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3-Benzoylmethylene-1-methyl-3,4-dihydroquinoxalin-2(1*H*)-one

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Key indicators

Single-crystal X-ray study
T = 288 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.107
Data-to-parameter ratio = 12.4

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

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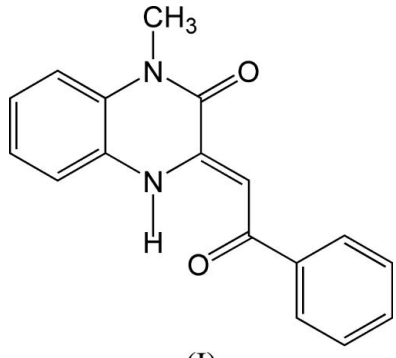
In the title compound, $C_{17}H_{14}N_2O_2$, the quinoxaline ring is essentially planar and makes a dihedral angle of 9.1 (2)° with the benzene ring of the benzoylmethylene group. There are intramolecular N—H···O hydrogen bonds and intermolecular C—H···O interactions.

Comment

The photochemical reactions of compounds containing the C=N double bond have not been as extensively investigated as those of carbonyl compounds. Nishio (1984) discovered that irradiation of quinoxalin-2-ones with alkenes results in regioselective [2+2]-cycloaddition to the C=N double bond. However, photoreactions of quinoxalin-2-ones with alkynes have not been reported before. We have recently investigated photo-induced reactions between quinoxalin-2-ones and phenylacetylenes. The title compound, (I), a β-benzoylmethylene derivative of quinoxalin-2-one, was isolated from the photoreaction of *N*-methylquinoxalin-2-one with phenylacetylene.



(I)

The geometrical parameters of the quinoxaline moiety in (I) (N1/C9/C10/N2/C12–C17) are comparable to those of the related structures reported earlier (Stepien *et al.*, 1976). The quinoxaline moiety is essentially planar (Fig. 1), mainly owing to the C=O and C—N conjugation, the dihedral angle between the planes of its heterocyclic and benzene ring (C12–C17) being 0.7 (2)°. The acetyl group (O1/C7/C8) bonded to atom C9 is twisted out of the quinoxaline plane by a small angle of 0.7 (2)°, thus indicating that the acetyl group tends to be coplanar with the quinoxaline system, obviously as a result of the π-conjugation involving the acetyl C=O bond. The C1–C6 benzene ring is almost coplanar with the quinoxaline moiety, with a dihedral angle of 9.1 (2)°. There are an intramolecular N1—H1*N*···O1 hydrogen bond and intermolecular C—H···O interactions (Table 2).

Experimental

Compound (I) was prepared by the photo-induced reaction of a benzene solution of *N*-methylquinoxaline-2-one with an excess amount of phenylacetylene, irradiated by the light of wavelength longer than 330 nm for 48 h, and was isolated by column chromatography of the reaction mixture after evaporation of the solvent on silica gel. Single crystals of (I) were obtained by slow evaporation from a petroleum ether–ethyl acetate (3:1) solvent system (yield 13.6%).

Crystal data

$C_{17}H_{14}N_2O_2$
M_r = 278.30
Monoclinic, $P2_1/n$
a = 9.6050 (19) Å
b = 10.767 (2) Å
c = 13.374 (3) Å
β = 104.36 (3)°
V = 1339.9 (5) Å^3
Z = 4
D_x = 1.380 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.09 mm^{-1}
T = 288 (2) K
Block, red
0.42 × 0.31 × 0.28 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (*XCAD4*; Harms & Wocadlo, 1995)
T_{min} = 0.946, T_{max} = 0.975
2510 measured reflections
2363 independent reflections
1356 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 25.0°
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -15 \rightarrow 15$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.107
S = 1.00
2363 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 0.49P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0139 (12)

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.256 (2)	N2—C12	1.408 (3)
O2—C10	1.225 (2)	N2—C11	1.468 (3)
N1—C9	1.345 (2)	C3—C7	1.499 (3)
N1—C17	1.389 (3)	C7—C8	1.423 (3)
N2—C10	1.373 (3)	C8—C9	1.375 (3)
C9—N1—C17	123.96 (19)	C8—C7—C3	120.2 (2)
C10—N2—C12	122.57 (19)	C9—C8—C7	123.4 (2)
O1—C7—C8	121.9 (2)	O2—C10—N2	121.5 (2)
O1—C7—C3	117.9 (2)	O2—C10—C9	121.2 (2)
C2—C3—C7—C8	−8.2 (3)	C7—C8—C9—N1	−0.1 (3)
C3—C7—C8—C9	178.0 (2)		

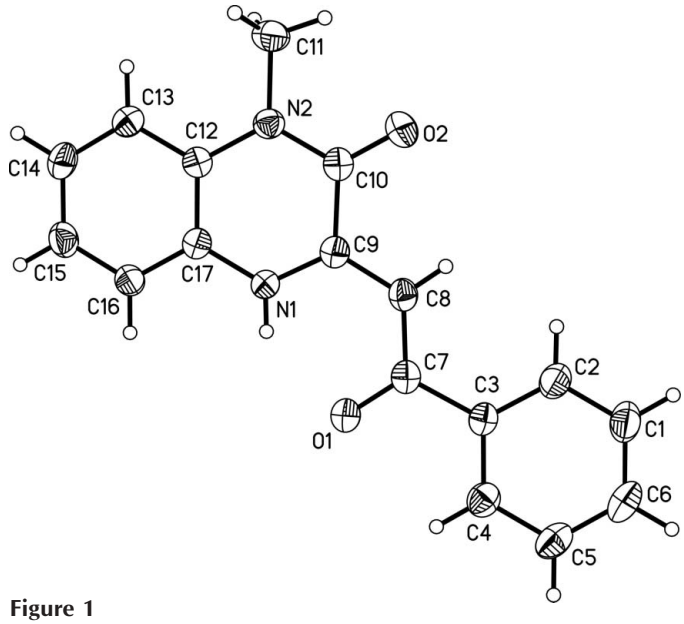


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O1	0.86	2.00	2.653 (2)	132
C6—H6···O1^{i}	0.93	2.55	3.382 (3)	149
C16—H16···O2^{ii}	0.93	2.50	3.356 (3)	153

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

The H atoms were positioned geometrically and were treated as riding, with C—H = 0.93–0.96 Å, N—H = 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(parent atom) or U_{iso}(H) = 1.5U_{eq}(C_{methyl}).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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* and *PLATON* (Spek, 2003).

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