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3-[(2,3-Diphenylquinoxalin-5-ylamino)-methylene]pentane-2,4-dione

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Key indicators

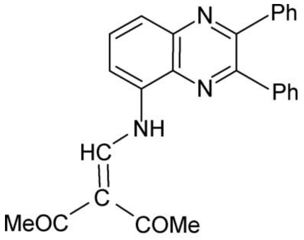
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.054
wR factor = 0.181
Data-to-parameter ratio = 16.8

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

In the title compound, $C_{26}H_{21}N_3O_2$, the enaminone portion of the molecule is almost exactly planar owing to strong conjugation across the ethylene double bond and is inclined at an angle of 20.8 (1)° to the mean plane of the quinoxaline heterocycle. The molecule has an intramolecular hydrogen bond between the NH and CO groups in the *Z* configuration, adopting the chelated form. The phenyl rings are twisted by *ca* 46° from the heterocyclic plane.

Comment

As a part of our project in biologically active compounds, we have focused our attention on quinoxaline derivatives having an enaminone group bonded to various positions of the heterocycle. These compounds are known to possess a wide spectrum of biological activities and can be easily prepared by a reaction of 3-ethoxymethylene-2,4-pentanedione with the corresponding heterocyclic amine (Černuchová *et al.*, 2005). In this communication we report the crystal structure of the title compound, (I), which consists of a diacetyleneamine group attached to the 5-position of 2,3-diphenylquinoxaline.



(I)

The molecular structure of (I) and the atom numbering is shown in Fig. 1. The most important feature of the structure concerns the degree of planarity of the molecule, *i.e.* the shape and orientation of the substituents with respect to the central quinoxaline ring system. First, for steric reasons, the two phenyl rings at C1 and C2 are twisted by 46.9 (1) and 46.2 (1)°, respectively, from the mean plane of the heterocycle [r.m.s. deviation 0.050 (2) Å]. As to the enaminone group, as revealed by the pattern of bond lengths (Table 1), the lone-pair electrons on the sp^2 hybridized N3 atom are delocalized across the ethylene bond as far as the carbonyl O atoms; as a result, a high degree of planarity is observed for this nine-atom molecular fragment [r.m.s. deviation 0.035 (2) Å]. Even though both carbonyl bonds lie in the plane of the C═C bond, they are non-equivalent; while the C23═O1 carbonyl (*Z* to the amine N atom) is oriented *syn* to the C═C bond, the conformation of the C25═O2 (*E* to N3) is *anti* (Fig. 1). The *syn* conformation of the *Z* acetyl group seems to be stabilized by an intramolecular hydrogen bond formed between N3/H and O1 [N$\cdots$O = 2.605 (2) Å]. Although to a much lesser extent, atom N3 is also involved in conjugation with the quinoxaline ring system, as indicated by the C4—N3 bond length [1.397 (2) Å], which is slightly but significantly shorter than the value of 1.425 (3) Å reported for a pure Csp^2—Nsp^2 single bond (Adler *et al.*, 1976). Consequently, the dihedral angle between the mean planes of the quinoxaline and enaminone groups is rather small [20.8 (1)°]. Nevertheless, based on our results, a low barrier to rotation around C4—N3 is predicted.

As the only hydrogen-bond donor of the molecule is utilized for intramolecular hydrogen bonding, the crystal packing is governed by ionic and van der Waals forces.

Experimental

5-Nitro-2,3-diphenylquinoxaline (10 mmol) in methanol (100 ml) was mixed with 3% Pd/C catalyst (350 mg) and hydrogenated (with magnetic stirring) at 120 kPa until 670 ml of hydrogen was consumed. The mixture was filtered into a methanol solution of 3-ethoxymethylene-2,4-pentanedione (10 mmol, 100 ml) and boiled with magnetic stirring for a short time. After cooling, the reaction mixture was evaporated *in vacuo* to dryness and the residue recrystallized from EtOH/benzene (1:1) to afford (I) as yellow crystals (51% yield; m.p. 474–476 K).

Crystal data

$C_{26}H_{21}N_3O_2$
M_r = 407.46
Monoclinic, $P2_1/n$
a = 12.326 (3) Å
b = 10.726 (2) Å
c = 15.999 (3) Å
β = 98.43 (3)°
V = 2092.4 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
Absorption correction: none
25458 measured reflections
4748 independent reflections
2246 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.181$
S = 0.94
4748 reflections
282 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C4—N3	1.397 (2)	C22—C25	1.468 (3)
C21—N3	1.324 (2)	C23—O1	1.225 (2)
C21—C22	1.386 (3)	C25—O2	1.217 (3)
C22—C23	1.463 (3)		
N1—C1—C9—C10	−47.1 (2)	C21—C22—C23—O1	−3.0 (3)
N2—C2—C15—C20	−48.1 (2)	C21—C22—C25—O2	179.1 (2)
N3—C21—C22—C23	4.7 (3)	C5—C4—N3—C21	8.4 (3)

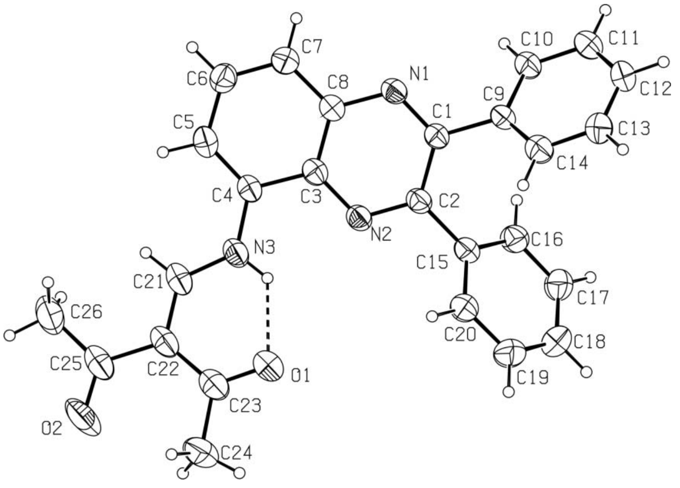


Figure 1
The molecular structure of (I), with the labelling scheme for the non-H atoms, which are drawn as 35% probability ellipsoids. The intramolecular hydrogen bond is illustrated by a dashed line.

H atoms were visible in difference maps and were subsequently treated as riding atoms with distances C—H = 0.93 Å (CH$_{\rm arom}$), 0.96 Å (CH$_3$) and N—H = 0.86 Å; $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N) (1.5 for the methyl H atoms).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2001); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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