

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# *N*<sup>2</sup>-[1-(2-Hydroxyphenyl)ethylidene]-*N*<sup>2'</sup>-(1*H*-indol-3-ylmethylene)carbonic dihydrazide

 Siti Munirah Saharin,<sup>a</sup> Hapipah Mohd Ali,<sup>a\*</sup> Ward T. Robinson<sup>a</sup> and A. A. Mahmood<sup>b</sup>
<sup>a</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and <sup>b</sup>Department of Molecular Medicine, Faculty of Medicine, University of Malaya, 50603 Kuala Lumpur, Malaysia

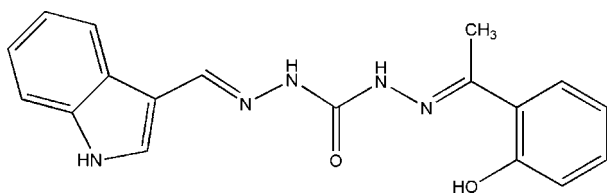
Correspondence e-mail: hapipah@um.edu.my

Received 23 October 2008; accepted 31 October 2008

 Key indicators: single-crystal X-ray study; *T* = 100 K; mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$ ; *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},  $\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2$ , the planar indole component is twisted at an angle of  $63.7 (10)^\circ$  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

 $\text{C}_{18}\text{H}_{17}\text{N}_5\text{O}_2$ 
 $M_r = 335.37$ 

 Monoclinic,  $P2_1/n$   
 $a = 7.0802 (8) \text{ \AA}$   
 $b = 9.5335 (11) \text{ \AA}$   
 $c = 25.110 (3) \text{ \AA}$   
 $\beta = 97.295 (2)^\circ$   
 $V = 1681.2 (3) \text{ \AA}^3$ 
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.09 \text{ mm}^{-1}$   
 $T = 100 (2) \text{ K}$   
 $0.42 \times 0.42 \times 0.16 \text{ mm}$ 

## 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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.014$ 

## 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  
 $\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$ 

Table 1

 Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
O1—H1A···N5	0.84	1.78	2.5121 (11)	145
N3—H3B···O2 <sup>i</sup>	0.88	1.99	2.8481 (13)	166
N1—H1B···O1 <sup>ii</sup>	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).

The authors thank the University of Malaya for funding this study (Science Fund Grants 12–02–03–2031, 12–02–03–2051 and PJP FS350–2008 A).

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

## References

- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
 Bruker (2007). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Dan, J., Seth, S. & Chakraborty, S. (1987). *Acta Cryst.* **C43**, 1114–1116.  
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Westrip, S. P. (2008). publCIF. In preparation.

**supplementary materials**

*Acta Cryst.* (2008). E64, o2357 [ doi:10.1107/S1600536808035757 ]

***N*<sup>2</sup>-[1-(2-Hydroxyphenyl)ethylidene]-*N*<sup>2'</sup>-(1*H*-indol-3-ylmethylene)carbonic dihydrazide**

**S. M. Saharin, H. Mohd Ali, W. T. Robinson and A. A. Mahmood**

**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**

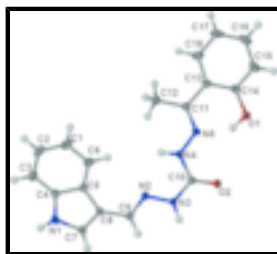


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of C<sub>18</sub>H<sub>17</sub>N<sub>5</sub>O<sub>2</sub> at 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius

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

*Crystal data*

C<sub>18</sub>H<sub>17</sub>N<sub>5</sub>O<sub>2</sub>

$M_r = 335.37$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 7.0802$  (8) Å

$F_{000} = 704$

$D_x = 1.325$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 6889 reflections

$\theta = 2.3$ – $30.5^\circ$

# supplementary materials

---

$b = 9.5335 (11) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 25.110 (3) \text{ \AA}$	$T = 100 (2) \text{ K}$
$\beta = 97.295 (2)^\circ$	Irregular, colourless
$V = 1681.2 (3) \text{ \AA}^3$	$0.42 \times 0.42 \times 0.16 \text{ mm}$
$Z = 4$	

## 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	$R_{\text{int}} = 0.014$
$T = 100(2) \text{ K}$	$\theta_{\text{max}} = 30.6^\circ$
$\omega$ scans	$\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 4$
$T_{\text{min}} = 0.821$ , $T_{\text{max}} = 0.986$	$k = -13 \rightarrow 12$
11912 measured reflections	$l = -34 \rightarrow 35$

## 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
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.7125P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
4740 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
228 parameters	$\Delta\rho_{\text{max}} = 0.43 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.32 \text{ e \AA}^{-3}$
	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 ( $\text{\AA}^2$ )

$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
-----	-----	-----	----------------------------------

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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

---

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)

## supplementary materials

---

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 ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1A $\cdots$ N5	0.84	1.78	2.5121 (11)	145
N3—H3B $\cdots$ O2 <sup>i</sup>	0.88	1.99	2.8481 (13)	166
N1—H1B $\cdots$ O1 <sup>ii</sup>	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

