IUCrData

ISSN 2414-3146

1-(3-Phenyl-1*H*-pyrazol-5-yl)-1,2,3,4-tetrahydroquinoxaline-2,3-dione

Joseph Tene Ghomsi Nathan,[a] Noureddine Hamou Ahabchane,[a]* Nada Kheira Sebbar,[a] El Mokhtar Essassi[a] and Joel T. Mague[b]

[a]Laboratoire de Chimie Organique Hétérocyclique URAC 21, Pôle de Compétence Pharmacochimie, Av. Ibn Battouta, BP 1014, Faculté des Sciences, Université Mohammed V, Rabat, Morocco, and [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA. *Correspondence e-mail: ahabchanenoureddine@gmail.com

Received 11 January 2017
Accepted 15 January 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

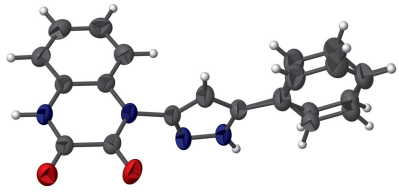
Keywords: crystal structure; quinoxaline; hydrogen bond; π–π stacking.

CCDC reference: 1527554

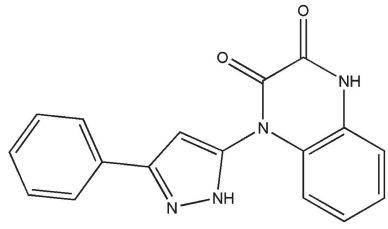
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{12}N_4O_2$, the mean plane of the pyrazole ring is nearly perpendicular to that of the tetrahydroquinoxalinedione moiety [dihedral angle = 86.92 (7)°]. The phenyl ring is disordered over two orientations in a 0.556 (4):0.444 (4) ratio. In the crystal, molecules are connected by bifurcated N—H···(N,O) and N—H···(O,O) hydrogen bonds, generating (100) sheets. Aromatic π–π stacking [shortest centroid–centroid separation = 3.5307 (8) Å] links the sheets into a three-dimensional network. A short N···O contact [2.8198 (19) Å] is present.

3D view



Chemical scheme



Structure description

Quinoxaline derivatives have various biological activities including antibacterial (Kleim *et al.*, 1995), antimicrobial (Vieira *et al.*, 2014) and anticancer (Noolvi *et al.*, 2011) properties. As part of our studies in this area, we now describe the synthesis and structure of the title compound (Fig. 1).

The phenyl ring attached to C11 is rotationally disordered over two orientations in a 0.556 (4):0.444 (4) ratio. The dihedral angle between the orientations is 26.6 (4)°. The dihedral angle between the mean planes of the C1–C6 and C1,C6,N1,C7,C8,N2 rings is 2.25 (9)° while that between the latter and the mean plane of the C9,C10,C11,N4,N3 ring is 86.34 (4)°.

In the crystal, the molecules are linked by N—H···(N,O) and N—H···(O,O) hydrogen bonds and C—H···O interactions (Table 1) and slipped π–π-stacking interactions (Figs. 2 and 3). The last involve the C9,C10,C11,N4,N3 ring and the C1,C6,N1,C7,C8,N2 ring at x, $\frac{3}{2}-y$, $\frac{1}{2}+z$ [centroid–centroid distance = 3.887 (1) Å, dihedral angle = 14.85 (8)°] as well

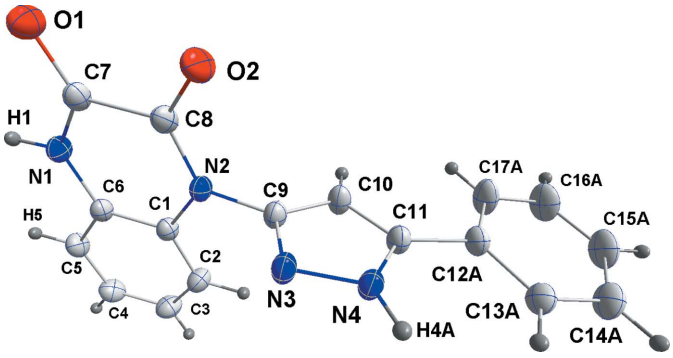


Figure 1
The title molecule, with 25% probability ellipsoids. Only the major orientation of the disordered phenyl ring is shown

as the latter ring and its counterpart at $1 - x$, $1 - y$, $-z$ [centroid–centroid distance = 3.531 (1) Å].

