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N^2-[1-(2-Hydroxyphenyl)ethylidene]-$N^{2'}$-(1*H*-indol-3-ylmethylene)carbonic dihydrazide

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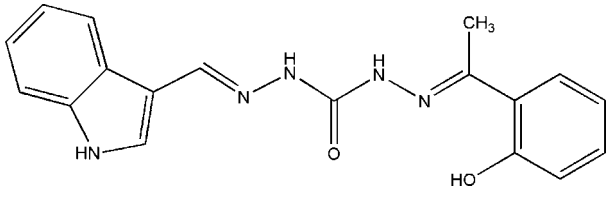
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.122; data-to-parameter ratio = 20.8.

In the crystal structure of the title compound {alternative name: 1-[1-(2-hydroxyphenyl)ethylideneamino]-3-(1*H*-indol-3-ylmethyleneamino)urea}, $C_{18}H_{17}N_5O_2$, the planar indole component is twisted at an angle of 63.7 (10)° with respect to the rest of the molecule. This compound is one of a series being studied for biological activity. The hydroxy groups are involved in both intramolecular (O—H···N) and intermolecular (N—H···O) hydrogen bonds.

Related literature

For a related compound, see: Dan *et al.* (1987).



Experimental

Crystal data

$C_{18}H_{17}N_5O_2$ M_r = 335.37
Monoclinic, $P2_1/n$
a = 7.0802 (8) Å
b = 9.5335 (11) Å
c = 25.110 (3) Å
β = 97.295 (2)°
V = 1681.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 (2) K
0.42 × 0.42 × 0.16 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.821, T_{max} = 0.986
11912 measured reflections
4740 independent reflections
4145 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.122
S = 1.03
4740 reflections
228 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N5	0.84	1.78	2.5121 (11)	145
N3—H3B···O2^{i}	0.88	1.99	2.8481 (13)	166
N1—H1B···O1^{ii}	0.88	2.14	2.8803 (13)	142

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2434).

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supplementary materials

Acta Cryst. (2008). E**64**, o2357 [**doi:10.1107/S1600536808035757**]

N^2-[1-(2-Hydroxyphenyl)ethylidene]-$N^{2'}$-(1*H*-indol-3-ylmethylene)carbonic dihydrazide

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Comment

X-ray structures, of Schiff bases derived from condensation of indole-3-carboxaldehyde and 2-hydroxyacetophenone, have not been investigated. The title compound (Fig. 1) appears to be the first example with the planar indole component is twisted at an angle of 116.3 (10)° with respect to the rest of the molecule. However, compound bis(salicylidene)carbonohydrazide (Dan *et al.*1987), which was reported previously shows planarity for the whole molecule.

Experimental

Indole-3-carboxaldehyde (0.30 g, 2.07 mmol), carbohydrazide (0.187 g, 2.07 mmol), and 2-Hydroxyacetophenone (0.24 ml, 2.07 mmol) were heated in acidified ethanol (20 ml) for 2 h. The solvent was removed and the product recrystallized from DMSO.

Refinement

Hydrogen atoms were placed at calculated positions (C—H 0.95, N—H 0.88 and O—H 0.84 Å), with U(H) set to 1.2–1.5 times U_{eq}(C,*N*,*O*).

Figures

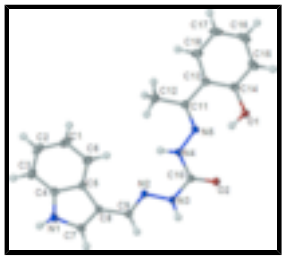


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{18}H_{17}N_5O_2$ at 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius

1-[1-(2-hydroxyphenyl)ethylideneamino]-3-(1*H*-indol-3- ylmethyleneamino)urea

Crystal data

$C_{18}H_{17}N_5O_2$	F_{000} = 704
M_r = 335.37	D_x = 1.325 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 6889 reflections
a = 7.0802 (8) Å	θ = 2.3–30.5º

b = 9.5335 (11) Å	μ = 0.09 mm^{-1}
c = 25.110 (3) Å	T = 100 (2) K
β = 97.295 (2)º	Irregular, colourless
V = 1681.2 (3) Å^3	0.42 × 0.42 × 0.16 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	4740 independent reflections
Radiation source: fine-focus sealed tube	4145 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.014
T = 100(2) K	θ_{max} = 30.6º
ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→4
T_{min} = 0.821, T_{max} = 0.986	k = −13→12
11912 measured reflections	l = −34→35

