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10-Acetonyl-10-hydroxyphenanthren-9(10*H*)-one

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Key indicators

Single-crystal X-ray study
T = 213 K
Mean σ(C–C) = 0.006 Å
R factor = 0.055
wR factor = 0.117
Data-to-parameter ratio = 6.7

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

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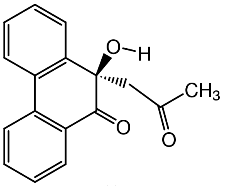
In the title compound, $C_{17}H_{14}O_3$, the central ring is not planar and tends towards an envelope conformation. The hydroxy and oxopropyl groups facilitate one O—H···O and one C—H···O interactions, which link the molecules into molecular chains parallel to the *c* direction. The packing is also stabilized by another C—H···O interaction, which interconnects the chains into a three-dimensional network.

Comment

Photoinduced cycloaddition reactions of phenanthrenequinone with several oxazole derivatives, which are of interest in heterocyclic chemistry, have been investigated (Zhang & Xu, 2002). The crystal structures of dioxino[2,3-*d*]oxazole derivatives resulting from such photo-induced reactions have been reported in our previous study (Usman *et al.*, 2002). In order to extend the photo-induced reactions of phenanthrenequinone, we have prepared and structurally analysed the title compound, (I), the crystal structure of which is presented here.



(I)

The bond lengths and angles in (I) (Fig. 1) are within normal ranges (Allen *et al.*, 1987). The values within the phenanthrene moiety (C1–C14) agree with the corresponding values in the related structure (Usman *et al.*, 2002), except for the bond lengths and angles around atoms C1 and C2. The ring containing these two atoms is distorted from planarity towards an envelope conformation, with atom C1 displaced by 0.483 (4) Å from the C2/C3/C8/C9/C14 plane. The ketone atom O2, attached at C2, deviates in the opposite direction from the plane by 0.403 (3) Å, although C2 is a Csp^2 atom. This is related to the configuration of atom C2, which is attached to the sp^3 atom C1 (average bond angle subtended at C1 is 109.5°). The mean plane of the ring involving atoms C1 and C2 makes dihedral angles of 8.7 (2) and 11.0 (2)°, respectively, with phenyl rings C3–C8 and C9–C14, while the two phenyl rings are twisted by an angle of 14.3 (2)° with respect to each other.

The oxopropyl moiety (O3/C15/C16/C17) is in a plane which makes a dihedral angle of 25.6 (3)° with the mean plane of the ring involving atoms C1 and C2.

The molecules are packed into infinite chains along the *c* direction (Fig. 2) by O1—H1···O3^{i} and C15—H15*B*···O2^{ii}

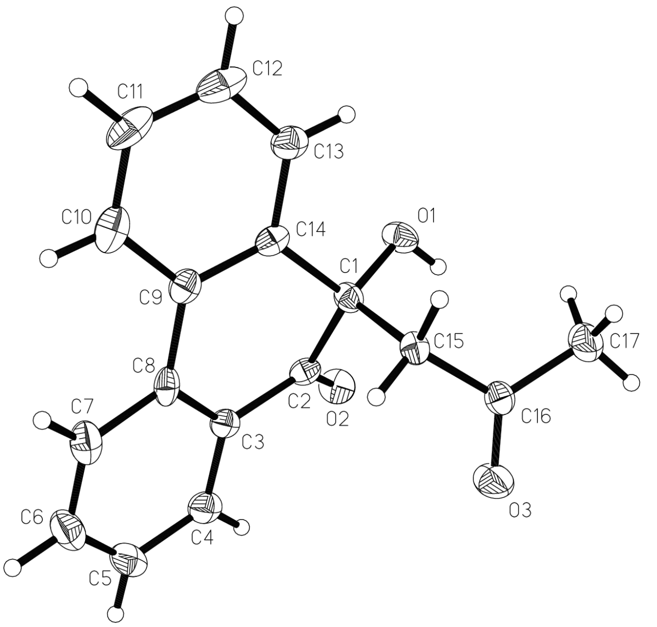


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

interactions [symmetry codes: (i) $\frac{1}{2} - x, y, z - \frac{1}{2}$; (ii) $\frac{1}{2} - x, y, \frac{1}{2} + z$], involving the oxopropyl and hydoxy substituents (see Table 2). The extended notation for a molecular chain comprising these rings is $C(5)[R_2^2(9)]$ (Bernstein *et al.*, 1995). The molecular chains are interconnected by a C17—H17*B*···O3^{iii} interaction [symmetry code: (iii) $x - \frac{1}{2}, 1 - y, z$] into a three-dimensional network. The packing is stabilized by these interactions along with the dipole–dipole and van der Waals interactions.