Synthesis and crystallization

0.01 mol of 3-*N*-(2-aminophenylamino)-5-phenylpyrazole was dissolved in 80 ml of ethyl oxalate. The reaction mixture was refluxed for 1 h. After cooling, the obtained precipitate was recrystallized from ethanol solution to afford crystals of the title compound.

Refinement

Crystal and refinement data are presented in Table 2. The pendant phenyl ring is rotationally disordered over two sets of sites in a 0.556 (4):0.444 (4) ratio. The two components of the disorder were refined as rigid hexagons with the H atoms included as riding contributions in calculated positions.

Acknowledgements

JTM thanks Tulane University for support of the Tulane Crystallography Laboratory.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.92 (2)	2.10 (2)	2.9448 (17)	152.1 (18)
N1—H1···N3^{ii}	0.92 (2)	2.45 (2)	3.0160 (18)	119.8 (17)
N4—H4*A*···O1^{iii}	0.917 (19)	2.276 (19)	3.1851 (18)	171.0 (16)
N4—H4*A*···O2^{iii}	0.917 (19)	2.399 (18)	2.9409 (18)	117.8 (14)
C5—H5···O2^{i}	0.982 (19)	2.552 (18)	3.311 (2)	134.1 (13)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{12}N_4O_2$
M_r	304.31
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	12.6320 (6), 9.3954 (4), 12.3733 (6)
β (°)	102.708 (1)
V (Å^3)	1432.52 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.30 × 0.29 × 0.11
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.86, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	26569, 3702, 2678
R_{int}	0.038
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.052, 0.153, 1.05
No. of reflections	3702
No. of parameters	231
No. of restraints	73
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.24

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

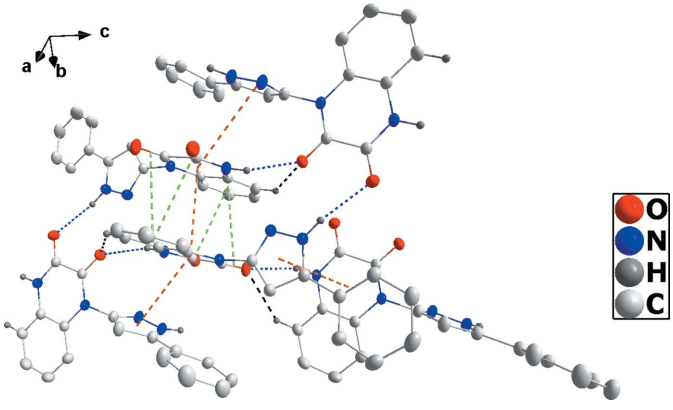


Figure 2
Detail of the hydrogen bonding (blue and black dotted lines), the π–π stacking interactions (orange dotted lines) and the π–π interactions of the carbonyl groups with the quinoxaline system (green dotted lines).

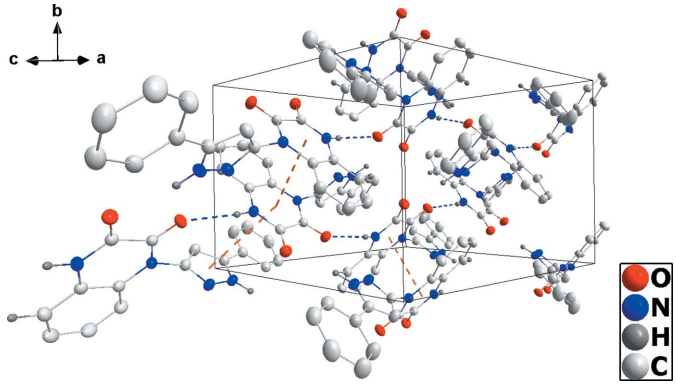


Figure 3
Packing projected onto (101) (key to intermolecular interactions as in Fig. 2).

