant product. The excess peracid was therefore decomposed with saturated Na_2SO_3 solution and the reaction mixture extracted thrice with dichloromethane. The combined extracts were washed with saturated $NaHCO_3$ solution and then dried over anhydrous Na_2SO_4. Evaporation of the solvent and purification of the residue by column chromatography over silica gel with 40% EtOAc-hexanes furnished the pure diepoxide **2** (0.170 g, 77%) as a colourless solid. M.p. 158 – 159 °C.

Single crystals of the diepoxide **2**, suitable for X-ray diffraction, were obtained by slow evaporation of its saturated solution in 1:2 EtOAc-hexanes.

S3. Refinement

H atoms were placed in geometrically idealized positions with C—H distances 0.93 or 0.98 Å and allowed to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$.
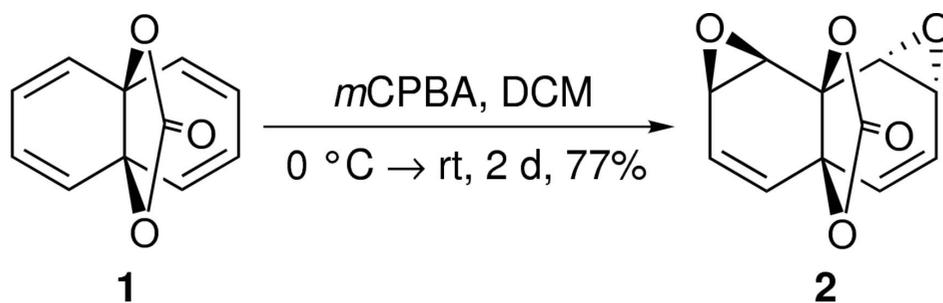
Figure 1

Preparation of the diepoxide **2** from the tetraene **1**.
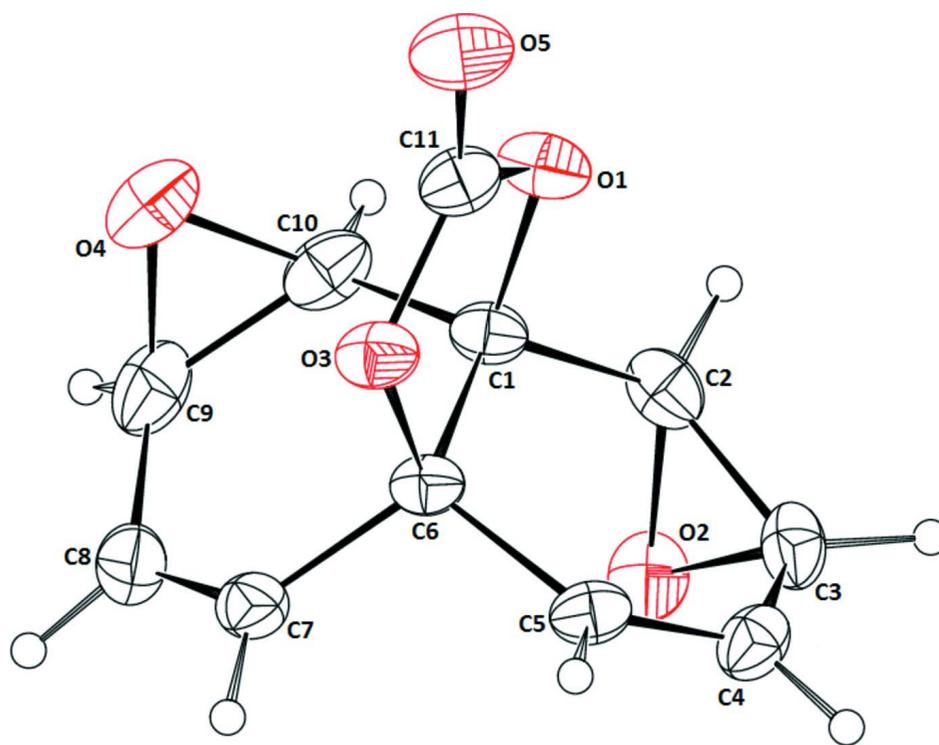
Figure 2

