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# Poly[[triaqua( $\mu_3$ -4-oxidopyridine-2,6-dicarboxylato)holmium(III)] mono-hydrate]

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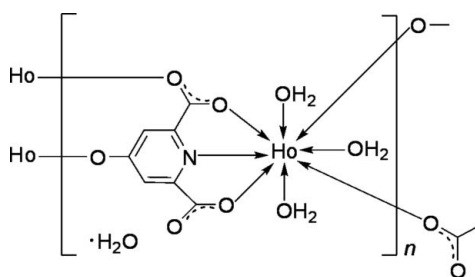
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; R factor = 0.021; wR factor = 0.047; data-to-parameter ratio = 10.2.

In the title coordination polymer,  $[\{\text{Ho}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3\} \cdot (\text{H}_2\text{O})]_n$ , the  $\text{Ho}^{\text{III}}$  atom is eight-coordinated by a tridentate 4-oxidopyridine-2,6-dicarboxylate trianion, two monodentate anions and three water molecules, forming a distorted bicapped trigonal-prismatic  $\text{HoNO}_7$  coordination geometry. The anions bridge adjacent  $\text{Ho}^{\text{III}}$  ions into double chains. Adjacent chains are further connected into sheets.  $\text{O}-\text{H} \cdots \text{O}$  hydrogen bonds involving both coordinated and uncoordinated water molecules generate a three-dimensional supramolecular framework.

## Related literature

For the structures and properties of lanthanide coordination compounds, see: Wang *et al.* (2007); Lv *et al.* (2010); Gao *et al.* (2006). For bond lengths and angles in other complexes with eight-coordinate  $\text{Ho}^{\text{III}}$ , see: Wang *et al.* (2007); Munoz *et al.* (2005).



## Experimental

### Crystal data

$[\text{Ho}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3] \cdot \text{H}_2\text{O}$   
 $M_r = 417.09$   
 Monoclinic,  $P2_1/n$   
 $a = 9.879$  (5) Å  
 $b = 7.557$  (4) Å  
 $c = 15.386$  (8) Å  
 $\beta = 105.386$  (5)°  
 $V = 1107.5$  (9) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 7.19$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.32 \times 0.29 \times 0.26$  mm

### Data collection

Bruker APEXII CCD diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2004)  
 $T_{\text{min}} = 0.207$ ,  $T_{\text{max}} = 0.257$   
 4776 measured reflections  
 2003 independent reflections  
 1646 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.021$   
 $wR(F^2) = 0.047$   
 $S = 1.02$   
 2003 reflections  
 196 parameters  
 12 restraints  
 H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.71$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.54$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O9}-\text{H8W} \cdots \text{O2}^i$	0.86 (1)	2.33 (4)	3.080 (5)	145 (6)
$\text{O9}-\text{H7W} \cdots \text{O4}$	0.86 (1)	1.84 (1)	2.693 (5)	170 (5)
$\text{O8}-\text{H6W} \cdots \text{O9}^{\text{ii}}$	0.86 (1)	2.28 (3)	3.026 (5)	145 (5)
$\text{O8}-\text{H5W} \cdots \text{O9}^{\text{iii}}$	0.86 (1)	1.84 (2)	2.689 (5)	168 (5)
$\text{O7}-\text{H4W} \cdots \text{O3}^{\text{iii}}$	0.86 (1)	2.08 (3)	2.788 (4)	139 (4)
$\text{O7}-\text{H3W} \cdots \text{O5}^{\text{iv}}$	0.87 (1)	1.92 (3)	2.711 (4)	152 (4)
$\text{O6}-\text{H2W} \cdots \text{O1}^{\text{v}}$	0.86 (1)	1.85 (2)	2.671 (4)	159 (5)
$\text{O6}-\text{H1W} \cdots \text{O4}^{\text{vi}}$	0.86 (1)	1.83 (1)	2.687 (4)	171 (5)

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $x + \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$ ; (iii)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (iv)  $-x + 1, -y + 1, -z + 2$ ; (v)  $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{5}{2}$ ; (vi)  $-x + 1, -y + 2, -z + 2$ .

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); 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.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5136).

