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3-{[4-(4-Methylphenylsulfonamido)benzoyl]methylidene}-3,4-dihydroquinoxalin-2(1*H*)-one

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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 = 0.127
Data-to-parameter ratio = 12.2

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

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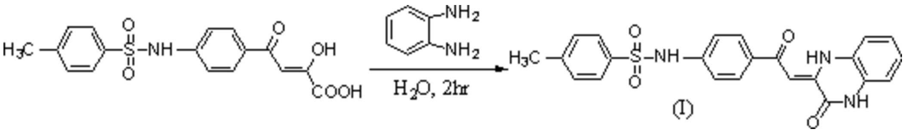
The title compound, $C_{23}H_{19}N_3O_4S$, a quinoxalone derivative, is the product of the condensation reaction of 4-[4-(4-methylphenylsulfonamido)phenyl]-2,4-dioxobutanoic acid with *o*-phenylenediamine. Intermolecular N—H···O hydrogen bonds and π–π stacking interactions stabilize the solid structure.

Comment

HIV-1 integrase (IN), one of three constitutive viral enzymes required for the retroviral replication cycle, has emerged as a promising target for chemotherapeutic intervention in the treatment of AIDS (Hazuda *et al.*, 1997; De Clercq, 2000). For the past few years, extensive efforts have been made resulting in a large number of HIV IN inhibitors, among which a new class of inhibitors, typified by a general diketo acid motif, was demonstrated to afford preferential inhibition of ST *versus* 3′-P reactions. The diketo acid unit is essential for the enzyme inhibitory activity (Hazuda *et al.*, 2000).



As part of our programme to search for viable replacements of the critical diketo acids with more drug-like characteristics, we have recently designed the 3-(2-oxo-2-phenylethylidene)-3,4-dihydro-1*H*-quinoxalin-2-one moiety as the bioisostere of such pharmacophores. We report here the synthetically efficient procedure (Ferro *et al.*, 2004; Saloutin *et al.*, 2000) and the characterization of the quinoxalone compound, (I).

The title compound consists of three molecular fragments: (i) a quinoxalone ring conjugated with the 2-oxoethylidene group, (ii) a central benzene ring and (iii) a *p*-methyloxyphenylsulfonamide group. The C1/C6/N2/C7/C8/N3 ring has a planar arrangement, with the largest deviation from the plane being 0.023 (2) Å, which implies that the two N atoms adopt sp^2 hybridization. This can also be deduced from the bond angles related to the two N atoms (Table 1), which are close to the standard angle of 120°. It should be noted that the bond lengths between each N atom and the neighbouring two C atoms are different; the lengths of the N2—C6 and N3—C1 bonds are longer than N2—C7 and N3—C8, respectively (Table 1). The quinoxalone ring and the 2-oxoethylidene unit are coplanar, the dihedral angle and the C7—C8—C9—C10 torsion angle being 1.9 (1)° and 177.9 (2)°, respectively. An intramolecular N3—H3*C*···O3 hydrogen bond forces the quinoxalone ring and 2-oxoethylidene unit to be in the same

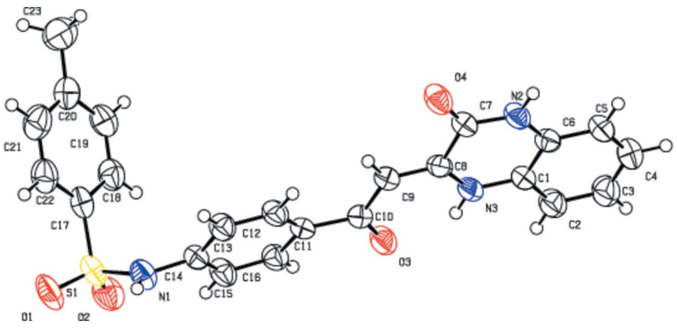


Figure 1
View of the title compound, showing 50% probability displacement ellipsoids.

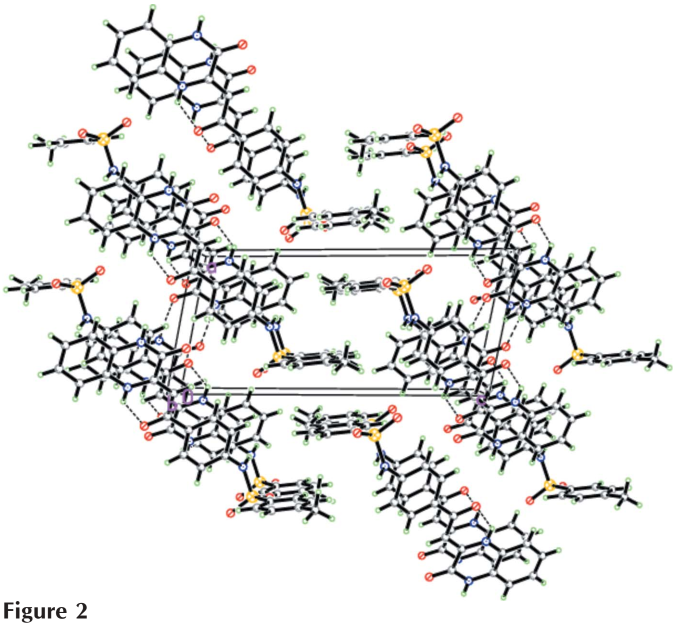


Figure 2
A three-dimensional view of the title compound along the *b* axis. Hydrogen bonds are illustrated by dashed lines.

plane. The 2-oxoethylidene unit is also conjugated with the central benzene ring, but the plane formed by the C9—C10—O3 group deviates from the benzene ring with a dihedral angle of 12.3 (1)°. The quinoxalone ring and the central benzene ring form a dihedral angle of 13.2 (1)°. There is an intermolecular N—H···O hydrogen bond (Table 2). In addition, there is a π–π stacking interaction between the two planes of a quinoxalone ring and the 2-oxoethylidene unit, with a centroid separation of 3.484 (2) Å.

