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(*E*)-Methyl *N*′-[(1*H*-indol-3-yl)methylidene]hydrazinecarboxylate 0.25-hydrate

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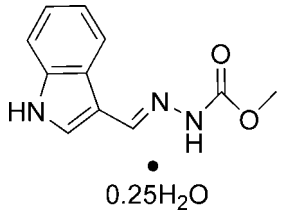
Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.124; data-to-parameter ratio = 13.6.

The asymmetric unit of the title compound, $C_{11}H_{11}N_3O_2\cdot 0.25H_2O$, contains two independent organic molecules and a water molecule, which lies on a twofold rotation axis. The side chains of the two molecules have slightly different orientations, the C=N—N—C torsion angle being −163.03 (15)° in one and −177.52 (14)° in the other, with each adopting a *trans* configuration with respect to the C=N bond. In the crystal, molecules are linked into chains extending along *b* by N—H···O, O—H···N and O—H···O hydrogen bonds and in addition, four intermolecular C—H···π interactions are present.

Related literature

For general background to Schiff bases, see: Cimerman *et al.* (1997); Offe *et al.* (1952); Richardson *et al.* (1988). For related structures, see: Shang *et al.* (2007); Tamboura *et al.* (2009).



Experimental

Crystal data

$C_{11}H_{11}N_3O_2\cdot 0.25H_2O$
M_r = 886.93
Monoclinic, $C2/c$
a = 27.842 (2) Å
b = 11.7574 (11) Å
c = 18.565 (2) Å
β = 130.558 (5)°
V = 4617.2 (8) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 223 K
0.21 × 0.17 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.977, T_{max} = 0.989
21333 measured reflections
4052 independent reflections
3083 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.124
S = 1.05
4052 reflections
298 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

*Cg*4 and *Cg*5 are the centroids of the C16–C19/N4 and C12–C17 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.15	2.9426 (18)	154
O1*W*—H1*WA*···N2^{ii}	0.93 (2)	2.19 (2)	3.0773 (17)	161 (2)
O1*W*—H1*WA*···O1^{ii}	0.93 (2)	2.60 (2)	3.2217 (14)	125 (2)
N6—H6···O1*W*	0.86	2.17	3.0087 (17)	166
C7—H7···*Cg*4^{iii}	0.93	2.85	3.594 (3)	137
C9—H9···*Cg*5^{iii}	0.93	2.74	3.537 (3)	145
C22—H22*A*···*Cg*4^{iv}	0.96	2.92	3.555 (4)	125
C22—H22*A*···*Cg*5^{iv}	0.96	2.86	3.796 (3)	125

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2120).

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supplementary materials

Acta Cryst. (2011). E**67**, o1956 [**doi:10.1107/S1600536811026249**]

(*E*)-Methyl *N*'-[(1*H*-indol-3-yl)methylidene]hydrazinecarboxylate 0.25-hydrate

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Comment

Schiff bases have attracted much attention due to their potential analytical applications (Cimerman *et al.*, 1997). They are also important ligands, which have been reported to have mild bacteriostatic activity and are used as potential oral iron-chelating drugs for genetic disorders such as thalassemia (Offe *et al.*, 1952; Richardson *et al.*, 1988). Metal complexes based on Schiff bases have received considerable attention because they can be utilized as model compounds of active centres in various complexes (Tamboura *et al.*, 2009). We report here the crystal structure of the title compound $C_{11}H_{11}N_3O_2$. $0.25H_2O$.

The title compound (Fig. 1) has two independent, but almost conformationally identical molecules in the asymmetric unit, together with a water molecule of solvation which lies on a twofold rotation axis. Each molecule adopts a *trans* configuration with respect to the C═ N bond. The N2/N3/O1/O2/C10/C11 and N5/N6/O3/O4/C21/C22 planes form dihedral angles of 20.39 (6)° and 16.57 (6)°, respectively, with the C1—C8/N1 and C12—C19/N4 planes. The dihedral angle between the two independent benzene rings is 64.06 (4)°. The bond lengths and angles are comparable to those observed for methyl *N*'-[(*E*)-4-methoxybenzylidene]hydrazinecarboxylate (Shang *et al.*, 2007).

