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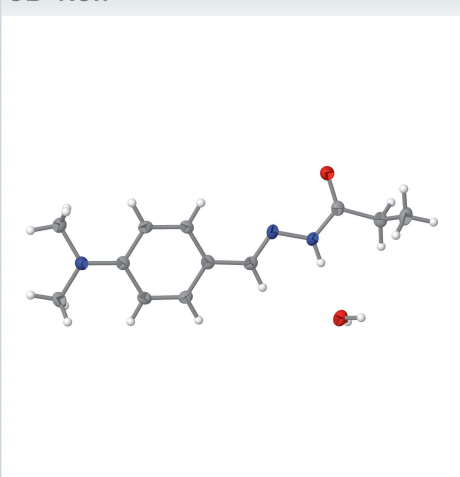
# (*E*)-*N*'-[4-(Dimethylamino)benzylidene]propionohydrazide monohydrate

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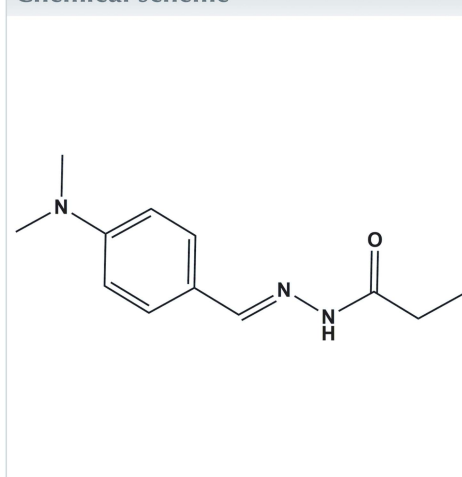
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In the title hydrated hydrazine compound, C<sub>12</sub>H<sub>17</sub>N<sub>3</sub>O·H<sub>2</sub>O, the C=N bond adopts an *E* conformation. In the crystal, water molecules bridge the hydrazine molecules, *via* N—H···O and O—H···O hydrogen bonds, forming sheets parallel to the *bc* plane. There are C—H··· $\pi$  interactions present within the sheets, and further C—H··· $\pi$  interactions link the sheets to form a three-dimensional structure.

## 3D view



## Chemical scheme



## Structure description

A number of industrial and biologically active compounds can be synthesized using Schiff bases as substrates *via* cycloaddition, ring closure and replacement reactions. In addition, Schiff bases are also known to have biological activities, such as antifungal (Singh & Dash, 1988), antimicrobial (El-Masry *et al.*, 2000) and antitumor (Desai *et al.*, 2001), and they have been used as herbicides. Schiff bases have also been employed as ligands for the complexation of metal ions (Aydogan *et al.*, 2001), since many of these complexes may be useful and serve as models for biologically important species (Dharmaraj *et al.*, 2001). In view of the importance of Schiff base hydrazones, we report herein on the synthesis and crystal structure of the title compound.

In the title compound (Fig. 1), the C9=N2 double bond adopts an *E* conformation. The methyleneformohydrazide unit [atoms O1/N2/N3/C9/C10; maximum deviation = 0.043 (1) Å for atom N2] is inclined to the benzene ring (C3–C8) by 8.94 (9)°. The solvent water molecule is linked to the hydrazine molecule by an N—H···O hydrogen bond (Fig. 1 and Table 1).

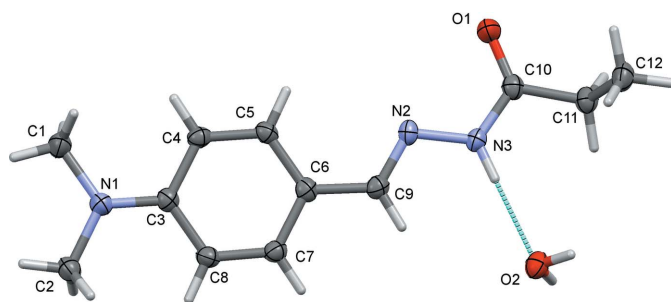
**Table 1**Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

Cg1 is the centroid of the C3–C8 ring.

