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## 3-Hydroxy-3-(2-oxo-2,3-dihydro-1*H*-indol-3-yl)-2,3-dihydro-1*H*-indol-2-one

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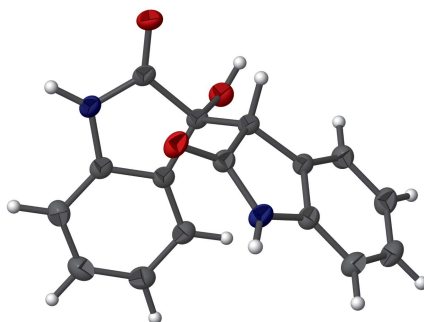
Keywords: crystal structure; hydrogen bond;  $\pi$ - $\pi$  stacking; indolinone; isatin; dimerization.

CCDC reference: 1529856

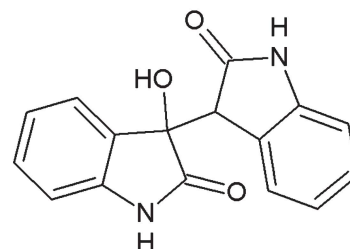
Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

The conformation of the title molecule, C<sub>16</sub>H<sub>12</sub>N<sub>2</sub>O<sub>3</sub>, is partly determined by an intramolecular C=O... $\pi$  interaction between one carbonyl group and the five-membered ring of the other indolinone moiety. The crystal packing consists of layers parallel to (001) formed by a combination of N—H...O and O—H...O hydrogen bonds and  $\pi$ - $\pi$  stacking interactions. Both the N—H...O and O—H...O hydrogen bonds generate inversion dimers.

### 3D view



### Chemical scheme



### Structure description

Indole scaffold compounds are synthetically important substrates that can be used for the synthesis of a large variety of heterocyclic compounds, and as raw material for drug synthesis (Grewal, 2014). Recently, isatin derivatives have attracted strong interest in organic and medicinal chemistry due to their potent biological and pharmacological activities including antitumor (Premanathan *et al.*, 2012; Havrylyuk *et al.*, 2011), antimicrobial (Singh *et al.*, 2010; Ali & Alam, 1994; Pandeya *et al.*, 1999*b*), anti-inflammatory and analgesic (Abele *et al.*, 2003; Mondal, *et al.*, 2010), antimycobacterial (Aboul-Fadl *et al.*, 2010; Sriram *et al.*, 2006), anticonvulsant (Malawska, 2005), antiviral (Selvam *et al.*, 2006; Selvam *et al.*, 2008; Abbas *et al.*, 2013), anthelmintic (Suresh *et al.*, 2011), anti-HIV applications (Pandeya *et al.*, 1999*a*) and anti-oxidant (Andreani *et al.*, 2010; Kiran *et al.*, 2013). In this context, the present study reports the synthesis and crystal structure determination of the title bis-indole derivative.

**Table 1**

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1,C6,C7,C8,N1 ring.

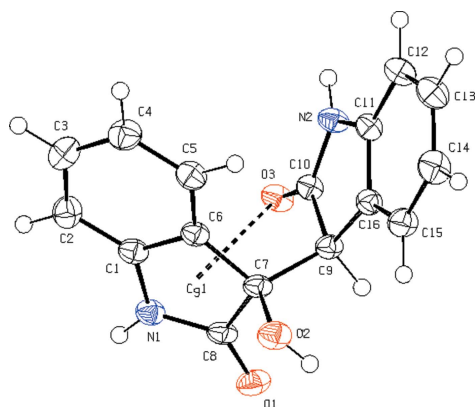
<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O2—H2A...O1 <sup>i</sup>	0.84	1.93	2.7575 (17)	167
N1—H1...O3 <sup>ii</sup>	0.94 (2)	1.96 (2)	2.8644 (17)	161 (2)
N2—H2B...O3 <sup>iii</sup>	0.90 (3)	2.00 (3)	2.8882 (18)	173 (2)
C10—O3...Cg1	1.24 (1)	2.92 (1)	3.0414 (13)	73 (1)

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

In the title compound (Fig. 1), the indolinone moieties are close to being planar, with r.m.s. deviations of the nine atoms from the mean plane being 0.025 Å for the ring system containing atom N1 and 0.014 Å for that containing atom N2. The dihedral angle between the mean planes of the two indolinone moieties is 58.69 (3)°. The conformation of the molecule is determined in part by a C=O... $\pi$  interaction between the C10=O3 carbonyl group and the C1/C6/C7/C8/N1 ring (Fig. 1 and Table 1).

