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3-(2-Amino-1-methyl-4-oxo-4,5-dihydro-1H-imidazol-5-yl)-3-hydroxyindolin-2-one monohydrate

Narsimha Reddy Penthala,^a Thirupathi Reddy Yerram Reddy,^a Sean Parkin^b and Peter A. Crooks^{a*}

^aDepartment of Pharmaceutical Sciences, College of Pharmacy, University of Kentucky, Lexington, KY 40536, USA, and ^bDepartment of Chemistry, University of Kentucky, Lexington, KY 40506, USA
Correspondence e-mail: pcrooks@email.uky.edu

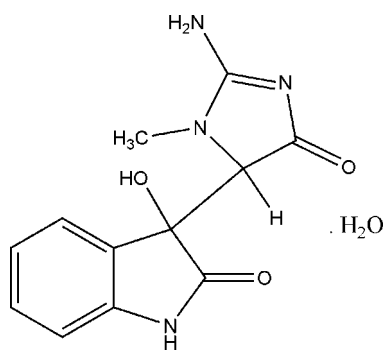
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Key indicators: single-crystal X-ray study; $T = 90$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.034; wR factor = 0.088; data-to-parameter ratio = 11.4.

Two chiral centres exist in the title compound, $\text{C}_{12}\text{H}_{12}\text{N}_4\text{O}_3 \cdot \text{H}_2\text{O}$. Molecules are linked into chains by series of intermolecular $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds, which causes supramolecular aggregation. Two chiral centres are formed in the title compound. The indole and creatinine moieties make a dihedral angle of 56.75 (4)°. The crystal structure of the compound indicates the presence of equimolar enantiomers (*RR* and *SS*) in the crystal structure.

Related literature

For 2-indol-3-yl-methylenequinuclidin-3-ols NADPH oxidase activity, see: Sekhar *et al.* (2003). For novel substituted (*Z*)-2-(*N*-benzylindol-3-ylmethylene)quinuclidin-3-one and (*Z*)-(\pm)-2-(*N*-benzylindol-3-ylmethylene)quinuclidin-3-ol derivatives as potent thermal sensitizing agents, see: Sonar *et al.* (2007). For the crystal and molecular structure of isatin, see: Frolova *et al.* (1988). For the structure of 1,1'-diacetyl-3-hydroxy-2,2',3,3'-tetrahydro-3,3'-bi(1*H*-indole)-2,2'-dione, see: Usman *et al.* (2002). The aldol condensation enolate mechanism by six-membered transition states has been described by Zimmerman & Traxler (1957).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{12}\text{N}_4\text{O}_3 \cdot \text{H}_2\text{O}$
 $M_r = 278.27$
Monoclinic, $P2_1/n$
 $a = 8.3514$ (1) Å
 $b = 10.7166$ (2) Å
 $c = 13.9679$ (2) Å
 $\beta = 104.755$ (1)°

$V = 1208.88$ (3) Å³
 $Z = 4$
Cu $K\alpha$ radiation
 $\mu = 0.99$ mm⁻¹
 $T = 90$ K
 $0.20 \times 0.15 \times 0.06$ mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2006)
 $T_{\min} = 0.780$, $T_{\max} = 0.943$

17255 measured reflections
2181 independent reflections
2121 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.088$
 $S = 1.04$
2181 reflections
191 parameters
3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.26$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|--|------------|--------------|--------------|----------------|
| $\text{N1}-\text{H1} \cdots \text{O1W}^i$ | 0.88 | 1.96 | 2.8128 (15) | 163 |
| $\text{O8}-\text{H8} \cdots \text{N12}^{ii}$ | 0.84 | 1.97 | 2.7984 (14) | 170 |
| $\text{N11}-\text{H11A} \cdots \text{O13}^{iii}$ | 0.88 | 2.17 | 3.0371 (15) | 171 |
| $\text{N11}-\text{H11B} \cdots \text{O1}^{iv}$ | 0.88 | 2.13 | 2.8678 (15) | 141 |
| $\text{O1W}-\text{H1W} \cdots \text{O8}$ | 0.847 (18) | 2.049 (18) | 2.8812 (14) | 167.4 (19) |
| $\text{O1W}-\text{H2W} \cdots \text{O13}^v$ | 0.861 (18) | 2.233 (18) | 3.0702 (14) | 164.1 (19) |

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *APEX2* and *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* and local procedures.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2475).