View of the diepoxide **2**, with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radii.
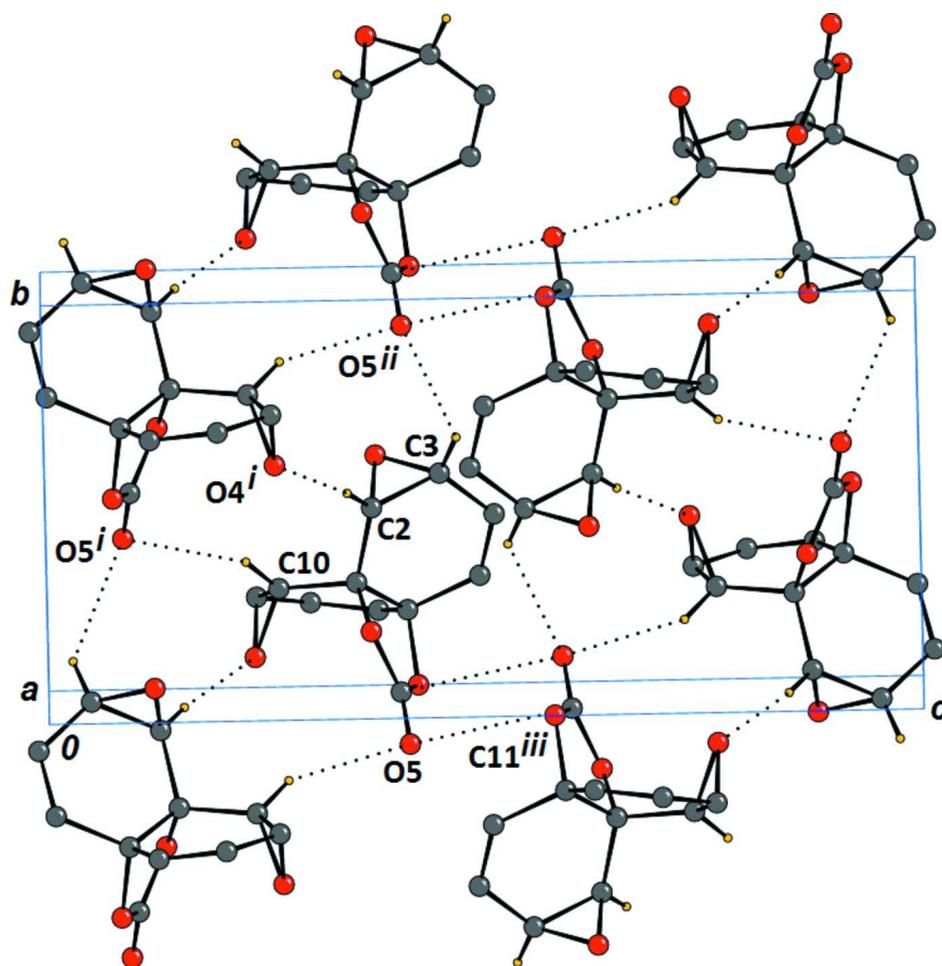
Figure 3

The molecular packing of the diepoxide **2**. Non-interacting hydrogen atoms have been omitted for the sake of clarity. Dotted lines indicate the C—H···O hydrogen bonds and C···O short contacts. Symmetry codes: (i) $-x + 1$, $y + 1/2$, $-z + 1/2$; (ii) $x, y + 1, z$; (iii) $-x + 1, -y, -z + 1$.
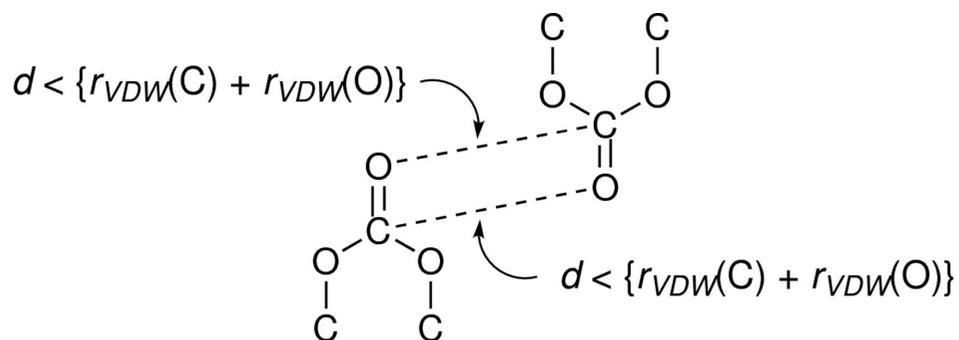
Figure 4