References

Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Bruker (2016). *APEX3*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Kleim, J. P., Bender, R., Kirsch, R., Meichsner, C., Paessens, A., Rösner, M., Rübsamen-Waigmann, H., Kaiser, R., Wichers, M., Schneweis, K. E., *et al.* (1995). *Antimicrob. Agents Chemother.* **39**, 2253–2257.

Noolvi, M. N., Patel, H. M., Bhardwaj, V. & Chauhan, A. (2011). *Eur. J. Med. Chem.* **46**, 2327–2346.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Vieira, M., Pinheiro, C., Fernandes, R., Noronha, J. P. & Prudêncio, C. (2014). *Microbiol. Res.* **169**, 287–293.

full crystallographic data

IUCrData (2017). **2**, x170073 [https://doi.org/10.1107/S2414314617000736]

1-(3-Phenyl-1*H*-pyrazol-5-yl)-1,2,3,4-tetrahydroquinoxaline-2,3-dione

Joseph Tene Ghomsi Nathan, Noureddine Hamou Ahabchane, Nada Kheira Sebbar, El Mokhtar Essassi and Joel T. Mague

1-(3-Phenyl-1*H*-pyrazol-5-yl)-1,2,3,4-tetrahydroquinoxaline-2,3-dione

Crystal data

$C_{17}H_{12}N_4O_2$
M_r = 304.31
Monoclinic, $P2_1/c$
a = 12.6320 (6) Å
b = 9.3954 (4) Å
c = 12.3733 (6) Å
β = 102.708 (1)°
V = 1432.52 (11) Å^3
Z = 4
$F(000)$ = 632
D_x = 1.411 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6591 reflections
θ = 2.7–24.9°
μ = 0.10 mm^{-1}
T = 296 K
Plate, colourless
0.30 × 0.29 × 0.11 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*; Bruker, 2016)
T_{min} = 0.86, T_{max} = 0.99
26569 measured reflections
3702 independent reflections
2678 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 28.7°, θ_{min} = 1.7°
h = −17→17
k = −12→12
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.153
S = 1.05
3702 reflections
231 parameters
73 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.2844P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, colllected 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 25 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. The pendant phenyl ring is rotationally disordered over two sites in a 56:44 ratio. The two components of the disorder were refined as rigid hexagons with the H-atoms included as riding contributions in calculated positions.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.43419 (10)	0.84212 (13)	−0.06590 (9)	0.0540 (3)	