The molecules are linked into chains extending along the *b* axis by N—H···O, O—H···N and O—H···O hydrogen bonds (Table 1 and Fig. 2). In addition, four intermolecular C—H···π interactions are present.

Experimental

1H-Indole-3-carbaldehyde (1.45 g, 0.01 mol) and methyl hydrazinecarboxylate (0.90g, 0.01 mol) were dissolved in stirred methanol (15 ml) and left for 3.5 h at room temperature. The resulting solid was filtered off and recrystallized from ethanol to give the title compound in 93% yield. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution at room temperature (m.p. 465–467 K).

Refinement

The water H atom was located in a difference Fourier and both positional and isotropic displacement parameters were refined. Other H atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93 or 0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{eq}(C_{methyl}). A rotating group model was used for the methyl H atoms.

Figures

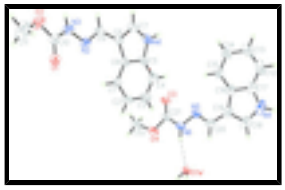

Fig. 1. The molecular conformation and atom numbering scheme for the two independent organic molecules and the water molecule of solvation in the asymmetric unit of the title compound. The water molecule lies on a twofold rotation axis and the hydrogen bond is shown as a dashed line. Displacement ellipsoids are drawn at the 30% probability level.

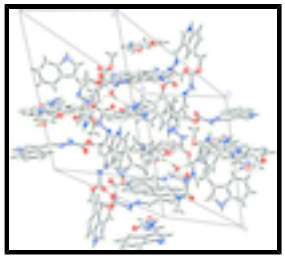

Fig. 2. Part of the crystal packing of the title compound in the unit cell. Hydrogen bonds are shown as dashed lines.

(*E*)-Methyl *N*'-[(1*H*-indol-3-yl)methylidene]hydrazinecarboxylate 0.25-hydrate

Crystal data

$C_{11}H_{11}N_3O_2 \cdot 0.25H_2O$	$F(000) = 1864$
$M_r = 886.93$	$D_x = 1.276$ Mg m^{-3}
Monoclinic, $C2/c$	Melting point = 465–467 K
Hall symbol: -C 2yc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 27.842$ (2) Å	Cell parameters from 4052 reflections
$b = 11.7574$ (11) Å	$\theta = 1.9$–$25.0°$
$c = 18.565$ (2) Å	$\mu = 0.09$ mm^{-1}
$\beta = 130.558$ (5)°	$T = 223$ K
$V = 4617.2$ (8) Å^3	Block, colourless
$Z = 16$	0.21 × 0.17 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4052 independent reflections
Radiation source: fine-focus sealed tube	3083 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.029$
φ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 1.9°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$h = -33 \rightarrow 33$
$T_{min} = 0.977$, $T_{max} = 0.989$	$k = -13 \rightarrow 13$
21333 measured reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.124$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0709P)^2 + 0.7592P]$ where $P = (F_o^2 + 2F_c^2)/3$
4052 reflections	$(\Delta/\sigma)_{max} < 0.001$
298 parameters	$\Delta\rho_{max} = 0.16$ e Å^{-3}