The centrosymmetric C···O interaction quadrilateral, employed in the CSD search.
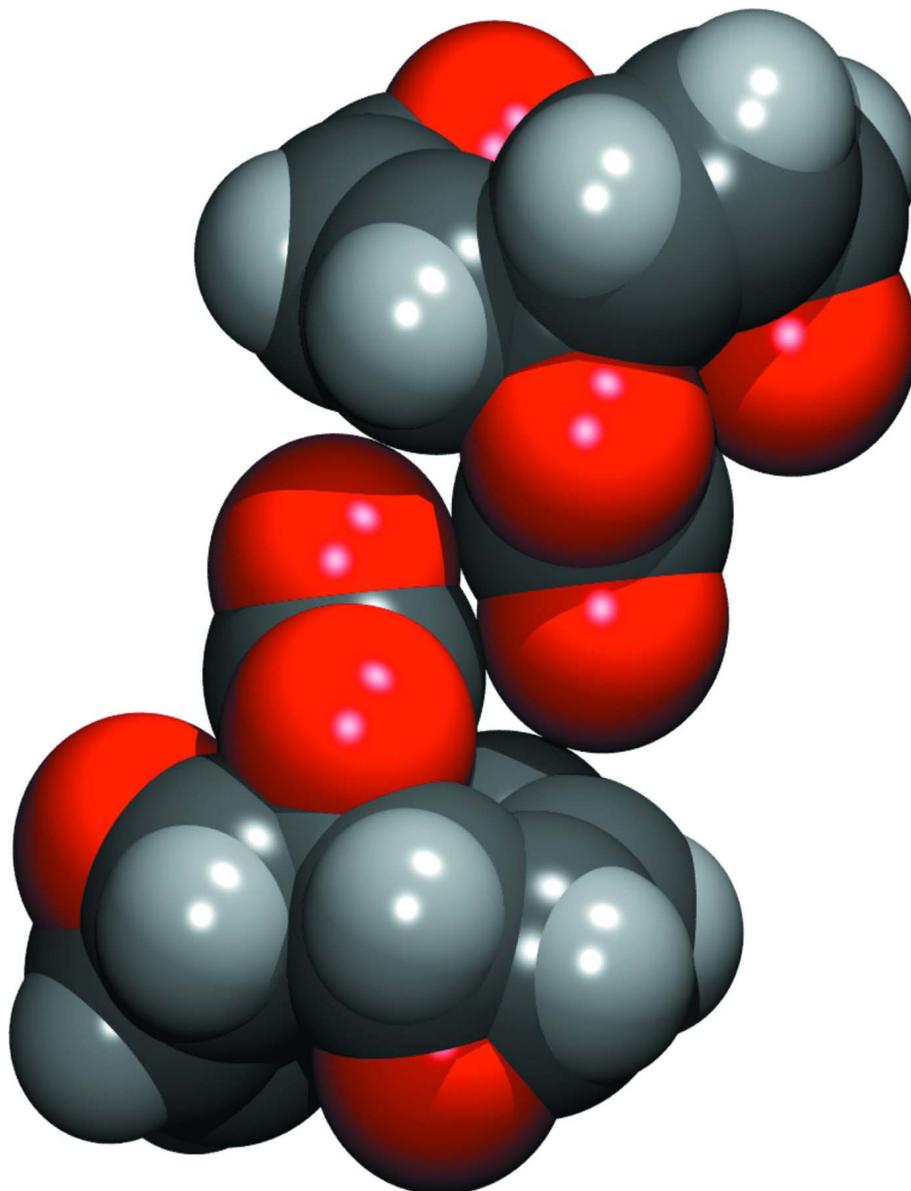
Figure 5

