

**(4*a*,4*a* $\beta$ ,9*a* $\beta$ )-4*a*-Acetoxy-4-methoxy-3,4,4*a*,9*a*-tetrahydro-1*H*-anthracene-2,9,10-trione**

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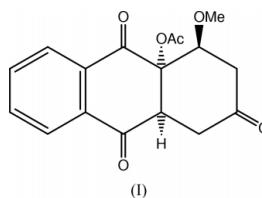
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## Key indicators

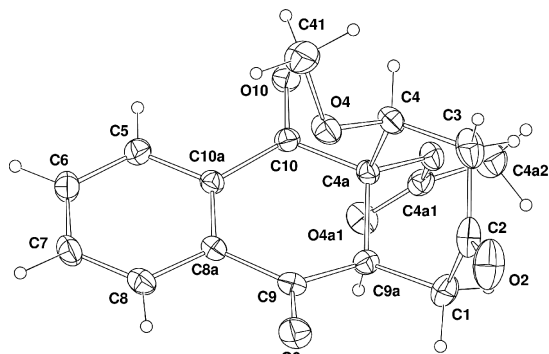
Single-crystal X-ray study  
*T* = 293 K  
Mean  $\sigma(\text{C}-\text{C})$  = 0.004 Å  
*R* factor = 0.052  
*wR* factor = 0.054  
Data-to-parameter ratio = 9.0For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The relative stereochemistry of the title compound is defined  
by a room-temperature single-crystal X-ray study.Received 15 September 2003  
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## Comment

A recent report describes the determination of a pair of tricyclic adducts of 'Danishefsky's diene'  $\text{H}_2\text{C}=\text{C}(\text{OSiMe}_3)-\text{CH}=\text{CH}(\text{OMe})$  with variously substituted 1,4-benzoquinones (Cameron *et al.*, 2003), a feature of interest being the possibility of interaction between axial methoxy substituents of the peripheral rings with the carboxyl groups of the central ring, provoking an extension of the work to a study of the present compound, a structure determination of which was undertaken (*a*) to define its stereochemistry and (*b*) to explore the possibility of similar interactions.



The results of the room-temperature, single-crystal X-ray structure determination are consistent with the stoichiometry, connectivity and stereochemistry of the title compound, (I), as displayed in Fig. 1. Despite the planarity of the associated peripheral aromatic ring and of the two carbonyl moieties, the central ring is far from planar, the torsion in the C9–C9a–C4a–C10 string being  $-46.2(3)^\circ$ ; dihedral angles between the  $\text{C}_2\text{CO}$  planes about C9,10 and the aromatic  $\text{C}_6$  plane are  $15.0(1)^\circ$  and  $5.9(1)^\circ$ , and between the carbonyl planes  $20.8(1)^\circ$ , *i.e.* they bow towards each other, with torsion angles O9–C9–C9a–C4a and O10–C10–C4a–C9a being  $-142.8(3)^\circ$  and  $-159.3(3)^\circ$ , respectively. The saturated peripheral ring is a 'boat', with C4a2 at the prow; torsion angles outward from C4a in either direction pairwise are:  $-52.8(3)^\circ$ ,  $53.2(3)^\circ$ ;  $51.3(3)^\circ$ ,  $-51.6(4)^\circ$ ,  $-50.4(4)^\circ$  and  $50.6(4)^\circ$ . Bond lengths and angles are as expected (Table 1); C–C distances to either side of the carbonyl groups are similar, excepting those to the aromatic ring which are slightly shorter. Of interest in previous contexts are the distances of O4 (O4a1) to C9 (C10) of  $2.783(4) \text{ \AA}$  [ $2.731(4) \text{ \AA}$ ], seemingly without impact on the planarity at the latter [angle sums about C9,10 are  $359.8^\circ (\times 2)$ ]. The crystal packing is unremarkable. The only other structurally characterized similar tricyclic system with similar saturation and trione substitution has an otherwise very different substitution pattern (Gordon *et al.*, 1992).



**Figure 1**

Projection of (I) normal to its aromatic ring plane, shown with 20% probability displacement ellipsoids for the non-H atoms, H atoms having arbitrary radii of 0.1 Å.