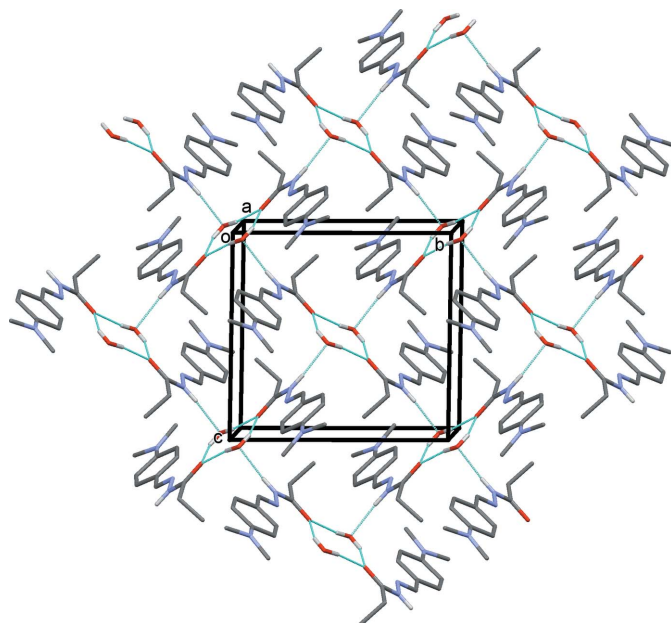
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N3-H8\cdots O2$	0.86	1.98	2.8330 (18)	175
$O2-H18\cdots O1^i$	0.83 (2)	2.21 (2)	2.9272 (18)	144 (2)
$O2-H18\cdots N2^i$	0.83 (2)	2.45 (2)	3.1684 (18)	145 (2)
$O2-H19\cdots O1^{ii}$	0.82 (3)	1.97 (2)	2.7845 (18)	172 (2)
$C1-H14\cdots Cg1^{iii}$	0.96	2.83	3.567 (2)	135
$C2-H16\cdots Cg1^{iv}$	0.96	2.91	3.701 (2)	140

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

In the crystal, water molecules bridge the hydrazine molecules *via*  $N-H\cdots O$  and  $O-H\cdots O$  hydrogen bonds, forming sheets parallel to the  $bc$  plane (Table 1 and Fig. 2). There are  $C-H\cdots\pi$  interactions present within the sheets, and further

**Figure 1**

A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level and the  $N-H\cdots O$  hydrogen bond is shown as a dashed line (see Table 1).

**Figure 2**

A view along the  $a$  axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1) and, for clarity, H atoms not involved in hydrogen bonding have been omitted.

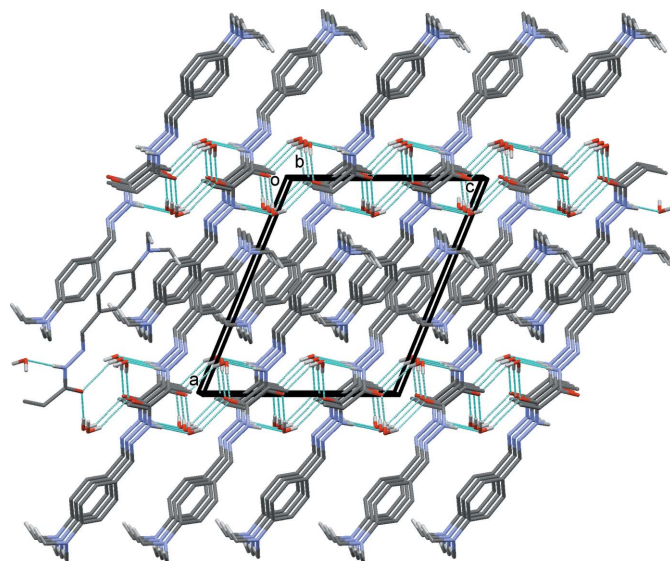
**Table 2**

Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{17}N_3O\cdot H_2O$
$M_r$	237.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
$a, b, c$ ( $\text{\AA}$ )	12.5756 (7), 10.5214 (6), 10.5737 (6)
$\beta$ ( $^\circ$ )	112.279 (3)
$V$ ( $\text{\AA}^3$ )	1294.60 (13)
$Z$	4
Radiation type	Cu $K\alpha$
$\mu$ ( $\text{mm}^{-1}$ )	0.69
Crystal size (mm)	$0.29 \times 0.26 \times 0.22$
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (SADABS; Bruker, 2013)
$T_{\min}, T_{\max}$	0.826, 0.864
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	10783, 2072, 1799
$R_{\text{int}}$	0.053
$(\sin \theta/\lambda)_{\text{max}}$ ( $\text{\AA}^{-1}$ )	0.581
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.043, 0.141, 1.11
No. of reflections	2072
No. of parameters	163
No. of restraints	3
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ ( $e \text{\AA}^{-3}$ )	0.21, $-0.24$

Computer programs: APEX2 and SAINT (Bruker, 2013), SHELXS97 and SHELXL97 (Sheldrick, 2008), PLATON (Spek, 2009) and Mercury (Macrae *et al.*, 2008).