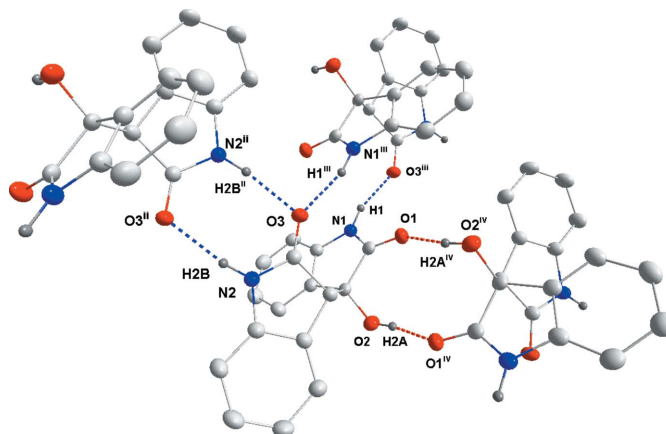
The bond lengths and angles of the title compound are comparable with those reported for related compounds such as (3*E*)-3-[(4-butylphenyl)imino]-1,3-dihydro-2*H*-indol-2-one (Akkurt *et al.*, 2003), 2-oxo-2,3-dihydro-1*H*-indol-3-one nicotinoylhydrazone (Ali *et al.*, 2005) and 3-(2-amino-1-methyl-4,4,5-dihydro-1*H*-imidazol-5-yl)-3-hydroxy-1-phenylindolin-2-one ethanol solvate (Penthala *et al.*, 2009).

The N2—H2B...O3 and the O2—H2A...O1 hydrogen bonds (Table 1) generate inversion dimers with  $R_2^2(8)$  and  $R_2^2(10)$  ring motifs, respectively (Fig. 2). The other N1—H1...O3 contact also forms an inversion dimer but, in this case, with an  $R_2^2(14)$  motif. A combination of N1—H1...O3 and N2—H2B...O3 hydrogen bonds generate zigzag chains of molecules running parallel to the *b*-axis direction which are formed into sheets parallel to (001) by pairwise O2—H2A...O1 hydrogen bonds (Figs. 2 and 3). The layer formation is assisted by complementary  $\pi$ - $\pi$  stacking interactions [ $Cg2 \cdots Cg3 = 3.571$  (1) Å] between the C11—C16 and C16/C9/C10/C11/N2 rings (Fig. 4).



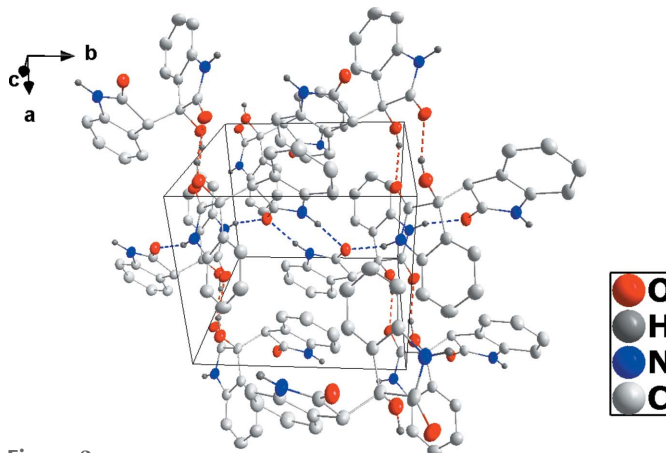
**Figure 1**

The title molecule with the atom-labeling scheme and 50% probability ellipsoids. The C=O... $\pi$ (ring) interaction is shown as a dotted line. Cg1 is the centroid of the C1/C6/C7/C8/N1 ring.