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## supporting information

*Acta Cryst.* (2011). E67, m740 [doi:10.1107/S1600536811016953]

**Poly[[triqua( $\mu_3$ -4-oxidopyridine-2,6-dicarboxylato)holmium(III)] monohydrate]****Zhu-Qing Gao, Dong-Yu Lv, Jin-Zhong Gu and Hong-Jin Li****S1. Comment**

Lanthanide coordination polymers have shown not only versatile architectures but also desirable properties, *e.g.*, luminescent, magnetic, catalytic, and gas absorption and separation properties (Wang *et al.*, 2007; Lv *et al.*, 2010). In order to extend our investigations in this field, we designed and synthesized one lanthanide coordination polymer  $\{[\text{Ho}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$  by choosing 4-oxidopyridine-2,6-dicarboxylic acid as a functional ligand, and report its structure here.

The title compound is isotopic with its Dy (Gao *et al.*, 2006) and Eu (Lv *et al.*, 2010) analogues. As shown in Fig.1, the asymmetric unit of the cell contains one Ho(III) ion, one 4-oxidopyridine-2,6-dicarboxylate anion, three coordinated water molecules, and one water molecule of crystallization. The Ho atom is eight-coordinated by seven oxygen atoms from three anions and three coordinated water molecules and by one nitrogen atom from one tridentate anion (the other two anions are monodentate), forming a distorted bicapped trigonal-prismatic coordination environment.

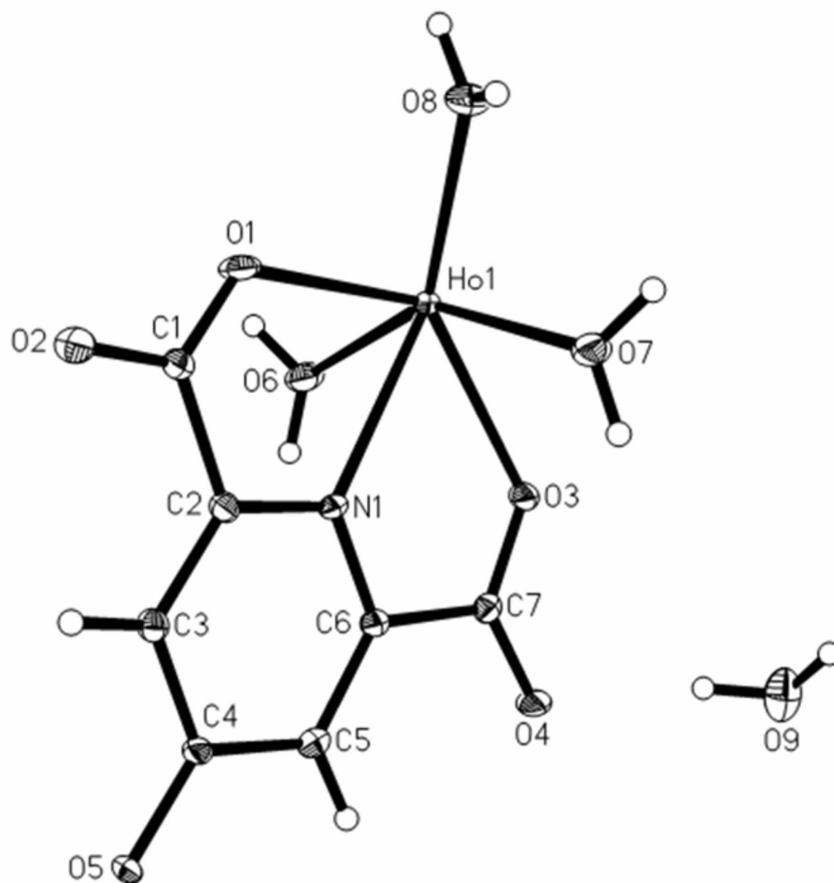
Important bond distances are presented in Table 1. The Ho–O bond lengths [2.279 (3) to 2.400 (3) Å] are shorter than the Ho–N bond length [2.450 (3) Å], which is in agreement with the bond lengths observed in other Ho(III) complexes (Wang *et al.*, 2007; Munoz *et al.*, 2005). The anion adopts a  $\mu_3$ -pentadentate coordination mode, as shown in Fig.1. The anions bridge the adjacent Ho<sup>III</sup> ions to form infinite double chains (Fig.2). Adjacent chains are further connected by the coordination of the anions and Ho(III) ions into a two-dimensional sheet (Fig.3), which are further extended into a three-dimensional supramolecular framework through O–H $\cdots$ O hydrogen-bonding interactions including both coordinated and uncoordinated water molecules (Table 2).