$C-H\cdots\pi$  interactions link the sheets to form a three-dimensional structure (Table 1 and Fig. 3).

**Figure 3**

A view along the  $b$  axis of the crystal packing of the title compound. The hydrogen bonds are shown as dashed lines (see Table 1) and, for clarity, H atoms not involved in the various intermolecular interactions have been omitted.

### Synthesis and crystallization

A mixture of 4-(dimethylamino)benzaldehyde (0.01 mol) and hydrazine hydrate (0.01 mol) in 15 ml of propanoic acid was refluxed for *ca* 2 h. On cooling, the solid that separated was filtered off and recrystallized from dimethylformamide (DMF). Colourless block-like crystals were grown from DMF by slow evaporation of the solvent (yield 82%, m.p. 409 K).

### Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The water H atoms were located in a difference Fourier map and refined, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

### Acknowledgements

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## full crystallographic data

*IUCrData* (2016). **1**, x161716 [<https://doi.org/10.1107/S2414314616017168>]

**(*E*)-*N'*-[4-(Dimethylamino)benzylidene]propionohydrazide monohydrate**

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**(*E*)-*N'*-[4-(Dimethylamino)benzylidene]propionohydrazide monohydrate***Crystal data*

$C_{12}H_{17}N_3O \cdot H_2O$

$M_r = 237.30$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 12.5756$  (7) Å

$b = 10.5214$  (6) Å

$c = 10.5737$  (6) Å

$\beta = 112.279$  (3)°

$V = 1294.60$  (13) Å<sup>3</sup>

$Z = 4$

$F(000) = 512$

$D_x = 1.217$  Mg m<sup>-3</sup>

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

Cell parameters from 2072 reflections

$\theta = 3.8$ – $63.7^\circ$

$\mu = 0.69$  mm<sup>-1</sup>

$T = 296$  K

Block, colourless

$0.29 \times 0.26 \times 0.22$  mm

*Data collection*

Bruker X8 Proteum

diffractometer

Radiation source: Bruker MicroStar microfocus

rotating anode

Helios multilayer optics monochromator

Detector resolution: 18.4 pixels mm<sup>-1</sup>

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2013)

$T_{\min} = 0.826$ ,  $T_{\max} = 0.864$

10783 measured reflections

2072 independent reflections

1799 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.053$

$\theta_{\max} = 63.7^\circ$ ,  $\theta_{\min} = 3.8^\circ$

$h = -14 \rightarrow 14$

$k = -12 \rightarrow 11$

$l = -11 \rightarrow 12$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.141$

$S = 1.11$

2072 reflections

163 parameters

3 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

$w = 1/[\sigma^2(F_o^2) + (0.0887P)^2 + 0.250P]$

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

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

$\Delta\rho_{\max} = 0.21$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

*Special details*

**Geometry.** Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

**Refinement.** Refinement on  $F^2$  for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of  $F^2 > 2\sigma(F^2)$  is used only for calculating -R-factor-obs 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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.01204 (10)	0.62894 (12)	0.61531 (12)	0.0317 (4)
N1	0.66806 (12)	0.86484 (13)	0.57578 (14)	0.0262 (4)
N2	0.20753 (11)	0.76812 (13)	0.69793 (13)	0.0223 (4)
N3	0.12447 (11)	0.78061 (13)	0.75446 (13)	0.0223 (4)
C1	0.67145 (15)	0.78096 (18)	0.46817 (17)	0.0300 (5)
C2	0.74862 (15)	0.97087 (16)	0.61820 (18)	0.0276 (5)
C3	0.57562 (14)	0.86170 (15)	0.61455 (15)	0.0214 (5)
C4	0.49094 (14)	0.76498 (15)	0.56646 (15)	0.0218 (5)
C5	0.39942 (13)	0.75970 (15)	0.60754 (15)	0.0213 (5)
C6	0.38583 (14)	0.85007 (15)	0.69790 (15)	0.0215 (5)
C7	0.46896 (14)	0.94588 (15)	0.74484 (16)	0.0229 (5)
C8	0.56171 (14)	0.95285 (15)	0.70483 (16)	0.0232 (5)
C9	0.29009 (14)	0.84864 (15)	0.74319 (15)	0.0222 (5)
C10	0.03017 (14)	0.70707 (15)	0.70870 (16)	0.0235 (5)
C11	−0.05031 (14)	0.72381 (16)	0.78285 (17)	0.0261 (5)
C12	−0.03320 (16)	0.61625 (18)	0.88521 (18)	0.0314 (6)
O2	0.16326 (10)	0.94926 (11)	0.97640 (12)	0.0275 (4)
H1	0.67440	0.69420	0.49750	0.0450*
H2	0.04330	0.62020	0.95350	0.0470*
H3	0.80850	0.95850	0.58410	0.0410*
H4	0.49740	0.70410	0.50610	0.0260*
H5	0.34550	0.69500	0.57480	0.0260*
H6	0.61510	1.01800	0.73770	0.0280*
H7	0.46170	1.00680	0.80480	0.0270*
H8	0.13350	0.83510	0.81830	0.0270*
H9	−0.03550	0.80470	0.83030	0.0310*
H10	−0.12920	0.72380	0.71760	0.0310*
H11	−0.04420	0.53610	0.83860	0.0470*
H12	−0.08790	0.62460	0.92800	0.0470*
H13	0.28860	0.90830	0.80740	0.0270*
H14	0.60380	0.79370	0.38730	0.0450*
H15	0.73840	0.79950	0.44860	0.0450*
H16	0.70880	1.04870	0.58230	0.0410*
H17	0.78160	0.97520	0.71620	0.0410*
H18	0.1452 (18)	0.9041 (17)	1.0300 (19)	0.0410*

