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# 1*H*-Imidazo[4,5-*f*][1,10]phenanthrolin-7-ium perchlorate monohydrate

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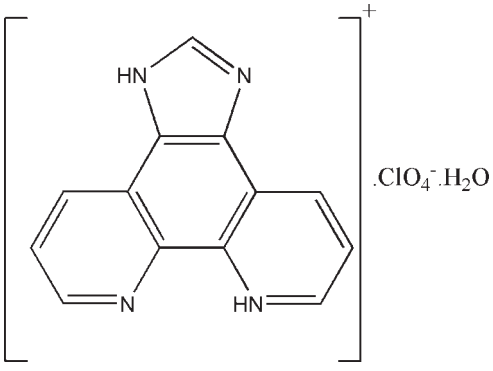
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.057; $wR$ factor = 0.197; data-to-parameter ratio = 12.2.

In the title crystal structure, $C_{13}H_9N_4^+\cdot ClO_4^-\cdot H_2O$, cations, anions and water molecules are linked through intermolecular N—H···O, O—H···N and O—H···O hydrogen bonds, forming layers parallel to (001). In addition, there are weak $\pi$–$\pi$ stacking interactions between the layers, involving the cations and with centroid–centroid distances in the range 3.584 (2)–3.662 (2) Å, forming a three-dimensional network.

## Related literature

For background to 1*H*-imidazo[4,5-*f*][1,10]-phenanthroline and its use as a molecular building block, see: Xiong *et al.* (1999); Yu *et al.* (2009); Liu *et al.* (2009).




## Experimental

### Crystal data

$C_{13}H_9N_4^+\cdot ClO_4^-\cdot H_2O$ $M_r$ = 338.71
Monoclinic, $P2_1/c$
$a$ = 11.401 (2) Å
$b$ = 18.475 (3) Å
$c$ = 6.7163 (13) Å
$\beta$ = 90.179 (3)°
$V$ = 1414.7 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.30 mm$^{-1}$
$T$ = 298 K
0.30 × 0.26 × 0.17 mm

### Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.914, $T_{max}$ = 0.950
7051 measured reflections
2534 independent reflections
1734 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.197
$S$ = 1.01
2534 reflections
208 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O1$W^{i}$ | 0.86 | 1.90 | 2.713 (4) | 156 |
| N3—H3$A$···O3$^{ii}$ | 0.86 | 1.99 | 2.825 (4) | 162 |
| O1$W$—H1$WB$···N4 | 0.84 | 2.02 | 2.852 (4) | 177 |
| O1$W$—H1$WA$···O2 | 0.84 | 2.25 | 3.018 (5) | 154 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2890).

## References

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, J. Q., Zhang, Y. N., Wang, Y. Y., Jin, J. C., Lermontova, E. K. & Shi, Q. Z. (2009). *Dalton Trans.* pp. 5365–5378.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xiong, Y., He, X. F., Zhou, X. H., Wu, J. Z., Chen, X. M., Ji, L. N., Li, R. H., Zhou, J. Y. & Yu, K. B. (1999). *J. Chem. Soc. Dalton Trans.* pp. 19–24.
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# supporting information

*Acta Cryst.* (2009). E**65**, o2332 [doi:10.1107/S1600536809034576]

## 1*H*-Imidazo[4,5-*f*][1,10]phenanthrolin-7-ium perchlorate monohydrate

**Su-Mei Shen**

### S1. Comment

1H-imidazo[4,5-f][1,10]-phenanthroline (IP) is an important derivative of 1,10-phenanthroline that has been used to recognize the secondary structure of DNA in an Ru(II) complex (Xiong *et al.*, 1999). IP is a good molecular building block and has been used to construct some interesting structures (Yu *et al.*, 2009, Liu *et al.*, 2009). In an attempt to form a Zn(II) complex with IP, we adventitiously formed the title compound (I) and its crystal structure is determined herein.

The asymmetric unit of (I) is shown in Fig 1. In the crystal structure N-H···O, O-H···N and O-H···O hydrogen bonds link cations, water molecules and perchlorate anions into a 2-D network (Fig. 2). Details of the hydrogen-bonding geometry are given in Table 1. In addition, there are weak $\pi$–$\pi$ stacking interactions between layers, involving cations with centroid to centroid distances in the range 3.584 (2)-3.662 (2)Å forming a three-dimensional network.

