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2-[(*E*)-2-(3,4-Dichlorobenzylidene)-hydrazin-1-yl]quinoxalineThais C. M. Nogueira,^a Alexandra C. Pinheiro,^a Marcus V. N. de Souza,^a James L. Wardell^{b‡} & Edward R. T. Tiekink^{c*}^aFundação Oswaldo Cruz, Instituto de Tecnologia em Fármacos—Farmanguinhos, R. Sizenando Nabuco, 100, Manguinhos 21041-250, Rio de Janeiro, RJ, Brazil, ^bChemistry Department, University of Aberdeen, Old Aberdeen AB24 3UE, Scotland, and ^cDepartment of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
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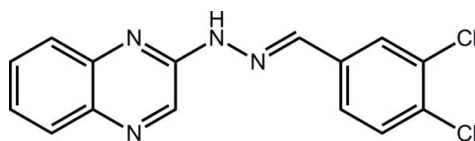
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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.060; wR factor = 0.219; data-to-parameter ratio = 12.4.

The 21 non-H atoms of the title compound, $\text{C}_{15}\text{H}_{10}\text{Cl}_2\text{N}_4$, are almost planar (r.m.s. deviation = 0.032 Å); the conformation about the $\text{N}=\text{C}$ bond [1.277 (6) Å] is *E*. In the crystal, zigzag supramolecular chains along the *c* axis (glide symmetry) are formed via $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds. These associate along the *b* axis by $\pi-\pi$ interactions between the fused and terminal benzene rings [intercentroid distance = 3.602 (3) Å] so that layers form in the *bc* plane.

Related literature

For the use of quinoxaline compounds as dyestuffs and biological agents, see: Mielcke *et al.* (2012); Mamedov & Zhukova (2012); Rodrigues *et al.* (2014). For a related hydrazone structure, see: de Souza *et al.* (2013).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{10}\text{Cl}_2\text{N}_4$ $V = 1380.12$ (16) Å³
 $M_r = 317.17$ $Z = 4$
Monoclinic, $P2_1/c$ $\text{Mo K}\alpha$ radiation
 $a = 16.0284$ (11) Å $\mu = 0.47$ mm⁻¹
 $b = 6.9756$ (4) Å $T = 120$ K
 $c = 12.4127$ (9) Å $0.20 \times 0.13 \times 0.03$ mm
 $\beta = 96.043$ (7)°

Data collection

Rigaku RAXIS conversion 7037 measured reflections
diffractometer 2385 independent reflections
Absorption correction: multi-scan 1670 reflections with $I > 2\sigma(I)$
(*CrystalClear-SM Expert*; Rigaku, $R_{\text{int}} = 0.043$
2012)
 $T_{\text{min}} = 0.654$, $T_{\text{max}} = 1.000$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.060$ H atoms treated by a mixture of
 $wR(F^2) = 0.219$ independent and constrained
 $S = 1.20$ refinement
2385 reflections $\Delta\rho_{\text{max}} = 0.76$ e Å⁻³
193 parameters $\Delta\rho_{\text{min}} = -0.65$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2N}\cdots\text{N4}^i$	0.92 (5)	2.10 (5)	3.013 (5)	171 (4)

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2012); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The use of the EPSRC X-ray crystallographic service (Coles & Gale, 2012) at the University of Southampton, England, and the valuable assistance of the staff there is gratefully acknowledged. JLW acknowledges support from CAPES (Brazil). Structural studies are supported by the Ministry of Higher Education (Malaysia) and the University of Malaya through the High-Impact Research scheme (UM.C/HIR/MOHE/SC/3).

Supporting information for this paper is available from the IUCr electronic archives (Reference: HG5372).

