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A Stable Ozonide

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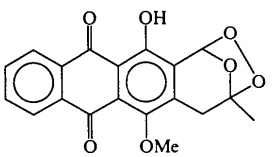
Abstract

The title compound, 13-hydroxy-6-methoxy-4-methyl-4,5-dihydro-1,4-epoxy-1*H*-anthra[2,3-*d*][1,2]dioxepine-7,12(7*H*,12*H*)-dione, $C_{19}H_{14}O_7$, is an example of an ozonide which shows unusual stability. In contrast to other ozonides it is very resistant to reduction.

Comment

Anthracyclinones have emerged in recent years as important chemotherapeutic agents for the treatment of a wide range of carcinomas. These compounds contain the anthroquinone chromophore as part of a tetracyclic unit linked to a carbohydrate. In a series of experiments in this department directed towards the elaboration of an anthroquinone into daunomycin (Cambie, Larsen, Rutledge & Woodgate, 1987), an ozonide was obtained that proved to be extremely resistant to reduction. We report here the structure of that ozonide, (I).



(I)

An *ORTEP* (Johnson, 1965) diagram of the title compound showing the numbering scheme used is given in Fig. 1. The structure has the expected planar arrangement of the aromatic system and there is a strong intramolecular hydrogen bond between the phenolic proton and the quinone O atom [O(1)···O(4) 2.538 Å], which is typical of hydroxyanthraquinone systems. There are structural details for 24 ozonides in the Cambridge Structural Database (Allen *et al.*, 1987) and analysis of these structures yields the following average dimensions: O—O 1.473 Å, C—(O—O) 1.443 Å and C—O 1.418 Å. These can be compared with the values obtained for the present structure: O—O 1.463 (3) Å, mean C—(O—O) 1.440 (2) Å and mean C—O 1.414 (3) Å. Many structures show marked asymmetry in the C—O bonds to the peroxy group and a similar feature is seen here: C(15)—O(5) 1.420 (4) Å and C(16)—O(6) 1.459 (3) Å. This asymmetry appears to be associated with other features of the molecule; here the shorter bond is to the C atom bound to the aromatic ring. Other distances and angles are within the normally expected range.

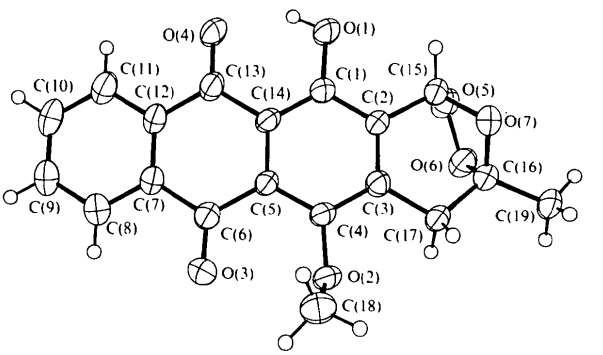


Fig. 1. The molecular structure (*ORTEP*; Johnson, 1965) showing 50% probability ellipsoids. H atoms are omitted for clarity.

Experimental

Crystal data

$C_{19}H_{14}O_7$
M_r = 354.30
Monoclinic
$P2_1/c$
a = 8.713 (4) Å
b = 11.5530 (10) Å
c = 15.612 (6) Å
β = 107.94 (5)°
V = 1495.1 (9) Å^3
Z = 4
D_x = 1.574 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54180 Å
Cell parameters from 25 reflections
θ = 9.4–11.5°
μ = 1.029 mm^{-1}
T = 293 (2) K
Plates
0.28 × 0.22 × 0.08 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
R_{int} = 0.0249
θ_{max} = 50°
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 17$