**Figure 2**

Detail of the O—H...O (red dotted lines) and N—H...O (blue dotted lines) hydrogen bonds. [Symmetry codes: (ii)  $1 - x, 1 - y, 1 - z$ ; (iii)  $1 - x, 2 - y, 1 - z$ ; (iv)  $2 - x, 2 - y, 1 - z$ .]



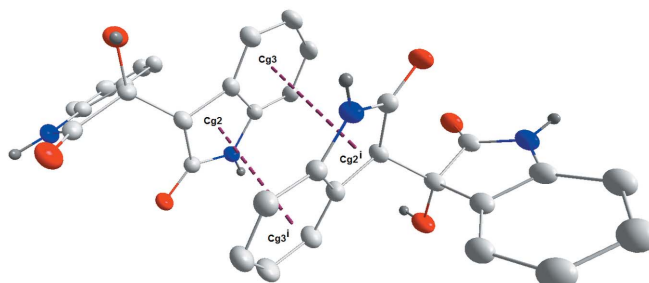
**Figure 3**

Packing showing one of the N—H...O hydrogen bonded (blue dotted lines) chains.

### Synthesis and crystallization

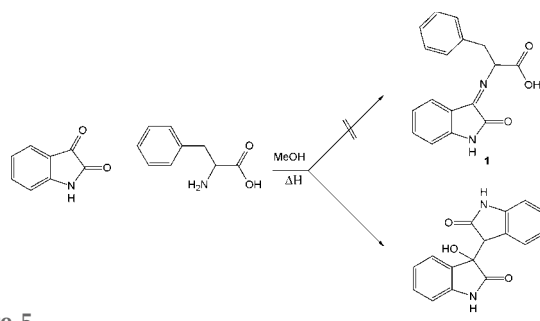
The title compound was obtained (Fig. 5) as a major product during an attempt to synthesize a new tridentate ONO-dibasic hydrazone (**1**) derived from the condensation of phenyl alanine with isatin by the following procedures:

A methanolic solution (15 ml) of isatin (1 mmol) was added dropwise to an aqueous/methanolic (1:1) (10 ml) solution of



**Figure 4**

Details of the  $\pi$ - $\pi$  stacking interactions. Cg2 and Cg3 are the centroids of the C9/C10/N3/C11/C16 and C11—C16 rings, respectively. [Symmetry code: (i)  $2 - x, 1 - y, 1 - z$ .]



**Figure 5**  
A scheme showing the reaction leading to the formation of the title compound.

phenyl alanine at room temperature. The reaction mixture was refluxed at 353 K (monitored by TLC) for 2 h, resulting in the formation of a dark-red precipitate. The precipitate was extracted by filtration and washed many times with water and diethyl ether then dried in an oven. The final product was recrystallized in hot methanol to furnish good quality dark-red crystals of 3-hydroxy-3-(2-oxo-2,3-dihydro-1H-indol-3-yl)-2,3-dihydro-1H-indol-2-one (**2**) in 84% yield. Phenyl alanine may act as a Lewis base to activate the isatin causing dimerization in the aqueous/methanolic media.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydroxyl hydrogen did not refine satisfactorily although definitely located in a difference map, possibly due to unresolved disorder. Consequently it was placed in a calculated position (*SHELXL* HFIX 147 instruction) and included as a riding contribution.

## Acknowledgements

The support of NSF–MRI Grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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**Table 2**

Experimental details.