## Experimental

Compound (I) was synthesized by the reaction of Danishefsky's diene and 2-acetoxy-1,4-naphthoquinone and recrystallized from diethyl ether (Cameron *et al.*, 1980), m.p. 451–453 K (decomposition). Analysis found: C 64.7, H 5.3%;  $C_{17}H_{16}O_6$  requires: C 64.5, H 5.1%;  $\lambda_{\max}$ ,  $\log(\epsilon)$ : 227, 258, 299 nm (4.55, 4.05, 3.59);  $\nu_{\max}$ : 1766 (*br*), 1760, 1596, 1240  $cm^{-1}$ ;  $\delta$  2.46 (*dd*,  $J = 15.5, 7.5$  Hz, H1 $\beta$ ), 2.73 (*ddd*,  $J = 15.5, 3, 2$  Hz, H3 $\alpha$ ), 2.85 (*dd*,  $J = 15.5, 3$  Hz, H3 $\beta$ ), 2.93 (*s*, OCH<sub>3</sub>), 3.39 (*ddd*,  $J = 15.5, 2, 1.5$  Hz, H1 $\alpha$ ), 4.02 (*brtd*,  $J = 3, 1$  Hz, H4), 4.29 (*br d*,  $J = 7.5$  Hz, H9a), 7.69–8.09 (*m*, H5–8). *m/z*: 316 ( $M^+$ , 1%), 274 (28), 216 (54), 188 (100). Like its congeners recorded elsewhere (Cameron *et al.*, 2003), the material decomposes readily; it was shipped rapidly and data measured within a restricted  $2\theta$  range within a few hours.

### Crystal data

$C_{17}H_{16}O_6$	$D_x = 1.358$ Mg m <sup>-3</sup>
$M_r = 316.31$	Mo K $\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 9 reflections
$a = 13.645$ (5) Å	$\theta = 9.9$ – $17.2^\circ$
$b = 8.293$ (4) Å	$\mu = 0.10$ mm <sup>-1</sup>
$c = 14.112$ (5) Å	$T = 293$ (2) K
$\beta = 104.40$ (2) $^\circ$	Block, colourless
$V = 1546.7$ (11) Å <sup>3</sup>	$0.34 \times 0.21 \times 0.20$ mm
$Z = 4$	

### Data collection

Syntex $P2_1$ diffractometer	$h = 0 \rightarrow 14$
$2\theta$ – $\omega$ scans	$k = 0 \rightarrow 8$
2035 measured reflections	$l = -15 \rightarrow 14$
2035 independent reflections	6 standard reflections
1878 reflections with $I > \sigma(I)$	frequency: 60 min
$\theta_{\max} = 22.5^\circ$	intensity decay: none

### Refinement

Refinement on $F$	H-atom parameters not refined
$R = 0.052$	$w = 1/(\sigma^2(F) + 0.0005F^2)$
$wR = 0.054$	$(\Delta/\sigma)_{\max} = 0.006$
$S = 1.07$	$\Delta\rho_{\max} = 0.25$ e Å <sup>-3</sup>
1878 reflections	$\Delta\rho_{\min} = -0.18$ e Å <sup>-3</sup>
208 parameters	

**Table 1**

Selected geometric parameters (Å,  $^\circ$ ).

C1–C2	1.493 (5)	O4–C41	1.407 (5)
C1–C9a	1.535 (3)	C4a–O4a	1.453 (3)
C2–O2	1.216 (4)	C4a–C9a	1.532 (4)
C2–C3	1.503 (5)	C8a–C9	1.484 (3)
C3–C4	1.530 (4)	C8a–C10a	1.403 (3)
C4–O4	1.415 (4)	C9–O9	1.211 (3)
C4–C4a	1.535 (4)	C10–O10	1.214 (3)
C2–C1–C9a	112.2 (3)	C4–C4a–O4a	103.8 (2)
C1–C2–O2	122.5 (3)	C4a–O4a–C4a1	117.9 (2)
O2–C2–C3	122.5 (3)	O4a–C4a1–O4a1	123.0 (3)
C3–C4–O4	109.7 (2)	O9–C9–C9a	121.7 (2)
O4–C4–C4a	105.1 (2)	O10–C10–C10a	122.1 (2)

The H atoms were located in difference Fourier maps and placed at idealized positions (C–H = 0.95 Å).

Data collection:  $P2_1$  *Diffractometer Control Software* (Syntex, 1973); cell refinement:  $P2_1$  *Diffractometer Control Software*; data reduction: *Xtal3.5* (Hall *et al.*, 1995); program(s) used to solve structure: *Xtal3.5*; program(s) used to refine structure: *Xtal3.5* *CRYLSQ*; molecular graphics: *Xtal3.5*; software used to prepare material for publication: *Xtal3.5 BONDLA CIFIO*.

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