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supporting information

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2-[(*E*)-2-(3,4-Dichlorobenzylidene)hydrazin-1-yl]quinoxaline

Thais C. M. Nogueira, Alexandra C. Pinheiro, Marcus V. N. de Souza, James L. Wardell and Edward R. T. Tiekink

S1. Experimental**S1.1. Synthesis and crystallization**

A solution of 2-hydrazinylquinoxaline (1 mmol) and 3,4-dichlorobenzaldehyde (1.05 mmol) in EtOH (10 ml) was stirred at room temperature for 24 h and rotary evaporated. The residue was washed with ice-cold EtOH (3 ×) and recrystallized from its MeOCH₂CH₂OH solution. Yield: 95%. M.Pt: 528–529 K. ¹H NMR (400 MHz, DMSO-*d*₆): δ 11.86 (1H, s, NH); 9.15 (1H, s, H3); 8.11 (1H, s, CH); 8.04 (1H, d, *J* = 2.0; H2'); 7.93 (1H, d, *J* = 8.2, H5); 7.77 (1H, dd, *J* = 8.4 and *J* = 2.0, H6'); 7.68 (3H, m, H5', H7 and H8); 7.51 (1H, dt, *J* = 8.2 and *J* = 2.0, H6) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆): δ 150.0; 140.7; 139.0; 138.0; 136.4; 135.5; 131.6; 131.1; 130.8; 130.3; 128.7; 127.8; 126.3; 126.2; 125.3 ppm. MS/ESI (M—H): 314.9. IR (cm⁻¹, KBr): 3437 ν(N—H); 1585 ν(C=N).

S1.2. Refinement

Intensity data was collected at the National Crystallographic Service, England (Coles & Gale, 2012). The C-bound H atoms were geometrically placed (C—H = 0.95 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The N-bound H atom was located from a difference map and refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

S2. Results and discussion

Quinoxaline compounds have found uses, mainly as biologically active compounds, but also as dyestuffs (Mielcke *et al.*, 2012; Mamedov & Zhukova, 2012). The biological activities of quinoxaline compounds include anti-bacterial, anti-tubercular, anti-microbial, anti-fungal, anti-malarial, anti-inflammatory, anti-leishmanial, anti-tumour, herbicidal and insecticidal (Mielcke *et al.*, 2012; Mamedov & Zhukova, 2012). In a recent study, an evaluation of the anti-cancer activities of a series of (*E*)-2-(2-benzylidene)hydrazinylquinoxaline derivatives was reported (Rodrigues *et al.*, 2014). As part of our continuing studies on the structures of biologically active hydrazones (de Souza *et al.*, 2013), we now report the crystal structure of the title compound, (*E*)-2-(2-(3,4-dichlorobenzylidene)hydrazinyl)-quinoxaline, (I).

In (I), Fig. 1, the 21 non-hydrogen atoms comprising the molecule are co-planar with the r.m.s. deviation = 0.032 Å; the maximum deviations from the least-squares plane are 0.066 (5) Å for atom C5 and -0.057 (1) Å for atom Cl2. The conformation about the N1=C7 bond [1.277 (6) Å] is *E*.

In the crystal packing, zigzag supramolecular chains (glide symmetry) are formed *via* N—H⋯N hydrogen bonds, Fig. 2 and Table 1. These chains along the *c* direction are consolidated into supramolecular layers in the *bc* plane by π—π interactions between the (C1—C6) and (C9—C14)^{*i*} rings [inter-centroid distance = 3.602 (3) Å, inter-planar angle = 2.4 (2)° for symmetry operation *i*: 1-*x*, 1-*y*, -*z*] formed along the *b* direction, Fig. 3.

