Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Synthesis, crystal structure and Hirshfeld surface analysis of 1-[3-(2-oxo-3-phenyl-1,2-dihydroquinoxalin-1-yl)propyl]-3-phenyl-1,2-dihydroquinoxalin-2-one

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Received 7 May 2024
Accepted 10 May 2024

Edited by L. Van Meervelt, Katholieke Universiteit Leuven, Belgium

Keywords: crystal structure; hydrogen bond; dihydroquinoxaline; π-stacking; C—H$\cdots\pi$(ring) interaction.

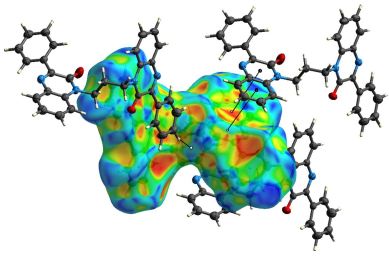
CCDC reference: 2354488

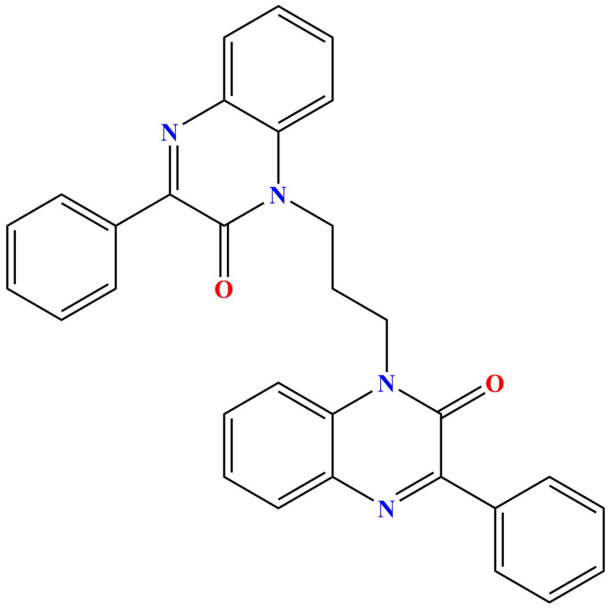
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compound, $C_{31}H_{24}N_4O_2$, the dihydroquinoxaline units are both essentially planar with the dihedral angle between their mean planes being 64.82 (4)°. The attached phenyl rings differ significantly in their rotational orientations with respect to the dihydroquinoxaline planes. In the crystal, one set of C—H$\cdots$O hydrogen bonds form chains along the *b*-axis direction, which are connected in pairs by a second set of C—H$\cdots$O hydrogen bonds. Two sets of π-stacking interactions and C—H$\cdots\pi$(ring) interactions join the double chains into the final three-dimensional structure.

1. Chemical context

The family of nitrogenous drugs, notably those containing the quinoxaline moiety, is important in medicinal chemistry because of the wide range of pharmacological activities exhibited, including antibacterial, antituberculosis, anti-inflammatory, antifungal anti-glycation, anti-analgesic and anticancer properties. In particular, quinoxalin-2-one derivatives are a class of heterocyclic compounds with different applications in various fields (Ramli *et al.*, 2014). They have been studied intensively as an important heterocyclic system for the synthesis of biologically active compounds ranging from herbicides and fungicides to therapeutically usable drugs (Ramli & Essassi, 2015). These chemicals are active anti-tumor agents with tyrosine kinase receptor inhibition properties (Galal *et al.*, 2014). They can also selectively antagonize the glycoprotein in cancer cells (Sun *et al.*, 2009). Quinoxalin-2-one derivatives are also potential antagonist ligands for imaging the A2A adenosine receptor by positron emission tomography (PET) (Holschbach et *al.*, 2005). Given the wide range of therapeutic applications for such compounds, we have previously reported a route for the preparation of quinoxalin-2-one derivatives using *N*-alkylation reactions carried out with di-halogenated carbon chains (Missioui *et al.* 2022; Abad *et al.*, 2024). A similar approach yielded the title compound, $C_{31}H_{24}N_4O_2$ (Fig. 1). In addition to the synthesis, we also report the molecular and crystal structure along with a Hirshfeld surface analysis.





2. Structural commentary

The title compound crystallizes in the triclinic space group $P\overline{1}$ with one molecule in the asymmetric unit (Fig. 2). The dihydroquinoxaline unit containing N1 is planar to within 0.038 (1) Å (r.m.s. deviation of fitted atoms = 0.0209 Å) while that containing N3 is planar to within 0.021 (1) Å (r.m.s. deviation = 0.0124 Å). The dihedral angle between their mean planes is 64.82 (4)°. The C9–C14 benzene ring is inclined to the plane of the dihydroquinoxaline unit containing N1 by 7.35 (5)°, which is due in part to an intramolecular C10—H10···O1 hydrogen bond (Table 1). The corresponding angle on the other half of the molecule is 37.63 (5)°. The greater out-of-plane orientation of the latter phenyl ring may be the result of its participation in C—H···π(ring) interactions (Table 1 and Fig. 3). There are close contacts of H29*A* with O1 (2.31 Å) and H31*B* with O2 (2.32 Å), which might be considered additional hydrogen-bond interactions although the C—H···O angles are only 102°. The central C—C—C unit extends out from N3 in an all-*trans* conformation with a C29—C30—C31—N3 torsion angle of −175.74 (9)° but this does not continue to the second quinoxaline unit as the N1—C29—C30—C31 torsion angle is −69.98 (13)°.

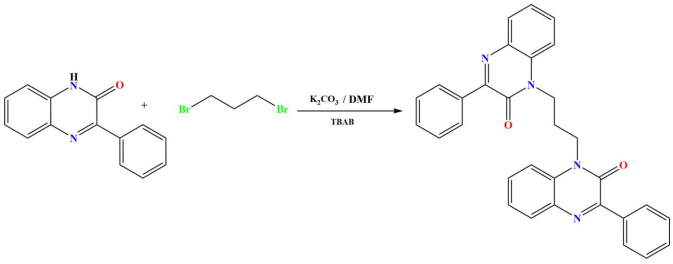


Figure 1
Synthesis of the title compound.

