Ethyl 2-(acetylamino)-2-(9-hydroxy-10-oxo-9,10-dihydro-9-phenanthrenyl)acetate

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In the title compound, $C_{20}H_{19}NO_2$, the phenanthrene moiety is non-planar, with the dihydrobenzene ring distorted towards a twisted envelope conformation. The molecules are linked into chains along the a axis by O—H···O hydrogen bonds; two other intermolecular hydrogen bonds interconnect these chains into a three-dimensional framework.

Comment

In our recent investigation on the photoinduced reactions of phenanthrenequinone with oxazole derivatives, we have carried out photoreactions of phenanthrenequinone with 2-methyl-5-ethoxyoxazole and obtained the title compound, (I), as one of the products. This compound is formed by the hydrolysis of the oxetane product derived from [2 + 2]-photocycloaddition of the two reactants.

(I)
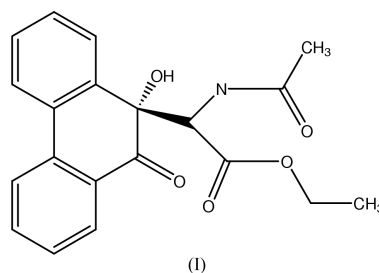
The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The values within the phenanthrene moiety (C1–C14) agree with the corresponding values in related structures (Usman *et al.*, 2002; Wang *et al.*, 2003). As observed in the previously reported compounds, the dihydrobenzene of the phenanthrene moiety is distorted towards a twisted envelope conformation, with atom C14 0.503 (5) Å from the C1/C6/C7/C12/C13 plane. Ketone atom O2 is displaced in the opposite direction by −0.488 (8) Å. The dihedral angle between rings C1–C6 and C7–C12 is 16.4 (1)°. This values is comparable to that in the related compound reported by Usman *et al.* (2002).

The two fragments attached to atom C15, *viz.* N1/C19/C20/O5 and C16–C18/O3/O4, are each planar, and the dihedral angle between these fragments is 74.9 (1)°.

The molecules in (I) are linked by an intermolecular O1—H1A···O5 hydrogen bond into chains along the a axis (Table 2 and Fig. 2), and two other intermolecular interactions, N1—H2A···O3 and C20—H20C···O1, interconnect the chains into a three-dimensional framework. These interactions, together with van der Waals forces, stabilize the molecular and packing structure in the crystal.
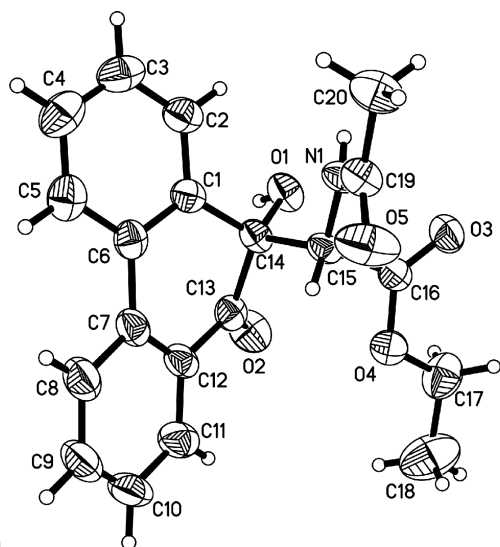
Figure 1
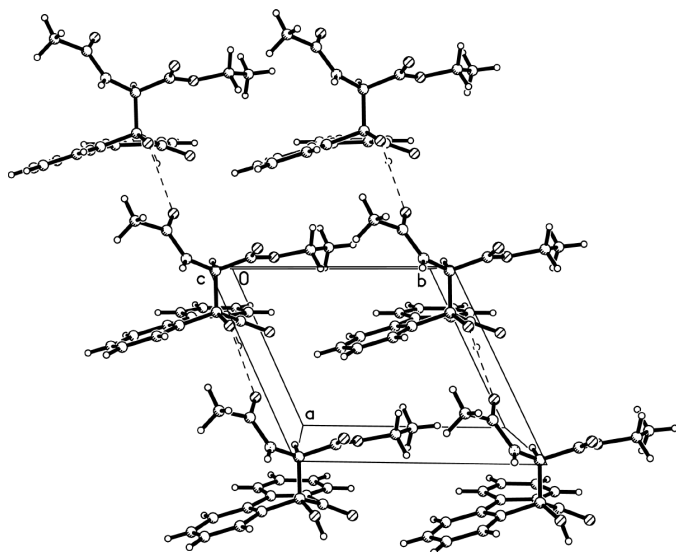View of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Figure 2
Packing diagram of (I), viewed down the *c* axis, showing extended chains along the *a* direction. Dashed lines denote intermolecular interactions.

Experimental

The title compound was prepared by the irradiation of a benzene solution of 9,10-phenanthrenequinone in the presence of an excess amount of oxazole, followed by silica-gel chromatographic separation of the reaction mixture. Single crystals suitable for X-ray crystallographic analysis were prepared by slow evaporation of a dichloromethane–acetone solution.

Crystal data

$C_{20}H_{19}NO_5$	$Z = 2$
$M_r = 353.36$	$D_x = 1.360$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.7205$ (5) Å	Cell parameters from 3047
$b = 8.8797$ (6) Å	reflections
$c = 14.1801$ (10) Å	$\theta = 2.6–28.3°$
$\alpha = 82.795$ (1)°	$\mu = 0.10$ mm^{-1}
$\beta = 80.241$ (1)°	$T = 293$ (2) K
$\gamma = 64.415$ (1)°	Block, pale yellow
$V = 862.63$ (10) Å^3	$0.48 \times 0.22 \times 0.18$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	2990 independent reflections
	2553 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.011$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$
(*SADABS*; Sheldrick, 1996)	$h = -5 \rightarrow 9$
$T_{min} = 0.954$, $T_{max} = 0.983$	$k = -10 \rightarrow 10$
4335 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.042$	$+ 0.2776P]$
$wR(F^2) = 0.117$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
2990 reflections	$\Delta\rho_{max} = 0.29$ e Å^{-3}
248 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
H atoms: see below	

Table 1
Selected geometric parameters (Å, °).

O1—C14	1.4080 (19)	C13—C14	1.534 (2)
O2—C13	1.209 (2)	C14—C15	1.572 (2)
C1—C14	1.529 (2)		
O1—C14—C1	112.05 (13)	O1—C14—C15	106.05 (13)
O1—C14—C13	111.47 (13)	C1—C14—C15	108.84 (12)
C1—C14—C13	109.74 (13)	C13—C14—C15	108.54 (13)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$A\cdots$O5^i	0.85 (3)	2.02 (3)	2.850 (2)	167 (2)
N1—H2$A\cdots$O3^{ii}	0.77 (2)	2.36 (2)	3.106 (2)	164 (2)
C20—H20$C\cdots$O1^{ii}	0.96	2.43	3.343 (3)	159

Symmetry codes: (i) $x - 1, y, z$; (ii) $2 - x, -y, -z$.

H atoms were placed geometrically and treated as riding on their parent C atoms, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) [for methyl H, U_{iso} = 1.5U_{eq}(C)], except for the H atoms at C15, O1 and N1, which were located in difference Fourier maps and refined isotropically. Owing to the large fraction of weak data at higher angles, the 2θ maximum was limited to 50°.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; structure solution: *SHELXTL* (Sheldrick, 1997); structure refinement: *SHELXTL*; molecular graphics: *SHELXTL*; preparation for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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