organic papers

1-Ethyl-4-phenylethyl-1,4-dihydroquinoxaline-2,3-dione

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean σ(C–C) = 0.004 Å

R factor = 0.048

wR factor = 0.089

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.

The title compound, $C_{18}H_{18}N_2O_2$, was synthesized by heating 2,4-dioxo-1,3-diphenyl-7,8-benzo-6-ethyl-9-(2-phenylethyl)-1,3,6,9-tetrazaspiro[4.4]nonane in toluene. The quinoxaline ring system is planar and the dihedral angle between the phenyl and piperazine rings is 28.6 (1)°. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds.

Comment

Electron-rich olefins are very reactive compounds and they easily react with many organic reagents. The reaction of electron-rich olefins with phenyl isocyanate gives parabanic acid derivatives (Hocker & Merten, 1971). 1,4-Diphenyl-quionoxaline-2,3-dione was first synthesized from N,N'-diphenyl-o-phenylenediamine and oxalyl chloride (Bourson, 1971). We have also synthesized and reported some parabanic acid derivatives of bis(1,3-dimethylbenzimidazol-2-ylidene) (Küçükbay *et al.*, 1995, 2003; Küçükbay & Durmaz, 1997). According to the literature, the title compound, (I), was was first synthesized from an electron-rich olefin *via* a parabanic acid derivative. The aim of this study was to explain the rearrangement of the parabanic acid derivative to the quionoxaline compound and elucidate the crystal structure of the title compound.

(I)
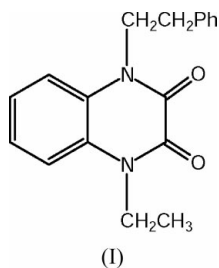
The bond lengths and angles observed in (I) show normal values (Allen *et al.*, 1987). The quinoxaline ring system is planar within 0.057 (2) Å, with a dihedral angle of 3.0 (1)° between the piperazine (N1/N2/C3–C8) and benzene (C13–C18) rings; the dihedral angle between the piperazine and phenyl rings is 28.6 (1)°.

The piperazine ring and the phenyl ring of the symmetry-related molecule at $(\frac{1}{2} - x,\ y - \frac{1}{2},\ z)$ are stacked, with a centroid–centroid distance of 3.467 (2) Å. In the crystal structure, intermolecular C—H···O hydrogen bonds form a network structure (Table 2 and Fig. 2).

Experimental

2,4-Dioxy-1,3-diphenyl-7,8-benzo-6-ethyl-9-(2-phenylethyl)-1,3,6,9-tetrazaspiro[4.4]nonane (Küçükbay *et al.*, 2003) (0.67 g, 1.37 mmol) was refluxed in non-dried toluene (5 ml) for half an hour. The
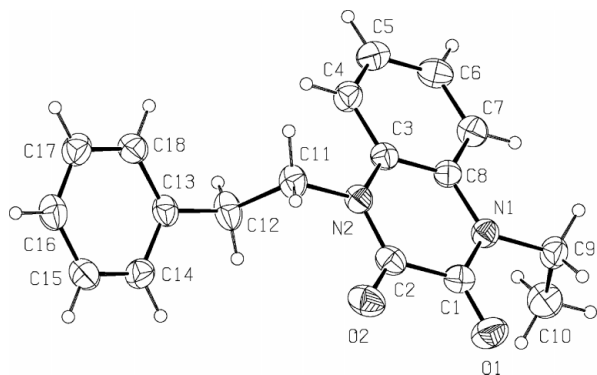
Figure 1
An *ORTEP*-3 (Farrugia, 1997) view of the title compound, showing the atom-numbering scheme and 30% probability displacement ellipsoids.
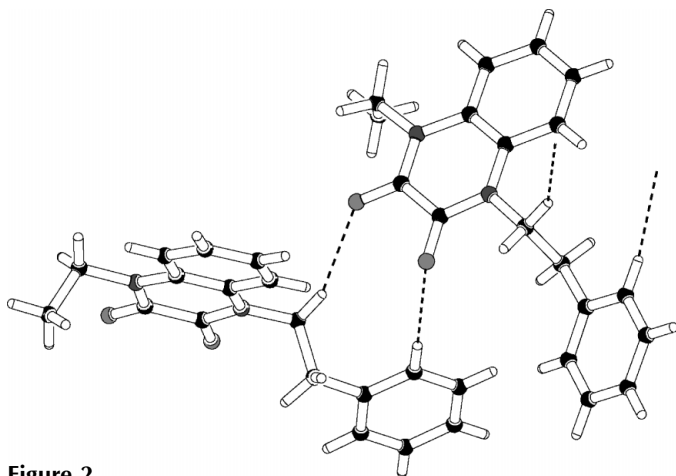