1 restraint $\Delta\rho_{min} = -0.19$ e Å^{-3}

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.37219 (7)	0.37491 (12)	0.56024 (11)	0.0531 (4)
H1A	0.4010	0.3162	0.5814	0.064*
C2	0.30987 (8)	0.35174 (15)	0.51235 (14)	0.0696 (5)
H2	0.2965	0.2765	0.5014	0.083*
C3	0.26622 (9)	0.43864 (17)	0.47977 (16)	0.0820 (6)
H3	0.2242	0.4201	0.4476	0.098*
C4	0.28342 (9)	0.55074 (16)	0.49370 (16)	0.0789 (5)
H4	0.2539	0.6085	0.4715	0.095*
C5	0.34667 (8)	0.57480 (12)	0.54227 (13)	0.0584 (4)
C6	0.39200 (7)	0.48869 (12)	0.57692 (10)	0.0475 (3)
C7	0.43931 (9)	0.65934 (14)	0.61492 (13)	0.0665 (5)
H7	0.4696	0.7161	0.6391	0.080*
C8	0.45160 (7)	0.54508 (13)	0.62345 (11)	0.0522 (4)
C9	0.51368 (7)	0.50072 (14)	0.67094 (11)	0.0552 (4)
H9	0.5470	0.5521	0.7009	0.066*
C10	0.60747 (8)	0.27236 (15)	0.71437 (11)	0.0607 (4)
C11	0.69308 (11)	0.1737 (2)	0.7468 (2)	0.1228 (10)
H11A	0.7380	0.1807	0.7843	0.184*
H11B	0.6730	0.1700	0.6809	0.184*
H11C	0.6839	0.1056	0.7644	0.184*
C12	0.10157 (13)	0.8858 (2)	0.10052 (18)	0.0940 (8)
H12	0.0845	0.9202	0.0432	0.113*
C13	0.15456 (13)	0.9284 (2)	0.1845 (2)	0.0928 (7)
H13	0.1743	0.9920	0.1842	0.111*
C14	0.17960 (9)	0.87807 (16)	0.27089 (15)	0.0760 (5)
H14	0.2158	0.9085	0.3271	0.091*
C15	0.15154 (7)	0.78446 (14)	0.27389 (12)	0.0591 (4)
H15	0.1682	0.7524	0.3318	0.071*
C16	0.09771 (8)	0.73753 (14)	0.18920 (11)	0.0575 (4)
C17	0.07411 (10)	0.78978 (18)	0.10343 (13)	0.0741 (5)
C18	0.01498 (10)	0.6371 (2)	0.06843 (15)	0.0889 (7)

