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9,10-Dimethyl-9,10-peroxy-9,10-dihydroanthracene

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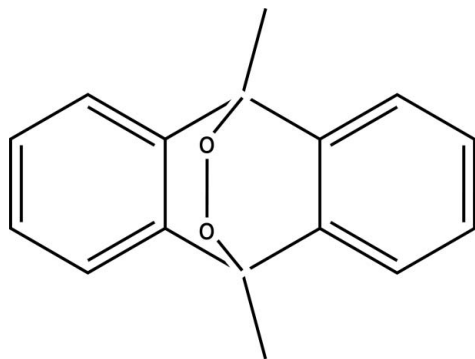
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.043; wR factor = 0.107; data-to-parameter ratio = 7.4.

The structure of the title compound, $\text{C}_{16}\text{H}_{14}\text{O}_2$, contains one half-molecule in the asymmetric unit and the molecule is located on a mirror plane. The dihedral angle between the two benzene ring planes is $53.07(6)^\circ$. The crystal structure involves intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related literature, see: Burrows *et al.* (1999); Gable *et al.* (1996); Karolak-Wojciechowska *et al.* (1998); Larson (1970); Price (1946); Simpson *et al.* (2004).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{14}\text{O}_2$
 $M_r = 238.29$
Orthorhombic, $Cmc2_1$

$a = 12.9873(7)$ Å
 $b = 11.0810(8)$ Å
 $c = 8.8368(8)$ Å

$V = 1271.72(16)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.08$ mm⁻¹
 $T = 296(1)$ K
 $0.40 \times 0.35 \times 0.30$ mm

Data collection

Rigaku R-Axis RAPID diffractometer
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.958$, $T_{\max} = 0.976$

5690 measured reflections
812 independent reflections
585 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.083$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.107$
 $S = 1.00$
699 reflections

94 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C4}-\text{H4}\cdots\text{O2}^i$	0.93	2.64	3.526 (2)	159

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WW2092).

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supplementary materials

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9,10-Dimethyl-9,10-peroxy-9,10-dihydroanthracene

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Comment

The Friedel–Crafts reaction of an alkyl halide with an aromatic hydrocarbon in the presence of aluminium chloride yields a substituted phenyl compound (Price, 1946). The reaction does not, however, stop at the stage of mono-substitution, an unpredictable compound was reported and determined by X-ray crystal structure analysis. The molecule of the title compound lie on a crystallographic mirror plane, which pass through atoms C1/O1/O2/C8/C9/C10 (Fig. 1). The geometrical parameters for (1) are similar to those of related 9,10-bridged anthracene derivatives (Simpson *et al.*, 2004; Gable *et al.*, 1996; Burrows *et al.*, 1999). Atoms C1, C8 are almost coplanar with the benzene ring plane, and the deviating distance from the benzene ring are 0.0074 Å, −0.066 Å respectively. The dihedral angle between the plane of bridge atoms C1—O1—O2—C8 and the benzene ring plane is 63.45 (5)°. The benzene ring plane and its symmetry-related one form the dihedral angle of 53.07 (6)°, which is smaller than that of 9,10-bridged anthracene systems, *e.g.* the corresponding dihedral angle in 11,12-bis(*N,N*-dimethyl-aminomethyl)-9,10-dihydro-9,10-ethanoanthracene (Karolak-Wojciechowska *et al.*, 1998) is 58.8 (2)°. The three six-membered rings of the bicycle core of (1) [C1—C2—C7—C8—C7ⁱ—C2ⁱ, C1—C2—C7—C8—O2—O1, C1—C2ⁱ—C7ⁱ—C8—O2—O1, symmetric code (i): 1 − *x,y,z*] are all forced into boat forms. Intermolecular weak interactions, C4—H4⋯O2ⁱⁱ [symmetric code (ii): 1/2 + *x*, 1/2 − *y*, −1/2 + *z*] and C6—H6⋯C4ⁱⁱⁱ [symmetric code (iii): *x*, 1 − *y*, 1/2 + *z*], link the molecules into circles (Fig. 2). The bridged O2 atom attached to H4 atom of neighbouring benzene ring may result in the longer distance O2—C8, compared with the bond length C1—O1 (Table 1).