**S2. Experimental**

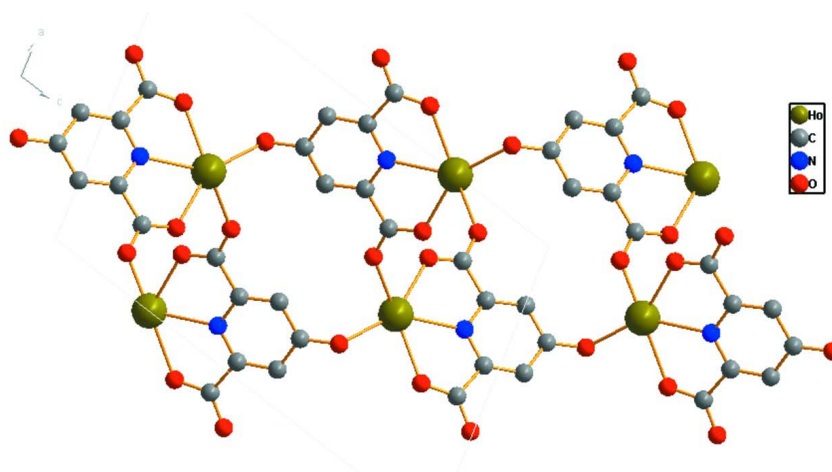
To a solution of holmium(III)nitrate hexahydrate (0.138 g, 0.3 mmol) in water (5 ml) was added an aqueous solution (5 ml) of the ligand (0.060 g, 0.3 mmol) and a drop of triethylamine. The reactants were sealed in a 25-ml Teflon-lined, stainless-steel Parr bomb. The bomb was heated at 433 K for 3 days. The cool solution yielded colorless single crystals in *ca* 75% yield. Anal. Calcd for  $\text{C}_7\text{H}_{10}\text{TbNO}_9$ : C, 20.16; H, 2.42; N, 3.36. Found: C, 20.45; H, 2.17; N, 3.64.

**S3. Refinement**

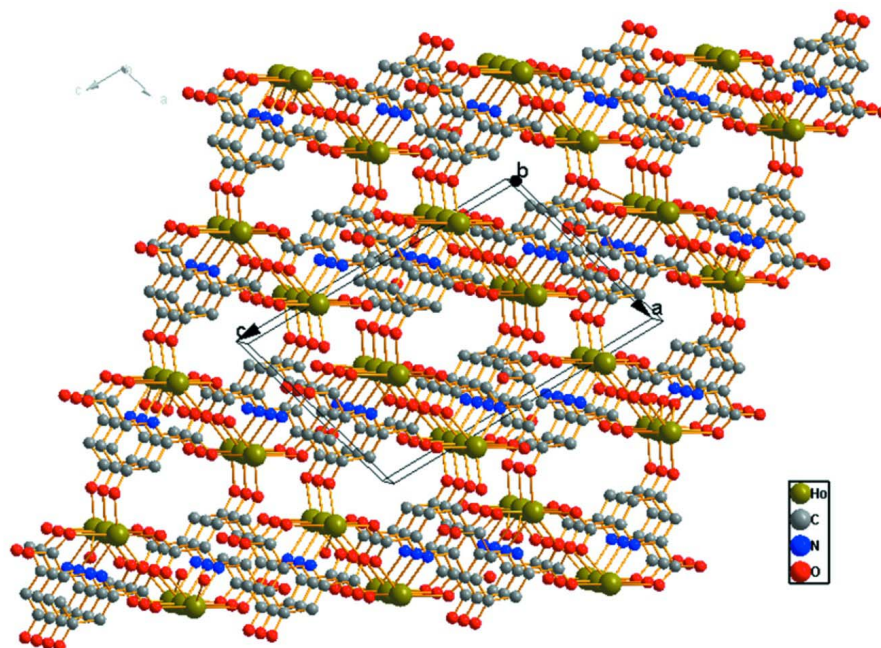
The coordinated water H atoms were located in a different Fourier map and refined with distance constraints of O–H = 0.83 (3) Å. The free water H atoms were placed at calculated positions and refined with a riding model, considering the position of oxygen atoms and the quantity of H atoms. The carbon-bound H atoms were placed in geometrically idealized positions, with C–H = 0.93 Å and constrained to ride on their respective parent atoms, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ .

