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# 4′,5a′-Diphenyl-10-oxospiro[phenanthrene-[9,2′]oxeto[5,4-*b*]oxazole]

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.042
*wR* factor = 0.117
Data-to-parameter ratio = 11.0

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

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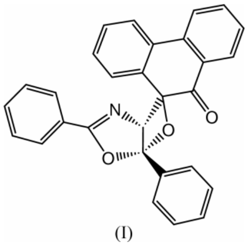
In the title compound, $C_{29}H_{19}NO_3$, the phenanthrene is out of planarity with its dihydrobenzene ring being distorted towards a half-chair conformation. The relative configuration of the planar oxetane and oxazole rings is conditioned by the C$sp^3$ state of the shared atoms.

## Comment

In our ongoing research on the photocycloaddition reactions of α-dicarbonyl compounds with oxazole derivatives (Zhang *et al.*, 2003), we have carried out the photoreaction of 9,10-phenanthrenedione with 2,5-diphenyloxazole and isolated the title compound, (I). We have also structurally analysed (I) and the results are presented here.



(I)

The bond lengths and angles in (I) (Fig. 1) are within normal ranges (Allen *et al.*, 1987). Those within the fused four/five-membered rings (C1/C16/O2/C3/O1/C2/N1) are comparable to the corresponding values in related structures, *viz.* spiro[indole-3,2′-(2′a*H*,5′a*H*)oxeto[5,4-*b*]oxazol]-2(1*H*)-one derivatives (Usman *et al.*, 2003). As in these structures, there is elongation of the C1—C16 bond and planarity of the oxetane ring. The dihedral angle between the O2/C3/C1 and O2/C1/C16 planes is 177.0 (1)° [*cf.* 176.9 (3)–178.6 (2)°; Usman *et al.*, 2003].

The planes through the oxetane and oxazole rings make a dihedral angle of 113.4 (1)°. This is a consequence of the $sp^3$ hydridization of the shared atoms C1 and C3. The relative configurations of the phenyl rings C4–C9 and C10–C15 with respect to the oxetane-oxazole system are determined by their attached atoms C3 ($sp^3$) and C2 ($sp^2$), respectively. The two phenyl rings make dihedral angles of 68.1 (1) and 22.4 (1)°, respectively, with the oxazole ring.

The phenanthrene is out of planarity with its dihydrobenzene ring (C16–C18/C23/C24/C29) having a half-chair conformation. Atoms C16 and C17 deviate by 0.209 (2) and 0.374 (2) Å in opposite directions from the C18/C23/C24/C29 plane.

## Experimental

The title compound was prepared by photolysis of phenanthrenequine (0.05 *M*) in the presence of an excess amount of 2,5-

diphenyloxazole in acetonitrile and was separated by column chromatography on silica gel, with petroleum ether–ethyl acetate as eluants. Single crystals suitable for X-ray diffraction measurement were obtained by slow evaporation of a petroleum ether–acetone solution.

*Crystal data*

$C_{29}H_{19}NO_3$
$M_r = 429.45$
Triclinic, $P\bar{1}$
$a = 8.3475$ (4) Å
$b = 11.8671$ (6) Å
$c = 11.9106$ (6) Å
$\alpha = 74.148$ (1)°
$\beta = 89.736$ (1)°
$\gamma = 72.344$ (1)°
$V = 1077.59$ (9) Å$^3$

$Z = 2$
$D_x = 1.324$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 4070 reflections
$\theta = 2.6$–28.3°
$\mu = 0.09$ mm$^{-1}$
$T = 293$ (2) K
Block, yellow
0.50 × 0.40 × 0.30 mm

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
5771 measured reflections
4110 independent reflections

3482 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\rm max} = 26.0°$
$h = -10 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -13 \rightarrow 14$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.117$
$S = 1.08$
4110 reflections
374 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.1949P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.20$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.19$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| O1—C2 | 1.376 (2) | N1—C1 | 1.449 (2) |
| O1—C3 | 1.440 (2) | C1—C3 | 1.543 (2) |
| O2—C3 | 1.427 (2) | C1—C16 | 1.586 (2) |
| O2—C16 | 1.453 (2) | C16—C29 | 1.506 (2) |
| N1—C2 | 1.273 (2) | C16—C17 | 1.519 (2) |
| | | | |
| C16—C1—C3—O2 | −2.0 (1) | N1—C1—C16—C29 | 13.38 (18) |
| N1—C1—C3—O1 | 0.8 (1) | | |

All H atoms were located from a difference Fourier map and refined isotropically [C—H = 0.92 (2)–1.02 (2) Å]. Owing to the large fraction of weak data at higher angles, the 2$\theta$ maximum was limited to 52°. The maximum and minimum electron-density peaks are located 0.98 Å from O3 and 1.29 Å from C6, respectively.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); 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, 2003).

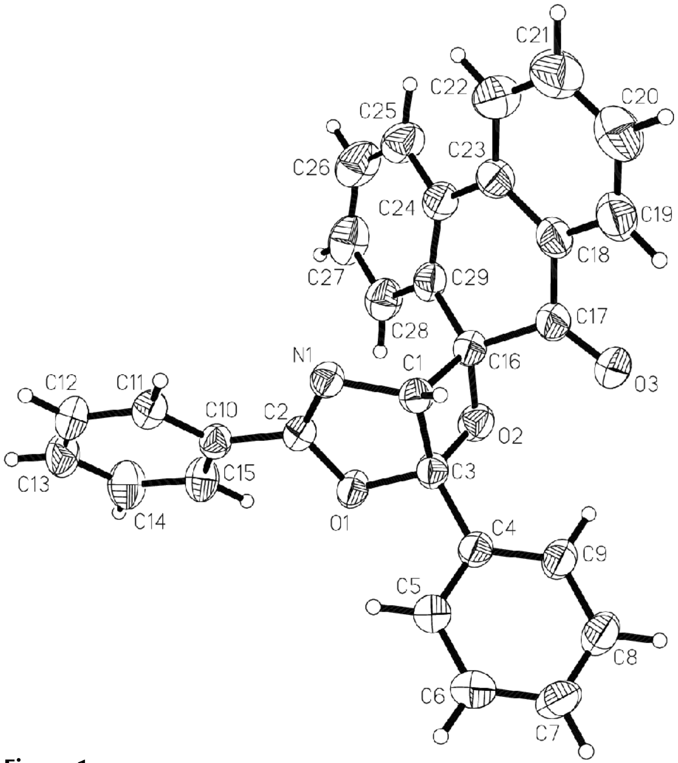


**Figure 1**
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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