Experimental

Ethyl 2-bromo-2-methylpropionate (3.84 g, 20 mmol) was added to a 50 ml flask equipped with a reflux condenser and large magnetic stirrer. Anhydrous benzene (20 ml) was added to the flask, followed by fresh anhydrous AlCl₃ (9.00 g, 67.5 mmol) in small portions. The solution was then slowly heated to the reflux temperature and at this time the exit of the reflux condenser was connected to a flowing-water HBr trap. The mixture was heated a total of 24 h without interruption. The reaction mixture was then cooled to 278k. and treated with 20 ml of 50/50 (by volume) conc. HCl/H₂O to decompose the catalyst complex. The benzene layer was then separated, washed once with ice-cold H₂O (12 ml) and twice with dilute aqueous sodium hydroxide. The organic phase was evaporate. After 6 days, a single-crystal suitable for X-ray analysis was obtained by recrystallization from ethanol.

Refinement

All H atoms were placed in calculated positions with C—H = 0.93–0.96 Å and included in the refinement in riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$. In the absence of anomalous scatterers, no attempt was made to establish the absolute configuration of the title compound.

Figures

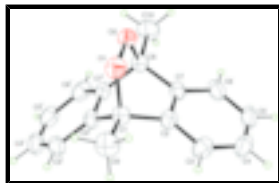


Fig. 1. A view of (1). Displacement ellipsoids are drawn at 30% probability level and H atoms are shown as small circles of arbitrary radii. Symmetry code (i): $1 - x, y, z$

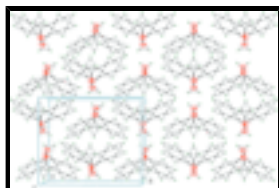


Fig. 2. Packing arrangement for (1).

9,10-Dimethyl-9,10-peroxy-9,10-dihydroanthracene

Crystal data

$C_{16}H_{14}O_2$

$M_r = 238.29$

Orthorhombic, $Cmc2_1$

Hall symbol: C 2c -2

$a = 12.9873$ (7) Å

$b = 11.0810$ (8) Å

$c = 8.8368$ (8) Å

$V = 1271.72$ (16) Å³

$Z = 4$

$F_{000} = 504.00$

$D_x = 1.244$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71069$ Å

Cell parameters from 2234 reflections

$\theta = 2.4$ – 27.3°

$\mu = 0.08$ mm⁻¹

$T = 296$ (1) K

Chunk, yellow

$0.40 \times 0.35 \times 0.30$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer

Detector resolution: 10.00 pixels mm⁻¹

ω scans

Absorption correction: multi-scan
(ABSCOR; Higashi, 1995)

$T_{\min} = 0.958$, $T_{\max} = 0.976$

5690 measured reflections

812 independent reflections

585 reflections with $F^2 > 2\sigma(F^2)$

$R_{\text{int}} = 0.083$

$\theta_{\text{max}} = 27.5^\circ$

$h = -16 \rightarrow 16$

$k = -14 \rightarrow 12$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.107$

$S = 1.00$

$w = 1/[0.0011F_o^2 + \sigma(F_o^2)]/(4F_o^2)$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.23$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

699 reflections
 94 parameters
 H-atom parameters constrained

Extinction correction: (Larson, 1970)
 Extinction coefficient: 132 (34)

Special details

Refinement. Refinement using all reflections. The weighted R -factor (wR) and goodness of fit (S) are based on F^2 . R -factor (gt) are based on F . The threshold expression of $F^2 > 2.0\sigma(F^2)$ is used only for calculating R -factor (gt).