**Figure 1**

The asymmetric unit of the title complex, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

View along the *b* axis, showing the polymeric double chain.



**Figure 3**

View along the *a* axis, showing the sheet structure of  $\{[\text{Ho}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}\}_n$ .

**Poly[[triaqua( $\mu_3$ -4-oxidopyridine-2,6-dicarboxylato)holmium(III)] monohydrate]**

*Crystal data*

$[\text{Ho}(\text{C}_7\text{H}_2\text{NO}_5)(\text{H}_2\text{O})_3]\cdot\text{H}_2\text{O}$

$M_r = 417.09$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2y/n$

$a = 9.879\ (5)\ \text{\AA}$

$b = 7.557\ (4)\ \text{\AA}$

$c = 15.386\ (8)\ \text{\AA}$

$\beta = 105.386\ (5)^\circ$

$V = 1107.5\ (9)\ \text{\AA}^3$

$Z = 4$

$F(000) = 792$

$D_x = 2.502\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2292 reflections

$\theta = 2.2\text{--}28.2^\circ$

$\mu = 7.19\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colorless

$0.32 \times 0.29 \times 0.26\ \text{mm}$

*Data collection*

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2004)

$T_{\min} = 0.207$ ,  $T_{\max} = 0.257$

4776 measured reflections

2003 independent reflections

1646 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 25.5^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -11 \rightarrow 7$

$k = -8 \rightarrow 9$

$l = -16 \rightarrow 17$

Refinement

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.021$   
 $wR(F^2) = 0.047$   
 $S = 1.02$   
 2003 reflections  
 196 parameters  
 12 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0176P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 0.71 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$   
 Extinction correction: *SHELXL97* (Sheldrick,  
 2008),  $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$   
 Extinction coefficient: 0.0119 (3)

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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Ho1	0.501337 (17)	0.82185 (2)	0.747304 (11)	0.01332 (10)
C1	0.7829 (4)	0.5891 (5)	0.8379 (3)	0.0184 (9)
C2	0.7250 (4)	0.6133 (5)	0.9175 (3)	0.0158 (9)
C3	0.7940 (4)	0.5582 (5)	1.0018 (3)	0.0172 (9)
H3	0.8780	0.4963	1.0112	0.021*
C4	0.7379 (4)	0.5951 (5)	1.0743 (2)	0.0163 (9)
C5	0.6087 (4)	0.6828 (5)	1.0537 (3)	0.0193 (9)
H5	0.5652	0.7073	1.0991	0.023*
C6	0.5460 (4)	0.7328 (5)	0.9665 (3)	0.0157 (9)
C7	0.4083 (4)	0.8304 (5)	0.9374 (3)	0.0162 (9)
H1W	0.613 (6)	1.087 (8)	0.8854 (11)	0.08 (2)*
H2W	0.661 (5)	1.128 (7)	0.806 (3)	0.08 (2)*
H3W	0.332 (5)	0.547 (8)	0.7874 (18)	0.09 (2)*
H4W	0.313 (4)	0.523 (6)	0.6913 (15)	0.040 (15)*
H5W	0.484 (6)	0.5513 (17)	0.606 (4)	0.08 (2)*
H6W	0.524 (8)	0.714 (7)	0.569 (3)	0.12 (3)*
H7W	0.1510 (15)	0.841 (6)	0.945 (3)	0.057 (19)*
H8W	0.032 (5)	0.745 (8)	0.888 (4)	0.16 (4)*
N1	0.6032 (3)	0.7004 (4)	0.8981 (2)	0.0151 (7)
O1	0.7200 (3)	0.6723 (4)	0.76661 (19)	0.0274 (8)
O2	0.8899 (3)	0.4962 (4)	0.84514 (18)	0.0234 (7)
O3	0.3689 (3)	0.8732 (4)	0.85503 (18)	0.0201 (6)
O4	0.3418 (3)	0.8625 (4)	0.99379 (18)	0.0249 (7)