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

*Cg*6 is the centroid of the C23–C28 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.58	3.3871 (16)	146
C10—H10···O1	0.93	2.22	2.8531 (16)	124
C27—H27···O2^{ii}	0.93	2.59	3.4603 (18)	155
C30—H30*A*···*Cg*6^{iii}	0.97	2.75	3.6013 (14)	147

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

3. Supramolecular features

In the crystal, chains of molecules extending along the *b*-axis direction are formed by C3—H3···O1 hydrogen bonds and are linked in pairs into a Z-shaped motif by C27—H27···O2 hydrogen bonds (Table 1 and Fig. 3). The paired chains are joined by π-stacking interactions between inversion-related dihydroquinoxaline moieties containing N1 (symmetry code: $-x+1, -y+1, -z$) with a distance of 3.5676 (7) Å between the centroids of the N1/C6/C1/N2/C8/C7 and C1–C6 rings as

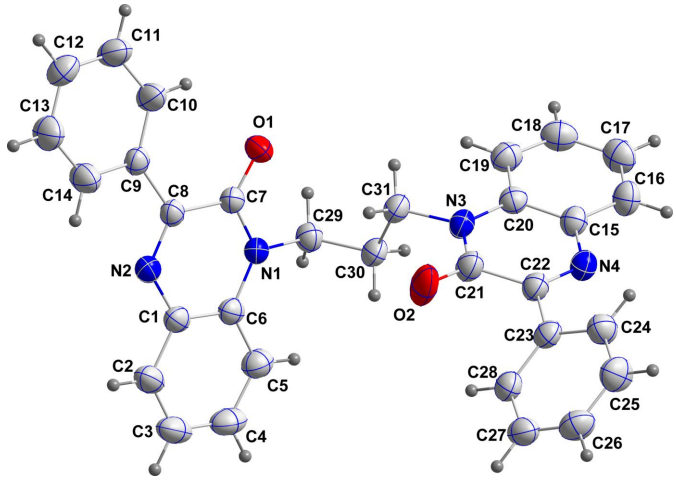


Figure 2
The title molecule with labeling scheme and 50% probability ellipsoids.

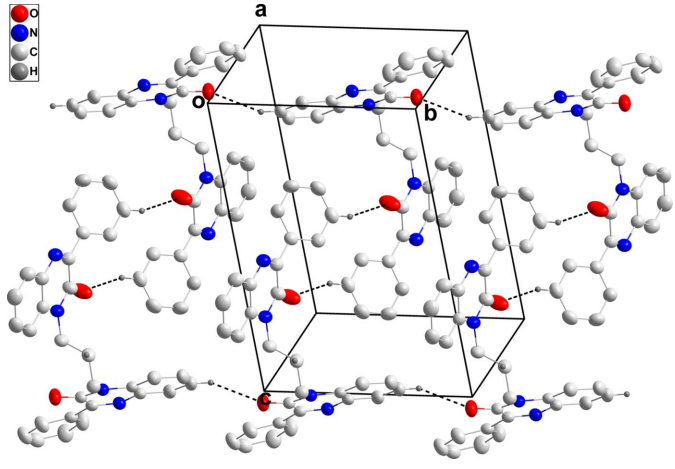


Figure 3
Perspective view of the chains formed by C—H···O hydrogen bonds (dashed lines).

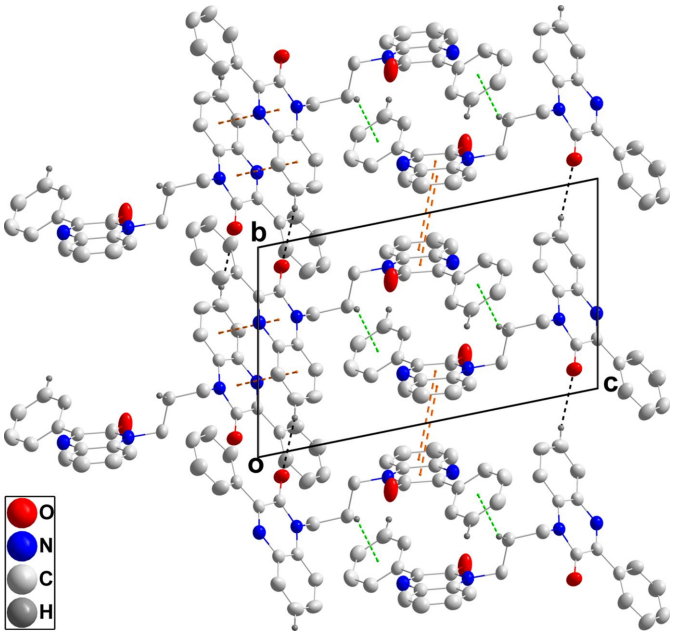


Figure 4
Packing viewed along the *a*-axis direction with C—H···O hydrogen bonds shown as black dashed lines and π-stacking and C—H···π(ring) interactions shown as orange and green dashed lines, respectively.

well as by corresponding interactions between those containing N3 (symmetry code: $-x+1, -y+2, -z+1$) with a distance of 3.8641 (7) Å between the centroids of the N3/C20/C15/N4/C22/C21 and C15–C20 rings (Fig. 4). These interactions are accompanied by inversion-related C30—H30*A*···*Cg*6 interactions (Table 1 and Fig. 4; *Cg*6 is the centroid of ring C23–C28).