Space filling representation of two molecules of the diepoxide **2**, involved in the cyclic centrosymmetric C···O interaction motif.
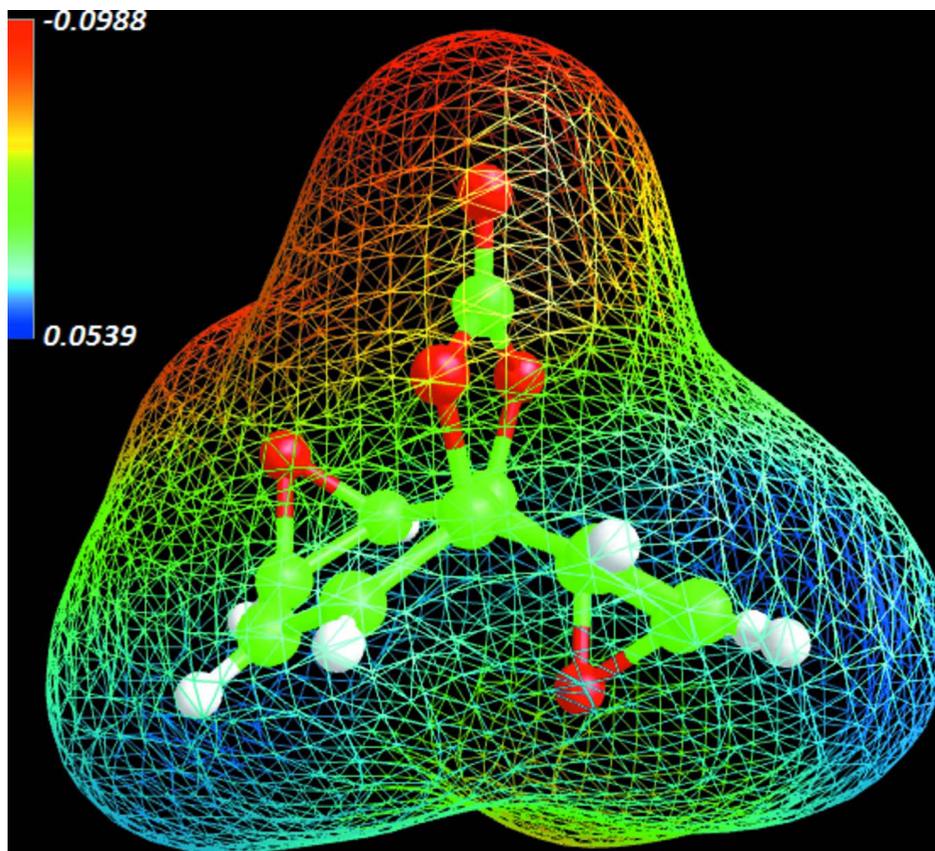
Figure 6

Molecular electrostatic potential (MEP) surface diagram of the diepoxide **2**, as generated by VEGA ZZ 3.0.0 (Pedretti *et al.*, 2004). The MEP calculation was performed after the semi-empirical charges were assigned by a single point MOPAC calculation, employing the MNDO method.

(2*R**,4*R**,7*S**,10*R**,12*R**)-3,11,13,15-Tetraoxapentacyclo[5.5.3.0^{1,7}.0^{2,4}.0^{10,12}]pentadeca-5,8-dien-14-one

Crystal data

$C_{11}H_8O_5$	$F(000) = 456$
$M_r = 220.17$	$D_x = 1.599$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2221 reflections
$a = 7.170$ (3) Å	$\theta = 2.9–25.2°$
$b = 7.809$ (3) Å	$\mu = 0.13$ mm^{-1}
$c = 16.357$ (6) Å	$T = 291$ K
$\beta = 93.301$ (6)°	Block, colorless
$V = 914.3$ (6) Å^3	$0.23 \times 0.17 \times 0.13$ mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
Radiation source: fine-focus sealed tube	$T_{min} = 0.971$, $T_{max} = 0.984$
Graphite monochromator	6504 measured reflections
φ and ω scans	1691 independent reflections
	1319 reflections with $I > 2\sigma(I)$