2306 measured reflections
2213 independent reflections
1626 observed reflections
$[I > 2\sigma(I)]$
3 standard reflections
monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0606$
$wR(F^2) = 0.1666$
$S = 1.157$
2213 reflections
238 parameters
H atoms refined with riding model
$w = 1/[\sigma^2(F_o^2) + (0.1349P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.561$
$\Delta\rho_{max} = 0.284$ e Å^{-3}
$\Delta\rho_{min} = -0.389$ 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}
C(1)	0.6725 (3)	0.3233 (2)	0.2109 (2)	0.0382 (7)
C(2)	0.6876 (3)	0.2023 (2)	0.2094 (2)	0.0355 (6)
C(3)	0.5701 (3)	0.1370 (2)	0.1498 (2)	0.0345 (6)
C(4)	0.4330 (3)	0.1911 (2)	0.0923 (2)	0.0355 (7)
C(5)	0.4167 (3)	0.3118 (2)	0.0904 (2)	0.0338 (6)
C(6)	0.2820 (3)	0.3712 (2)	0.0232 (2)	0.0400 (7)
C(7)	0.2819 (3)	0.5005 (2)	0.0202 (2)	0.0371 (7)
C(8)	0.1583 (4)	0.5576 (3)	−0.0429 (2)	0.0458 (7)
C(9)	0.1550 (4)	0.6773 (3)	−0.0450 (2)	0.0533 (8)
C(10)	0.2750 (4)	0.7404 (3)	0.0160 (2)	0.0547 (9)
C(11)	0.3981 (4)	0.6845 (2)	0.0793 (2)	0.0473 (8)
C(12)	0.4026 (3)	0.5645 (2)	0.0822 (2)	0.0392 (7)
C(13)	0.5340 (3)	0.5056 (2)	0.1507 (2)	0.0392 (7)
C(14)	0.5398 (3)	0.3778 (2)	0.1521 (2)	0.0361 (7)
C(15)	0.8373 (3)	0.1420 (2)	0.2688 (2)	0.0423 (7)
C(16)	0.7639 (3)	−0.0247 (2)	0.2020 (2)	0.0390 (7)
C(17)	0.5941 (3)	0.0084 (2)	0.1452 (2)	0.0385 (7)
C(18)	0.1785 (4)	0.0952 (3)	0.0520 (2)	0.0603 (9)
C(19)	0.7972 (4)	−0.1527 (2)	0.2109 (2)	0.0511 (8)
O(1)	0.7941 (3)	0.3804 (2)	0.2705 (2)	0.0560 (7)
O(2)	0.3254 (2)	0.1191 (2)	0.03280 (14)	0.0453 (6)
O(3)	0.1718 (3)	0.3191 (2)	−0.0304 (2)	0.0630 (7)
O(4)	0.6390 (3)	0.5626 (2)	0.20516 (15)	0.0542 (6)
O(5)	0.9512 (2)	0.1249 (2)	0.22160 (15)	0.0528 (6)
O(6)	0.8750 (2)	0.0304 (2)	0.16112 (14)	0.0507 (6)
O(7)	0.7990 (2)	0.0275 (2)	0.28752 (13)	0.0455 (6)

Table 2. *Selected geometric parameters* (Å)

C(1)—O(1)	1.348 (3)	C(8)—C(9)	1.383 (4)
C(1)—C(14)	1.386 (4)	C(9)—C(10)	1.385 (5)
C(1)—C(2)	1.405 (4)	C(10)—C(11)	1.375 (4)
C(2)—C(3)	1.377 (4)	C(11)—C(12)	1.387 (4)
C(2)—C(15)	1.518 (4)	C(12)—C(13)	1.471 (4)
C(3)—C(4)	1.401 (4)	C(13)—O(4)	1.231 (3)
C(3)—C(17)	1.505 (4)	C(13)—C(14)	1.477 (4)
C(4)—O(2)	1.377 (3)	C(15)—O(7)	1.416 (3)
C(4)—C(5)	1.401 (4)	C(15)—O(5)	1.420 (4)
C(5)—C(14)	1.423 (4)	C(16)—O(7)	1.411 (3)
C(5)—C(6)	1.480 (4)	C(16)—O(6)	1.459 (3)
C(6)—O(3)	1.220 (3)	C(16)—C(19)	1.506 (4)
C(5)—C(7)	1.495 (4)	C(16)—C(17)	1.521 (4)
C(7)—C(8)	1.383 (4)	C(18)—O(2)	1.429 (4)
C(7)—C(12)	1.401 (4)	O(5)—O(6)	1.463 (3)

H atoms were included in calculated positions and allowed to ride on the atom to which they were attached. H-atom displacement parameters were taken as 20% greater than those of the parent atom.

Data collection: CAD-4 software. Cell refinement: CAD-4 software. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965).

We thank Dr D. Larsen for provision of the crystals.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: FG1034). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A [5.5.5.5]Fenestrane Derivative

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Abstract

The title compound, ethyl *rel*-(1*R*,2*S*,4*R*,7*R*,10*R*,12*S*)-1-hydroxy-3-oxo-2-oxapentacyclo[7.4.2.0^{4,14}.0^{12,15}.0^{6,15}]-pentadecane-6-carboxylate, $C_{17}H_{22}O_5$, has been synthe-