O2	0.52330 (11)	0.80657 (13)	0.15125 (9)	0.0590 (3)	
N1	0.54348 (10)	0.66824 (14)	−0.10784 (10)	0.0437 (3)	
H1	0.5128 (17)	0.676 (2)	−0.1819 (18)	0.074 (6)*	
N2	0.63137 (10)	0.62897 (13)	0.11388 (9)	0.0411 (3)	
N3	0.60756 (11)	0.51255 (14)	0.27572 (10)	0.0470 (3)	
N4	0.66080 (11)	0.50551 (14)	0.38248 (10)	0.0447 (3)	
H4A	0.6303 (15)	0.4513 (19)	0.4292 (16)	0.054 (5)*	
C1	0.66635 (12)	0.54086 (16)	0.03612 (12)	0.0404 (3)	
C2	0.74269 (14)	0.43396 (19)	0.06845 (15)	0.0535 (4)	
H2	0.7758 (15)	0.420 (2)	0.1484 (16)	0.062 (5)*	
C3	0.77174 (16)	0.3479 (2)	−0.01032 (17)	0.0611 (5)	
H3	0.8262 (19)	0.274 (2)	0.0132 (18)	0.082 (6)*	
C4	0.72460 (15)	0.3660 (2)	−0.12091 (16)	0.0581 (4)	
H4	0.7446 (17)	0.304 (2)	−0.1770 (18)	0.074 (6)*	
C5	0.64992 (14)	0.47250 (18)	−0.15432 (14)	0.0499 (4)	
H5	0.6160 (15)	0.4852 (19)	−0.2331 (16)	0.057 (5)*	
C6	0.62063 (11)	0.56096 (15)	−0.07603 (12)	0.0400 (3)	
C7	0.50519 (11)	0.75326 (16)	−0.03792 (11)	0.0401 (3)	
C8	0.55497 (12)	0.73284 (15)	0.08487 (11)	0.0406 (3)	
C9	0.66757 (12)	0.59926 (17)	0.23003 (12)	0.0431 (3)	
C10	0.75853 (14)	0.6487 (2)	0.30459 (13)	0.0517 (4)	
H10	0.8141 (16)	0.712 (2)	0.2907 (16)	0.065 (5)*	
C11	0.75230 (12)	0.58549 (17)	0.40387 (12)	0.0449 (3)	
C12A	0.8249 (3)	0.6043 (4)	0.5144 (2)	0.0476 (5)	0.556 (4)
C13A	0.8158 (3)	0.5171 (5)	0.6026 (3)	0.0483 (9)	0.556 (4)
H13A	0.7596	0.4514	0.5945	0.058*	0.556 (4)
C14A	0.8907 (4)	0.5282 (5)	0.7030 (3)	0.0612 (9)	0.556 (4)
H14A	0.8846	0.4699	0.7620	0.073*	0.556 (4)
C15A	0.9748 (3)	0.6265 (5)	0.7151 (2)	0.0748 (10)	0.556 (4)
H15A	1.0249	0.6339	0.7823	0.090*	0.556 (4)
C16A	0.9839 (3)	0.7136 (5)	0.6269 (3)	0.0811 (13)	0.556 (4)
H16A	1.0401	0.7794	0.6350	0.097*	0.556 (4)
C17A	0.9090 (3)	0.7026 (5)	0.5265 (2)	0.0713 (12)	0.556 (4)
H17A	0.9151	0.7609	0.4674	0.086*	0.556 (4)