$R_{int} = 0.040$

$\theta_{max} = 25.4°$, $\theta_{min} = 2.5°$

$h = -8 \rightarrow 8$

$k = -9 \rightarrow 9$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.072$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.162$	
$S = 1.18$	H-atom parameters constrained
1691 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.6586P]$
145 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.33$ e Å^{-3}
	$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. IR (KBr, v cm^{-1}) 3057, 2203, 1805, 1524, 1204, 1263, 1032, 943, 854, 758, 725, 665, 619; ^{1}H NMR (400 MHz, CDCl$_3$, δ, p.p.m.) 6.46 (dd, $J = 8$, 3 Hz, 1H), 6.34 (dd, $J = 8$, 3 Hz, 1H), 5.91 (d, $J = 8$ Hz, 1H), 5.84 (dd, $J = 8$, 1 Hz, 1H), 4.00 (d, $J = 3$ Hz, 1H), 3.85 (d, $J = 3$ Hz, 1H), 3.73 (ddd appearing as a dt, $J = 3$, 1 Hz, 1H), 3.57 (ddd appearing as a dt, $J = 3$, 1 Hz, 1H); ^{13}C NMR (100 MHz, CDCl$_3$, δ, p.p.m.) 151.7, 130.9, 129.8, 128.1, 125.4, 78.3, 76.2, 56.8, 51.8, 49.5, 46.6.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.5301 (3)	0.1663 (3)	0.36989 (14)	0.0500 (7)
O2	0.7967 (3)	0.5656 (3)	0.37864 (14)	0.0514 (7)
O3	0.7693 (3)	0.0174 (3)	0.42208 (14)	0.0432 (6)
O4	0.7672 (4)	0.0902 (3)	0.23628 (15)	0.0688 (8)
O5	0.4863 (4)	−0.1009 (3)	0.41171 (16)	0.0638 (8)
C1	0.6909 (4)	0.2708 (4)	0.35416 (19)	0.0373 (8)
C2	0.6407 (5)	0.4528 (4)	0.3733 (2)	0.0447 (8)
C3	0.6966 (5)	0.5281 (4)	0.4515 (2)	0.0468 (9)
C4	0.7949 (5)	0.4205 (4)	0.5129 (2)	0.0445 (8)
C5	0.8586 (4)	0.2693 (4)	0.49495 (19)	0.0400 (8)
C6	0.8441 (4)	0.1914 (4)	0.41165 (18)	0.0340 (7)
C7	1.0317 (5)	0.1766 (4)	0.3782 (2)	0.0430 (8)
C8	1.0625 (5)	0.1947 (5)	0.3007 (2)	0.0538 (10)
C9	0.9122 (6)	0.2221 (5)	0.2393 (2)	0.0593 (11)
C10	0.7260 (5)	0.2568 (4)	0.2652 (2)	0.0511 (9)
C11	0.5881 (5)	0.0159 (4)	0.4026 (2)	0.0442 (8)
H10	0.6466	0.3279	0.2281	0.061*