Crystal data	
Chemical formula	C <sub>16</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub>
<i>M<sub>r</sub></i>	280.28
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	150
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.7193 (3), 8.6977 (3), 9.5907 (4)
$\alpha$ , $\beta$ , $\gamma$ (°)	90.457 (2), 95.070 (2), 90.627 (2)
<i>V</i> (Å <sup>3</sup> )	641.34 (4)
<i>Z</i>	2
Radiation type	Cu <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.84
Crystal size (mm)	0.14 × 0.07 × 0.05
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2016)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.89, 0.96
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	8464, 2487, 2137
<i>R<sub>int</sub></i>	0.033
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.618
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.039, 0.101, 1.06
No. of reflections	2487
No. of parameters	235
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.19, −0.28

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

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## full crystallographic data

*IUCrData* (2017). **2**, x170139 [https://doi.org/10.1107/S2414314617001390]

3-Hydroxy-3-(2-oxo-2,3-dihydro-1*H*-indol-3-yl)-2,3-dihydro-1*H*-indol-2-one

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3-Hydroxy-3-(2-oxo-2,3-dihydro-1*H*-indol-3-yl)-2,3-dihydro-1*H*-indol-2-one*Crystal data*

$C_{16}H_{12}N_2O_3$

$M_r = 280.28$

Triclinic,  $P\bar{1}$

$a = 7.7193$  (3) Å

$b = 8.6977$  (3) Å

$c = 9.5907$  (4) Å

$\alpha = 90.457$  (2)°

$\beta = 95.070$  (2)°

$\gamma = 90.627$  (2)°

$V = 641.34$  (4) Å<sup>3</sup>

$Z = 2$

$F(000) = 292$

$D_x = 1.451$  Mg m<sup>-3</sup>

Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å

Cell parameters from 6334 reflections

$\theta = 4.6$ – $72.4$ °

$\mu = 0.84$  mm<sup>-1</sup>

$T = 150$  K

Column, dark yellow-orange

$0.14 \times 0.07 \times 0.05$  mm

*Data collection*

Bruker D8 VENTURE PHOTON 100 CMOS  
diffractometer

Radiation source: INCOATEC  $I\mu$ S micro-focus  
source

Mirror monochromator

Detector resolution: 10.4167 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2016)

$T_{\min} = 0.89$ ,  $T_{\max} = 0.96$

8464 measured reflections

2487 independent reflections

2137 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 72.4$ °,  $\theta_{\min} = 4.6$ °

$h = -9 \rightarrow 9$

$k = -10 \rightarrow 10$

$l = -11 \rightarrow 11$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.101$

$S = 1.06$

2487 reflections

235 parameters

0 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

$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.3252P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.19$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.28$  e Å<sup>-3</sup>