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supporting information

Acta Cryst. (2009). E65, o552 [doi:10.1107/S1600536809004875]

3-(2-Amino-1-methyl-4-oxo-4,5-dihydro-1*H*-imidazol-5-yl)-3-hydroxyindolin-2-one monohydrate

Narsimha Reddy Penthala, Thirupathi Reddy Yerram Reddy, Sean Parkin and Peter A. Crooks

S1. Comment

In our endeavor to design and synthesize novel radiosensitizers such as (*Z*)-2-(*N*-benzylindol-3-ylmethylene)quinuclidin-3-one and (*Z*)-(\pm)-2-(*N*-benzylindol-3-ylmethylene)quinuclidin-3-ol derivatives (Sekhar *et al.*, 2003; Sonar *et al.*, 2007), we have undertaken the design, synthesis and structural analysis of a series of 3-(2-amino-1-methyl-4-oxo-4,5-dihydro-1*H*-imidazol-5-yl)-3-hydroxyindolin-2-one analogs with different substituents on the indole moiety. The primary goal for X-ray analysis of the title compound is to confirm the stereochemistry of the molecule and to obtain detailed information on the structural conformation that may be useful in structure–activity relationship (SAR) analysis. The title compound was prepared by the aldol condensation of indol-2,3-dione (isatin) with 2-amino-1-methyl-1*H*-imidazol-4(5*H*)-one (creatinine) in the presence of sodium acetate in acetic acid under microwave irradiation. The compound was crystallized from 2% aqueous glycol. This aldol condensation reaction proceeds by the formation of the *E*-enolate, as per the Zimmerman–Traxler model (Zimmerman & Traxler, 1957), which favors *anti* products, and leads to the formation of equimolar *RR* and *SS* enantiomers. The molecular structure and the atom-numbering scheme are shown in Fig. 1. The isatin ring is planar (r.m.s. deviation = 0.0112 (10) Å) with bond distances and angles comparable with those previously reported for other isatin derivatives (Frolova *et al.*, 1988; Usman *et al.*, 2002). Atoms C₈ and C₉ are the two chiral centers of the title compound. The X-ray studies revealed that the obtained compound is racemic (having equimolar *RR* and *SS* enantiomers). The indole and creatinine moieties make a dihedral angle of 56.75 (4)°. Intermolecular N—H⋯O and O—H⋯N hydrogen bonds stabilize the crystal structure and form a supramolecular architecture.

S2. Experimental

A mixture of isatin (1 mmol), creatinine (1.1 mmol) and sodium acetate (1.2 mmol) in acetic acid (1 ml) were irradiated in a domestic microwave oven for 40 sec with intermittent cooling every 10 sec. The reaction mixture was allowed to cool to room temperature, 10 ml of saturated sodium bicarbonate solution was added, and the mixture was stirred for 10 minutes. The precipitate thus obtained was collected by filtration, washed with cold water and dried, to afford the crude product. Crystallization from 2% aqueous glycol gave a light yellow crystalline product of 3-(2-amino-1-methyl-4-oxo-4,5-dihydro-1*H*-imidazol-5-yl)-3-hydroxyindolin-2-one monohydrate that was suitable for X-ray analysis. ¹H NMR (DMSO-*d*₆): δ 3.13 (*s*, 3H), 4.06 (*s*, 1H), 6.37 (*s*, 1H, OH), 6.73–6.75 (*d*, *J* = 7.5 Hz, 1H), 6.84–6.89 (*t*, *J* = 7.5 Hz, 1H), 7.04–7.06 (*d*, *J* = 7.5 Hz, 1H), 7.15–7.21 (*t*, *J* = 7.8 Hz, 1H), 7.51 (*bs*, 2H, NH₂), 10.23 (*s*, 1H, NH) p.p.m.; ¹³C NMR (DMSO-*d*₆): δ 32.59, 69.44, 76.28, 109.49, 121.1, 123.95, 127.98, 129.34, 142.66, 171.76, 175.71, 182.26 p.p.m.