Experimental

A mixture of 4-[4-(4-methylphenylsulfonamido)phenyl]-2,4-dioxobutanoic acid (3 mmol), *o*-phenylenediamine (3.6 mmol) and water (30 ml) was refluxed with stirring for 2–4 h. A yellow sticky solid appeared during the reaction time. The reaction mixture was cooled to room temperature, and the product was collected by filtration and washed with water and then with diethyl ether to remove the residual diamine. The compound was purified by flash column chromatography (eluant: methanol/$CHCl_3$, 1:10). Yield 60.8%, m.p. 545 K. ^1H NMR (DMSO-d_6, p.p.m.): δ 2.32 (1H, *s*, CH_3), 6.71 (1H, *s*, COCH), 7.09 (3H, *m*, Ar—H), 7.22 (2H, *d*, J = 7.52 Hz, Ar—H), 7.35 (2H, *d*, J = 7.96 Hz, Ar—H), 7.45 (1H, *m*, Ar—H), 7.71 (2H, *d*, J = 6.92 Hz, Ar—H), 7.86 (2H, *d*, J = 7.72 Hz, Ar—H), 10.72 (1H, *s*, CONH), 11.98 (1H, *s*, NH), 13.60 (1H, *s*, OH); ^{13}C NMR (DMSO-d_6, p.p.m.): δ 187.9, 156.2, 145.8, 144.1, 144.8, 137.0, 134.1, 130.3, 128.9, 127.19, 127.06, 124.6, 124.3, 124.1, 118.8, 116.9, 115.8, 89.4, 39.97. Single crystals of the title compound were obtained from a methanol solution by slow evaporation.

Crystal data

$C_{23}H_{19}N_3O_4S$
$M_r = 433.47$
Triclinic, $P\overline{1}$
$a = 7.485$ (3) Å
$b = 8.509$ (4) Å
$c = 16.377$ (7) Å
$\alpha = 82.938$ (6)°
$\beta = 78.218$ (6)°
$\gamma = 83.095$ (6)°
$V = 1008.5$ (8) Å^3
$Z = 2$
$D_x = 1.427$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1347 reflections
$\theta = 2.6$–25.2°
$\mu = 0.20$ mm^{-1}
$T = 293$ (2) K
Block, pale red
0.28 × 0.22 × 0.16 mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.590$, $T_{\max} = 0.969$
5245 measured reflections
3431 independent reflections
2356 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$
$\theta_{\max} = 25.0°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 6$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.127$
$S = 1.08$
3431 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.1035P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.4210 (19)	N2—C6	1.392 (3)
S1—O1	1.4259 (19)	N2—C7	1.352 (3)
S1—N1	1.619 (2)	N3—C1	1.381 (3)
S1—C17	1.752 (3)	N3—C8	1.349 (3)
N1—C14	1.426 (3)		
C7—N2—C6	124.63 (19)	N2—C7—C8	116.3 (2)
C1—N3—C8	124.57 (19)	N3—C8—C7	117.4 (2)
N3—C1—C2	121.2 (2)	N3—C8—C9	123.4 (2)
N3—C1—C6	118.5 (2)	O3—C10—C9	121.7 (2)
C1—C6—N2	118.4 (2)		
N3—C1—C6—C5	179.1 (2)	N2—C7—C8—C9	177.1 (2)
C7—N2—C6—C1	−2.0 (3)	C8—C9—C10—O3	−0.7 (4)
N2—C7—C8—N3	−3.8 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O4$^{\rm i}$	0.86	1.98	2.830 (3)	171
N3—H3*C*···O3	0.86	1.95	2.608 (3)	132

Symmetry code: (i) $-x+3, -y+1, -z$.

Acta Cryst. (2005). E**61**, o3802–o3804 Xu *et al.* • $C_{23}H_{19}N_3O_4S$ **o3803**

H atoms were treated as riding, with N—H and C—H distances of 0.86 and 0.93–0.96 Å, respectively, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C,N})$ and $1.5U_{\rm eq}({\rm C_{methyl}})$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (2002). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

De Clercq, E. (2000). *Rev. Med. Virol.* **10**, 255–277.

Ferro, S., Rao, A., Zappal, M., Chimirri, A., Bavreca, M. L., Witvrouw, M., Debyser, Z. & Monforte, P. (2004). *Heterocycles*, **63**, 2727–2734.

Hazuda, D. J., Felock, P. J., Hastings, J. C., Pramanik, B. & Wolfe, A. L. (1997). *Drug Des. Discov.* **15**, 17–24.

Hazuda, D. J., Felock, P., Witmer, M., Wolfe, A., Stillmock, K., Grobler, J. A., Espeseth, A., Gabryelski, L., Schleif, W., Blau, C. & Miller, M. D. (2000). *Science*, **287**, 646–650.

Saloutin,V. I., Burgart, Y. V., Kappe, C. O. & Chupahin, O. N. (2000). *Heterocycles*, **52**, 1411–1434.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

o3804 Xu *et al.* • $C_{23}H_{19}N_3O_4S$ *Acta Cryst.* (2005). E**61**, o3802–o3804