4. Database survey

A search of the Cambridge Structural Database (CSD, updated to March 2024; Groom *et al.*, 2016) with the search fragment shown in Fig. 5 (*R* = anything) yielded five hits. These contain *R* = *n*-pentyl (AZAZEC; Abad *et al.*, 2021*b*), 2-oxy-3-phenylquinoxaline (KOPKAF; Abad *et al.*, 2024), OH (RIRBOM; Abad *et al.*, 2018), *n*-hexyl (UDAMIZ; Abad *et al.*, 2021*a*) and Et (UFITEM; Abad *et al.*, 2023). In AZAZEC, the quinoxaline unit is planar with the exception of the nitrogen bearing the alkyl chain while in the others, the unit shows somewhat greater deviations from planarity. The dihedral angle between the mean planes of the quinoxaline unit and the attached phenyl ring vary from 12.90 (4)° (AZAZEC) to 44.89 (3)° (RIRBOM) with the lower values resulting from intramolecular C—H···O hydrogen bonding. In AZAZEC, RIRBOM and UFITEM there are C—H···π(ring) interactions, which help stabilize the crystal packing, while in UFITEM and KOPKAF there are π-stacking interactions between inversion-related quinoxaline moieties as in the present case. In UFITEM there are C═O···π(ring) interactions as well. In the examples containing a single quinoxaline moiety, the absolute values of the N—C—C—C torsion angles vary from 178.73 (8)° (KOPKAF) to 168.64 (8)° (RIRBOM) while in KOPKAF and RIRBOM, the O2—C17—C16—C15 torsion angles are, respectively, −68.46 (12) and −63.85 (11)°. These conformations are quite similar to that in the present structure.

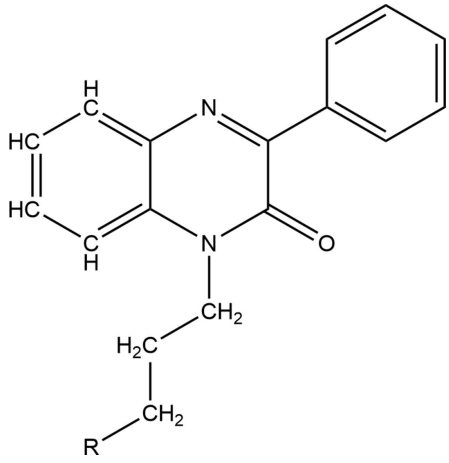


Figure 5
Search fragment used in the database survey.

5. Hirshfeld surface analysis

To quantify the intermolecular interactions, the Hirshfeld surface was calculated with *CrystalExplorer 21.5* (Spackman *et al.*, 2021). Descriptions of the plots generated and their interpretation have been published previously (Tan *et al.*, 2019). Fig. 6 shows the d_{norm} surface plotted over the range

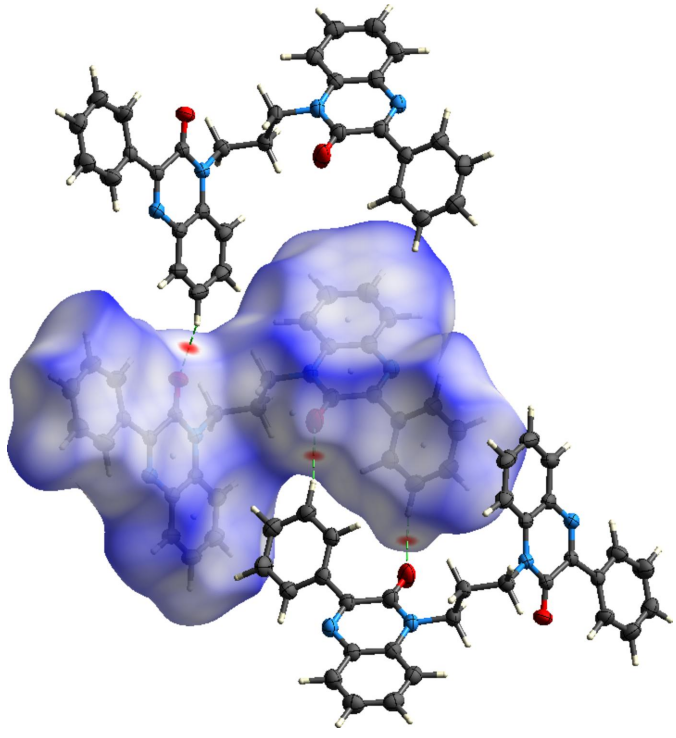


Figure 6
The Hirshfeld surface plotted over d_{norm} showing the C—H···O hydrogen bonds to neighboring molecules.

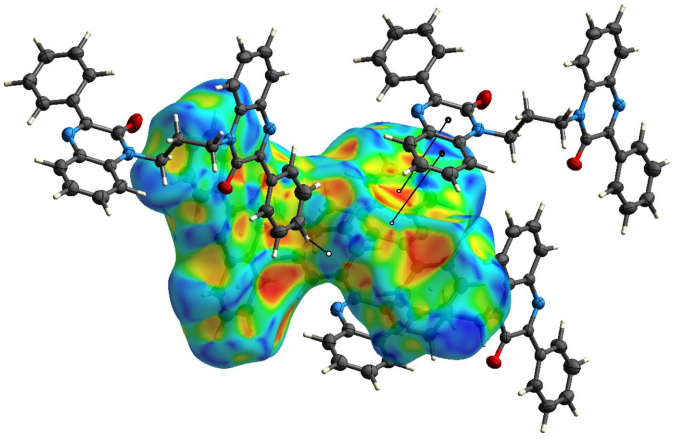


Figure 7
The Hirshfeld surface plotted over shape-index showing the π-stacking and C—H···π(ring) interactions to neighboring molecules.