S3. Refinement

Non-water H atoms were found in difference Fourier maps and subsequently placed in idealized positions with constrained distances of 0.98 Å (RCH₃), 1.00 Å (R₃CH), 0.95 Å (C_{Ar}H), 0.84 Å (O—H) and 0.88 Å (N—H) distances. $U_{\text{iso}}(\text{H})$ values set to either 1.2 U_{eq} or 1.5 U_{eq} (RCH₃, OH) of the attached atom. The water H atoms were refined subject to distance and angle restraints and assigned $U_{\text{iso}}(\text{H})$ values of 1.5 U_{eq} of the water oxygen atom.

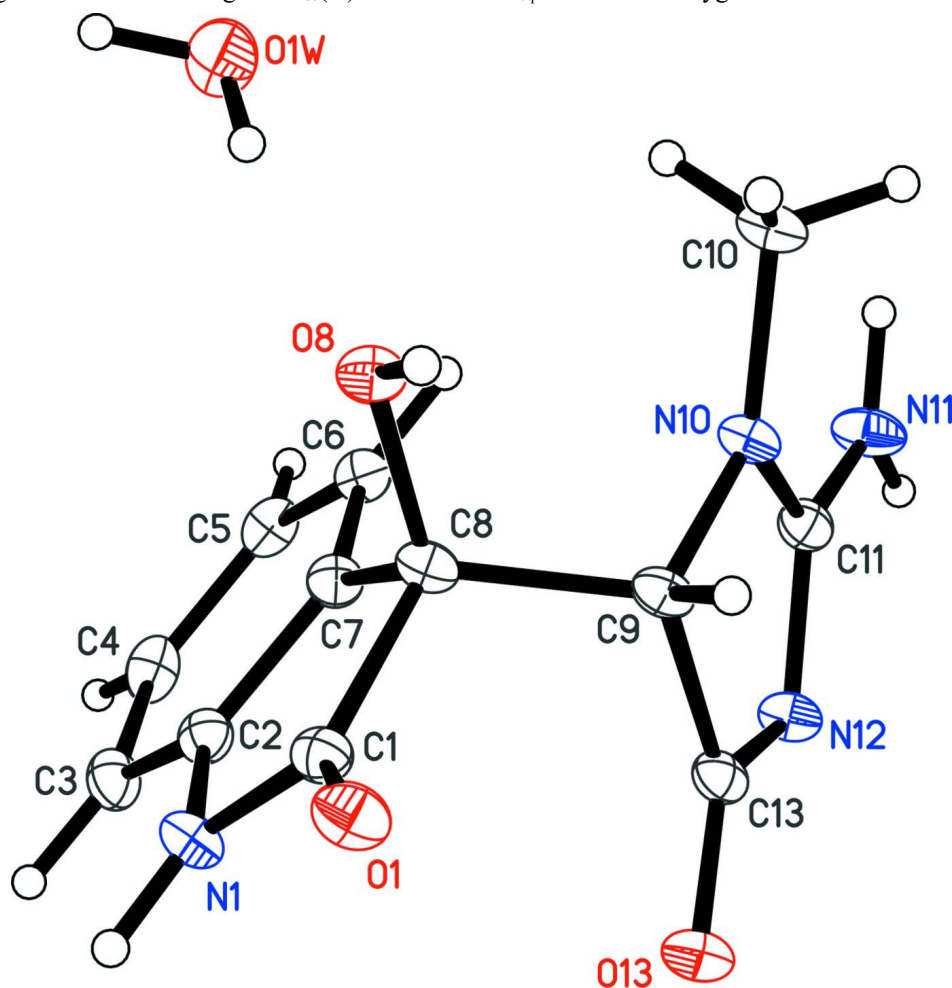


Figure 1

A view of the molecule with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

3-(2-Amino-1-methyl-4-oxo-4,5-dihydro-1*H*-imidazol-5-yl)-3-hydroxyindolin-2-one monohydrate*Crystal data*C₁₂H₁₂N₄O₃·H₂O $M_r = 278.27$ Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