Figure 2
A view of the C—H···O hydrogen bonds (dashed lines) in the title compound.

solution was then concentrated to half of the original volume. Upon cooling the solution to 253 K, crystals of the title compound were obtained (yield: 0.36 g, 89%; m.p. 459–460 K). ^{1}H NMR (CDCl$_3$): δ 1.2 (t, CH$_2$CH$_3$, 3H), 2.8 (t, CH$_2$CH$_2$Ph, 2H), 3.5 (t, CH$_2$CH$_2$Ph, 2H), 3.3 (q, CH$_2$CH$_3$, 2H), 6.2–7.6 (m, Ar-H, 19H). ^{13}C NMR (CDCl$_3$): δ 15.44, 36.66, 42.59, 48.69, 100.78, 105.08, 119.82, 120.06, 130.13, 132.36, 153.35, 154.45. $\nu_{(C=O)}$: 1725 cm^{-1}. Analysis calculated for C$_{18}$H$_{18}$N$_2$O$_2$: C 73.47, H 6.12, N 9.52%; found: C 74.17, H 6.48, N 11.25%.

Crystal data

C$_{18}$H$_{18}$N$_2$O$_2$	Mo Kα radiation
$M_r = 294.34$	Cell parameters from 6175
Orthorhombic, *Pbca*	reflections
$a = 13.369$ (5) Å	$\theta = 2.5$–25.3°
$b = 15.310$ (5) Å	$\mu = 0.09$ mm^{-1}
$c = 14.690$ (5) Å	$T = 293$ K
$V = 3006.7$ (18) Å^3	Prism, yellow
$Z = 8$	$0.42 \times 0.33 \times 0.24$ mm
$D_x = 1.301$ Mg m^{-3}	

Data collection

Stoe IPDS-II diffractometer	$R_{int} = 0.115$
ω scans	$\theta_{max} = 27.3°$
Absorption correction: none	$h = -14 \rightarrow 17$
18583 measured reflections	$k = -19 \rightarrow 19$
3351 independent reflections	$l = -18 \rightarrow 18$
1328 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.089$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.86$	$\Delta\rho_{max} = 0.12$ e Å^{-3}
3351 reflections	$\Delta\rho_{min} = -0.13$ e Å^{-3}
200 parameters	Extinction correction: *SHELXL*97
H-atom parameters constrained	Extinction coefficient: 0.0021 (3)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.222 (3)	N1—C9	1.476 (3)
O2—C2	1.230 (3)	N2—C2	1.363 (3)
N1—C1	1.356 (3)	N2—C3	1.401 (3)
N1—C8	1.404 (3)	N2—C11	1.473 (3)
C1—N1—C8	122.7 (2)	O2—C2—N2	123.2 (2)
C1—N1—C9	115.5 (2)	O2—C2—C1	119.0 (2)
C8—N1—C9	121.67 (19)	N2—C2—C1	117.9 (2)
C2—N2—C3	122.4 (2)	N2—C3—C4	121.5 (2)
C2—N2—C11	116.04 (19)	N2—C3—C8	119.6 (2)
C3—N2—C11	121.5 (2)	N1—C8—C3	119.2 (2)
O1—C1—N1	122.7 (2)	N1—C8—C7	121.5 (2)
O1—C1—C2	119.5 (2)	N1—C9—C10	111.5 (2)
N1—C1—C2	117.8 (2)	N2—C11—C12	112.50 (19)
C8—N1—C1—O1	175.0 (2)	O1—C1—C2—O2	−0.4 (4)
C8—N1—C9—C10	−89.2 (3)	N2—C11—C12—C13	164.0 (2)
C3—N2—C11—C12	86.7 (3)	C11—C12—C13—C14	−107.7 (3)
C3—N2—C2—O2	−175.4 (2)	C11—C12—C13—C18	70.5 (3)
C2—N2—C11—C12	−90.4 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O1[i]	0.97	2.47	3.336 (3)	149
C18—H18···O2[i]	0.93	2.54	3.453 (3)	167

Symmetry code: (i) $\frac{1}{2} + x, y, \frac{3}{2} - z$.

The crystals of the title compound diffracted very poorly, displaying broad weak reflections. Therefore, the measured intensities have a high standard uncertainty and, as a consequence, the R_{int} value is also relatively high. H atoms were placed in geometrically idealized positions (C—H = 0.93–0.97 Å) and allowed to ride on their parent C atoms, with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for the other H atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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