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H-atom parameters constrained
$wR(F^2)$ = 0.122	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.7125P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
4740 reflections	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
228 parameters	$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

	x	y	z	U_{iso}*/U_{eq}

N2	0.47231 (13)	0.13948 (10)	0.11232 (3)	0.01593 (18)
O2	0.38944 (12)	0.16315 (8)	−0.02799 (3)	0.01958 (18)
O1	0.22435 (14)	0.47449 (9)	−0.09575 (3)	0.0246 (2)
H1A	0.2598	0.4252	−0.0686	0.037*
N5	0.28865 (13)	0.41919 (9)	0.00270 (3)	0.01540 (18)
N3	0.45872 (14)	0.09630 (10)	0.05941 (4)	0.01774 (19)
H3B	0.4878	0.0099	0.0513	0.021*
C13	0.17966 (15)	0.64221 (11)	−0.02541 (4)	0.0159 (2)
N4	0.35380 (14)	0.31965 (9)	0.03933 (3)	0.01631 (19)
H4B	0.3663	0.3374	0.0740	0.020*
C11	0.25266 (15)	0.54484 (11)	0.01820 (4)	0.0150 (2)
C10	0.39925 (15)	0.19077 (11)	0.02031 (4)	0.0155 (2)
C9	0.57339 (15)	0.06194 (11)	0.14689 (4)	0.0160 (2)
H9	0.6346	−0.0193	0.1354	0.019*
C8	0.59385 (15)	0.09842 (11)	0.20307 (4)	0.0153 (2)
N1	0.69458 (15)	0.09189 (10)	0.29159 (4)	0.0203 (2)
H1B	0.7590	0.0676	0.3225	0.024*
C12	0.28004 (18)	0.59167 (13)	0.07571 (5)	0.0224 (2)
H12A	0.1593	0.5833	0.0907	0.034*
H12B	0.3223	0.6896	0.0777	0.034*
H12C	0.3764	0.5326	0.0963	0.034*
C14	0.16631 (16)	0.60243 (11)	−0.07989 (4)	0.0180 (2)
C15	0.09241 (18)	0.69504 (13)	−0.12025 (5)	0.0237 (2)
H15	0.0813	0.6664	−0.1568	0.028*
C7	0.71448 (17)	0.03294 (12)	0.24298 (4)	0.0192 (2)
H7	0.7986	−0.0418	0.2375	0.023*
C5	0.49227 (15)	0.20425 (11)	0.22913 (4)	0.0147 (2)
C18	0.12097 (18)	0.77799 (12)	−0.01393 (5)	0.0240 (2)
H18	0.1294	0.8076	0.0224	0.029*
C3	0.48431 (17)	0.27906 (12)	0.32274 (5)	0.0208 (2)
H3	0.5303	0.2721	0.3599	0.025*
C1	0.27606 (18)	0.38481 (13)	0.24917 (5)	0.0255 (3)
H1	0.1791	0.4509	0.2376	0.031*
C4	0.55764 (16)	0.19532 (11)	0.28456 (4)	0.0164 (2)
C2	0.34160 (18)	0.37266 (13)	0.30407 (5)	0.0247 (2)
H2	0.2868	0.4299	0.3290	0.030*
C17	0.05073 (19)	0.87046 (13)	−0.05440 (6)	0.0283 (3)
H17	0.0134	0.9625	−0.0457	0.034*
C6	0.35025 (16)	0.30202 (12)	0.21144 (5)	0.0200 (2)
H6	0.3056	0.3114	0.1743	0.024*
C16	0.03524 (18)	0.82812 (13)	−0.10749 (5)	0.0264 (3)
H16	−0.0148	0.8907	−0.1352	0.032*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0201 (4)	0.0163 (4)	0.0114 (4)	0.0015 (3)	0.0021 (3)	−0.0005 (3)
O2	0.0286 (4)	0.0170 (4)	0.0127 (3)	0.0047 (3)	0.0009 (3)	−0.0006 (3)