 $a = 8.3514$ (1) Å $b = 10.7166$ (2) Å $c = 13.9679$ (2) Å $\beta = 104.755$ (1)° $V = 1208.88$ (3) Å³ $Z = 4$ $F(000) = 584$ $D_x = 1.529$ Mg m⁻³Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å

Cell parameters from 9952 reflections

 $\theta = 5.3$ – 68.0 ° $\mu = 0.99$ mm⁻¹ $T = 90$ K

Block, colourless

 $0.20 \times 0.15 \times 0.06$ mm

Data collection

Bruker X8 Proteum
diffractometer

Radiation source: fine-focus rotating anode

Graded multilayer optics monochromator

Detector resolution: 5.6 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(*SADABS* in *APEX2*; Bruker, 2006)

$T_{\min} = 0.780$, $T_{\max} = 0.943$

17255 measured reflections

2181 independent reflections

2121 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 68.0^\circ$, $\theta_{\min} = 5.3^\circ$

$h = -10 \rightarrow 10$

$k = -12 \rightarrow 11$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.088$

$S = 1.04$

2181 reflections

191 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

$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.777P]$

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

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

$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kFc[1 + 0.001x\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0029 (7)

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|---------------|--------------|--------------|----------------------------------|
| O1 | -0.09392 (12) | 0.10429 (9) | 0.10391 (7) | 0.0184 (2) |
| N1 | -0.15262 (14) | 0.29911 (10) | 0.15528 (8) | 0.0158 (3) |
| H1 | -0.2381 | 0.3179 | 0.1060 | 0.019* |
| C1 | -0.06677 (16) | 0.19095 (12) | 0.16253 (9) | 0.0146 (3) |
| C2 | -0.08770 (16) | 0.37782 (12) | 0.23678 (9) | 0.0150 (3) |
| C3 | -0.14037 (16) | 0.49625 (13) | 0.25289 (10) | 0.0183 (3) |
| H3 | -0.2307 | 0.5349 | 0.2072 | 0.022* |
| C4 | -0.05487 (17) | 0.55663 (13) | 0.33926 (10) | 0.0192 (3) |
| H4 | -0.0875 | 0.6383 | 0.3527 | 0.023* |
| C5 | 0.07679 (16) | 0.49981 (13) | 0.40589 (10) | 0.0179 (3) |
| H5 | 0.1332 | 0.5433 | 0.4640 | 0.021* |
| C6 | 0.12728 (16) | 0.37988 (12) | 0.38869 (9) | 0.0161 (3) |
| H6 | 0.2163 | 0.3405 | 0.4348 | 0.019* |
| C7 | 0.04456 (16) | 0.31943 (12) | 0.30268 (9) | 0.0143 (3) |