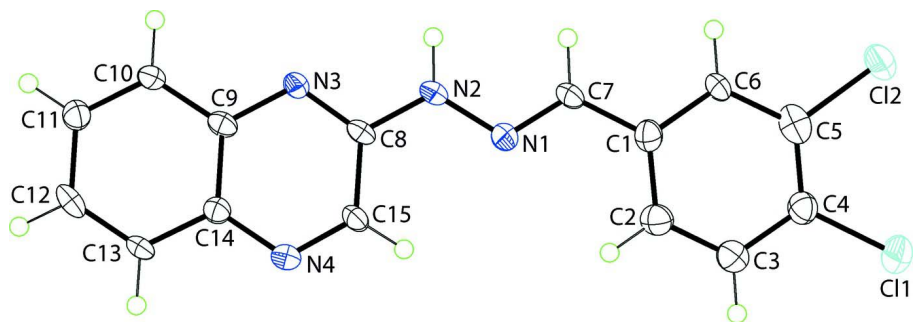


Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

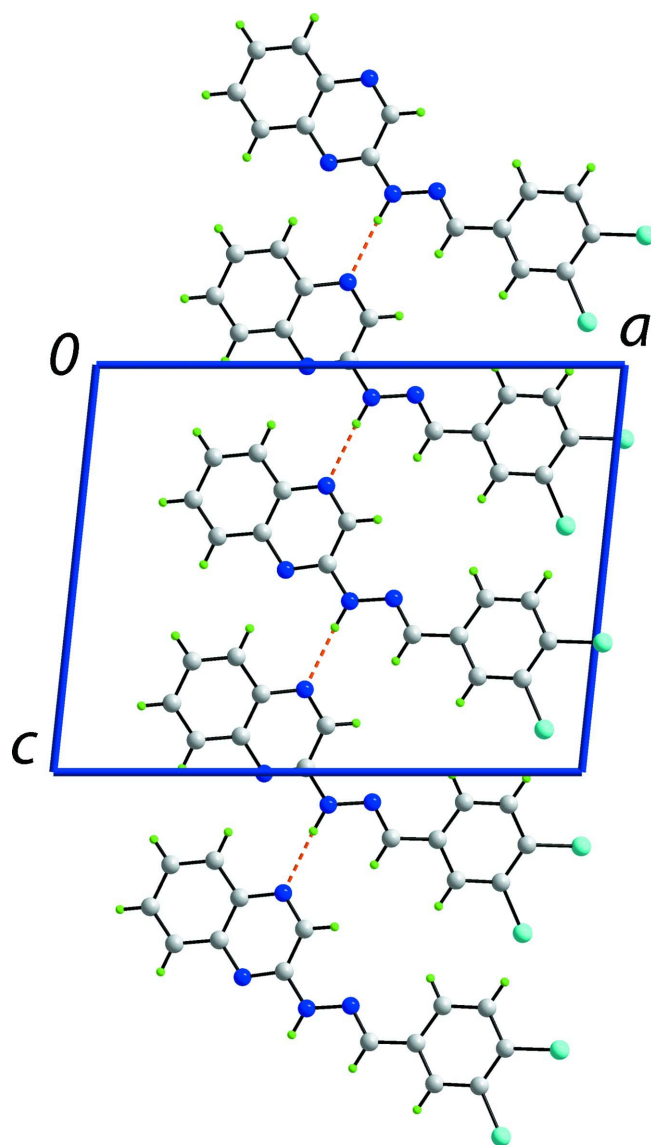


Figure 2

A view of the supramolecular zigzag chain along the *c* axis in (I). The N—H···H hydrogen bonds are shown as orange dashed lines.

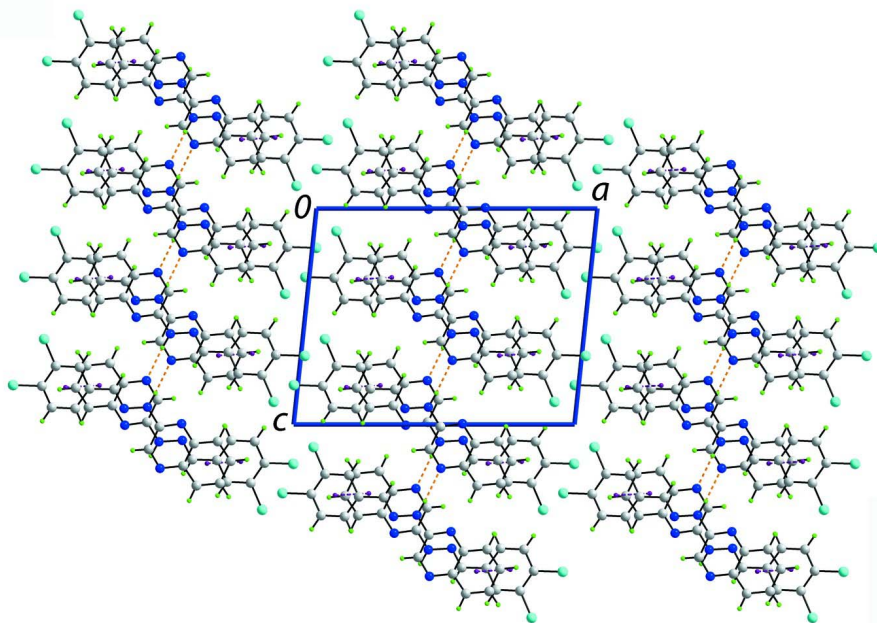


Figure 3

A view in projection down the b axis of the unit-cell contents for (I). The N—H \cdots H and π — π interactions are shown as orange and purple dashed lines, respectively.