C12B	0.8291 (4)	0.5833 (6)	0.5131 (3)	0.0476 (5)	0.444 (4)
C13B	0.7960 (3)	0.5408 (7)	0.6081 (4)	0.0483 (9)	0.444 (4)
H13B	0.7251	0.5104	0.6034	0.058*	0.444 (4)
C14B	0.8690 (4)	0.5437 (8)	0.7101 (3)	0.0612 (9)	0.444 (4)
H14B	0.8469	0.5153	0.7737	0.073*	0.444 (4)
C15B	0.9750 (4)	0.5892 (7)	0.7172 (3)	0.0748 (10)	0.444 (4)
H15B	1.0238	0.5911	0.7854	0.090*	0.444 (4)
C16B	1.0081 (3)	0.6316 (7)	0.6221 (4)	0.0811 (13)	0.444 (4)
H16B	1.0790	0.6620	0.6268	0.097*	0.444 (4)
C17B	0.9351 (4)	0.6287 (6)	0.5201 (3)	0.0713 (12)	0.444 (4)
H17B	0.9573	0.6571	0.4565	0.086*	0.444 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0589 (7)	0.0560 (7)	0.0458 (6)	0.0137 (5)	0.0084 (5)	0.0050 (5)
O2	0.0826 (9)	0.0582 (7)	0.0371 (6)	0.0166 (6)	0.0151 (6)	−0.0036 (5)
N1	0.0493 (7)	0.0513 (7)	0.0293 (6)	0.0045 (6)	0.0060 (5)	0.0000 (5)
N2	0.0471 (6)	0.0460 (7)	0.0296 (6)	0.0012 (5)	0.0070 (5)	0.0020 (5)
N3	0.0521 (7)	0.0532 (8)	0.0319 (6)	−0.0088 (6)	0.0013 (5)	0.0048 (5)
N4	0.0490 (7)	0.0501 (7)	0.0324 (6)	−0.0071 (6)	0.0030 (5)	0.0051 (5)
C1	0.0420 (7)	0.0428 (7)	0.0371 (7)	−0.0016 (6)	0.0104 (6)	0.0003 (6)
C2	0.0545 (9)	0.0577 (10)	0.0477 (9)	0.0102 (7)	0.0100 (7)	0.0074 (7)
C3	0.0614 (10)	0.0575 (10)	0.0668 (12)	0.0164 (9)	0.0195 (9)	0.0044 (9)
C4	0.0638 (10)	0.0565 (10)	0.0596 (11)	0.0047 (8)	0.0260 (9)	−0.0078 (8)
C5	0.0525 (9)	0.0563 (9)	0.0424 (8)	−0.0007 (7)	0.0138 (7)	−0.0060 (7)
C6	0.0403 (7)	0.0429 (8)	0.0371 (7)	−0.0031 (6)	0.0091 (6)	0.0006 (6)
C7	0.0429 (7)	0.0416 (7)	0.0353 (7)	−0.0019 (6)	0.0078 (6)	0.0016 (6)
C8	0.0488 (8)	0.0397 (7)	0.0343 (7)	−0.0007 (6)	0.0112 (6)	0.0012 (6)
C9	0.0467 (8)	0.0488 (8)	0.0328 (7)	−0.0010 (6)	0.0062 (6)	0.0024 (6)
C10	0.0512 (9)	0.0635 (10)	0.0390 (8)	−0.0133 (8)	0.0070 (7)	0.0023 (7)
C11	0.0443 (8)	0.0525 (8)	0.0360 (7)	−0.0044 (6)	0.0048 (6)	−0.0006 (6)
C12A	0.0449 (8)	0.0575 (14)	0.0383 (8)	−0.0024 (8)	0.0046 (6)	−0.0041 (8)
C13A	0.0435 (14)	0.0562 (18)	0.0426 (9)	0.0046 (14)	0.0035 (10)	0.0017 (10)
C14A	0.054 (2)	0.0836 (17)	0.0428 (10)	0.0090 (15)	0.0028 (10)	0.0052 (10)
C15A	0.0544 (11)	0.118 (3)	0.0456 (10)	−0.0025 (14)	−0.0038 (8)	−0.0115 (13)
C16A	0.0564 (18)	0.118 (4)	0.0652 (16)	−0.031 (2)	0.0049 (13)	−0.018 (2)
C17A	0.064 (2)	0.098 (3)	0.0485 (12)	−0.029 (2)	0.0059 (12)	−0.0059 (19)
C12B	0.0449 (8)	0.0575 (14)	0.0383 (8)	−0.0024 (8)	0.0046 (6)	−0.0041 (8)
C13B	0.0435 (14)	0.0562 (18)	0.0426 (9)	0.0046 (14)	0.0035 (10)	0.0017 (10)
C14B	0.054 (2)	0.0836 (17)	0.0428 (10)	0.0090 (15)	0.0028 (10)	0.0052 (10)
C15B	0.0544 (11)	0.118 (3)	0.0456 (10)	−0.0025 (14)	−0.0038 (8)	−0.0115 (13)
C16B	0.0564 (18)	0.118 (4)	0.0652 (16)	−0.031 (2)	0.0049 (13)	−0.018 (2)
C17B	0.064 (2)	0.098 (3)	0.0485 (12)	−0.029 (2)	0.0059 (12)	−0.0059 (19)