### S2. Experimental

IP (0.23 mg,0.1 mmol), $Zn(ClO_4)_2$ (0.27 mg, 0.1 mmol), were dissolved in methanol. The mixture was heated and stirred for ten hours under reflux. The resulting solid was then filtered off to give a pure solution which was treated with diethyl ether in a closed vessel. Five weeks later, single crystals were obtained.

### S3. Refinement

All H atoms were visible in difference Fourier maps but were subsequently placed in calculated positions treated as riding with C—H = 0.93, N—H == 0.86Å and with $U_{iso}(H) = 1.2U_{eq}(C,N)$. The H atoms of the water molecules were included in the subsequent refinement with O-H = 0.84Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

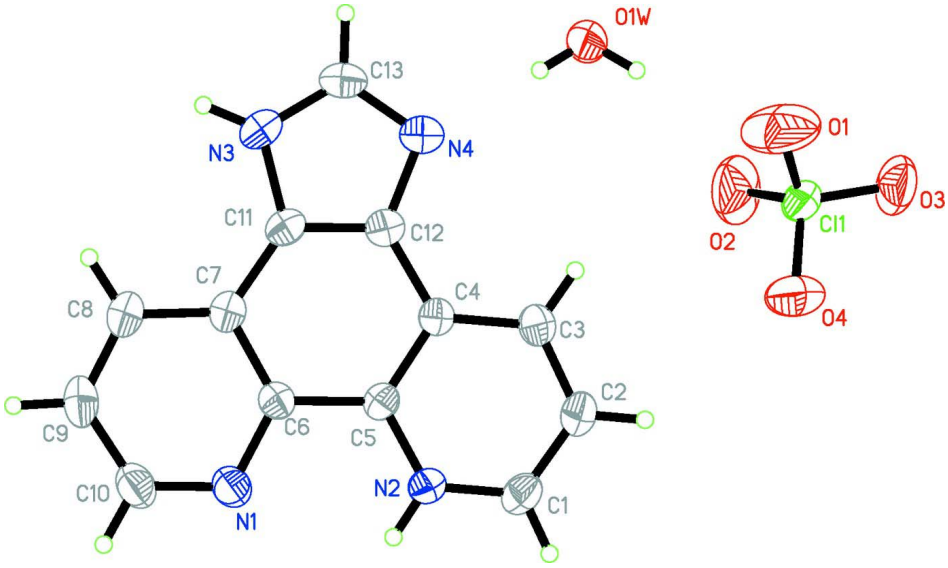


**Figure 1**

The asymmetric unit of (I) with displacement ellipdoids drawn at the the 30% probability level. H atoms are shown as spheres of arbitrary radii.

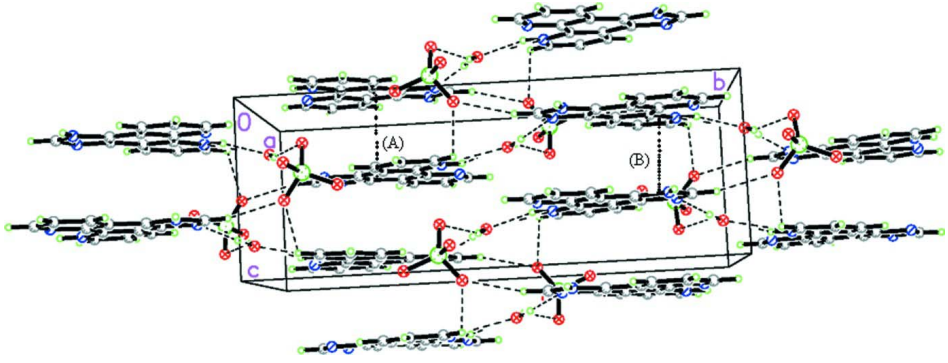


**Figure 2**

Part of the crystal structure of (I). Hydrogen bonds are drawn as dashed lines and $\pi$–$\pi$ stacking interactions are denoted by dashed lines along with labels (A) and (B).