H18	−0.0161	0.5817	0.0330	0.107*
C19	0.05859 (8)	0.63973 (15)	0.16554 (12)	0.0629 (4)
C20	0.06170 (8)	0.56012 (14)	0.22699 (14)	0.0649 (5)
H20	0.0370	0.4947	0.2013	0.078*
C21	0.13167 (8)	0.50520 (12)	0.46021 (14)	0.0601 (4)
C22	0.16560 (12)	0.40801 (19)	0.59737 (16)	0.0963 (7)
H22A	0.1588	0.3377	0.6159	0.144*
H22B	0.1545	0.4705	0.6174	0.144*
H22C	0.2093	0.4140	0.6265	0.144*
N1	0.37741 (8)	0.67819 (11)	0.56674 (12)	0.0742 (4)
H1	0.3599	0.7439	0.5534	0.089*
N2	0.52637 (6)	0.39465 (11)	0.67489 (9)	0.0563 (3)
N3	0.58987 (6)	0.37255 (13)	0.72462 (10)	0.0686 (4)
H3N	0.6178	0.4232	0.7620	0.082*
N4	0.02321 (9)	0.7252 (2)	0.03134 (11)	0.0964 (6)
H4A	0.0003	0.7393	−0.0282	0.116*
N5	0.09752 (6)	0.57712 (10)	0.31589 (11)	0.0603 (4)
N6	0.09703 (7)	0.49139 (11)	0.36687 (11)	0.0670 (4)
H6	0.0749	0.4308	0.3390	0.080*
O1	0.57234 (6)	0.19418 (10)	0.66782 (9)	0.0769 (4)
O2	0.66952 (6)	0.27132 (13)	0.76297 (11)	0.0956 (5)
O1W	0.0000	0.30761 (12)	0.2500	0.0550 (4)
O3	0.16253 (6)	0.58794 (9)	0.50562 (9)	0.0702 (3)
O4	0.12715 (6)	0.41067 (10)	0.49677 (10)	0.0790 (4)
H1WA	−0.0053 (11)	0.2590 (17)	0.2837 (14)	0.110 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0528 (9)	0.0430 (8)	0.0676 (10)	0.0006 (6)	0.0410 (8)	0.0033 (7)
C2	0.0606 (11)	0.0536 (10)	0.0968 (13)	−0.0079 (8)	0.0522 (11)	−0.0047 (9)
C3	0.0542 (11)	0.0768 (13)	0.1139 (16)	−0.0024 (9)	0.0541 (12)	−0.0064 (11)
C4	0.0640 (12)	0.0660 (11)	0.1093 (15)	0.0177 (9)	0.0574 (12)	0.0080 (10)
C5	0.0641 (11)	0.0464 (8)	0.0772 (11)	0.0035 (7)	0.0515 (10)	0.0014 (7)
C6	0.0533 (9)	0.0443 (8)	0.0554 (9)	0.0002 (6)	0.0401 (8)	0.0017 (6)
C7	0.0784 (12)	0.0476 (9)	0.0950 (13)	−0.0127 (8)	0.0659 (11)	−0.0078 (8)
C8	0.0590 (10)	0.0474 (8)	0.0620 (10)	−0.0076 (7)	0.0446 (9)	−0.0042 (7)
C9	0.0545 (10)	0.0576 (10)	0.0632 (10)	−0.0147 (7)	0.0425 (9)	−0.0093 (7)
C10	0.0535 (10)	0.0675 (11)	0.0551 (9)	0.0027 (8)	0.0327 (8)	0.0019 (8)
C11	0.0888 (17)	0.129 (2)	0.133 (2)	0.0305 (15)	0.0641 (16)	−0.0249 (17)
C12	0.123 (2)	0.1079 (18)	0.0932 (16)	0.0552 (16)	0.0886 (17)	0.0451 (14)
C13	0.1119 (19)	0.0849 (15)	0.130 (2)	0.0228 (13)	0.1002 (19)	0.0324 (14)
C14	0.0701 (12)	0.0744 (12)	0.0918 (14)	0.0033 (9)	0.0564 (11)	0.0109 (10)
C15	0.0549 (10)	0.0629 (10)	0.0582 (9)	0.0075 (7)	0.0362 (8)	0.0080 (8)
C16	0.0570 (10)	0.0627 (10)	0.0539 (9)	0.0217 (8)	0.0365 (8)	0.0073 (7)
C17	0.0815 (13)	0.0883 (14)	0.0601 (11)	0.0357 (11)	0.0494 (11)	0.0148 (10)
C18	0.0670 (13)	0.1005 (17)	0.0687 (13)	0.0171 (11)	0.0306 (11)	−0.0243 (12)
C19	0.0501 (9)	0.0642 (11)	0.0613 (10)	0.0093 (8)	0.0304 (9)	−0.0114 (8)

C20	0.0497 (10)	0.0529 (9)	0.0816 (13)	−0.0011 (7)	0.0380 (10)	−0.0145 (9)
C21	0.0554 (10)	0.0384 (8)	0.0911 (13)	−0.0019 (7)	0.0497 (10)	−0.0021 (8)
C22	0.1127 (18)	0.0771 (14)	0.0996 (17)	−0.0153 (12)	0.0692 (16)	0.0080 (11)
N1	0.0903 (11)	0.0386 (7)	0.1144 (12)	0.0056 (7)	0.0758 (11)	0.0035 (7)
N2	0.0464 (8)	0.0592 (8)	0.0643 (8)	−0.0069 (6)	0.0364 (7)	−0.0063 (6)
N3	0.0460 (8)	0.0722 (9)	0.0825 (10)	−0.0108 (7)	0.0394 (8)	−0.0202 (8)
N4	0.0883 (13)	0.1306 (17)	0.0476 (9)	0.0431 (12)	0.0341 (10)	0.0016 (10)
N5	0.0542 (8)	0.0436 (7)	0.0782 (10)	−0.0021 (6)	0.0410 (8)	−0.0039 (6)
N6	0.0647 (9)	0.0428 (7)	0.0899 (11)	−0.0131 (6)	0.0486 (9)	−0.0071 (7)
O1	0.0685 (8)	0.0574 (7)	0.0732 (8)	0.0012 (6)	0.0321 (7)	0.0016 (6)
O2	0.0550 (8)	0.1026 (11)	0.1118 (11)	0.0021 (7)	0.0465 (8)	−0.0286 (9)
O1W	0.0567 (9)	0.0410 (8)	0.0685 (10)	0.000	0.0412 (8)	0.000
O3	0.0713 (8)	0.0426 (6)	0.0865 (9)	−0.0090 (5)	0.0468 (7)	−0.0039 (6)
O4	0.0910 (10)	0.0490 (7)	0.1064 (11)	−0.0161 (6)	0.0684 (9)	−0.0004 (6)