Geometric parameters (Å, º)

O1—C7	1.2184 (18)	C10—H10	0.97 (2)
O2—C8	1.2079 (18)	C11—C12A	1.480 (2)
N1—C7	1.3434 (19)	C11—C12B	1.480 (2)
N1—C6	1.3971 (19)	C12A—C13A	1.3900
N1—H1	0.92 (2)	C12A—C17A	1.3900
N2—C8	1.3639 (19)	C13A—C14A	1.3900
N2—C1	1.4119 (19)	C13A—H13A	0.9300
N2—C9	1.4365 (18)	C14A—C15A	1.3900
N3—C9	1.3215 (19)	C14A—H14A	0.9300
N3—N4	1.3457 (17)	C15A—C16A	1.3900
N4—C11	1.355 (2)	C15A—H15A	0.9300
N4—H4A	0.917 (19)	C16A—C17A	1.3900
C1—C2	1.389 (2)	C16A—H16A	0.9300
C1—C6	1.394 (2)	C17A—H17A	0.9300
C2—C3	1.377 (3)	C12B—C13B	1.3900
C2—H2	0.995 (19)	C12B—C17B	1.3900
C3—C4	1.378 (3)	C13B—C14B	1.3900
C3—H3	0.97 (2)	C13B—H13B	0.9300
C4—C5	1.375 (2)	C14B—C15B	1.3900
C4—H4	0.98 (2)	C14B—H14B	0.9300
C5—C6	1.387 (2)	C15B—C16B	1.3900
C5—H5	0.982 (19)	C15B—H15B	0.9300
C7—C8	1.523 (2)	C16B—C17B	1.3900
C9—C10	1.386 (2)	C16B—H16B	0.9300
C10—C11	1.382 (2)	C17B—H17B	0.9300
C7—N1—C6	125.12 (12)	N4—C11—C12A	125.1 (2)
C7—N1—H1	117.6 (13)	C10—C11—C12A	128.7 (2)
C6—N1—H1	117.1 (13)	N4—C11—C12B	122.0 (2)
C8—N2—C1	123.34 (12)	C10—C11—C12B	131.6 (2)
C8—N2—C9	116.96 (12)	C13A—C12A—C17A	120.0
C1—N2—C9	119.30 (12)	C13A—C12A—C11	120.9 (3)
C9—N3—N4	103.91 (12)	C17A—C12A—C11	118.8 (3)
N3—N4—C11	112.65 (12)	C14A—C13A—C12A	120.0
N3—N4—H4A	117.3 (12)	C14A—C13A—H13A	120.0
C11—N4—H4A	130.0 (12)	C12A—C13A—H13A	120.0
C2—C1—C6	119.61 (14)	C13A—C14A—C15A	120.0
C2—C1—N2	121.96 (13)	C13A—C14A—H14A	120.0
C6—C1—N2	118.41 (13)	C15A—C14A—H14A	120.0
C3—C2—C1	119.81 (16)	C16A—C15A—C14A	120.0
C3—C2—H2	120.5 (11)	C16A—C15A—H15A	120.0
C1—C2—H2	119.7 (11)	C14A—C15A—H15A	120.0
C2—C3—C4	120.40 (17)	C17A—C16A—C15A	120.0
C2—C3—H3	119.1 (13)	C17A—C16A—H16A	120.0
C4—C3—H3	120.5 (13)	C15A—C16A—H16A	120.0
C5—C4—C3	120.46 (16)	C16A—C17A—C12A	120.0

C5—C4—H4	119.1 (13)	C16A—C17A—H17A	120.0
C3—C4—H4	120.5 (13)	C12A—C17A—H17A	120.0
C4—C5—C6	119.79 (16)	C13B—C12B—C17B	120.0
C4—C5—H5	120.5 (11)	C13B—C12B—C11	121.2 (4)
C6—C5—H5	119.7 (11)	C17B—C12B—C11	118.8 (4)
C5—C6—C1	119.90 (14)	C14B—C13B—C12B	120.0
C5—C6—N1	120.81 (14)	C14B—C13B—H13B	120.0
C1—C6—N1	119.27 (13)	C12B—C13B—H13B	120.0
O1—C7—N1	124.84 (13)	C13B—C14B—C15B	120.0
O1—C7—C8	119.10 (13)	C13B—C14B—H14B	120.0
N1—C7—C8	116.06 (13)	C15B—C14B—H14B	120.0
O2—C8—N2	123.54 (14)	C16B—C15B—C14B	120.0
O2—C8—C7	118.70 (13)	C16B—C15B—H15B	120.0
N2—C8—C7	117.74 (12)	C14B—C15B—H15B	120.0
N3—C9—C10	113.04 (13)	C15B—C16B—C17B	120.0
N3—C9—N2	117.48 (13)	C15B—C16B—H16B	120.0
C10—C9—N2	129.48 (14)	C17B—C16B—H16B	120.0
C11—C10—C9	104.29 (14)	C16B—C17B—C12B	120.0
C11—C10—H10	127.6 (12)	C16B—C17B—H17B	120.0
C9—C10—H10	128.1 (12)	C12B—C17B—H17B	120.0
N4—C11—C10	106.11 (13)		