*Special details*

**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  $F^2 > 2\sigma(F^2)$  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 hydroxyl hydrogen did not refine satisfactorily although definitely located in a difference map, possibly due to unresolved disorder. Consequently it was placed in a calculated position (SHELXL HFIX 147 instruction) and included as a riding contribution.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.78629 (14)	1.05555 (13)	0.52109 (12)	0.0311 (3)
O2	0.94772 (15)	0.94251 (13)	0.26711 (13)	0.0311 (3)
H2A	1.0265	0.9586	0.3319	0.047*
O3	0.58356 (14)	0.70275 (12)	0.52877 (12)	0.0272 (3)
N1	0.56106 (17)	1.03152 (15)	0.34911 (14)	0.0252 (3)
H1	0.491 (3)	1.105 (3)	0.388 (2)	0.052 (6)*
N2	0.69437 (18)	0.50298 (15)	0.40763 (14)	0.0248 (3)
H2B	0.613 (3)	0.433 (3)	0.423 (2)	0.051 (6)*
C1	0.5182 (2)	0.93711 (17)	0.23090 (16)	0.0243 (3)
C2	0.3649 (2)	0.9328 (2)	0.14575 (18)	0.0306 (4)
H2	0.266 (3)	1.000 (2)	0.162 (2)	0.033 (5)*
C3	0.3529 (2)	0.8279 (2)	0.03503 (19)	0.0341 (4)
H3	0.246 (3)	0.824 (2)	-0.025 (2)	0.042 (6)*
C4	0.4899 (2)	0.7319 (2)	0.01088 (18)	0.0334 (4)
H4	0.474 (3)	0.656 (2)	-0.070 (2)	0.037 (5)*
C5	0.6447 (2)	0.73867 (19)	0.09783 (17)	0.0281 (4)
H5	0.743 (3)	0.672 (2)	0.084 (2)	0.033 (5)*
C6	0.6578 (2)	0.84201 (17)	0.20856 (16)	0.0230 (3)
C7	0.80112 (19)	0.87379 (17)	0.32288 (16)	0.0224 (3)
C8	0.71796 (19)	0.99649 (17)	0.41294 (17)	0.0239 (3)
C9	0.85503 (19)	0.73274 (17)	0.41305 (16)	0.0225 (3)
H9	0.928 (2)	0.7736 (19)	0.5023 (18)	0.022 (4)*
C10	0.69513 (19)	0.64824 (17)	0.45866 (16)	0.0222 (3)
C11	0.8409 (2)	0.47612 (17)	0.33341 (16)	0.0233 (3)
C12	0.8850 (2)	0.34313 (19)	0.26664 (18)	0.0289 (4)
H12	0.812 (3)	0.251 (2)	0.267 (2)	0.033 (5)*
C13	1.0385 (2)	0.3456 (2)	0.20033 (19)	0.0326 (4)
H13	1.073 (3)	0.257 (2)	0.153 (2)	0.039 (5)*
C14	1.1432 (2)	0.4764 (2)	0.20282 (19)	0.0336 (4)
H14	1.251 (3)	0.476 (2)	0.159 (2)	0.039 (5)*
C15	1.0967 (2)	0.6096 (2)	0.27092 (18)	0.0288 (4)
H15	1.169 (2)	0.701 (2)	0.273 (2)	0.031 (5)*
C16	0.94318 (19)	0.60941 (17)	0.33510 (16)	0.0229 (3)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0273 (6)	0.0304 (6)	0.0357 (7)	-0.0045 (5)	0.0043 (5)	-0.0121 (5)
O2	0.0265 (6)	0.0299 (6)	0.0382 (7)	-0.0054 (5)	0.0111 (5)	-0.0011 (5)
O3	0.0281 (6)	0.0223 (5)	0.0331 (6)	0.0003 (4)	0.0133 (5)	-0.0023 (4)
N1	0.0236 (6)	0.0220 (6)	0.0306 (7)	0.0002 (5)	0.0065 (5)	-0.0039 (5)
N2	0.0262 (7)	0.0193 (6)	0.0302 (7)	-0.0027 (5)	0.0102 (5)	-0.0015 (5)
C1	0.0257 (8)	0.0217 (7)	0.0265 (8)	-0.0028 (6)	0.0076 (6)	-0.0003 (6)
C2	0.0259 (8)	0.0344 (9)	0.0321 (9)	0.0018 (7)	0.0057 (7)	0.0022 (7)
C3	0.0291 (8)	0.0430 (10)	0.0297 (9)	-0.0039 (7)	-0.0001 (7)	0.0009 (7)
C4	0.0391 (9)	0.0346 (9)	0.0263 (9)	-0.0015 (7)	0.0021 (7)	-0.0041 (7)
C5	0.0326 (8)	0.0258 (8)	0.0265 (8)	0.0017 (7)	0.0058 (6)	-0.0016 (6)
C6	0.0235 (7)	0.0203 (7)	0.0258 (8)	-0.0031 (6)	0.0058 (6)	0.0016 (6)
C7	0.0202 (7)	0.0195 (7)	0.0283 (8)	-0.0042 (6)	0.0075 (6)	-0.0019 (6)
C8	0.0229 (7)	0.0187 (7)	0.0310 (8)	-0.0052 (6)	0.0086 (6)	-0.0018 (6)
C9	0.0203 (7)	0.0214 (7)	0.0263 (8)	-0.0015 (6)	0.0050 (6)	-0.0033 (6)
C10	0.0233 (7)	0.0203 (7)	0.0235 (7)	-0.0001 (6)	0.0043 (6)	0.0001 (6)
C11	0.0247 (7)	0.0222 (7)	0.0236 (8)	0.0018 (6)	0.0053 (6)	0.0004 (6)
C12	0.0358 (9)	0.0218 (8)	0.0298 (8)	0.0012 (7)	0.0074 (7)	-0.0013 (6)
C13	0.0384 (9)	0.0281 (9)	0.0327 (9)	0.0070 (7)	0.0100 (7)	-0.0042 (7)
C14	0.0296 (9)	0.0364 (9)	0.0367 (9)	0.0052 (7)	0.0127 (7)	-0.0032 (7)
C15	0.0237 (8)	0.0287 (8)	0.0347 (9)	-0.0005 (7)	0.0067 (6)	-0.0019 (7)
C16	0.0226 (7)	0.0213 (7)	0.0250 (7)	0.0010 (6)	0.0030 (6)	-0.0014 (6)