−0.1072 to 1.3548 a.u. together with two neighboring molecules and the connecting C—H···O hydrogen bonds. The red spots on the surface clearly indicate the sites of these interactions. Fig. 7 shows the surface plotted over the shape-index with three neighboring molecules included. The pattern of blue and orange triangles marking a site of π-stacking interactions is clearly visible in the upper right of the surface with the interaction denoted by two lines. On the lower left, the C—H···π(ring) interaction is shown by a third line. The 2-D fingerprint plots (Fig. 8) show that the greatest contribution to the total intermolecular interactions is from H···H contacts at 49.6% (Fig. 8*a*), which is expected due to the significant hydrogen content and the fact that most of the hydrogen atoms are attached to aromatic rings. The other large contribution is from C···H/H···C contacts (23.0%, Fig. 8*b*), which come primarily from the C—H···π(ring) interactions. In addition, there are O···H/H···O contacts (7.4%, Fig. 8*c*), C···C contacts (5.8%, Fig. 8*d*) and N···H/H···N contacts (5.2%, Fig. 8*e*). The C···C contacts are primarily the π-stacking interactions.

6. Synthesis and crystallization

To a solution of 3-phenylquinoxalin-2(1*H*)-one (0.5 g, 2.25 mmol) in *N*,*N*-dimethylformamide (15 mL) were added 1,3-dibromopropane (0.12 ml, 1.125 mmol), sodium hydroxide (0.1 g, 2.25 mmol) and a catalytic quantity of tetra-*n*-butylammonium bromide. The reaction mixture was stirred at room temperature for 24 h. The solution was filtered and the solvent removed under reduced pressure. The residue obtained was chromatographed on a silica gel column using a hexane/ethyl acetate 9:1 mixture as eluent. The solid obtained upon solvent removal was recrystallized from ethanol to afford thick, colorless, plate-like crystals of the title compound with a yield of 30%, m.p. = 321–325 K, ^{1}H NMR (300 MHz, $CDCl_3$) δ ppm: 2.54 (quin, 2H, CH_2); 3.85 (*t*, 2H, N—CH_2, *J* = 6Hz); 3.96 (*t*, 2H, O—CH_2—N, *J* = 6Hz); 7.33–8.12 (*m*, 18H, CHarom).^{13}C NMR (75 MHz, $CDCl_3$) δ ppm: 22.16 (CH_2); 33.19 (N—CH_2); 34.87(N—CH_2); 113.43–134.23 (CHarom); 134.33–144.11 (Cq); 155.34 (C═O); 155.65 (C═O).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(H).

Acknowledgements

JTM thanks Tulane University for support of the Tulane Crystallography Laboratory. The contributions of the authors are as follows: conceptualization, EME and YR; methodology, AS; investigation, NA; writing (original draft), JTM and NA;

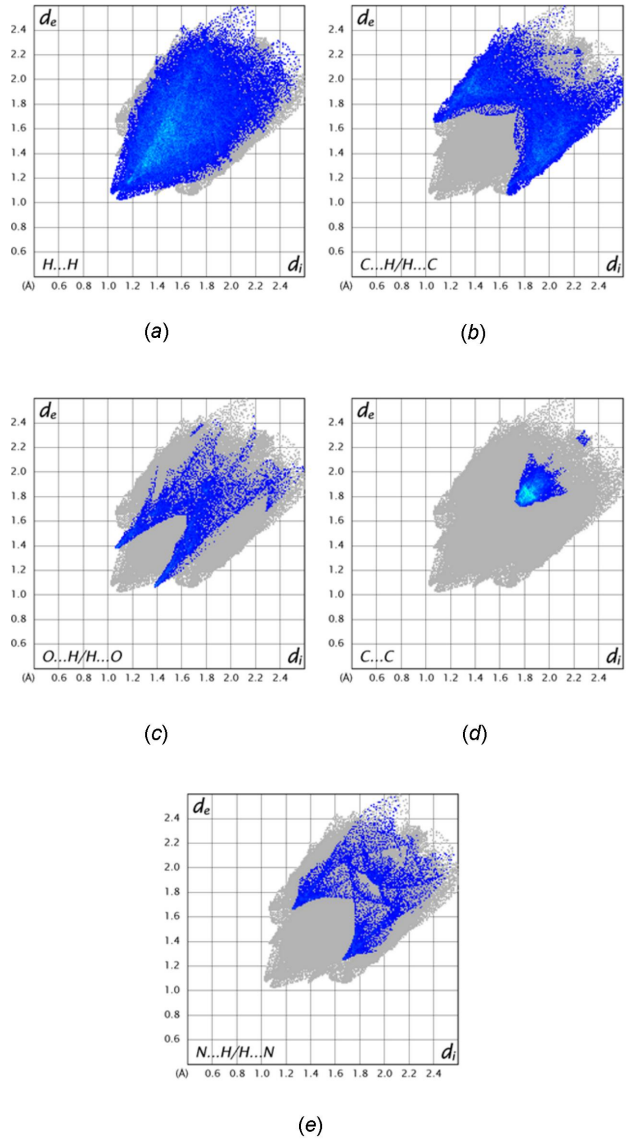


Figure 8
The 2-D fingerprint plots delineated into: (*a*) H···H interactions, (*b*) C···H/H···C interactions, (*c*) O···H/H···O interactions, (*d*) C···C interactions and (*e*) N···H/H···N interactions.

writing (review and editing of the manuscript), YR; formal analysis, YR; supervision, YR; crystal structure determination and validation, JTM; resources, AYAA

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Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{31}H_{24}N_4O_2$
M_r	484.54
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	298
a, b, c (Å)	9.0384 (3), 9.4484 (4), 14.9524 (6)
α, β, γ (°)	77.267 (1), 83.991 (1), 72.708 (1)
V (Å^3)	1188.16 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.35 × 0.30 × 0.10
Data collection	
Diffractometer	Bruker *SMART APEX* CCD
Absorption correction	Multi-scan (*SADABS*;Krause *et al.*, 2015)
T_{min}, T_{max}	0.89, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23105, 6318, 4347
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.686
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.140, 1.08
No. of reflections	6318
No. of parameters	334
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.16

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019/1* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

