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1-Oxo-1,2-dihydro-1,4-oxazino[2,3,4-*jk*]-carbazole-4-carbaldehyde

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Key indicators

Single-crystal X-ray study
T = 203 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.103
Data-to-parameter ratio = 17.5

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

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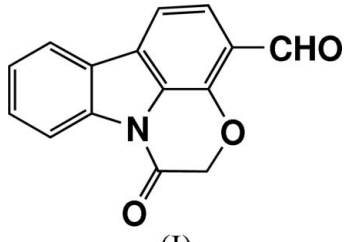
The carbazole unit of the title molecule, $C_{15}H_9NO_3$, is planar and forms dihedral angles of 3.08 (4)° with the attached carbaldehyde group and 0.75 (4)° with the fused oxazine ring. The dihedral angle between the two benzene rings is 2.09 (4)°. The structure is stabilized by inter- and intramolecular C—H···O hydrogen bonds.

Comment

Carbazole derivatives are well known for their pharmacological activities (Chowdhury *et al.*, 1978). Several reports have appeared on the syntheses of carbazole derivatives in connection with the search for newer physiologically active compounds. Carbazomycin A and carbazomycin B have been found to be useful as antibacterial and antifungal agents (Sakano *et al.*, 1980). It was reported that hetero-annulated carbazoles show marked anticancer (Kansal & Potier, 1986; Haider *et al.*, 1998; Hedin *et al.*, 2000) and anti-HIV activities (Hirata *et al.*, 1999). The discovery of the antineoplastic activity of the naturally occurring carbazole alkaloid ellipticine and its isomer olivacine has stimulated considerable research effort in the field of condensed systems (Haider, 2002). As synthetic materials, many carbazoles exhibit photoreactive, photoconductive and light-emitting properties (Thomas *et al.*, 2001; Van Dijken *et al.*, 2004). Carbazoles have also been recognized as a useful scaffold in anion-binding studies (Chmielewski *et al.*, 2004). Here, we present the crystal structure of such a heterofused carbazole, the title compound, (I).



(I)

The molecular structure of (I), with the atomic numbering scheme, is shown in Fig. 1. The carbazole unit is planar and forms dihedral angles of 3.08 (4)° with the attached carbaldehyde group at position 4 and 0.75 (4)° with the fused oxazine ring. The dihedral angle between the two benzene rings is 2.09 (4)°.

The structure is stabilized by inter- and intramolecular C—H···O hydrogen bonds (Table 1).

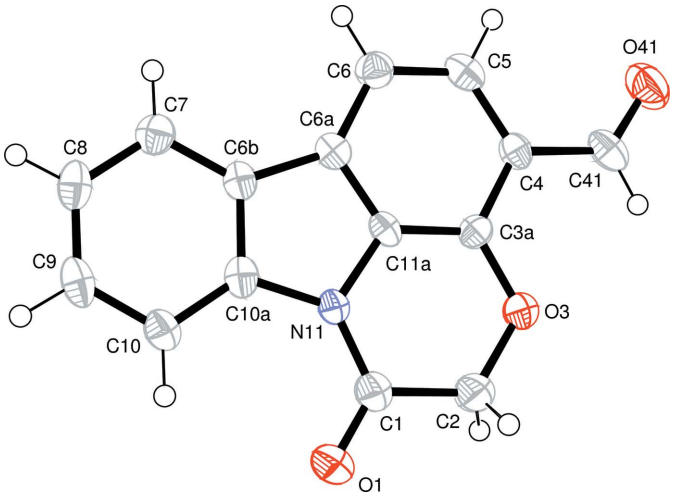


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

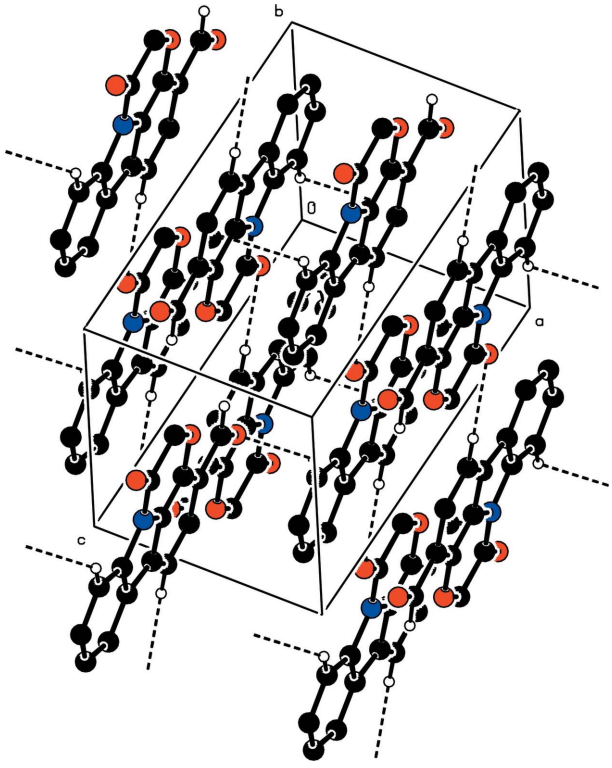


Figure 2
The molecular packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Experimental

A mixture of 1-hydroxycarbazole-2-carbaldehyde (211 mg, 0.001 mol), ethyl bromoacetate (166 mg, 0.001 mol) and ignited potassium carbonate (276 mg, 0.002 mol) in dry acetone (15 ml) was refluxed over a steam bath for 3 h. The reaction was monitored by thin-layer chromatography. After completion of the reaction, the solvent was removed by distillation and the mixture was poured on to crushed ice. It was extracted with ethyl acetate and dried over anhydrous sodium sulfate to obtain crude compound (I), which was purified by column chromatography over silica gel (60–120 mesh) using petroleum ether–ethyl acetate (95:5 *v*/*v*) as eluant (126 mg, 50%) and recrystallized from glacial acetic acid.

Crystal data

$C_{15}H_9NO_3$
M_r = 251.23
Triclinic, $P\overline{1}$
a = 6.811 (2) Å
b = 8.751 (2) Å
c = 10.066 (4) Å
α = 111.84 (3)°
β = 100.31 (3)°
γ = 91.98 (3)°
V = 544.6 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 203 (2) K
0.65 × 0.45 × 0.23 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*SCALE3 ABSPACK* in *CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.632, T_{max} = 1.000
7163 measured reflections
3013 independent reflections
1262 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.103
S = 0.82
3013 reflections
172 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^{i}	0.94	2.56	3.493 (3)	174
C10—H10···O41^{ii}	0.94	2.53	3.213 (3)	129
C41—H41···O3	0.94	2.53	2.861 (2)	101

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

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.94–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

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