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

O1—C8	1.2275 (19)	C5—C6	1.382 (2)
O2—C7	1.4217 (18)	C5—H5	0.97 (2)
O2—H2A	0.8400	C6—C7	1.509 (2)
O3—C10	1.2348 (18)	C7—C9	1.546 (2)
N1—C8	1.347 (2)	C7—C8	1.548 (2)
N1—C1	1.408 (2)	C9—C16	1.505 (2)
N1—H1	0.94 (2)	C9—C10	1.528 (2)
N2—C10	1.3510 (19)	C9—H9	1.039 (17)
N2—C11	1.4091 (19)	C11—C12	1.378 (2)
N2—H2B	0.90 (3)	C11—C16	1.395 (2)
C1—C2	1.378 (2)	C12—C13	1.394 (2)
C1—C6	1.396 (2)	C12—H12	0.97 (2)
C2—C3	1.391 (3)	C13—C14	1.387 (3)
C2—H2	0.99 (2)	C13—H13	0.95 (2)
C3—C4	1.388 (3)	C14—C15	1.392 (2)
C3—H3	0.97 (2)	C14—H14	0.97 (2)
C4—C5	1.396 (2)	C15—C16	1.383 (2)
C4—H4	1.02 (2)	C15—H15	0.96 (2)
C7—O2—H2A	109.5	O1—C8—N1	126.03 (14)
C8—N1—C1	111.47 (13)	O1—C8—C7	125.72 (14)