O5	0.8051 (3)	0.5493 (4)	1.15748 (17)	0.0200 (6)
O6	0.6030 (3)	1.0722 (4)	0.8286 (2)	0.0315 (8)
O7	0.3636 (3)	0.5656 (4)	0.7411 (2)	0.0295 (8)
O8	0.4963 (4)	0.6642 (4)	0.6113 (2)	0.0255 (7)
O9	0.0620 (4)	0.8221 (4)	0.9302 (3)	0.0392 (9)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ho1	0.01235 (13)	0.01734 (13)	0.01046 (14)	0.00009 (7)	0.00339 (8)	0.00057 (8)
C1	0.016 (2)	0.024 (2)	0.014 (2)	0.0019 (17)	0.0032 (17)	-0.0012 (17)
C2	0.015 (2)	0.018 (2)	0.014 (2)	0.0028 (16)	0.0033 (17)	-0.0011 (17)
C3	0.016 (2)	0.018 (2)	0.017 (2)	0.0040 (16)	0.0039 (17)	0.0012 (16)
C4	0.017 (2)	0.019 (2)	0.013 (2)	-0.0047 (16)	0.0028 (17)	0.0022 (17)
C5	0.019 (2)	0.022 (2)	0.019 (2)	-0.0011 (16)	0.0079 (18)	0.0022 (17)
C6	0.016 (2)	0.017 (2)	0.014 (2)	0.0005 (16)	0.0049 (17)	0.0004 (16)
C7	0.016 (2)	0.015 (2)	0.018 (2)	-0.0025 (15)	0.0059 (18)	-0.0032 (16)
N1	0.0145 (18)	0.0198 (19)	0.0126 (18)	0.0011 (13)	0.0063 (14)	0.0005 (13)
O1	0.0270 (17)	0.044 (2)	0.0138 (16)	0.0158 (14)	0.0108 (14)	0.0074 (13)
O2	0.0244 (17)	0.0273 (18)	0.0196 (16)	0.0151 (13)	0.0076 (13)	0.0013 (13)
O3	0.0150 (15)	0.0300 (17)	0.0150 (16)	0.0057 (12)	0.0033 (12)	0.0029 (12)
O4	0.0211 (16)	0.0410 (19)	0.0151 (16)	0.0065 (13)	0.0092 (13)	0.0003 (13)
O5	0.0158 (15)	0.0295 (17)	0.0124 (15)	-0.0036 (12)	-0.0005 (12)	0.0065 (12)
O6	0.036 (2)	0.041 (2)	0.021 (2)	-0.0177 (15)	0.0143 (16)	-0.0087 (15)
O7	0.0341 (19)	0.036 (2)	0.0202 (18)	-0.0165 (14)	0.0105 (16)	-0.0040 (15)
O8	0.036 (2)	0.0232 (19)	0.0186 (18)	-0.0012 (14)	0.0098 (15)	-0.0031 (13)
O9	0.027 (2)	0.029 (2)	0.059 (3)	-0.0001 (15)	0.0072 (19)	0.0033 (18)

*Geometric parameters (Å, °)*

Ho1—O5 <sup>i</sup>	2.279 (3)	C5—C6	1.373 (5)
Ho1—O6	2.342 (3)	C5—H5	0.9300
Ho1—O7	2.354 (3)	C6—N1	1.341 (5)
Ho1—O1	2.386 (3)	C6—C7	1.508 (5)
Ho1—O2 <sup>ii</sup>	2.393 (3)	C7—O4	1.243 (5)
Ho1—O8	2.397 (3)	C7—O3	1.265 (5)
Ho1—O3	2.400 (3)	O2—Ho1 <sup>iii</sup>	2.393 (3)
Ho1—N1	2.450 (3)	O5—Ho1 <sup>iv</sup>	2.279 (3)
C1—O2	1.249 (5)	O6—H1W	0.860 (10)
C1—O1	1.275 (5)	O6—H2W	0.858 (10)
C1—C2	1.493 (5)	O7—H3W	0.865 (10)
C2—N1	1.334 (5)	O7—H4W	0.861 (10)
C2—C3	1.361 (5)	O8—H5W	0.862 (10)
C3—C4	1.399 (5)	O8—H6W	0.861 (10)
C3—H3	0.9300	O9—H7W	0.860 (10)
C4—O5	1.322 (4)	O9—H8W	0.861 (10)
C4—C5	1.398 (5)		