O1	0.0389 (5)	0.0196 (4)	0.0140 (4)	0.0084 (4)	−0.0020 (3)	−0.0002 (3)
N5	0.0172 (4)	0.0140 (4)	0.0148 (4)	0.0021 (3)	0.0012 (3)	0.0020 (3)
N3	0.0264 (5)	0.0145 (4)	0.0121 (4)	0.0054 (3)	0.0016 (3)	−0.0001 (3)
C13	0.0141 (4)	0.0142 (4)	0.0192 (5)	0.0006 (4)	0.0015 (4)	0.0011 (4)
N4	0.0228 (5)	0.0140 (4)	0.0118 (4)	0.0046 (3)	0.0008 (3)	0.0009 (3)
C11	0.0144 (4)	0.0146 (4)	0.0162 (4)	0.0004 (4)	0.0027 (3)	−0.0004 (4)
C10	0.0175 (5)	0.0144 (4)	0.0143 (4)	0.0017 (4)	0.0013 (4)	0.0002 (3)
C9	0.0180 (5)	0.0149 (4)	0.0153 (4)	0.0021 (4)	0.0033 (4)	0.0004 (4)
C8	0.0172 (5)	0.0148 (4)	0.0139 (4)	0.0013 (4)	0.0024 (4)	0.0012 (3)
N1	0.0262 (5)	0.0209 (5)	0.0128 (4)	0.0031 (4)	−0.0008 (3)	0.0018 (3)
C12	0.0300 (6)	0.0201 (5)	0.0173 (5)	0.0019 (4)	0.0036 (4)	−0.0036 (4)
C14	0.0173 (5)	0.0162 (5)	0.0197 (5)	0.0010 (4)	−0.0007 (4)	0.0018 (4)
C15	0.0235 (6)	0.0242 (6)	0.0221 (5)	0.0013 (4)	−0.0028 (4)	0.0061 (4)
C7	0.0232 (5)	0.0187 (5)	0.0157 (5)	0.0044 (4)	0.0016 (4)	0.0015 (4)
C5	0.0161 (5)	0.0138 (4)	0.0143 (4)	−0.0022 (4)	0.0027 (3)	−0.0007 (3)
C18	0.0265 (6)	0.0172 (5)	0.0281 (6)	0.0045 (4)	0.0033 (5)	−0.0003 (4)
C3	0.0244 (6)	0.0212 (5)	0.0176 (5)	−0.0058 (4)	0.0052 (4)	−0.0048 (4)
C1	0.0210 (6)	0.0239 (6)	0.0310 (6)	0.0058 (4)	0.0013 (5)	−0.0083 (5)
C4	0.0188 (5)	0.0151 (5)	0.0156 (5)	−0.0030 (4)	0.0030 (4)	−0.0003 (4)
C2	0.0240 (6)	0.0244 (6)	0.0269 (6)	−0.0022 (5)	0.0076 (5)	−0.0108 (5)
C17	0.0284 (6)	0.0161 (5)	0.0402 (7)	0.0068 (4)	0.0034 (5)	0.0046 (5)
C6	0.0189 (5)	0.0199 (5)	0.0208 (5)	0.0031 (4)	0.0002 (4)	−0.0029 (4)
C16	0.0219 (6)	0.0218 (6)	0.0343 (6)	0.0021 (4)	−0.0013 (5)	0.0116 (5)

Geometric parameters (Å, °)

N2—C9	1.2862 (14)	C12—H12A	0.9800
N2—N3	1.3824 (12)	C12—H12B	0.9800
O2—C10	1.2344 (13)	C12—H12C	0.9800
O1—C14	1.3626 (13)	C14—C15	1.3949 (15)
O1—H1A	0.8400	C15—C16	1.3816 (18)
N5—C11	1.2948 (13)	C15—H15	0.9500
N5—N4	1.3608 (12)	C7—H7	0.9500
N3—C10	1.3591 (13)	C5—C6	1.4013 (15)
N3—H3B	0.8800	C5—C4	1.4125 (14)
C13—C18	1.4007 (15)	C18—C17	1.3887 (17)
C13—C14	1.4111 (15)	C18—H18	0.9500
C13—C11	1.4779 (14)	C3—C2	1.3844 (18)
N4—C10	1.3712 (13)	C3—C4	1.3973 (15)
N4—H4B	0.8800	C3—H3	0.9500
C11—C12	1.5003 (15)	C1—C6	1.3866 (16)
C9—C8	1.4424 (14)	C1—C2	1.4024 (18)
C9—H9	0.9500	C1—H1	0.9500
C8—C7	1.3807 (14)	C2—H2	0.9500
C8—C5	1.4431 (14)	C17—C16	1.384 (2)
N1—C7	1.3672 (14)	C17—H17	0.9500
N1—C4	1.3784 (14)	C6—H6	0.9500
N1—H1B	0.8800	C16—H16	0.9500
C9—N2—N3	116.25 (9)	C15—C14—C13	120.46 (10)