Experimental

A solution of 9,10-phenanthrenedione (0.05 *M*) in the presence of an excess of 2-methyl-4-phenyloxazole in benzene solution was irradiated with light having a wavelength longer than 400 nm. After completion of reaction, the solvent was removed *in vacuo* and the residue was separated by column chromatography on a silica gel to afford the title compound. Single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an acetone–petroleum ether solution (1:5 *v*/*v*).

Crystal data

$C_{17}H_{14}O_3$
$M_r = 266.28$
Orthorhombic, $Pca2_1$
$a = 9.2240$ (8) Å
$b = 15.2406$ (9) Å
$c = 9.2898$ (8) Å
$V = 1305.9$ (2) Å^3
$Z = 4$
$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3007 reflections
$\theta = 2.6$–$28.3°$
$\mu = 0.09$ mm^{-1}
$T = 213$ (2) K
Needle, colorless
0.50 × 0.18 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
5952 measured reflections
1226 independent reflections
980 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.120$
$\theta_{\rm max} = 25.0°$
$h = -10 \rightarrow 9$
$k = -18 \rightarrow 16$
$l = -11 \rightarrow 10$

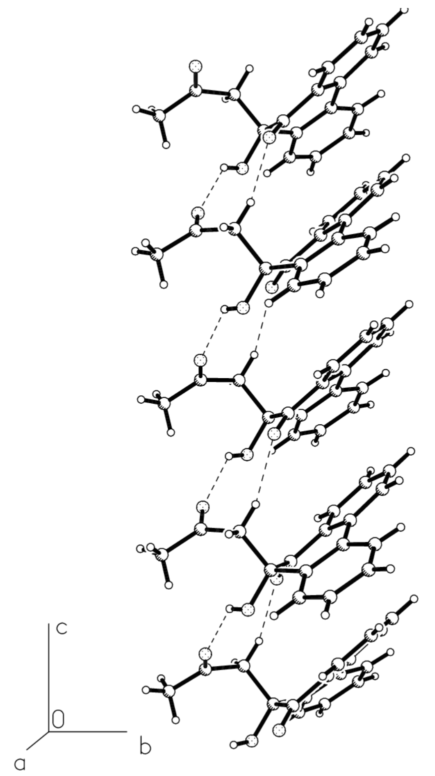


Figure 2
Packing diagram of the title compound viewed down the *a* axis, showing the infinite molecular chains. The dashed lines denote the C—H···O intermolecular interactions.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.117$
$S = 0.99$
1226 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.021 (4)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.402 (5)	C1—C2	1.526 (5)
O2—C2	1.217 (5)	C1—C15	1.556 (6)
C1—C14	1.519 (5)		
O1—C1—C14	109.3 (3)	O1—C1—C15	110.6 (3)
O1—C1—C2	110.5 (3)	C14—C1—C15	107.4 (3)
C14—C1—C2	110.9 (3)	C2—C1—C15	108.0 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.82	2.18	2.813 (4)	135
C15—H15*B*···O2^{ii}	0.97	2.55	3.444 (6)	153
C17—H17*B*···O3^{iii}	0.96	2.57	3.446 (6)	151

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

The H atoms were placed geometrically and treated as riding on their parent C and O atoms, with C—H = 0.93–0.97 Å, O—H = 0.82 Å, and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ and $1.5U_{\rm eq}({\rm O})$. In the absence of

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significant anomalous dispersion effects, 1065 Friedel pairs were merged and the absolute structure can not be determined from the crystallographic experiment. In the refinements, the 2θ maximum was limited to 50°, due to a large fraction of weak data at higher angles.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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