C8—N1—H1	120.7 (14)	N1—C8—C7	108.20 (13)
C1—N1—H1	127.6 (14)	C16—C9—C10	102.45 (12)
C10—N2—C11	111.35 (13)	C16—C9—C7	113.88 (13)
C10—N2—H2B	123.3 (14)	C10—C9—C7	110.84 (12)
C11—N2—H2B	125.3 (14)	C16—C9—H9	114.1 (9)
C2—C1—C6	122.38 (15)	C10—C9—H9	108.2 (10)
C2—C1—N1	127.82 (14)	C7—C9—H9	107.2 (9)
C6—C1—N1	109.80 (14)	O3—C10—N2	125.26 (14)
C1—C2—C3	117.15 (16)	O3—C10—C9	126.29 (14)
C1—C2—H2	122.0 (11)	N2—C10—C9	108.44 (13)
C3—C2—H2	120.9 (11)	C12—C11—C16	122.28 (15)
C4—C3—C2	121.50 (16)	C12—C11—N2	128.31 (15)
C4—C3—H3	120.7 (12)	C16—C11—N2	109.41 (13)
C2—C3—H3	117.8 (12)	C11—C12—C13	117.30 (16)
C3—C4—C5	120.50 (16)	C11—C12—H12	120.7 (11)
C3—C4—H4	118.5 (11)	C13—C12—H12	121.9 (11)
C5—C4—H4	121.0 (11)	C14—C13—C12	121.20 (15)
C6—C5—C4	118.51 (15)	C14—C13—H13	118.8 (13)
C6—C5—H5	119.3 (11)	C12—C13—H13	119.9 (13)
C4—C5—H5	122.2 (11)	C13—C14—C15	120.69 (16)
C5—C6—C1	119.95 (15)	C13—C14—H14	120.5 (12)
C5—C6—C7	131.78 (14)	C15—C14—H14	118.8 (12)
C1—C6—C7	108.24 (13)	C16—C15—C14	118.65 (16)
O2—C7—C6	110.72 (12)	C16—C15—H15	120.3 (11)
O2—C7—C9	111.02 (12)	C14—C15—H15	121.0 (11)
C6—C7—C9	114.49 (12)	C15—C16—C11	119.85 (14)
O2—C7—C8	107.89 (12)	C15—C16—C9	131.84 (14)
C6—C7—C8	102.04 (12)	C11—C16—C9	108.31 (13)
C9—C7—C8	110.15 (12)		
C8—N1—C1—C2	175.44 (16)	C6—C7—C9—C16	67.76 (16)
C8—N1—C1—C6	-4.44 (19)	C8—C7—C9—C16	-177.95 (12)
C6—C1—C2—C3	0.4 (2)	O2—C7—C9—C10	-173.39 (12)
N1—C1—C2—C3	-179.48 (16)	C6—C7—C9—C10	-47.12 (17)
C1—C2—C3—C4	-0.3 (3)	C8—C7—C9—C10	67.17 (15)
C2—C3—C4—C5	-0.2 (3)	C11—N2—C10—O3	-179.33 (14)
C3—C4—C5—C6	0.4 (3)	C11—N2—C10—C9	1.48 (17)
C4—C5—C6—C1	-0.3 (2)	C16—C9—C10—O3	178.65 (15)
C4—C5—C6—C7	177.50 (16)	C7—C9—C10—O3	-59.5 (2)
C2—C1—C6—C5	-0.1 (2)	C16—C9—C10—N2	-2.16 (16)
N1—C1—C6—C5	179.79 (14)	C7—C9—C10—N2	119.69 (14)
C2—C1—C6—C7	-178.38 (15)	C10—N2—C11—C12	-179.90 (16)
N1—C1—C6—C7	1.50 (17)	C10—N2—C11—C16	-0.08 (18)
C5—C6—C7—O2	68.9 (2)	C16—C11—C12—C13	0.4 (2)
C1—C6—C7—O2	-113.13 (14)	N2—C11—C12—C13	-179.79 (16)
C5—C6—C7—C9	-57.6 (2)	C11—C12—C13—C14	0.6 (3)
C1—C6—C7—C9	120.45 (14)	C12—C13—C14—C15	-0.6 (3)
C5—C6—C7—C8	-176.54 (17)	C13—C14—C15—C16	-0.5 (3)

C1—C6—C7—C8	1.48 (15)	C14—C15—C16—C11	1.5 (2)
C1—N1—C8—O1	-177.15 (15)	C14—C15—C16—C9	-178.43 (16)
C1—N1—C8—C7	5.31 (17)	C12—C11—C16—C15	-1.5 (2)
O2—C7—C8—O1	-65.0 (2)	N2—C11—C16—C15	178.67 (14)
C6—C7—C8—O1	178.37 (15)	C12—C11—C16—C9	178.46 (15)
C9—C7—C8—O1	56.38 (19)	N2—C11—C16—C9	-1.37 (17)
O2—C7—C8—N1	112.59 (14)	C10—C9—C16—C15	-177.95 (17)
C6—C7—C8—N1	-4.09 (15)	C7—C9—C16—C15	62.3 (2)
C9—C7—C8—N1	-126.08 (13)	C10—C9—C16—C11	2.10 (16)
O2—C7—C9—C16	-58.50 (16)	C7—C9—C16—C11	-117.65 (14)

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

Cg1 is the centroid of the C1,C6,C7,C8,N1 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2A $\cdots$ O1 <sup>i</sup>	0.84	1.93	2.7575 (17)	167
N1—H1 $\cdots$ O3 <sup>ii</sup>	0.94 (2)	1.96 (2)	2.8644 (17)	161 (2)
N2—H2B $\cdots$ O3 <sup>iii</sup>	0.90 (3)	2.00 (3)	2.8882 (18)	173 (2)
C10—O3 $\cdots$ Cg1	1.24 (1)	2.92 (1)	3.0414 (13)	73 (1)

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