C14—O1—H1A	109.5	C16—C15—C14	120.45 (11)
C11—N5—N4	120.31 (9)	C16—C15—H15	119.8
C10—N3—N2	118.31 (9)	C14—C15—H15	119.8
C10—N3—H3B	120.8	N1—C7—C8	109.75 (10)
N2—N3—H3B	120.8	N1—C7—H7	125.1
C18—C13—C14	117.56 (10)	C8—C7—H7	125.1
C18—C13—C11	120.87 (10)	C6—C5—C4	118.98 (10)
C14—C13—C11	121.57 (9)	C6—C5—C8	134.45 (10)
N5—N4—C10	117.64 (9)	C4—C5—C8	106.55 (9)
N5—N4—H4B	121.2	C17—C18—C13	121.61 (12)
C10—N4—H4B	121.2	C17—C18—H18	119.2
N5—C11—C13	114.95 (9)	C13—C18—H18	119.2
N5—C11—C12	123.94 (10)	C2—C3—C4	117.05 (10)
C13—C11—C12	121.10 (9)	C2—C3—H3	121.5
O2—C10—N3	122.82 (10)	C4—C3—H3	121.5
O2—C10—N4	123.15 (9)	C6—C1—C2	121.18 (11)
N3—C10—N4	114.02 (9)	C6—C1—H1	119.4
N2—C9—C8	119.98 (10)	C2—C1—H1	119.4
N2—C9—H9	120.0	N1—C4—C3	129.59 (10)
C8—C9—H9	120.0	N1—C4—C5	107.85 (9)
C7—C8—C9	125.24 (10)	C3—C4—C5	122.54 (10)
C7—C8—C5	106.56 (9)	C3—C2—C1	121.48 (11)
C9—C8—C5	128.18 (9)	C3—C2—H2	119.3
C7—N1—C4	109.28 (9)	C1—C2—H2	119.3
C7—N1—H1B	125.4	C16—C17—C18	119.80 (11)
C4—N1—H1B	125.4	C16—C17—H17	120.1
C11—C12—H12A	109.5	C18—C17—H17	120.1
C11—C12—H12B	109.5	C1—C6—C5	118.75 (11)
H12A—C12—H12B	109.5	C1—C6—H6	120.6
C11—C12—H12C	109.5	C5—C6—H6	120.6
H12A—C12—H12C	109.5	C15—C16—C17	120.09 (11)
H12B—C12—H12C	109.5	C15—C16—H16	120.0
O1—C14—C15	116.97 (10)	C17—C16—H16	120.0
O1—C14—C13	122.57 (9)		
C9—N2—N3—C10	−162.57 (10)	C5—C8—C7—N1	−0.35 (13)
C11—N5—N4—C10	−176.52 (10)	C7—C8—C5—C6	−178.47 (12)
N4—N5—C11—C13	−179.00 (9)	C9—C8—C5—C6	−0.3 (2)
N4—N5—C11—C12	0.64 (16)	C7—C8—C5—C4	−0.58 (12)
C18—C13—C11—N5	174.99 (10)	C9—C8—C5—C4	177.62 (10)
C14—C13—C11—N5	−5.00 (15)	C14—C13—C18—C17	0.46 (18)
C18—C13—C11—C12	−4.66 (16)	C11—C13—C18—C17	−179.52 (11)
C14—C13—C11—C12	175.35 (10)	C7—N1—C4—C3	177.00 (11)
N2—N3—C10—O2	177.29 (10)	C7—N1—C4—C5	−1.54 (13)
N2—N3—C10—N4	−1.86 (15)	C2—C3—C4—N1	−178.03 (11)
N5—N4—C10—O2	2.37 (16)	C2—C3—C4—C5	0.33 (17)
N5—N4—C10—N3	−178.48 (9)	C6—C5—C4—N1	179.57 (10)
N3—N2—C9—C8	−179.42 (9)	C8—C5—C4—N1	1.29 (12)
N2—C9—C8—C7	−172.45 (11)	C6—C5—C4—C3	0.90 (16)
N2—C9—C8—C5	9.66 (17)	C8—C5—C4—C3	−177.38 (10)