2-[(*E*)-2-(3,4-Dichlorobenzylidene)hydrazin-1-yl]quinoxaline

Crystal data

$C_{15}H_{10}Cl_2N_4$

$M_r = 317.17$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 16.0284$ (11) Å

$b = 6.9756$ (4) Å

$c = 12.4127$ (9) Å

$\beta = 96.043$ (7)°

$V = 1380.12$ (16) Å³

$Z = 4$

$F(000) = 648$

$D_x = 1.526$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5450 reflections

$\theta = 3.2$ – 29.1 °

$\mu = 0.47$ mm⁻¹

$T = 120$ K

Prism, yellow

$0.20 \times 0.13 \times 0.03$ mm

Data collection

Rigaku RAXIS conversion
diffractometer

Radiation source: Sealed Tube

Graphite monochromator

Detector resolution: 10.0000 pixels mm⁻¹

profile data from ω -scans

Absorption correction: multi-scan

(*CrystalClear-SM Expert*; Rigaku, 2012)

$T_{\min} = 0.654$, $T_{\max} = 1.000$

7037 measured reflections

2385 independent reflections

1670 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.043$

$\theta_{\max} = 25.0$ °, $\theta_{\min} = 3.2$ °

$h = -19$ → 17

$k = -8$ → 7

$l = -14$ → 14

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.219$
 $S = 1.20$
 2385 reflections
 193 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.1005P)^2 + 2.9896P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.76 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s 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 $F^2 > 2\sigma(F^2)$ 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.92037 (8)	0.4044 (2)	0.39515 (10)	0.0391 (4)
Cl2	1.01377 (8)	0.3943 (2)	0.18221 (11)	0.0391 (4)
N1	0.6096 (2)	0.6684 (6)	0.0737 (3)	0.0234 (9)
N2	0.5269 (2)	0.7147 (6)	0.0801 (3)	0.0249 (9)
H2N	0.504 (3)	0.718 (7)	0.145 (4)	0.030*
N3	0.3991 (2)	0.8161 (5)	0.0034 (3)	0.0206 (9)
N4	0.4593 (2)	0.8165 (6)	-0.2031 (3)	0.0247 (9)
C1	0.7406 (3)	0.5710 (7)	0.1655 (4)	0.0256 (11)
C2	0.7839 (3)	0.5206 (7)	0.2653 (4)	0.0244 (11)
H2	0.7560	0.5212	0.3291	0.029*
C3	0.8691 (3)	0.4690 (7)	0.2706 (4)	0.0307 (12)
C4	0.9107 (3)	0.4672 (7)	0.1782 (4)	0.0281 (11)
C5	0.8680 (3)	0.5232 (7)	0.0788 (4)	0.0301 (12)
H5	0.8963	0.5262	0.0154	0.036*
C6	0.7837 (3)	0.5744 (7)	0.0738 (4)	0.0291 (11)
H6	0.7549	0.6125	0.0064	0.035*
C7	0.6523 (3)	0.6206 (7)	0.1623 (4)	0.0235 (11)
H7	0.6259	0.6175	0.2273	0.028*
C8	0.4770 (3)	0.7668 (6)	-0.0107 (3)	0.0203 (10)
C9	0.3477 (3)	0.8652 (6)	-0.0881 (3)	0.0215 (10)
C10	0.2636 (3)	0.9113 (7)	-0.0786 (4)	0.0225 (10)
H10	0.2431	0.9085	-0.0095	0.027*
C11	0.2105 (3)	0.9607 (7)	-0.1694 (4)	0.0253 (11)
H11	0.1536	0.9921	-0.1625	0.030*