Data collection(812 indenpent reflections but 699 in refinement): The author of the software, Dr Lee Daniels (ldaniels@RigakuMSC.com) explain this problem (see following). The number of reflections used to refine the cell is taken from the diffractometer program, which uses all available reflections measured in different scans. Even though some reflections are used more than once, they measured at different psi angles and therefore represent independent observations for determination of the cell dimensions.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.5000	0.1212 (2)	0.7053 (3)	0.0584 (7)
O2	0.5000	0.1937 (2)	0.8453 (2)	0.0547 (6)
C1	0.5000	0.1992 (2)	0.5707 (4)	0.0488 (9)
C2	0.59500 (13)	0.27732 (18)	0.5847 (2)	0.0466 (6)
C3	0.67877 (17)	0.2829 (2)	0.4883 (3)	0.0631 (8)
C4	0.76037 (19)	0.3580 (2)	0.5212 (3)	0.0750 (10)
C5	0.75816 (19)	0.4289 (3)	0.6492 (3)	0.0769 (9)
C6	0.67600 (18)	0.4228 (2)	0.7475 (3)	0.0655 (8)
C7	0.59468 (14)	0.3466 (2)	0.7153 (2)	0.0482 (5)
C8	0.5000	0.3253 (3)	0.8122 (4)	0.0502 (9)
C9	0.500000 (10)	0.1104 (4)	0.4404 (5)	0.0734 (12)
C10	0.500000 (10)	0.3808 (4)	0.9660 (5)	0.0786 (13)
H3	0.6800	0.2359	0.4011	0.076*
H4	0.8170	0.3608	0.4569	0.090*
H5	0.8125	0.4811	0.6693	0.092*
H6	0.6753	0.4696	0.8349	0.079*
H101	0.5603	0.3555	1.0201	0.094*
H102	0.5000	0.4671	0.9567	0.094*
H901	0.5604	0.0606	0.4462	0.088*
H902	0.5000	0.1538	0.3464	0.088*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0637 (13)	0.0364 (11)	0.0750 (17)	0.0000	0.0000	0.0017 (13)
O2	0.0565 (13)	0.0537 (13)	0.0538 (15)	0.0000	0.0000	0.0087 (11)
C1	0.0523 (17)	0.0373 (15)	0.057 (2)	0.0000	0.0000	-0.0067 (16)
C2	0.0403 (12)	0.0385 (10)	0.0608 (15)	0.0054 (8)	0.0021 (10)	0.0028 (10)
C3	0.0594 (14)	0.0578 (15)	0.072 (2)	0.0127 (11)	0.0150 (14)	0.0089 (14)
C4	0.0450 (13)	0.081 (2)	0.099 (2)	0.0072 (13)	0.0146 (15)	0.032 (2)

supplementary materials

C5	0.0440 (13)	0.0784 (19)	0.108 (2)	-0.0167 (13)	-0.0131 (16)	0.037 (2)
C6	0.0569 (14)	0.0614 (15)	0.078 (2)	-0.0120 (12)	-0.0224 (14)	0.0061 (14)
C7	0.0400 (10)	0.0440 (10)	0.0606 (14)	0.0016 (8)	-0.0061 (10)	0.0019 (12)
C8	0.0492 (16)	0.0492 (18)	0.052 (2)	0.0000	0.0000	-0.0027 (15)
C9	0.089 (2)	0.054 (2)	0.077 (2)	0.0000	0.0000	-0.019 (2)
C10	0.089 (2)	0.089 (2)	0.058 (2)	0.0000	0.0000	-0.017 (2)

Geometric parameters (Å, °)

O1—O2	1.475 (3)	C7—C8	1.517 (3)
O1—C1	1.470 (4)	C8—C10	1.492 (6)
O2—C8	1.487 (4)	C3—H3	0.930
C1—C2	1.512 (2)	C4—H4	0.930
C1—C2 ⁱ	1.512 (2)	C5—H5	0.930
C1—C9	1.514 (5)	C6—H6	0.930
C2—C3	1.383 (3)	C9—H901	0.960
C2—C7	1.386 (3)	C9—H901 ⁱ	0.960
C3—C4	1.378 (3)	C9—H902	0.960
C4—C5	1.377 (4)	C10—H101	0.960
C5—C6	1.378 (3)	C10—H101 ⁱ	0.960
C6—C7	1.382 (3)	C10—H102	0.960
O2—O1—C1	111.0 (2)	C7—C8—C10	116.76 (19)
O1—O2—C8	111.6 (2)	C7 ⁱ —C8—C10	116.75 (19)
O1—C1—C2	105.68 (18)	C2—C3—H3	119.9
O1—C1—C2 ⁱ	105.68 (18)	C4—C3—H3	119.9
O1—C1—C9	103.5 (2)	C3—C4—H4	119.9
C2—C1—C2 ⁱ	109.3 (2)	C5—C4—H4	119.9
C2—C1—C9	115.72 (18)	C4—C5—H5	119.8
C2 ⁱ —C1—C9	115.72 (18)	C6—C5—H5	119.8
C1—C2—C3	128.1 (2)	C5—C6—H6	120.3
C1—C2—C7	112.5 (2)	C7—C6—H6	120.3
C3—C2—C7	119.37 (19)	C1—C9—H901	109.5
C2—C3—C4	120.1 (2)	C1—C9—H901 ⁱ	109.5
C3—C4—C5	120.1 (2)	C1—C9—H902	109.5
C4—C5—C6	120.4 (2)	H901—C9—H901 ⁱ	109.5
C5—C6—C7	119.4 (2)	H901—C9—H902	109.5
C2—C7—C6	120.5 (2)	H901 ⁱ —C9—H902	109.5
C2—C7—C8	112.7 (2)	C8—C10—H101	109.5
C6—C7—C8	126.7 (2)	C8—C10—H101 ⁱ	109.5
O2—C8—C7	105.30 (19)	C8—C10—H102	109.5
O2—C8—C7 ⁱ	105.30 (19)	H101—C10—H101 ⁱ	109.4
O2—C8—C10	103.0 (3)	H101—C10—H102	109.5
C7—C8—C7 ⁱ	108.3 (2)	H101 ⁱ —C10—H102	109.5
O2—O1—C1—C2	57.93 (16)	C1—C2—C7—C6	179.4 (2)
O2—O1—C1—C2 ⁱ	-57.93 (16)	C1—C2—C7—C8	0.8 (2)
O1—O2—C8—C7	-57.16 (18)	C3—C2—C7—C6	1.5 (3)