Geometric parameters (Å, °)

C1—C2	1.367 (2)	C13—H13	0.9300
C1—C6	1.403 (2)	C14—C15	1.372 (2)
C1—H1A	0.9300	C14—H14	0.9300
C2—C3	1.390 (3)	C15—C16	1.399 (2)
C2—H2	0.9300	C15—H15	0.9300
C3—C4	1.368 (3)	C16—C17	1.409 (2)
C3—H3	0.9300	C16—C19	1.442 (2)
C4—C5	1.389 (2)	C17—N4	1.382 (3)
C4—H4	0.9300	C18—N4	1.344 (3)
C5—N1	1.382 (2)	C18—C19	1.371 (3)
C5—C6	1.406 (2)	C18—H18	0.9300
C6—C8	1.441 (2)	C19—C20	1.434 (3)
C7—N1	1.347 (2)	C20—N5	1.272 (2)
C7—C8	1.370 (2)	C20—H20	0.9300
C7—H7	0.9300	C21—O3	1.2055 (18)
C8—C9	1.434 (2)	C21—N6	1.336 (2)
C9—N2	1.285 (2)	C21—O4	1.3502 (19)
C9—H9	0.9300	C22—O4	1.427 (2)
C10—O1	1.2038 (19)	C22—H22A	0.9600
C10—O2	1.331 (2)	C22—H22B	0.9600
C10—N3	1.336 (2)	C22—H22C	0.9600
C11—O2	1.447 (3)	N1—H1	0.8600
C11—H11A	0.9600	N2—N3	1.3868 (18)
C11—H11B	0.9600	N3—H3N	0.8600
C11—H11C	0.9600	N4—H4A	0.8600
C12—C13	1.367 (3)	N5—N6	1.3887 (19)
C12—C17	1.384 (3)	N6—H6	0.8600
C12—H12	0.9300	O1W—H1WA	0.928 (15)
C13—C14	1.398 (3)		
C2—C1—C6	118.95 (14)	C13—C14—H14	119.5
C2—C1—H1A	120.5	C14—C15—C16	119.54 (16)
C6—C1—H1A	120.5	C14—C15—H15	120.2