| | | | | |
|------|--------------|--------------|-------------|------------|
| O8 | 0.03982 (11) | 0.09668 (8) | 0.32447 (6) | 0.0154 (2) |
| H8 | 0.0481 | 0.0270 | 0.2986 | 0.023* |
| C8 | 0.07032 (16) | 0.19298 (12) | 0.26163 (9) | 0.0142 (3) |
| C9 | 0.24325 (16) | 0.17641 (12) | 0.24175 (9) | 0.0134 (3) |
| H9 | 0.2513 | 0.0945 | 0.2090 | 0.016* |
| C10 | 0.41472 (17) | 0.10072 (12) | 0.41347 (9) | 0.0171 (3) |
| H10A | 0.5352 | 0.0920 | 0.4366 | 0.026* |
| H10B | 0.3655 | 0.0195 | 0.3909 | 0.026* |
| H10C | 0.3705 | 0.1309 | 0.4678 | 0.026* |
| N10 | 0.37459 (13) | 0.18921 (10) | 0.33208 (8) | 0.0138 (3) |
| C11 | 0.45660 (16) | 0.29528 (12) | 0.32705 (9) | 0.0148 (3) |
| N11 | 0.57702 (14) | 0.33716 (11) | 0.40073 (8) | 0.0195 (3) |
| H11A | 0.6067 | 0.2948 | 0.4564 | 0.023* |
| H11B | 0.6276 | 0.4075 | 0.3942 | 0.023* |
| N12 | 0.40787 (14) | 0.35577 (10) | 0.23870 (8) | 0.0156 (3) |
| O13 | 0.21974 (12) | 0.30271 (8) | 0.09262 (7) | 0.0174 (2) |
| C13 | 0.28724 (16) | 0.28445 (12) | 0.18112 (9) | 0.0140 (3) |
| O1W | 0.03730 (13) | 0.14500 (11) | 0.52700 (8) | 0.0256 (3) |
| H1W | 0.023 (2) | 0.1260 (19) | 0.4666 (13) | 0.038* |
| H2W | -0.058 (2) | 0.1453 (19) | 0.5402 (14) | 0.038* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|-------------|-------------|
| O1 | 0.0209 (5) | 0.0161 (5) | 0.0156 (5) | -0.0002 (4) | -0.0002 (4) | -0.0028 (4) |
| N1 | 0.0151 (5) | 0.0148 (6) | 0.0149 (5) | 0.0014 (4) | -0.0010 (4) | 0.0007 (4) |
| C1 | 0.0150 (6) | 0.0141 (6) | 0.0140 (6) | -0.0015 (5) | 0.0025 (5) | 0.0010 (5) |
| C2 | 0.0149 (6) | 0.0147 (6) | 0.0155 (6) | -0.0017 (5) | 0.0043 (5) | 0.0001 (5) |
| C3 | 0.0161 (6) | 0.0153 (7) | 0.0238 (7) | 0.0021 (5) | 0.0058 (5) | 0.0026 (5) |
| C4 | 0.0205 (7) | 0.0123 (6) | 0.0278 (7) | -0.0005 (5) | 0.0117 (6) | -0.0025 (5) |
| C5 | 0.0201 (7) | 0.0169 (7) | 0.0182 (7) | -0.0046 (5) | 0.0079 (5) | -0.0044 (5) |
| C6 | 0.0170 (6) | 0.0160 (7) | 0.0153 (6) | -0.0013 (5) | 0.0040 (5) | -0.0002 (5) |
| C7 | 0.0157 (6) | 0.0120 (6) | 0.0151 (6) | -0.0003 (5) | 0.0038 (5) | 0.0003 (5) |
| O8 | 0.0198 (5) | 0.0106 (5) | 0.0151 (5) | -0.0008 (4) | 0.0032 (4) | 0.0000 (3) |
| C8 | 0.0164 (7) | 0.0116 (6) | 0.0128 (6) | 0.0004 (5) | 0.0007 (5) | 0.0007 (5) |
| C9 | 0.0155 (6) | 0.0111 (6) | 0.0115 (6) | 0.0006 (5) | -0.0004 (5) | -0.0004 (5) |
| C10 | 0.0201 (7) | 0.0139 (6) | 0.0146 (6) | 0.0003 (5) | -0.0006 (5) | 0.0034 (5) |
| N10 | 0.0153 (5) | 0.0113 (5) | 0.0124 (5) | -0.0002 (4) | -0.0009 (4) | 0.0013 (4) |
| C11 | 0.0156 (6) | 0.0132 (6) | 0.0147 (6) | 0.0008 (5) | 0.0026 (5) | -0.0001 (5) |
| N11 | 0.0228 (6) | 0.0159 (6) | 0.0156 (6) | -0.0059 (5) | -0.0029 (5) | 0.0029 (4) |
| N12 | 0.0186 (6) | 0.0129 (5) | 0.0134 (5) | -0.0008 (4) | 0.0006 (4) | 0.0011 (4) |
| O13 | 0.0215 (5) | 0.0154 (5) | 0.0128 (5) | 0.0004 (4) | -0.0003 (4) | 0.0010 (3) |
| C13 | 0.0157 (6) | 0.0116 (6) | 0.0140 (6) | 0.0030 (5) | 0.0024 (5) | 0.0000 (5) |
| O1W | 0.0214 (5) | 0.0353 (6) | 0.0184 (5) | -0.0051 (4) | 0.0019 (4) | -0.0057 (4) |