O5 <sup>i</sup> —Ho1—O6	98.86 (11)	C3—C2—C1	122.7 (3)
O5 <sup>i</sup> —Ho1—O7	86.39 (12)	C2—C3—C4	119.7 (4)
O6—Ho1—O7	147.96 (12)	C2—C3—H3	120.2
O5 <sup>i</sup> —Ho1—O1	150.97 (10)	C4—C3—H3	120.2
O6—Ho1—O1	94.04 (12)	O5—C4—C5	122.5 (4)
O7—Ho1—O1	96.31 (12)	O5—C4—C3	121.1 (4)
O5 <sup>i</sup> —Ho1—O2 <sup>ii</sup>	81.27 (10)	C5—C4—C3	116.5 (4)
O6—Ho1—O2 <sup>ii</sup>	71.09 (11)	C6—C5—C4	119.9 (4)
O7—Ho1—O2 <sup>ii</sup>	140.78 (10)	C6—C5—H5	120.0
O1—Ho1—O2 <sup>ii</sup>	78.49 (10)	C4—C5—H5	120.0
O5 <sup>i</sup> —Ho1—O8	82.29 (11)	N1—C6—C5	122.8 (4)
O6—Ho1—O8	140.74 (12)	N1—C6—C7	113.0 (3)
O7—Ho1—O8	71.19 (12)	C5—C6—C7	124.2 (4)
O1—Ho1—O8	71.43 (11)	O4—C7—O3	124.8 (4)
O2 <sup>ii</sup> —Ho1—O8	70.33 (11)	O4—C7—C6	119.4 (4)
O5 <sup>i</sup> —Ho1—O3	79.50 (10)	O3—C7—C6	115.8 (3)
O6—Ho1—O3	74.68 (11)	C2—N1—C6	117.2 (3)
O7—Ho1—O3	75.28 (11)	C2—N1—Ho1	121.0 (3)
O1—Ho1—O3	129.20 (9)	C6—N1—Ho1	121.4 (2)
O2 <sup>ii</sup> —Ho1—O3	137.27 (9)	C1—O1—Ho1	124.0 (2)
O8—Ho1—O3	142.60 (11)	C1—O2—Ho1 <sup>iii</sup>	139.4 (3)
O5 <sup>i</sup> —Ho1—N1	143.51 (10)	C7—O3—Ho1	124.8 (2)
O6—Ho1—N1	77.69 (12)	C4—O5—Ho1 <sup>iv</sup>	127.1 (2)
O7—Ho1—N1	79.53 (11)	Ho1—O6—H1W	124 (3)
O1—Ho1—N1	64.77 (10)	Ho1—O6—H2W	115 (3)
O2 <sup>ii</sup> —Ho1—N1	129.28 (10)	H1W—O6—H2W	116 (2)
O8—Ho1—N1	123.29 (11)	Ho1—O7—H3W	116 (3)
O3—Ho1—N1	64.44 (10)	Ho1—O7—H4W	123 (3)
O2—C1—O1	124.2 (4)	H3W—O7—H4W	114.8 (19)
O2—C1—C2	119.7 (3)	Ho1—O8—H5W	123 (3)
O1—C1—C2	116.1 (3)	Ho1—O8—H6W	120 (4)
N1—C2—C3	123.9 (4)	H5W—O8—H6W	115.3 (19)
N1—C2—C1	113.4 (3)	H7W—O9—H8W	116 (2)
O2—C1—C2—N1	-173.7 (4)	O3—Ho1—N1—C2	-179.5 (3)
O1—C1—C2—N1	8.7 (5)	O5 <sup>i</sup> —Ho1—N1—C6	-15.3 (4)
O2—C1—C2—C3	9.1 (6)	O6—Ho1—N1—C6	72.9 (3)
O1—C1—C2—C3	-168.6 (4)	O7—Ho1—N1—C6	-84.4 (3)
N1—C2—C3—C4	-1.0 (6)	O1—Ho1—N1—C6	173.5 (3)
C1—C2—C3—C4	175.9 (4)	O2 <sup>ii</sup> —Ho1—N1—C6	125.6 (3)
C2—C3—C4—O5	-177.4 (4)	O8—Ho1—N1—C6	-143.5 (3)
C2—C3—C4—C5	2.3 (6)	O3—Ho1—N1—C6	-5.8 (3)
O5—C4—C5—C6	177.7 (4)	O2—C1—O1—Ho1	172.6 (3)
C3—C4—C5—C6	-2.0 (6)	C2—C1—O1—Ho1	-9.9 (5)
C4—C5—C6—N1	0.3 (6)	O5 <sup>i</sup> —Ho1—O1—C1	-163.5 (3)
C4—C5—C6—C7	-178.9 (4)	O6—Ho1—O1—C1	80.1 (3)
N1—C6—C7—O4	177.2 (3)	O7—Ho1—O1—C1	-69.5 (3)
C5—C6—C7—O4	-3.4 (6)	O2 <sup>ii</sup> —Ho1—O1—C1	149.8 (3)