### 1*H*-Imidazo[4,5-*f*][1,10]phenanthrolin-7-ium perchlorate monohydrate

*Crystal data*

$C_{13}H_9N_4^+\cdot ClO_4^-\cdot H_2O$
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$c = 6.7163\ (13)$ Å
$\beta = 90.179\ (3)°$
$V = 1414.7\ (4)$ Å$^3$
$Z = 4$
$F(000) = 696$
$D_x = 1.590$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2521 reflections
$\theta = 1.8–25.2°$
$\mu = 0.30$ mm$^{-1}$
$T = 298$ K
Block, colorless
0.30 × 0.26 × 0.17 mm

*Data collection*

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.914, $T_{max}$ = 0.950
7051 measured reflections
2534 independent reflections
1734 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.031
$\theta_{max}$ = 25.2°, $\theta_{min}$ = 1.8°
$h$ = −13→11
$k$ = −21→22
$l$ = −7→8

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.197
$S$ = 1.01
2534 reflections
208 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1261P)^2 + 0.1912P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.7565 (2) | 0.41426 (15) | 0.3687 (4) | 0.0563 (7) |
| N2 | 0.5235 (2) | 0.38399 (13) | 0.3439 (4) | 0.0494 (7) |
| H2A | 0.5510 | 0.4273 | 0.3390 | 0.059* |
| N3 | 0.8115 (3) | 0.14741 (15) | 0.3782 (4) | 0.0539 (7) |
| H3A | 0.8855 | 0.1388 | 0.3832 | 0.065* |
| N4 | 0.6190 (3) | 0.12600 (14) | 0.3669 (4) | 0.0537 (7) |
| C1 | 0.4079 (3) | 0.37511 (19) | 0.3383 (5) | 0.0583 (9) |
| H1 | 0.3590 | 0.4153 | 0.3301 | 0.070* |
| C2 | 0.3602 (3) | 0.30690 (19) | 0.3445 (5) | 0.0577 (9) |
| H2 | 0.2792 | 0.3008 | 0.3435 | 0.069* |
| C3 | 0.4328 (3) | 0.24803 (18) | 0.3521 (4) | 0.0508 (8) |
| H3 | 0.4011 | 0.2016 | 0.3527 | 0.061* |
| C4 | 0.5552 (2) | 0.25749 (16) | 0.3589 (4) | 0.0423 (7) |
| C5 | 0.5997 (2) | 0.32858 (16) | 0.3569 (4) | 0.0424 (7) |
| C6 | 0.7246 (3) | 0.34396 (16) | 0.3670 (4) | 0.0437 (7) |
| C7 | 0.8053 (3) | 0.28627 (17) | 0.3736 (4) | 0.0463 (7) |