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supporting information

Acta Cryst. (2024). E**80**, 610-614 [https://doi.org/10.1107/S2056989024004377]

Synthesis, crystal structure and Hirshfeld surface analysis of 1-[3-(2-oxo-3-phenyl-1,2-dihydroquinoxalin-1-yl)propyl]-3-phenyl-1,2-dihydroquinoxalin-2-one

Nadeem Abad, Joel T. Mague, Abdulsalam Alsubari, El Mokhtar Essassi, Abdullah Yahya Abdullah Alzahrani and Youssef Ramli

Computing details

1-[3-(2-Oxo-3-phenyl-1,2-dihydroquinoxalin-1-yl)propyl]-3-phenyl-1,2-dihydroquinoxalin-2-one

Crystal data

$C_{31}H_{24}N_4O_2$
$M_r = 484.54$
Triclinic, $P\overline{1}$
$a = 9.0384$ (3) Å
$b = 9.4484$ (4) Å
$c = 14.9524$ (6) Å
$\alpha = 77.267$ (1)°
$\beta = 83.991$ (1)°
$\gamma = 72.708$ (1)°
$V = 1188.16$ (8) Å^3
$Z = 2$
$F(000) = 508$
$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7750 reflections
$\theta = 2.4$–$28.6°$
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Thick plate, colourless
0.35 × 0.30 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*;Krause *et al.*, 2015)
$T_{min} = 0.89$, $T_{max} = 0.99$
23105 measured reflections
6318 independent reflections
4347 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 29.2°$, $\theta_{min} = 2.3°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -20 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.140$
$S = 1.08$
6318 reflections
334 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0793P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, collected at φ = 0.00, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ, collected at ω = –30.00 and 210.00°. The scan time was 20 sec/frame.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å). All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.39620 (10)	0.88674 (9)	0.06918 (6)	0.0490 (2)
O2	0.21475 (12)	0.71620 (14)	0.39102 (6)	0.0692 (3)
N1	0.44699 (10)	0.63291 (10)	0.11355 (6)	0.0352 (2)
N2	0.21498 (11)	0.64096 (11)	0.00336 (6)	0.0396 (2)
N3	0.43990 (11)	0.77981 (11)	0.38219 (6)	0.0395 (2)
N4	0.42341 (11)	0.74089 (11)	0.57324 (7)	0.0408 (2)
C1	0.28939 (13)	0.50787 (13)	0.06015 (8)	0.0379 (3)
C2	0.24951 (16)	0.37603 (15)	0.05881 (9)	0.0485 (3)
H2	0.169960	0.381198	0.022410	0.058*
C3	0.32574 (17)	0.23990 (15)	0.11018 (10)	0.0546 (4)
H3	0.299792	0.152567	0.108083	0.066*
C4	0.44215 (17)	0.23383 (15)	0.16538 (10)	0.0544 (3)
H4	0.493638	0.141281	0.200596	0.065*
C5	0.48393 (15)	0.36109 (14)	0.16963 (9)	0.0475 (3)
H5	0.561308	0.354575	0.207998	0.057*
C6	0.40876 (13)	0.49995 (12)	0.11567 (7)	0.0362 (3)
C7	0.36729 (13)	0.77086 (13)	0.06313 (7)	0.0349 (2)
C8	0.24934 (12)	0.76485 (13)	0.00243 (7)	0.0338 (2)
C9	0.16777 (12)	0.90221 (13)	−0.06329 (7)	0.0359 (3)
C10	0.20206 (15)	1.03996 (15)	−0.08165 (9)	0.0502 (3)
H10	0.280946	1.051677	−0.051211	0.060*
C11	0.11914 (16)	1.15982 (16)	−0.14513 (10)	0.0563 (4)
H11	0.142651	1.251697	−0.156303	0.068*
C12	0.00292 (14)	1.14611 (16)	−0.19197 (9)	0.0492 (3)
H12	−0.051781	1.227421	−0.234579	0.059*
C13	−0.03084 (16)	1.01039 (16)	−0.17471 (10)	0.0579 (4)
H13	−0.108010	0.998776	−0.206648	0.070*
C14	0.04859 (16)	0.89079 (15)	−0.11040 (10)	0.0529 (3)
H14	0.021810	0.800486	−0.098289	0.063*
C15	0.54198 (13)	0.78976 (13)	0.52169 (8)	0.0390 (3)
C16	0.65404 (14)	0.81762 (15)	0.56774 (9)	0.0480 (3)