C1—C2—C3	121.17 (16)	C16—C15—H15	120.2
C1—C2—H2	119.4	C15—C16—C17	118.01 (18)
C3—C2—H2	119.4	C15—C16—C19	134.57 (15)
C4—C3—C2	121.77 (17)	C17—C16—C19	107.38 (17)
C4—C3—H3	119.1	N4—C17—C12	130.9 (2)
C2—C3—H3	119.1	N4—C17—C16	106.6 (2)
C3—C4—C5	117.33 (16)	C12—C17—C16	122.4 (2)
C3—C4—H4	121.3	N4—C18—C19	110.9 (2)
C5—C4—H4	121.3	N4—C18—H18	124.6
N1—C5—C4	130.18 (15)	C19—C18—H18	124.6
N1—C5—C6	107.65 (14)	C18—C19—C20	125.10 (19)
C4—C5—C6	122.17 (15)	C18—C19—C16	105.46 (19)
C1—C6—C5	118.61 (14)	C20—C19—C16	129.43 (15)
C1—C6—C8	134.87 (14)	N5—C20—C19	121.37 (15)
C5—C6—C8	106.51 (13)	N5—C20—H20	119.3
N1—C7—C8	110.71 (14)	C19—C20—H20	119.3
N1—C7—H7	124.6	O3—C21—N6	126.17 (16)
C8—C7—H7	124.6	O3—C21—O4	124.73 (18)
C7—C8—C9	122.58 (14)	N6—C21—O4	109.10 (14)
C7—C8—C6	106.16 (14)	O4—C22—H22A	109.5
C9—C8—C6	131.26 (14)	O4—C22—H22B	109.5
N2—C9—C8	124.40 (14)	H22A—C22—H22B	109.5
N2—C9—H9	117.8	O4—C22—H22C	109.5
C8—C9—H9	117.8	H22A—C22—H22C	109.5
O1—C10—O2	124.77 (17)	H22B—C22—H22C	109.5
O1—C10—N3	124.98 (16)	C7—N1—C5	108.96 (13)
O2—C10—N3	110.25 (15)	C7—N1—H1	125.5
O2—C11—H11A	109.5	C5—N1—H1	125.5
O2—C11—H11B	109.5	C9—N2—N3	113.83 (13)
H11A—C11—H11B	109.5	C10—N3—N2	119.82 (14)
O2—C11—H11C	109.5	C10—N3—H3N	120.1
H11A—C11—H11C	109.5	N2—N3—H3N	120.1
H11B—C11—H11C	109.5	C18—N4—C17	109.66 (16)
C13—C12—C17	117.93 (19)	C18—N4—H4A	125.2
C13—C12—H12	121.0	C17—N4—H4A	125.2
C17—C12—H12	121.0	C20—N5—N6	115.05 (14)
C12—C13—C14	121.1 (2)	C21—N6—N5	118.48 (13)
C12—C13—H13	119.4	C21—N6—H6	120.8
C14—C13—H13	119.4	N5—N6—H6	120.8
C15—C14—C13	120.9 (2)	C10—O2—C11	115.48 (17)
C15—C14—H14	119.5	C21—O4—C22	116.13 (15)
C6—C1—C2—C3	0.4 (3)	C15—C16—C17—C12	−1.0 (2)
C1—C2—C3—C4	0.2 (3)	C19—C16—C17—C12	−179.00 (16)
C2—C3—C4—C5	−0.2 (3)	N4—C18—C19—C20	177.95 (16)
C3—C4—C5—N1	−179.76 (19)	N4—C18—C19—C16	−1.3 (2)
C3—C4—C5—C6	−0.5 (3)	C15—C16—C19—C18	−176.42 (17)
C2—C1—C6—C5	−1.0 (2)	C17—C16—C19—C18	1.06 (17)
C2—C1—C6—C8	179.73 (16)	C15—C16—C19—C20	4.4 (3)
N1—C5—C6—C1	−179.47 (14)	C17—C16—C19—C20	−178.17 (15)

C4—C5—C6—C1	1.1 (2)	C18—C19—C20—N5	−169.80 (16)
N1—C5—C6—C8	−0.04 (17)	C16—C19—C20—N5	9.3 (3)
C4—C5—C6—C8	−179.45 (17)	C8—C7—N1—C5	0.4 (2)
N1—C7—C8—C9	179.48 (14)	C4—C5—N1—C7	179.13 (19)
N1—C7—C8—C6	−0.42 (19)	C6—C5—N1—C7	−0.2 (2)
C1—C6—C8—C7	179.56 (17)	C8—C9—N2—N3	179.82 (14)
C5—C6—C8—C7	0.28 (17)	O1—C10—N3—N2	−4.1 (3)
C1—C6—C8—C9	−0.3 (3)	O2—C10—N3—N2	175.87 (15)
C5—C6—C8—C9	−179.62 (16)	C9—N2—N3—C10	−163.03 (15)
C7—C8—C9—N2	−175.11 (16)	C19—C18—N4—C17	1.1 (2)
C6—C8—C9—N2	4.8 (3)	C12—C17—N4—C18	178.02 (19)
C17—C12—C13—C14	−1.2 (3)	C16—C17—N4—C18	−0.4 (2)
C12—C13—C14—C15	−0.3 (3)	C19—C20—N5—N6	−178.25 (13)
C13—C14—C15—C16	1.1 (3)	O3—C21—N6—N5	2.6 (2)
C14—C15—C16—C17	−0.5 (2)	O4—C21—N6—N5	−177.00 (13)
C14—C15—C16—C19	176.80 (17)	C20—N5—N6—C21	−177.52 (14)
C13—C12—C17—N4	−176.32 (18)	O1—C10—O2—C11	8.4 (3)
C13—C12—C17—C16	1.8 (3)	N3—C10—O2—C11	−171.5 (2)
C15—C16—C17—N4	177.52 (13)	O3—C21—O4—C22	−4.5 (2)
C19—C16—C17—N4	−0.45 (17)	N6—C21—O4—C22	175.15 (16)