N1—C6—C7—O3	-2.4 (5)	O8—Ho1—O1—C1	-137.3 (3)
C5—C6—C7—O3	176.9 (4)	O3—Ho1—O1—C1	6.6 (4)
C3—C2—N1—C6	-0.8 (6)	N1—Ho1—O1—C1	5.7 (3)
C1—C2—N1—C6	-178.0 (3)	O1—C1—O2—Ho1 <sup>iii</sup>	-28.4 (7)
C3—C2—N1—Ho1	173.2 (3)	C2—C1—O2—Ho1 <sup>iii</sup>	154.1 (3)
C1—C2—N1—Ho1	-4.0 (5)	O4—C7—O3—Ho1	177.4 (3)
C5—C6—N1—C2	1.1 (6)	C6—C7—O3—Ho1	-3.0 (5)
C7—C6—N1—C2	-179.6 (3)	O5 <sup>i</sup> —Ho1—O3—C7	178.8 (3)
C5—C6—N1—Ho1	-172.8 (3)	O6—Ho1—O3—C7	-78.9 (3)
C7—C6—N1—Ho1	6.5 (4)	O7—Ho1—O3—C7	89.8 (3)
O5 <sup>i</sup> —Ho1—N1—C2	171.0 (3)	O1—Ho1—O3—C7	3.6 (3)
O6—Ho1—N1—C2	-100.7 (3)	O2 <sup>ii</sup> —Ho1—O3—C7	-116.6 (3)
O7—Ho1—N1—C2	101.9 (3)	O8—Ho1—O3—C7	116.6 (3)
O1—Ho1—N1—C2	-0.2 (3)	N1—Ho1—O3—C7	4.5 (3)
O2 <sup>ii</sup> —Ho1—N1—C2	-48.1 (3)	C5—C4—O5—Ho1 <sup>iv</sup>	-106.4 (4)
O8—Ho1—N1—C2	42.8 (3)	C3—C4—O5—Ho1 <sup>iv</sup>	73.3 (4)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O9—H8W $\cdots$ O2 <sup>v</sup>	0.86 (1)	2.33 (4)	3.080 (5)	145 (6)
O9—H7W $\cdots$ O4	0.86 (1)	1.84 (1)	2.693 (5)	170 (5)
O8—H6W $\cdots$ O9 <sup>vi</sup>	0.86 (1)	2.28 (3)	3.026 (5)	145 (5)
O8—H5W $\cdots$ O9 <sup>vii</sup>	0.86 (1)	1.84 (2)	2.689 (5)	168 (5)
O7—H4W $\cdots$ O3 <sup>viii</sup>	0.86 (1)	2.08 (3)	2.788 (4)	139 (4)
O7—H3W $\cdots$ O5 <sup>viii</sup>	0.87 (1)	1.92 (3)	2.711 (4)	152 (4)
O6—H2W $\cdots$ O1 <sup>ii</sup>	0.86 (1)	1.85 (2)	2.671 (4)	159 (5)
O6—H1W $\cdots$ O4 <sup>ix</sup>	0.86 (1)	1.83 (1)	2.687 (4)	171 (5)

Symmetry codes: (ii)  $-x+3/2, y+1/2, -z+3/2$ ; (v)  $x-1, y, z$ ; (vi)  $x+1/2, -y+3/2, z-1/2$ ; (vii)  $-x+1/2, y-1/2, -z+3/2$ ; (viii)  $-x+1, -y+1, -z+2$ ; (ix)  $-x+1, -y+2, -z+2$ .