C9—N3—N4—C11	−0.11 (18)	C1—N2—C9—C10	90.1 (2)
C8—N2—C1—C2	−178.61 (14)	N3—C9—C10—C11	0.1 (2)
C9—N2—C1—C2	−6.0 (2)	N2—C9—C10—C11	−179.94 (15)
C8—N2—C1—C6	0.0 (2)	N3—N4—C11—C10	0.16 (19)
C9—N2—C1—C6	172.63 (13)	N3—N4—C11—C12A	177.1 (2)
C6—C1—C2—C3	−0.7 (2)	N3—N4—C11—C12B	−174.1 (3)
N2—C1—C2—C3	177.98 (16)	C9—C10—C11—N4	−0.13 (18)
C1—C2—C3—C4	−0.8 (3)	C9—C10—C11—C12A	−176.9 (3)
C2—C3—C4—C5	1.6 (3)	C9—C10—C11—C12B	173.4 (3)
C3—C4—C5—C6	−0.9 (3)	N4—C11—C12A—C13A	13.0 (4)
C4—C5—C6—C1	−0.5 (2)	C10—C11—C12A—C13A	−170.8 (2)
C4—C5—C6—N1	−178.86 (15)	N4—C11—C12A—C17A	−172.5 (2)
C2—C1—C6—C5	1.3 (2)	C10—C11—C12A—C17A	3.7 (4)
N2—C1—C6—C5	−177.39 (13)	C17A—C12A—C13A—C14A	0.0
C2—C1—C6—N1	179.67 (14)	C11—C12A—C13A—C14A	174.4 (3)
N2—C1—C6—N1	1.0 (2)	C12A—C13A—C14A—C15A	0.0
C7—N1—C6—C5	175.77 (14)	C13A—C14A—C15A—C16A	0.0
C7—N1—C6—C1	−2.6 (2)	C14A—C15A—C16A—C17A	0.0
C6—N1—C7—O1	−176.34 (14)	C15A—C16A—C17A—C12A	0.0
C6—N1—C7—C8	2.8 (2)	C13A—C12A—C17A—C16A	0.0
C1—N2—C8—O2	178.50 (14)	C11—C12A—C17A—C16A	−174.5 (3)
C9—N2—C8—O2	5.8 (2)	N4—C11—C12B—C13B	−22.4 (4)
C1—N2—C8—C7	0.3 (2)	C10—C11—C12B—C13B	164.9 (3)
C9—N2—C8—C7	−172.48 (12)	N4—C11—C12B—C17B	159.5 (3)
O1—C7—C8—O2	−0.7 (2)	C10—C11—C12B—C17B	−13.2 (5)
N1—C7—C8—O2	−179.93 (14)	C17B—C12B—C13B—C14B	0.0

O1—C7—C8—N2	177.60 (13)	C11—C12B—C13B—C14B	−178.1 (4)
N1—C7—C8—N2	−1.6 (2)	C12B—C13B—C14B—C15B	0.0
N4—N3—C9—C10	0.02 (19)	C13B—C14B—C15B—C16B	0.0
N4—N3—C9—N2	−179.97 (13)	C14B—C15B—C16B—C17B	0.0
C8—N2—C9—N3	83.11 (18)	C15B—C16B—C17B—C12B	0.0
C1—N2—C9—N3	−89.94 (18)	C13B—C12B—C17B—C16B	0.0
C8—N2—C9—C10	−96.9 (2)	C11—C12B—C17B—C16B	178.1 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.92 (2)	2.10 (2)	2.9448 (17)	152.1 (18)
N1—H1···N3^{ii}	0.92 (2)	2.45 (2)	3.0160 (18)	119.8 (17)
N4—H4*A*···O1^{iii}	0.917 (19)	2.276 (19)	3.1851 (18)	171.0 (16)
N4—H4*A*···O2^{iii}	0.917 (19)	2.399 (18)	2.9409 (18)	117.8 (14)
C5—H5···O2^{i}	0.982 (19)	2.552 (18)	3.311 (2)	134.1 (13)

Symmetry codes: (i) x, $-y+3/2$, $z-1/2$; (ii) $-x+1$, $-y+1$, $-z$; (iii) $-x+1$, $y-1/2$, $-z+1/2$.