Hydrogen-bond geometry (Å, °)

Cg4 and Cg5 are the centroids of the C16–C19/N4 and C12–C17 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.86	2.15	2.9426 (18)	154.
O1W—H1WA···N2^{ii}	0.93 (2)	2.19 (2)	3.0773 (17)	161.(2)
O1W—H1WA···O1^{ii}	0.93 (2)	2.60 (2)	3.2217 (14)	125.(2)
N6—H6···O1W	0.86	2.17	3.0087 (17)	166.
C7—H7···Cg4^{iii}	0.93	2.85	3.594 (3)	137
C9—H9···Cg5^{iii}	0.93	2.74	3.537 (3)	145
C22—H22A···Cg4^{iv}	0.96	2.92	3.555 (4)	125
C22—H22A···Cg5^{iv}	0.96	2.86	3.796 (3)	125

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

Fig. 1

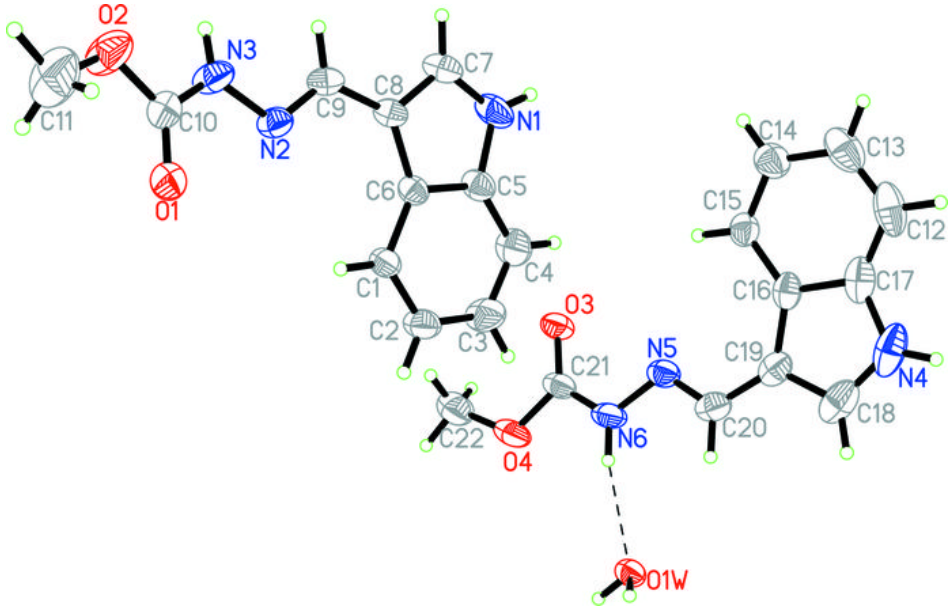
O2
N3
C11
C10
C9
C8
C7
N1
N2
O1
C6
C5
C14
C13
C15
C12
C16
C17
C4
C1
C2
C3
O3
C21
N5
C19
N4
C22
O4
N6
C20
C18
O1W

Fig. 2