H19      0.1159 (16)      1.0074 (15)      0.955 (2)      0.0410\*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0339 (7)	0.0360 (7)	0.0292 (7)	−0.0111 (5)	0.0165 (5)	−0.0072 (5)
N1	0.0251 (7)	0.0296 (8)	0.0265 (8)	−0.0065 (6)	0.0126 (6)	−0.0058 (6)
N2	0.0233 (7)	0.0256 (7)	0.0212 (7)	0.0004 (5)	0.0120 (6)	0.0020 (5)
N3	0.0242 (7)	0.0253 (7)	0.0206 (7)	−0.0012 (5)	0.0120 (6)	−0.0019 (5)
C1	0.0277 (9)	0.0365 (10)	0.0292 (9)	−0.0041 (7)	0.0145 (7)	−0.0067 (7)
C2	0.0262 (9)	0.0271 (9)	0.0309 (9)	−0.0042 (7)	0.0125 (7)	0.0007 (7)
C3	0.0223 (8)	0.0228 (8)	0.0177 (8)	−0.0001 (6)	0.0061 (6)	0.0033 (6)
C4	0.0259 (9)	0.0204 (8)	0.0178 (8)	−0.0005 (6)	0.0068 (6)	−0.0007 (6)
C5	0.0218 (8)	0.0211 (8)	0.0192 (8)	−0.0027 (6)	0.0056 (6)	0.0018 (6)
C6	0.0231 (8)	0.0221 (8)	0.0184 (8)	0.0007 (6)	0.0069 (6)	0.0049 (6)
C7	0.0281 (9)	0.0209 (8)	0.0202 (8)	−0.0009 (6)	0.0098 (6)	−0.0012 (6)
C8	0.0252 (8)	0.0208 (8)	0.0226 (8)	−0.0042 (6)	0.0078 (6)	−0.0011 (6)
C9	0.0264 (9)	0.0217 (8)	0.0183 (8)	0.0012 (6)	0.0081 (6)	0.0020 (6)
C10	0.0254 (9)	0.0235 (8)	0.0227 (8)	0.0012 (6)	0.0104 (7)	0.0045 (6)
C11	0.0228 (9)	0.0281 (9)	0.0284 (9)	0.0024 (7)	0.0109 (7)	0.0015 (7)
C12	0.0354 (10)	0.0341 (10)	0.0323 (10)	0.0016 (8)	0.0216 (8)	0.0028 (7)
O2	0.0310 (7)	0.0269 (7)	0.0278 (7)	0.0054 (5)	0.0148 (5)	0.0026 (5)