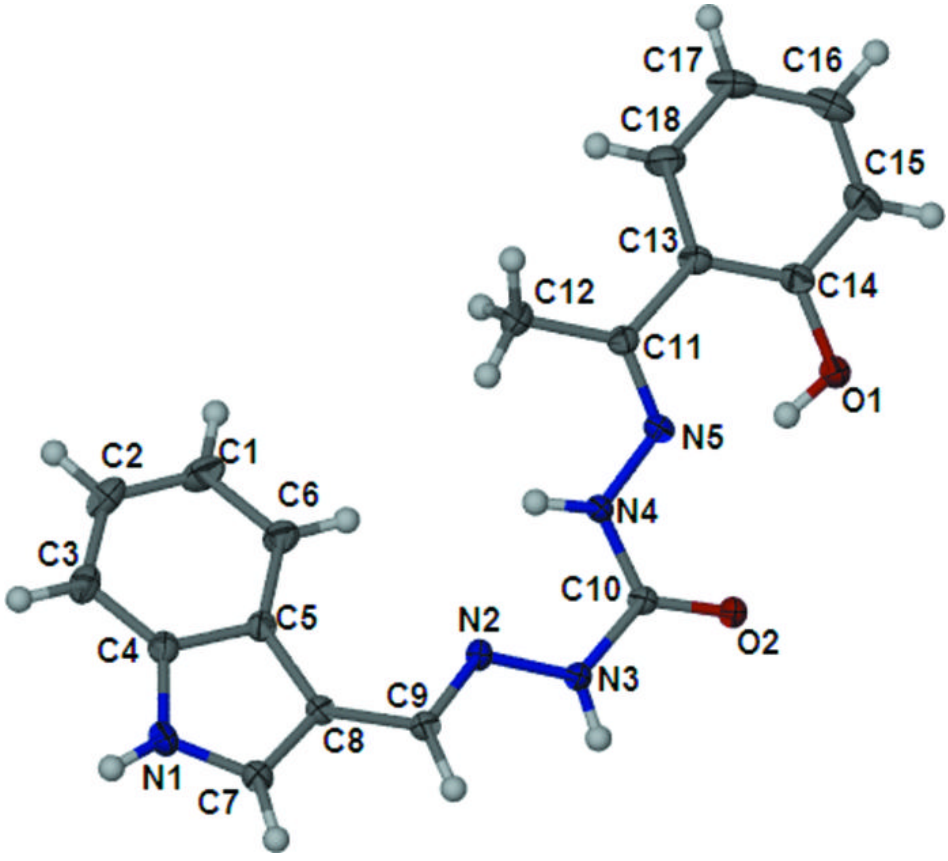
C18—C13—C14—O1	178.09 (11)	C4—C3—C2—C1	−1.19 (18)
C11—C13—C14—O1	−1.93 (17)	C6—C1—C2—C3	0.8 (2)
C18—C13—C14—C15	−1.62 (16)	C13—C18—C17—C16	0.9 (2)
C11—C13—C14—C15	178.37 (10)	C2—C1—C6—C5	0.44 (18)
O1—C14—C15—C16	−178.26 (11)	C4—C5—C6—C1	−1.26 (16)
C13—C14—C15—C16	1.46 (18)	C8—C5—C6—C1	176.42 (12)
C4—N1—C7—C8	1.19 (13)	C14—C15—C16—C17	−0.10 (19)
C9—C8—C7—N1	−178.62 (10)	C18—C17—C16—C15	−1.1 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···N5	0.84	1.78	2.5121 (11)	145
N3—H3B···O2^{i}	0.88	1.99	2.8481 (13)	166
N1—H1B···O1^{ii}	0.88	2.14	2.8803 (13)	142

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) *x*+1/2, −*y*+1/2, *z*+1/2.

Fig. 1


C17
C16
C18
C15
C13
C12
C14
C11
O1
N5
C2
C1
C6
N4
C3
C10
C5
N2
O2
C4
N3
C9
C8
N1
C7