H16	0.645671	0.805913	0.631306	0.058*
C17	0.77608 (14)	0.86201 (16)	0.52021 (10)	0.0522 (3)
H17	0.850311	0.880335	0.551234	0.063*
C18	0.78793 (15)	0.87935 (15)	0.42545 (10)	0.0513 (3)
H18	0.871213	0.908817	0.393312	0.062*
C19	0.67912 (14)	0.85389 (14)	0.37802 (9)	0.0465 (3)
H19	0.688651	0.866859	0.314411	0.056*
C20	0.55402 (13)	0.80842 (12)	0.42573 (8)	0.0371 (3)
C21	0.31637 (14)	0.73619 (15)	0.43057 (8)	0.0439 (3)
C22	0.31797 (13)	0.71537 (13)	0.53167 (8)	0.0387 (3)
C23	0.19375 (13)	0.66197 (13)	0.58906 (8)	0.0383 (3)
C24	0.13789 (15)	0.71825 (15)	0.66841 (9)	0.0473 (3)
H24	0.175355	0.791637	0.683015	0.057*
C25	0.02711 (16)	0.66568 (17)	0.72557 (10)	0.0569 (4)
H25	−0.010397	0.704779	0.778051	0.068*
C26	−0.02799 (16)	0.55617 (16)	0.70545 (10)	0.0543 (3)
H26	−0.102757	0.521512	0.744151	0.065*
C27	0.02727 (15)	0.49781 (14)	0.62819 (9)	0.0485 (3)
H27	−0.008983	0.422527	0.615101	0.058*
C28	0.13690 (14)	0.55079 (14)	0.56969 (8)	0.0432 (3)
H28	0.172843	0.511759	0.517023	0.052*
C29	0.58258 (13)	0.62965 (14)	0.16149 (8)	0.0400 (3)
H29A	0.619669	0.715413	0.131354	0.048*
H29B	0.664531	0.538571	0.155101	0.048*
C30	0.55317 (14)	0.63387 (13)	0.26271 (8)	0.0424 (3)
H30A	0.652121	0.610476	0.290478	0.051*
H30B	0.504683	0.555402	0.291637	0.051*
C31	0.45142 (14)	0.78405 (14)	0.28279 (8)	0.0419 (3)
H31A	0.495241	0.864656	0.251294	0.050*
H31B	0.348760	0.804628	0.260318	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0647 (6)	0.0385 (5)	0.0502 (5)	−0.0216 (4)	−0.0196 (4)	−0.0048 (4)
O2	0.0685 (6)	0.1113 (9)	0.0408 (5)	−0.0518 (6)	−0.0143 (4)	−0.0002 (5)
N1	0.0409 (5)	0.0362 (5)	0.0305 (5)	−0.0135 (4)	−0.0033 (4)	−0.0064 (4)
N2	0.0472 (6)	0.0418 (6)	0.0344 (5)	−0.0199 (4)	−0.0024 (4)	−0.0068 (4)
N3	0.0441 (5)	0.0432 (6)	0.0304 (5)	−0.0122 (4)	−0.0047 (4)	−0.0042 (4)
N4	0.0399 (5)	0.0469 (6)	0.0350 (5)	−0.0121 (4)	−0.0046 (4)	−0.0060 (4)
C1	0.0469 (6)	0.0379 (6)	0.0317 (6)	−0.0176 (5)	0.0048 (5)	−0.0081 (5)
C2	0.0617 (8)	0.0467 (7)	0.0462 (7)	−0.0289 (6)	0.0051 (6)	−0.0129 (6)
C3	0.0732 (9)	0.0407 (7)	0.0558 (8)	−0.0274 (7)	0.0155 (7)	−0.0146 (6)
C4	0.0675 (9)	0.0340 (7)	0.0549 (9)	−0.0119 (6)	0.0089 (7)	−0.0037 (6)
C5	0.0539 (7)	0.0389 (7)	0.0451 (7)	−0.0096 (6)	−0.0013 (6)	−0.0043 (6)
C6	0.0431 (6)	0.0357 (6)	0.0307 (6)	−0.0137 (5)	0.0054 (4)	−0.0084 (5)
C7	0.0418 (6)	0.0362 (6)	0.0293 (6)	−0.0148 (5)	−0.0013 (4)	−0.0071 (4)
C8	0.0377 (6)	0.0388 (6)	0.0278 (5)	−0.0148 (5)	0.0008 (4)	−0.0083 (5)

C9	0.0386 (6)	0.0415 (6)	0.0285 (6)	−0.0130 (5)	0.0001 (4)	−0.0072 (5)
C10	0.0562 (8)	0.0510 (8)	0.0483 (7)	−0.0274 (6)	−0.0160 (6)	0.0032 (6)
C11	0.0634 (9)	0.0497 (8)	0.0571 (9)	−0.0278 (7)	−0.0138 (7)	0.0082 (6)
C12	0.0469 (7)	0.0509 (8)	0.0427 (7)	−0.0106 (6)	−0.0065 (5)	0.0028 (6)
C13	0.0578 (8)	0.0569 (9)	0.0609 (9)	−0.0166 (7)	−0.0258 (7)	−0.0042 (7)
C14	0.0584 (8)	0.0439 (7)	0.0602 (9)	−0.0176 (6)	−0.0219 (7)	−0.0052 (6)
C15	0.0366 (6)	0.0405 (6)	0.0378 (6)	−0.0074 (5)	−0.0046 (5)	−0.0067 (5)
C16	0.0451 (7)	0.0571 (8)	0.0416 (7)	−0.0124 (6)	−0.0085 (5)	−0.0096 (6)
C17	0.0408 (7)	0.0564 (8)	0.0619 (9)	−0.0145 (6)	−0.0105 (6)	−0.0122 (7)
C18	0.0433 (7)	0.0488 (8)	0.0613 (9)	−0.0154 (6)	0.0035 (6)	−0.0095 (6)
C19	0.0490 (7)	0.0451 (7)	0.0428 (7)	−0.0126 (6)	0.0025 (5)	−0.0067 (6)
C20	0.0364 (6)	0.0340 (6)	0.0387 (6)	−0.0065 (5)	−0.0038 (5)	−0.0065 (5)
C21	0.0448 (7)	0.0508 (7)	0.0360 (6)	−0.0163 (6)	−0.0080 (5)	−0.0015 (5)
C22	0.0407 (6)	0.0391 (6)	0.0342 (6)	−0.0093 (5)	−0.0048 (5)	−0.0044 (5)
C23	0.0375 (6)	0.0391 (6)	0.0345 (6)	−0.0082 (5)	−0.0063 (4)	−0.0005 (5)
C24	0.0518 (7)	0.0475 (7)	0.0453 (7)	−0.0173 (6)	0.0014 (5)	−0.0118 (6)
C25	0.0626 (9)	0.0630 (9)	0.0471 (8)	−0.0218 (7)	0.0145 (6)	−0.0172 (7)
C26	0.0537 (8)	0.0551 (8)	0.0530 (8)	−0.0226 (6)	0.0049 (6)	−0.0018 (7)
C27	0.0515 (7)	0.0432 (7)	0.0509 (8)	−0.0183 (6)	−0.0108 (6)	0.0011 (6)
C28	0.0474 (7)	0.0441 (7)	0.0369 (6)	−0.0111 (5)	−0.0091 (5)	−0.0045 (5)
C29	0.0394 (6)	0.0434 (7)	0.0382 (6)	−0.0126 (5)	−0.0060 (5)	−0.0069 (5)
C30	0.0516 (7)	0.0405 (7)	0.0337 (6)	−0.0118 (5)	−0.0117 (5)	−0.0019 (5)
C31	0.0501 (7)	0.0429 (7)	0.0303 (6)	−0.0119 (5)	−0.0057 (5)	−0.0021 (5)