C12	0.2405 (3)	0.9648 (7)	-0.2726 (4)	0.0272 (11)
H12	0.2038	0.9993	-0.3347	0.033*
C13	0.3230 (3)	0.9187 (7)	-0.2832 (3)	0.0233 (10)
H13	0.3430	0.9218	-0.3526	0.028*
C14	0.3777 (3)	0.8671 (6)	-0.1917 (4)	0.0214 (10)
C15	0.5066 (3)	0.7679 (7)	-0.1146 (3)	0.0222 (10)
H15	0.5631	0.7319	-0.1202	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0337 (8)	0.0532 (9)	0.0288 (7)	0.0025 (6)	-0.0040 (5)	0.0098 (6)
C12	0.0250 (7)	0.0496 (9)	0.0422 (8)	0.0038 (6)	0.0012 (6)	0.0045 (6)
N1	0.022 (2)	0.029 (2)	0.0190 (19)	0.0013 (17)	0.0001 (16)	-0.0014 (17)
N2	0.025 (2)	0.037 (2)	0.0130 (19)	0.0015 (19)	0.0014 (16)	0.0009 (17)
N3	0.020 (2)	0.026 (2)	0.0155 (18)	-0.0015 (17)	-0.0006 (15)	-0.0013 (16)
N4	0.026 (2)	0.030 (2)	0.0177 (19)	0.0012 (19)	0.0034 (16)	-0.0016 (17)
C1	0.025 (3)	0.024 (2)	0.027 (3)	0.003 (2)	0.000 (2)	0.000 (2)
C2	0.024 (2)	0.032 (3)	0.015 (2)	0.003 (2)	-0.0031 (18)	0.000 (2)
C3	0.033 (3)	0.021 (2)	0.035 (3)	-0.009 (2)	-0.007 (2)	0.002 (2)
C4	0.024 (2)	0.030 (3)	0.029 (3)	-0.002 (2)	0.000 (2)	0.001 (2)
C5	0.031 (3)	0.033 (3)	0.027 (3)	0.000 (2)	0.002 (2)	0.000 (2)
C6	0.028 (3)	0.033 (3)	0.026 (3)	-0.005 (2)	0.002 (2)	-0.003 (2)
C7	0.027 (3)	0.030 (3)	0.013 (2)	0.002 (2)	-0.0005 (18)	-0.0013 (19)
C8	0.027 (2)	0.021 (2)	0.012 (2)	-0.001 (2)	0.0000 (18)	-0.0011 (17)
C9	0.027 (2)	0.022 (2)	0.015 (2)	-0.002 (2)	0.0009 (18)	-0.0037 (18)
C10	0.023 (2)	0.029 (3)	0.015 (2)	-0.001 (2)	0.0025 (18)	-0.0006 (19)
C11	0.023 (2)	0.028 (3)	0.024 (3)	0.001 (2)	0.0002 (19)	-0.002 (2)
C12	0.035 (3)	0.027 (3)	0.017 (2)	-0.002 (2)	-0.009 (2)	0.0006 (19)
C13	0.027 (3)	0.032 (3)	0.011 (2)	0.002 (2)	0.0001 (18)	-0.0005 (19)
C14	0.022 (2)	0.024 (2)	0.019 (2)	0.001 (2)	0.0012 (18)	-0.0025 (19)
C15	0.025 (2)	0.028 (2)	0.014 (2)	0.003 (2)	-0.0018 (17)	-0.0006 (19)

Geometric parameters (Å, °)

C11—C3	1.733 (5)	C5—C6	1.393 (7)
C12—C4	1.725 (5)	C5—H5	0.9500
N1—C7	1.277 (6)	C6—H6	0.9500
N1—N2	1.375 (5)	C7—H7	0.9500
N2—C8	1.361 (6)	C8—C15	1.421 (6)
N2—H2N	0.92 (5)	C9—C10	1.402 (6)
N3—C8	1.323 (6)	C9—C14	1.420 (6)
N3—C9	1.375 (6)	C10—C11	1.383 (6)
N4—C15	1.312 (6)	C10—H10	0.9500
N4—C14	1.378 (6)	C11—C12	1.414 (6)
C1—C6	1.392 (7)	C11—H11	0.9500
C1—C2	1.400 (6)	C12—C13	1.381 (7)
C1—C7	1.453 (6)	C12—H12	0.9500