| | | | | |
|---|---|---|---|---|
| C8 | 0.9257 (3) | 0.3041 (2) | 0.3816 (5) | 0.0575 (9) |
| H8 | 0.9826 | 0.2681 | 0.3859 | 0.069* |
| C9 | 0.9563 (3) | 0.3750 (2) | 0.3827 (5) | 0.0676 (10) |
| H9 | 1.0350 | 0.3880 | 0.3883 | 0.081* |
| C10 | 0.8704 (3) | 0.4282 (2) | 0.3753 (5) | 0.0656 (10) |
| H10 | 0.8942 | 0.4763 | 0.3751 | 0.079* |
| C11 | 0.7577 (3) | 0.21528 (16) | 0.3725 (4) | 0.0462 (7) |
| C12 | 0.6393 (3) | 0.20018 (16) | 0.3648 (4) | 0.0451 (7) |
| C13 | 0.7259 (3) | 0.09872 (19) | 0.3745 (5) | 0.0584 (9) |
| H13 | 0.7402 | 0.0492 | 0.3770 | 0.070* |
| Cl1 | 0.15290 (7) | 0.11627 (5) | 0.40411 (14) | 0.0643 (4) |
| O1 | 0.2077 (4) | 0.07296 (19) | 0.5458 (6) | 0.1414 (17) |
| O2 | 0.2199 (3) | 0.1137 (2) | 0.2286 (6) | 0.1356 (15) |
| O3 | 0.0402 (2) | 0.08894 (19) | 0.3635 (5) | 0.0990 (10) |
| O4 | 0.1451 (3) | 0.18729 (16) | 0.4828 (6) | 0.1099 (12) |
| O1W | 0.4428 (2) | 0.02582 (13) | 0.2434 (5) | 0.0871 (9) |
| H1WB | 0.4934 | 0.0564 | 0.2771 | 0.131* |
| H1WA | 0.3755 | 0.0409 | 0.2713 | 0.131* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0558 (17) | 0.0523 (17) | 0.0608 (16) | −0.0063 (13) | −0.0055 (13) | 0.0023 (12) |
| N2 | 0.0472 (15) | 0.0436 (15) | 0.0572 (16) | 0.0054 (11) | −0.0031 (12) | 0.0011 (11) |
| N3 | 0.0536 (16) | 0.0575 (17) | 0.0506 (15) | 0.0156 (14) | 0.0007 (12) | 0.0021 (12) |
| N4 | 0.0655 (18) | 0.0469 (16) | 0.0488 (15) | 0.0036 (13) | 0.0019 (13) | 0.0006 (11) |
| C1 | 0.050 (2) | 0.064 (2) | 0.061 (2) | 0.0124 (16) | −0.0055 (15) | −0.0016 (15) |
| C2 | 0.0421 (17) | 0.069 (2) | 0.062 (2) | 0.0021 (16) | −0.0014 (15) | −0.0016 (16) |
| C3 | 0.0482 (18) | 0.057 (2) | 0.0470 (17) | −0.0054 (15) | −0.0012 (13) | 0.0004 (14) |
| C4 | 0.0434 (17) | 0.0471 (17) | 0.0364 (15) | −0.0003 (13) | 0.0031 (12) | −0.0003 (12) |
| C5 | 0.0421 (16) | 0.0479 (17) | 0.0372 (15) | 0.0035 (13) | −0.0024 (12) | 0.0004 (12) |
| C6 | 0.0469 (17) | 0.0445 (17) | 0.0396 (15) | −0.0030 (13) | −0.0012 (12) | 0.0032 (11) |
| C7 | 0.0434 (16) | 0.057 (2) | 0.0381 (15) | 0.0013 (14) | 0.0004 (12) | 0.0020 (12) |
| C8 | 0.0431 (18) | 0.071 (2) | 0.0581 (19) | 0.0031 (16) | −0.0005 (14) | 0.0023 (16) |
| C9 | 0.044 (2) | 0.086 (3) | 0.074 (2) | −0.0140 (18) | −0.0041 (17) | 0.0049 (19) |
| C10 | 0.062 (2) | 0.059 (2) | 0.076 (2) | −0.0161 (18) | −0.0063 (18) | 0.0054 (17) |
| C11 | 0.0507 (18) | 0.0493 (19) | 0.0386 (15) | 0.0098 (14) | 0.0016 (13) | 0.0023 (12) |
| C12 | 0.0511 (18) | 0.0476 (18) | 0.0366 (15) | 0.0031 (14) | 0.0033 (12) | 0.0001 (12) |
| C13 | 0.078 (2) | 0.0406 (18) | 0.0570 (19) | 0.0052 (18) | −0.0009 (17) | −0.0016 (14) |
| Cl1 | 0.0453 (5) | 0.0575 (6) | 0.0902 (7) | 0.0079 (4) | −0.0048 (4) | −0.0043 (4) |
| O1 | 0.157 (4) | 0.089 (2) | 0.178 (4) | 0.033 (2) | −0.096 (3) | 0.005 (2) |