Geometric parameters (Å, º)

O1—C7	1.2222 (13)	C13—H13	0.9300
O2—C21	1.2227 (13)	C14—H14	0.9300
N1—C7	1.3815 (14)	C15—C16	1.3978 (15)
N1—C6	1.3917 (13)	C15—C20	1.4031 (16)
N1—C29	1.4726 (13)	C16—C17	1.3701 (18)
N2—C8	1.2943 (14)	C16—H16	0.9300
N2—C1	1.3787 (15)	C17—C18	1.3864 (19)
N3—C21	1.3830 (15)	C17—H17	0.9300
N3—C20	1.3946 (14)	C18—C19	1.3748 (17)
N3—C31	1.4714 (14)	C18—H18	0.9300
N4—C22	1.2959 (14)	C19—C20	1.3988 (16)
N4—C15	1.3856 (15)	C19—H19	0.9300
C1—C2	1.4006 (15)	C21—C22	1.4819 (16)
C1—C6	1.4039 (16)	C22—C23	1.4818 (16)
C2—C3	1.367 (2)	C23—C28	1.3920 (16)
C2—H2	0.9300	C23—C24	1.3922 (17)
C3—C4	1.384 (2)	C24—C25	1.3818 (17)
C3—H3	0.9300	C24—H24	0.9300
C4—C5	1.3788 (18)	C25—C26	1.3727 (19)
C4—H4	0.9300	C25—H25	0.9300
C5—C6	1.3971 (17)	C26—C27	1.373 (2)
C5—H5	0.9300	C26—H26	0.9300

C7—C8	1.4916 (14)	C27—C28	1.3849 (17)
C8—C9	1.4879 (16)	C27—H27	0.9300
C9—C14	1.3891 (16)	C28—H28	0.9300
C9—C10	1.3892 (16)	C29—C30	1.5162 (16)
C10—C11	1.3840 (18)	C29—H29A	0.9700
C10—H10	0.9300	C29—H29B	0.9700
C11—C12	1.3732 (17)	C30—C31	1.5178 (17)
C11—H11	0.9300	C30—H30A	0.9700
C12—C13	1.3691 (19)	C30—H30B	0.9700
C12—H12	0.9300	C31—H31A	0.9700
C13—C14	1.3785 (18)	C31—H31B	0.9700

C7—N1—C6	122.56 (9)	C15—C16—H16	119.7
C7—N1—C29	116.94 (9)	C16—C17—C18	119.41 (12)
C6—N1—C29	120.40 (9)	C16—C17—H17	120.3
C8—N2—C1	120.56 (10)	C18—C17—H17	120.3
C21—N3—C20	122.01 (10)	C19—C18—C17	121.36 (12)
C21—N3—C31	116.59 (9)	C19—C18—H18	119.3
C20—N3—C31	121.26 (10)	C17—C18—H18	119.3
C22—N4—C15	119.21 (10)	C18—C19—C20	119.80 (12)
N2—C1—C2	118.84 (11)	C18—C19—H19	120.1
N2—C1—C6	121.69 (10)	C20—C19—H19	120.1
C2—C1—C6	119.39 (11)	N3—C20—C19	122.91 (11)
C3—C2—C1	120.99 (13)	N3—C20—C15	118.01 (10)
C3—C2—H2	119.5	C19—C20—C15	119.08 (11)
C1—C2—H2	119.5	O2—C21—N3	121.12 (11)
C2—C3—C4	119.07 (12)	O2—C21—C22	123.53 (11)
C2—C3—H3	120.5	N3—C21—C22	115.35 (10)
C4—C3—H3	120.5	N4—C22—C23	117.70 (10)
C5—C4—C3	121.87 (13)	N4—C22—C21	123.37 (11)
C5—C4—H4	119.1	C23—C22—C21	118.92 (10)
C3—C4—H4	119.1	C28—C23—C24	118.47 (11)
C4—C5—C6	119.28 (13)	C28—C23—C22	122.47 (11)
C4—C5—H5	120.4	C24—C23—C22	118.95 (10)
C6—C5—H5	120.4	C25—C24—C23	120.36 (12)
N1—C6—C5	123.06 (11)	C25—C24—H24	119.8
N1—C6—C1	117.58 (10)	C23—C24—H24	119.8
C5—C6—C1	119.37 (11)	C26—C25—C24	120.48 (13)
O1—C7—N1	120.38 (10)	C26—C25—H25	119.8
O1—C7—C8	124.38 (10)	C24—C25—H25	119.8
N1—C7—C8	115.24 (9)	C25—C26—C27	119.99 (12)
N2—C8—C9	117.15 (9)	C25—C26—H26	120.0
N2—C8—C7	122.00 (10)	C27—C26—H26	120.0
C9—C8—C7	120.84 (9)	C26—C27—C28	120.14 (12)
C14—C9—C10	117.63 (11)	C26—C27—H27	119.9
C14—C9—C8	117.09 (11)	C28—C27—H27	119.9
C10—C9—C8	125.28 (10)	C27—C28—C23	120.55 (12)
C11—C10—C9	120.18 (11)	C27—C28—H28	119.7