C2—C3	1.406 (7)	C13—C14	1.406 (6)
C2—H2	0.9500	C13—H13	0.9500
C3—C4	1.385 (7)	C15—H15	0.9500
C4—C5	1.401 (7)		
C7—N1—N2	116.3 (4)	C1—C7—H7	119.4
C8—N2—N1	120.1 (4)	N3—C8—N2	116.1 (4)
C8—N2—H2N	118 (3)	N3—C8—C15	121.9 (4)
N1—N2—H2N	122 (3)	N2—C8—C15	122.0 (4)
C8—N3—C9	116.6 (4)	N3—C9—C10	119.1 (4)
C15—N4—C14	116.8 (4)	N3—C9—C14	121.3 (4)
C6—C1—C2	119.0 (4)	C10—C9—C14	119.6 (4)
C6—C1—C7	122.6 (4)	C11—C10—C9	120.2 (4)
C2—C1—C7	118.3 (4)	C11—C10—H10	119.9
C1—C2—C3	119.6 (4)	C9—C10—H10	119.9
C1—C2—H2	120.2	C10—C11—C12	120.3 (4)
C3—C2—H2	120.2	C10—C11—H11	119.9
C4—C3—C2	120.8 (4)	C12—C11—H11	119.9
C4—C3—C11	120.8 (4)	C13—C12—C11	120.1 (4)
C2—C3—C11	118.4 (4)	C13—C12—H12	119.9
C3—C4—C5	119.5 (5)	C11—C12—H12	119.9
C3—C4—C12	121.4 (4)	C12—C13—C14	120.3 (4)
C5—C4—C12	119.0 (4)	C12—C13—H13	119.9
C6—C5—C4	119.5 (5)	C14—C13—H13	119.9
C6—C5—H5	120.2	N4—C14—C13	120.0 (4)
C4—C5—H5	120.2	N4—C14—C9	120.5 (4)
C1—C6—C5	121.4 (5)	C13—C14—C9	119.5 (4)
C1—C6—H6	119.3	N4—C15—C8	122.9 (4)
C5—C6—H6	119.3	N4—C15—H15	118.5
N1—C7—C1	121.1 (4)	C8—C15—H15	118.5
N1—C7—H7	119.4		
C7—N1—N2—C8	-179.9 (4)	N1—N2—C8—C15	2.6 (7)
C6—C1—C2—C3	-1.9 (7)	C8—N3—C9—C10	177.5 (4)
C7—C1—C2—C3	179.0 (4)	C8—N3—C9—C14	-1.5 (6)
C1—C2—C3—C4	-0.1 (7)	N3—C9—C10—C11	-179.8 (4)
C1—C2—C3—C11	-179.2 (4)	C14—C9—C10—C11	-0.8 (7)
C2—C3—C4—C5	2.0 (7)	C9—C10—C11—C12	0.2 (7)
C11—C3—C4—C5	-178.9 (4)	C10—C11—C12—C13	0.2 (7)
C2—C3—C4—C12	-177.1 (4)	C11—C12—C13—C14	0.1 (7)
C11—C3—C4—C12	1.9 (6)	C15—N4—C14—C13	-179.2 (4)
C3—C4—C5—C6	-1.9 (8)	C15—N4—C14—C9	-0.1 (7)
C12—C4—C5—C6	177.2 (4)	C12—C13—C14—N4	178.3 (4)
C2—C1—C6—C5	2.0 (7)	C12—C13—C14—C9	-0.8 (7)
C7—C1—C6—C5	-179.0 (5)	N3—C9—C14—N4	1.0 (7)
C4—C5—C6—C1	-0.1 (8)	C10—C9—C14—N4	-178.0 (4)
N2—N1—C7—C1	-179.3 (4)	N3—C9—C14—C13	-179.8 (4)
C6—C1—C7—N1	0.7 (7)	C10—C9—C14—C13	1.2 (7)

C2—C1—C7—N1	179.8 (4)	C14—N4—C15—C8	-0.2 (7)
C9—N3—C8—N2	-178.7 (4)	N3—C8—C15—N4	-0.3 (7)
C9—N3—C8—C15	1.2 (6)	N2—C8—C15—N4	179.6 (4)
N1—N2—C8—N3	-177.5 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2N \cdots N4 ⁱ	0.92 (5)	2.10 (5)	3.013 (5)	171 (4)

Symmetry code: (i) $x, -y+3/2, z+1/2$.