| O2 | 0.100 (3) | 0.167 (4) | 0.140 (3) | −0.013 (2) | 0.059 (2) | −0.016 (3) |
| O3 | 0.0470 (15) | 0.117 (2) | 0.133 (3) | −0.0055 (15) | −0.0056 (16) | −0.028 (2) |
| O4 | 0.107 (3) | 0.0571 (18) | 0.166 (3) | 0.0161 (16) | −0.005 (2) | −0.0134 (19) |
| O1W | 0.0592 (16) | 0.0560 (16) | 0.146 (3) | −0.0021 (12) | 0.0052 (16) | −0.0208 (15) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C10 | 1.324 (4) | C5—C6 | 1.454 (4) |
| N1—C6 | 1.349 (4) | C6—C7 | 1.408 (4) |
| N2—C1 | 1.329 (4) | C7—C8 | 1.413 (4) |
| N2—C5 | 1.345 (4) | C7—C11 | 1.419 (4) |
| N2—H2A | 0.8600 | C8—C9 | 1.355 (5) |
| N3—C13 | 1.327 (4) | C8—H8 | 0.9300 |
| N3—C11 | 1.396 (4) | C9—C10 | 1.388 (5) |
| N3—H3A | 0.8600 | C9—H9 | 0.9300 |
| N4—C13 | 1.320 (4) | C10—H10 | 0.9300 |
| N4—C12 | 1.390 (4) | C11—C12 | 1.379 (4) |
| C1—C2 | 1.373 (5) | C13—H13 | 0.9300 |
| C1—H1 | 0.9300 | Cl1—O1 | 1.390 (3) |
| C2—C3 | 1.367 (5) | Cl1—O3 | 1.406 (3) |
| C2—H2 | 0.9300 | Cl1—O2 | 1.407 (4) |
| C3—C4 | 1.408 (4) | Cl1—O4 | 1.417 (3) |
| C3—H3 | 0.9300 | O1W—H1WB | 0.8379 |
| C4—C5 | 1.408 (4) | O1W—H1WA | 0.8377 |
| C4—C12 | 1.429 (4) | | |
| | | | |
| C10—N1—C6 | 116.9 (3) | C6—C7—C11 | 116.7 (3) |
| C1—N2—C5 | 123.2 (3) | C8—C7—C11 | 126.0 (3) |
| C1—N2—H2A | 118.4 | C9—C8—C7 | 118.4 (3) |
| C5—N2—H2A | 118.4 | C9—C8—H8 | 120.8 |
| C13—N3—C11 | 106.6 (3) | C7—C8—H8 | 120.8 |
| C13—N3—H3A | 126.7 | C8—C9—C10 | 120.2 (3) |
| C11—N3—H3A | 126.7 | C8—C9—H9 | 119.9 |
| C13—N4—C12 | 102.9 (3) | C10—C9—H9 | 119.9 |
| N2—C1—C2 | 120.3 (3) | N1—C10—C9 | 123.7 (3) |
| N2—C1—H1 | 119.8 | N1—C10—H10 | 118.1 |
| C2—C1—H1 | 119.8 | C9—C10—H10 | 118.1 |
| C3—C2—C1 | 119.4 (3) | C12—C11—N3 | 104.4 (3) |
| C3—C2—H2 | 120.3 | C12—C11—C7 | 124.2 (3) |
| C1—C2—H2 | 120.3 | N3—C11—C7 | 131.4 (3) |
| C2—C3—C4 | 120.2 (3) | C11—C12—N4 | 111.2 (3) |
| C2—C3—H3 | 119.9 | C11—C12—C4 | 120.5 (3) |
| C4—C3—H3 | 119.9 | N4—C12—C4 | 128.3 (3) |
| C3—C4—C5 | 118.2 (3) | N4—C13—N3 | 114.9 (3) |
| C3—C4—C12 | 125.0 (3) | N4—C13—H13 | 122.6 |
| C5—C4—C12 | 116.8 (3) | N3—C13—H13 | 122.6 |
| N2—C5—C4 | 118.6 (3) | O1—Cl1—O3 | 109.5 (2) |
| N2—C5—C6 | 119.1 (3) | O1—Cl1—O2 | 108.1 (3) |
| C4—C5—C6 | 122.3 (3) | O3—Cl1—O2 | 108.9 (2) |
| N1—C6—C7 | 123.5 (3) | O1—Cl1—O4 | 107.8 (2) |
| N1—C6—C5 | 116.9 (3) | O3—Cl1—O4 | 110.2 (2) |
| C7—C6—C5 | 119.5 (3) | O2—Cl1—O4 | 112.2 (2) |
| C6—C7—C8 | 117.3 (3) | H1WB—O1W—H1WA | 110.3 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···O1*W*$^{i}$ | 0.86 | 1.90 | 2.713 (4) | 156 |
| N3—H3*A*···O3$^{ii}$ | 0.86 | 1.99 | 2.825 (4) | 162 |
| O1*W*—H1*WB*···N4 | 0.84 | 2.02 | 2.852 (4) | 177 |
| O1*W*—H1*WA*···O2 | 0.84 | 2.25 | 3.018 (5) | 154 |

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