H2	0.5258	0.4974	0.3458	0.054*
H3	0.6167	0.6189	0.4716	0.056*
H4	0.8127	0.4607	0.5663	0.053*
H5	0.9171	0.2057	0.5371	0.048*
H7	1.1331	0.1531	0.4145	0.052*
H8	1.1847	0.1899	0.2846	0.065*
H9	0.9455	0.2714	0.1870	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0374 (13)	0.0479 (14)	0.0635 (15)	−0.0075 (11)	−0.0084 (11)	0.0113 (12)
O2	0.0623 (17)	0.0365 (13)	0.0565 (15)	−0.0047 (11)	0.0147 (12)	0.0086 (11)
O3	0.0452 (14)	0.0288 (12)	0.0544 (14)	−0.0040 (10)	−0.0083 (10)	0.0050 (10)
O4	0.090 (2)	0.0617 (17)	0.0536 (16)	−0.0063 (16)	−0.0078 (14)	−0.0158 (13)
O5	0.0650 (18)	0.0488 (15)	0.0770 (19)	−0.0260 (14)	−0.0016 (14)	0.0047 (13)
C1	0.0344 (18)	0.0367 (18)	0.0401 (18)	−0.0036 (14)	−0.0049 (13)	0.0074 (14)
C2	0.0398 (19)	0.0388 (19)	0.056 (2)	0.0083 (15)	0.0026 (15)	0.0143 (16)
C3	0.050 (2)	0.0364 (18)	0.056 (2)	0.0000 (15)	0.0219 (17)	−0.0005 (16)
C4	0.055 (2)	0.0409 (19)	0.0387 (18)	−0.0115 (16)	0.0104 (15)	−0.0048 (15)
C5	0.0436 (19)	0.0398 (19)	0.0358 (17)	−0.0093 (15)	−0.0051 (14)	0.0055 (14)
C6	0.0356 (17)	0.0268 (15)	0.0388 (17)	−0.0038 (13)	−0.0050 (13)	0.0055 (13)
C7	0.0393 (19)	0.0357 (18)	0.053 (2)	0.0008 (15)	−0.0044 (15)	−0.0055 (15)
C8	0.051 (2)	0.050 (2)	0.061 (3)	−0.0007 (18)	0.0114 (19)	−0.0129 (18)
C9	0.083 (3)	0.056 (2)	0.039 (2)	−0.006 (2)	0.014 (2)	−0.0057 (17)
C10	0.071 (3)	0.042 (2)	0.0390 (19)	−0.0004 (18)	−0.0117 (17)	0.0021 (16)
C11	0.051 (2)	0.0364 (19)	0.045 (2)	−0.0097 (17)	−0.0016 (16)	0.0011 (15)
O1	0.0374 (13)	0.0479 (14)	0.0635 (15)	−0.0075 (11)	−0.0084 (11)	0.0113 (12)
O2	0.0623 (17)	0.0365 (13)	0.0565 (15)	−0.0047 (11)	0.0147 (12)	0.0086 (11)
O3	0.0452 (14)	0.0288 (12)	0.0544 (14)	−0.0040 (10)	−0.0083 (10)	0.0050 (10)
O4	0.090 (2)	0.0617 (17)	0.0536 (16)	−0.0063 (16)	−0.0078 (14)	−0.0158 (13)
O5	0.0650 (18)	0.0488 (15)	0.0770 (19)	−0.0260 (14)	−0.0016 (14)	0.0047 (13)

Geometric parameters (Å, º)

O1—C1	1.448 (4)	C4—C3	1.460 (5)
O1—C11	1.346 (4)	C4—H4	0.9300
O2—C2	1.422 (4)	C5—C4	1.305 (5)
O2—C3	1.456 (4)	C5—C6	1.490 (4)
O3—C11	1.320 (4)	C5—H5	0.9300
O3—C6	1.474 (3)	C6—C1	1.535 (4)
O4—C10	1.421 (4)	C6—C7	1.486 (4)
O4—C9	1.462 (5)	C7—C8	1.307 (5)
O5—C11	1.183 (4)	C7—H7	0.9300
C1—C10	1.495 (5)	C8—C9	1.446 (6)
C2—C1	1.504 (4)	C8—H8	0.9300
C2—C3	1.443 (5)	C9—H9	0.9800
C2—H2	0.9800	C10—C9	1.449 (5)