C11—C10—H10	119.9	C23—C28—H28	119.7
C9—C10—H10	119.9	N1—C29—C30	115.05 (9)
C12—C11—C10	121.47 (12)	N1—C29—H29A	108.5
C12—C11—H11	119.3	C30—C29—H29A	108.5
C10—C11—H11	119.3	N1—C29—H29B	108.5
C13—C12—C11	118.69 (12)	C30—C29—H29B	108.5
C13—C12—H12	120.7	H29A—C29—H29B	107.5
C11—C12—H12	120.7	C29—C30—C31	114.50 (10)
C12—C13—C14	120.56 (12)	C29—C30—H30A	108.6
C12—C13—H13	119.7	C31—C30—H30A	108.6
C14—C13—H13	119.7	C29—C30—H30B	108.6
C13—C14—C9	121.44 (12)	C31—C30—H30B	108.6
C13—C14—H14	119.3	H30A—C30—H30B	107.6
C9—C14—H14	119.3	N3—C31—C30	110.11 (9)
N4—C15—C16	118.36 (11)	N3—C31—H31A	109.6
N4—C15—C20	121.96 (10)	C30—C31—H31A	109.6
C16—C15—C20	119.66 (11)	N3—C31—H31B	109.6
C17—C16—C15	120.69 (12)	C30—C31—H31B	109.6
C17—C16—H16	119.7	H31A—C31—H31B	108.2

C8—N2—C1—C2	−179.66 (10)	C20—C15—C16—C17	−0.43 (19)
C8—N2—C1—C6	−2.80 (17)	C15—C16—C17—C18	0.0 (2)
N2—C1—C2—C3	176.47 (11)	C16—C17—C18—C19	0.4 (2)
C6—C1—C2—C3	−0.46 (18)	C17—C18—C19—C20	−0.5 (2)
C1—C2—C3—C4	1.1 (2)	C21—N3—C20—C19	−179.43 (11)
C2—C3—C4—C5	−0.4 (2)	C31—N3—C20—C19	5.04 (17)
C3—C4—C5—C6	−1.1 (2)	C21—N3—C20—C15	0.85 (17)
C7—N1—C6—C5	−177.01 (10)	C31—N3—C20—C15	−174.67 (10)
C29—N1—C6—C5	6.80 (16)	C18—C19—C20—N3	−179.64 (11)
C7—N1—C6—C1	3.35 (15)	C18—C19—C20—C15	0.07 (18)
C29—N1—C6—C1	−172.84 (10)	N4—C15—C20—N3	1.57 (17)
C4—C5—C6—N1	−177.90 (10)	C16—C15—C20—N3	−179.89 (11)
C4—C5—C6—C1	1.74 (18)	N4—C15—C20—C19	−178.15 (10)
N2—C1—C6—N1	1.83 (16)	C16—C15—C20—C19	0.38 (18)
C2—C1—C6—N1	178.67 (10)	C20—N3—C21—O2	177.67 (12)
N2—C1—C6—C5	−177.83 (10)	C31—N3—C21—O2	−6.61 (18)
C2—C1—C6—C5	−0.99 (17)	C20—N3—C21—C22	−2.68 (17)
C6—N1—C7—O1	173.86 (10)	C31—N3—C21—C22	173.04 (10)
C29—N1—C7—O1	−9.82 (16)	C15—N4—C22—C23	179.48 (10)
C6—N1—C7—C8	−6.86 (15)	C15—N4—C22—C21	−0.21 (18)
C29—N1—C7—C8	169.45 (9)	O2—C21—C22—N4	−177.92 (12)
C1—N2—C8—C9	177.83 (9)	N3—C21—C22—N4	2.43 (18)
C1—N2—C8—C7	−1.19 (16)	O2—C21—C22—C23	2.39 (19)
O1—C7—C8—N2	−174.88 (11)	N3—C21—C22—C23	−177.26 (10)
N1—C7—C8—N2	5.88 (16)	N4—C22—C23—C28	−139.78 (12)
O1—C7—C8—C9	6.14 (17)	C21—C22—C23—C28	39.93 (16)
N1—C7—C8—C9	−173.10 (9)	N4—C22—C23—C24	36.31 (16)
N2—C8—C9—C14	7.41 (16)	C21—C22—C23—C24	−143.98 (12)

C7—C8—C9—C14	−173.56 (11)	C28—C23—C24—C25	−0.83 (19)
N2—C8—C9—C10	−172.08 (11)	C22—C23—C24—C25	−177.07 (11)
C7—C8—C9—C10	6.96 (18)	C23—C24—C25—C26	0.7 (2)
C14—C9—C10—C11	−0.2 (2)	C24—C25—C26—C27	0.2 (2)
C8—C9—C10—C11	179.29 (12)	C25—C26—C27—C28	−1.0 (2)
C9—C10—C11—C12	−0.6 (2)	C26—C27—C28—C23	0.87 (19)
C10—C11—C12—C13	0.2 (2)	C24—C23—C28—C27	0.03 (17)
C11—C12—C13—C14	1.1 (2)	C22—C23—C28—C27	176.13 (11)
C12—C13—C14—C9	−2.0 (2)	C7—N1—C29—C30	99.27 (12)
C10—C9—C14—C13	1.5 (2)	C6—N1—C29—C30	−84.34 (12)
C8—C9—C14—C13	−178.05 (13)	N1—C29—C30—C31	−69.98 (13)
C22—N4—C15—C16	179.59 (11)	C21—N3—C31—C30	−92.34 (13)
C22—N4—C15—C20	−1.86 (17)	C20—N3—C31—C30	83.41 (13)
N4—C15—C16—C17	178.15 (12)	C29—C30—C31—N3	−175.74 (9)

Hydrogen-bond geometry (Å, º)

*Cg*6 is the centroid of the C23–C28 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.58	3.3871 (16)	146
C10—H10···O1	0.93	2.22	2.8531 (16)	124
C27—H27···O2^{ii}	0.93	2.59	3.4603 (18)	155
C30—H30*A*···*Cg*6^{iii}	0.97	2.75	3.6013 (14)	147

Symmetry codes: (i) *x*, *y*−1, *z*; (ii) −*x*, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*+1.