Geometric parameters (Å, °)

| | | | |
|----------|-------------|---------------|-------------|
| O1—C1 | 1.2206 (16) | C8—C9 | 1.5488 (18) |
| N1—C1 | 1.3531 (17) | C9—N10 | 1.4522 (16) |
| N1—C2 | 1.4096 (17) | C9—C13 | 1.5334 (17) |
| N1—H1 | 0.8800 | C9—H9 | 1.0000 |
| C1—C8 | 1.5557 (17) | C10—N10 | 1.4525 (16) |
| C2—C3 | 1.3803 (19) | C10—H10A | 0.9800 |
| C2—C7 | 1.3932 (18) | C10—H10B | 0.9800 |
| C3—C4 | 1.395 (2) | C10—H10C | 0.9800 |
| C3—H3 | 0.9500 | N10—C11 | 1.3382 (17) |
| C4—C5 | 1.387 (2) | C11—N11 | 1.3209 (17) |
| C4—H4 | 0.9500 | C11—N12 | 1.3612 (17) |
| C5—C6 | 1.3925 (19) | N11—H11A | 0.8800 |
| C5—H5 | 0.9500 | N11—H11B | 0.8800 |
| C6—C7 | 1.3847 (18) | N12—C13 | 1.3540 (17) |
| C6—H6 | 0.9500 | O13—C13 | 1.2366 (16) |
| C7—C8 | 1.5080 (17) | O1W—H1W | 0.847 (18) |
| O8—C8 | 1.4192 (15) | O1W—H2W | 0.861 (18) |
| O8—H8 | 0.8400 | | |
| | | | |
| C1—N1—C2 | 111.39 (11) | C7—C8—C1 | 101.94 (10) |
| C1—N1—H1 | 124.3 | C9—C8—C1 | 110.33 (10) |
| C2—N1—H1 | 124.3 | N10—C9—C13 | 100.04 (10) |
| O1—C1—N1 | 126.67 (12) | N10—C9—C8 | 111.46 (10) |
| O1—C1—C8 | 125.20 (11) | C13—C9—C8 | 112.21 (10) |
| N1—C1—C8 | 108.08 (10) | N10—C9—H9 | 110.9 |
| C3—C2—C7 | 122.50 (12) | C13—C9—H9 | 110.9 |
| C3—C2—N1 | 127.51 (12) | C8—C9—H9 | 110.9 |
| C7—C2—N1 | 109.99 (11) | N10—C10—H10A | 109.5 |
| C2—C3—C4 | 116.96 (12) | N10—C10—H10B | 109.5 |
| C2—C3—H3 | 121.5 | H10A—C10—H10B | 109.5 |
| C4—C3—H3 | 121.5 | N10—C10—H10C | 109.5 |
| C5—C4—C3 | 121.36 (12) | H10A—C10—H10C | 109.5 |
| C5—C4—H4 | 119.3 | H10B—C10—H10C | 109.5 |
| C3—C4—H4 | 119.3 | C11—N10—C9 | 108.54 (10) |
| C4—C5—C6 | 120.82 (12) | C11—N10—C10 | 125.20 (11) |
| C4—C5—H5 | 119.6 | C9—N10—C10 | 126.23 (10) |
| C6—C5—H5 | 119.6 | N11—C11—N10 | 123.09 (12) |
| C7—C6—C5 | 118.39 (12) | N11—C11—N12 | 122.48 (12) |
| C7—C6—H6 | 120.8 | N10—C11—N12 | 114.41 (11) |
| C5—C6—H6 | 120.8 | C11—N11—H11A | 120.0 |
| C6—C7—C2 | 119.96 (12) | C11—N11—H11B | 120.0 |
| C6—C7—C8 | 131.47 (12) | H11A—N11—H11B | 120.0 |
| C2—C7—C8 | 108.56 (11) | C13—N12—C11 | 105.97 (11) |
| C8—O8—H8 | 109.5 | O13—C13—N12 | 125.82 (12) |
| O8—C8—C7 | 110.67 (10) | O13—C13—C9 | 124.01 (11) |
| O8—C8—C9 | 110.48 (10) | N12—C13—C9 | 110.17 (10) |