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

O1—C10	1.238 (2)	C11—C12	1.524 (2)
O2—H18	0.83 (2)	C1—H1	0.9600
O2—H19	0.823 (18)	C1—H15	0.9600
N1—C1	1.453 (2)	C1—H14	0.9600
N1—C3	1.372 (2)	C2—H16	0.9600
N1—C2	1.459 (2)	C2—H17	0.9600
N2—N3	1.393 (2)	C2—H3	0.9600
N2—C9	1.284 (2)	C4—H4	0.9300
N3—C10	1.343 (2)	C5—H5	0.9300
N3—H8	0.8600	C7—H7	0.9300
C3—C4	1.420 (2)	C8—H6	0.9300
C3—C8	1.410 (2)	C9—H13	0.9300
C4—C5	1.377 (3)	C11—H10	0.9700
C5—C6	1.404 (2)	C11—H9	0.9700
C6—C7	1.401 (2)	C12—H12	0.9600
C6—C9	1.455 (3)	C12—H2	0.9600
C7—C8	1.385 (3)	C12—H11	0.9600
C10—C11	1.507 (3)		
H18—O2—H19	105 (2)	N1—C1—H15	109.00
C1—N1—C2	118.82 (15)	N1—C2—H3	109.00
C1—N1—C3	119.94 (14)	N1—C2—H16	109.00
C2—N1—C3	119.67 (14)	H3—C2—H16	109.00

N3—N2—C9	114.20 (13)	H3—C2—H17	109.00
N2—N3—C10	119.64 (13)	N1—C2—H17	109.00
N2—N3—H8	120.00	H16—C2—H17	109.00
C10—N3—H8	120.00	C5—C4—H4	119.00
C4—C3—C8	117.45 (16)	C3—C4—H4	119.00
N1—C3—C8	121.51 (15)	C4—C5—H5	119.00
N1—C3—C4	121.03 (14)	C6—C5—H5	119.00
C3—C4—C5	121.26 (15)	C6—C7—H7	119.00
C4—C5—C6	121.30 (15)	C8—C7—H7	119.00
C7—C6—C9	119.37 (14)	C7—C8—H6	120.00
C5—C6—C9	123.14 (15)	C3—C8—H6	120.00
C5—C6—C7	117.49 (16)	C6—C9—H13	119.00
C6—C7—C8	122.11 (15)	N2—C9—H13	119.00
C3—C8—C7	120.38 (15)	C10—C11—H9	110.00
N2—C9—C6	122.51 (14)	C12—C11—H9	110.00
O1—C10—N3	122.71 (17)	C12—C11—H10	110.00
N3—C10—C11	114.87 (14)	C10—C11—H10	110.00
O1—C10—C11	122.39 (16)	H9—C11—H10	108.00
C10—C11—C12	109.93 (15)	C11—C12—H11	109.00
N1—C1—H1	109.00	C11—C12—H12	109.00
N1—C1—H14	109.00	C11—C12—H2	109.00
H1—C1—H14	109.00	H2—C12—H12	110.00
H1—C1—H15	109.00	H11—C12—H12	109.00
H14—C1—H15	109.00	H2—C12—H11	109.00
C1—N1—C3—C4	9.6 (2)	C4—C3—C8—C7	0.6 (2)
C1—N1—C3—C8	−171.26 (15)	C3—C4—C5—C6	0.4 (2)
C2—N1—C3—C4	175.19 (14)	C4—C5—C6—C7	0.0 (2)
C2—N1—C3—C8	−5.7 (2)	C4—C5—C6—C9	179.55 (15)
C9—N2—N3—C10	175.73 (14)	C5—C6—C7—C8	0.0 (2)
N3—N2—C9—C6	179.26 (13)	C9—C6—C7—C8	−179.60 (15)
N2—N3—C10—O1	−1.4 (2)	C5—C6—C9—N2	−3.8 (2)
N2—N3—C10—C11	176.70 (13)	C7—C6—C9—N2	175.81 (15)
N1—C3—C4—C5	178.48 (15)	C6—C7—C8—C3	−0.3 (2)
C8—C3—C4—C5	−0.7 (2)	O1—C10—C11—C12	77.2 (2)
N1—C3—C8—C7	−178.52 (15)	N3—C10—C11—C12	−100.88 (17)

### Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C3—C8 ring.

<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>
N3—H8 $\cdots$ O2	0.86	1.98	2.8330 (18)	175
O2—H18 $\cdots$ O1 <sup>i</sup>	0.83 (2)	2.21 (2)	2.9272 (18)	144 (2)
O2—H18 $\cdots$ N2 <sup>i</sup>	0.83 (2)	2.45 (2)	3.1684 (18)	145 (2)
O2—H19 $\cdots$ O1 <sup>ii</sup>	0.82 (3)	1.97 (2)	2.7845 (18)	172 (2)

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C1—H14...Cg1 <sup>iii</sup>	0.96	2.83	3.567 (2)	135
C2—H16...Cg1 <sup>iv</sup>	0.96	2.91	3.701 (2)	140

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