C3—H3	0.9800	C10—H10	0.9800

O1—C1—C10	108.2 (3)	C4—C3—H3	117.0
O1—C1—C2	107.1 (3)	C4—C5—C6	124.7 (3)
O1—C1—C6	102.3 (2)	C4—C5—H5	117.7
O2—C2—C1	113.7 (3)	C5—C4—C3	121.8 (3)
O2—C2—C3	61.1 (2)	C5—C4—H4	119.1
O2—C2—H2	116.4	C5—C6—C1	113.9 (3)
O2—C3—C4	116.0 (3)	C6—C5—H5	117.7
O2—C3—H3	117.0	C6—C7—H7	118.1
O3—C11—O1	111.4 (3)	C7—C6—C1	115.9 (3)
O3—C6—C1	101.0 (2)	C7—C6—C5	110.6 (3)
O3—C6—C5	106.1 (2)	C7—C8—C9	122.0 (4)
O3—C6—C7	108.3 (2)	C7—C8—H8	119.0
O4—C10—C1	116.1 (3)	C8—C7—C6	123.8 (3)
O4—C10—C9	61.3 (2)	C8—C7—H7	118.1
O4—C10—H10	115.9	C8—C9—C10	119.1 (3)
O4—C9—H9	117.1	C8—C9—H9	117.1
O5—C11—O1	122.9 (3)	C8—C9—O4	114.8 (3)
O5—C11—O3	125.6 (3)	C9—C10—C1	120.5 (3)
C1—C10—H10	115.9	C9—C10—H10	115.9
C1—C2—H2	116.4	C9—C8—H8	119.0
C2—C1—C6	115.2 (3)	C10—C1—C2	109.1 (3)
C2—C3—C4	118.3 (3)	C10—C1—C6	114.3 (3)
C2—C3—H3	117.0	C10—C9—H9	117.1
C2—C3—O2	58.8 (2)	C10—C9—O4	58.4 (2)
C2—O2—C3	60.2 (2)	C10—O4—C9	60.3 (2)
C3—C2—C1	120.8 (3)	C11—O1—C1	109.4 (2)
C3—C2—H2	116.4	C11—O3—C6	109.9 (2)
C3—C4—H4	119.1		

O1—C1—C10—C9	−131.7 (3)	C4—C5—C6—O3	−130.7 (3)
O1—C1—C10—O4	−61.1 (4)	C5—C4—C3—C2	11.0 (5)
O2—C2—C1—C10	−76.4 (3)	C5—C4—C3—O2	−55.9 (4)
O2—C2—C1—C6	53.7 (4)	C5—C6—C1—C10	153.6 (3)
O2—C2—C1—O1	166.7 (2)	C5—C6—C1—C2	26.0 (4)
O2—C2—C3—C4	−104.8 (3)	C5—C6—C1—O1	−89.7 (3)
O3—C6—C1—C10	−93.2 (3)	C5—C6—C7—C8	−145.4 (3)
O3—C6—C1—C2	139.2 (3)	C6—C1—C10—C9	−18.4 (4)
O3—C6—C1—O1	23.5 (3)	C6—C1—C10—O4	52.1 (4)
O3—C6—C7—C8	98.8 (4)	C6—C5—C4—C3	1.6 (5)
O4—C10—C9—C8	−102.8 (4)	C6—C7—C8—C9	−3.4 (5)
C1—C10—C9—C8	2.3 (5)	C6—O3—C11—O1	7.5 (4)
C1—C10—C9—O4	105.0 (3)	C6—O3—C11—O5	−172.8 (3)
C1—C2—C3—C4	−3.0 (5)	C7—C6—C1—C10	23.6 (4)
C1—C2—C3—O2	101.8 (3)	C7—C6—C1—C2	−104.0 (3)
C1—C6—C7—C8	−13.8 (4)	C7—C6—C1—O1	140.3 (3)
C1—O1—C11—O3	9.5 (4)	C7—C8—C9—C10	9.7 (5)

C1—O1—C11—O5	−170.2 (3)	C7—C8—C9—O4	−56.5 (5)
C2—C1—C10—C9	112.2 (4)	C9—O4—C10—C1	−112.1 (4)
C2—C1—C10—O4	−177.3 (3)	C10—O4—C9—C8	110.2 (3)
C2—O2—C3—C4	108.7 (3)	C11—O1—C1—C10	99.9 (3)
C3—C2—C1—C10	−145.7 (3)	C11—O1—C1—C2	−142.6 (3)
C3—C2—C1—C6	−15.6 (4)	C11—O1—C1—C6	−21.1 (3)
C3—C2—C1—O1	97.4 (3)	C11—O3—C6—C1	−19.8 (3)
C3—O2—C2—C1	−113.4 (3)	C11—O3—C6—C5	99.3 (3)
C4—C5—C6—C1	−20.5 (4)	C11—O3—C6—C7	−142.0 (3)
C4—C5—C6—C7	112.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4[i]	0.98	2.53	3.509 (5)	174
C3—H3···O5[ii]	0.98	2.55	3.313 (4)	134
C10—H10···O5[i]	0.98	2.49	3.379 (4)	151

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) *x*, *y*+1, *z*.