O1—O2—C8—C7 ⁱ	57.16 (18)	C3—C2—C7—C8	-177.1 (2)
O1—C1—C2—C3	118.2 (2)	C7—C2—C3—C4	-0.8 (3)
O1—C1—C2—C7	-59.5 (2)	C2—C3—C4—C5	-0.9 (4)
O1—C1—C2 ⁱ —C3 ⁱ	-118.2 (2)	C3—C4—C5—C6	1.9 (4)
O1—C1—C2 ⁱ —C7 ⁱ	59.5 (2)	C4—C5—C6—C7	-1.2 (4)
C2—C1—C2 ⁱ —C3 ⁱ	128.5 (2)	C5—C6—C7—C2	-0.5 (3)
C2—C1—C2 ⁱ —C7 ⁱ	-53.8 (3)	C5—C6—C7—C8	177.9 (2)
C2 ⁱ —C1—C2—C3	-128.5 (2)	C2—C7—C8—O2	57.3 (2)
C2 ⁱ —C1—C2—C7	53.8 (3)	C2—C7—C8—C10	170.8 (2)
C9—C1—C2—C3	4.3 (4)	C2—C7—C8—C7 ⁱ	-55.0 (3)
C9—C1—C2—C7	-173.3 (2)	C6—C7—C8—O2	-121.2 (2)
C9—C1—C2 ⁱ —C3 ⁱ	-4.3 (4)	C6—C7—C8—C10	-7.7 (4)
C9—C1—C2 ⁱ —C7 ⁱ	173.3 (2)	C6—C7—C8—C7 ⁱ	126.5 (2)
C1—C2—C3—C4	-178.4 (2)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C4—H4 \cdots O2 ⁱⁱ	0.93	2.64	3.526 (2)	159

Symmetry codes: (ii) $x+1/2, -y+1/2, z-1/2$.

Fig. 1

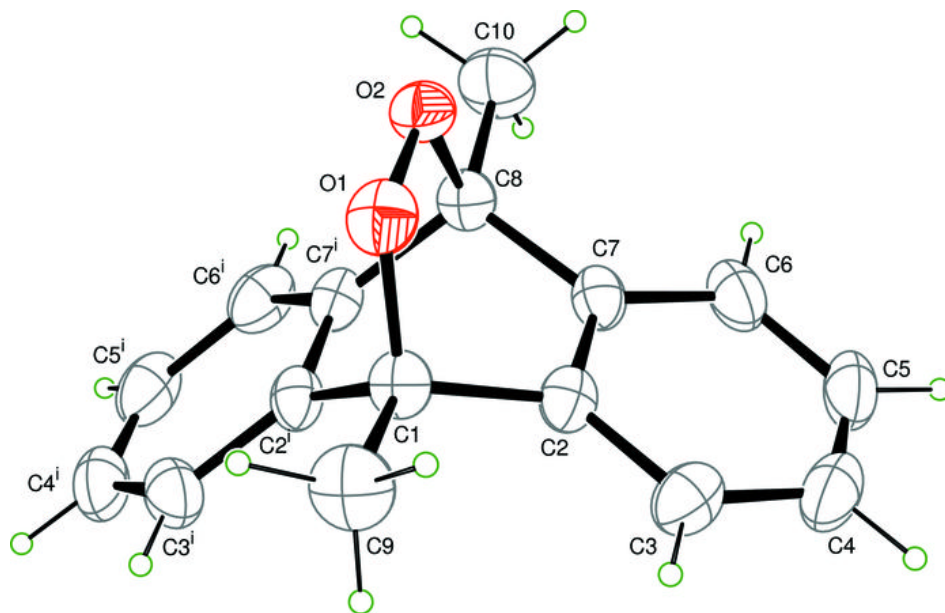


Fig. 2