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|-------------|--------------|-----------------|--------------|
| C7—C8—C9 | 113.64 (10) | H1W—O1W—H2W | 108.3 (17) |
| O8—C8—C1 | 109.44 (10) | | |
| C2—N1—C1—O1 | 179.49 (12) | O1—C1—C8—C9 | 59.80 (16) |
| C2—N1—C1—C8 | 1.73 (14) | N1—C1—C8—C9 | -122.39 (11) |
| C1—N1—C2—C3 | 177.98 (13) | O8—C8—C9—N10 | -64.03 (13) |
| C1—N1—C2—C7 | -1.42 (15) | C7—C8—C9—N10 | 61.06 (14) |
| C7—C2—C3—C4 | -0.05 (19) | C1—C8—C9—N10 | 174.83 (10) |
| N1—C2—C3—C4 | -179.38 (12) | O8—C8—C9—C13 | -175.30 (10) |
| C2—C3—C4—C5 | -0.16 (19) | C7—C8—C9—C13 | -50.22 (14) |
| C3—C4—C5—C6 | -0.3 (2) | C1—C8—C9—C13 | 63.55 (13) |
| C4—C5—C6—C7 | 1.00 (19) | C13—C9—N10—C11 | 8.33 (13) |
| C5—C6—C7—C2 | -1.19 (19) | C8—C9—N10—C11 | -110.49 (12) |
| C5—C6—C7—C8 | 178.47 (13) | C13—C9—N10—C10 | -169.97 (11) |
| C3—C2—C7—C6 | 0.7 (2) | C8—C9—N10—C10 | 71.21 (15) |
| N1—C2—C7—C6 | -179.82 (11) | C9—N10—C11—N11 | 175.73 (12) |
| C3—C2—C7—C8 | -179.00 (12) | C10—N10—C11—N11 | -5.9 (2) |
| N1—C2—C7—C8 | 0.44 (14) | C9—N10—C11—N12 | -5.46 (15) |
| C6—C7—C8—O8 | 64.51 (18) | C10—N10—C11—N12 | 172.86 (11) |
| C2—C7—C8—O8 | -115.79 (12) | N11—C11—N12—C13 | 178.05 (12) |
| C6—C7—C8—C9 | -60.47 (18) | N10—C11—N12—C13 | -0.77 (15) |
| C2—C7—C8—C9 | 119.23 (12) | C11—N12—C13—O13 | -174.16 (12) |
| C6—C7—C8—C1 | -179.17 (13) | C11—N12—C13—C9 | 6.41 (14) |
| C2—C7—C8—C1 | 0.53 (13) | N10—C9—C13—O13 | 171.44 (12) |
| O1—C1—C8—O8 | -61.96 (16) | C8—C9—C13—O13 | -70.29 (16) |
| N1—C1—C8—O8 | 115.85 (11) | N10—C9—C13—N12 | -9.12 (13) |
| O1—C1—C8—C7 | -179.17 (12) | C8—C9—C13—N12 | 109.15 (12) |
| N1—C1—C8—C7 | -1.36 (13) | | |

Hydrogen-bond geometry (Å, °)

| <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> |
|-------------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1...O1W ⁱ | 0.88 | 1.96 | 2.8128 (15) | 163 |
| O8—H8...N12 ⁱⁱ | 0.84 | 1.97 | 2.7984 (14) | 170 |
| N11—H11A...O13 ⁱⁱⁱ | 0.88 | 2.17 | 3.0371 (15) | 171 |
| N11—H11B...O1 ^{iv} | 0.88 | 2.13 | 2.8678 (15) | 141 |
| O1W—H1W...O8 | 0.85 (2) | 2.05 (2) | 2.8812 (14) | 167 (2) |
| O1W—H2W...O13 ^v | 0.86 (2) | 2.23 (2) | 3.0702 (14) | 164 (2) |

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