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catena-Poly[[[diaqua(6-carboxypyridine-2-carboxylato)holmium(III)]-μ-pyridine-2,6-dicarboxylato] tetrahydrate]

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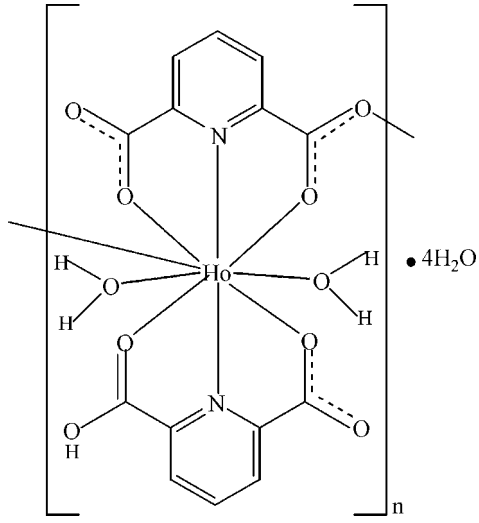
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.009 Å; R factor = 0.038; wR factor = 0.122; data-to-parameter ratio = 11.2.

The title compound, $\{[Ho(C_7H_3NO_4)(C_7H_4NO_4)(H_2O)_2]\cdot 4H_2O\}_n$, prepared by hydrothermal synthesis, is isostructural with its La^{III}-, Ce^{III}-, Pr^{III}-, Nd^{III}-, Sm^{III}-, Gd^{III}- and Tb^{III}-containing analogues. The Ho^{III} ion is nine-coordinated by four O and two N atoms from two independent pyridine-2,6-dicarboxylate groups, one carboxylate O atom belonging to a neighboring pyridine-2,6-dicarboxylate ligand and two water molecules. The bridging pyridine-2,6-dicarboxylate ligand gives rise to infinite chains. The crystal structure contains O—H···O hydrogen bonds, which connect the chains into a three-dimensional network.

Related literature

For related literature, see: Li *et al.* (1993); Gao *et al.* (2006); Go *et al.* (2004); An *et al.* (2000); Baroni *et al.* (1996); Hundal *et al.* (2002). Isostructural lanthanide compounds have been reported with La^{III} (Guerriero *et al.*, 1987; Ghosh & Bharadwaj, 2005), Ce^{III} (Okabe *et al.*, 2002; Ghosh & Bharadwaj, 2003; Rafizadeh *et al.*, 2005; Ramezanipour *et al.*, 2005), Pr^{III} (Ghosh & Bharadwaj, 2003; Zhao *et al.*, 2005), Nd^{III} (Miao *et al.*, 1992), Sm^{III} (Liu *et al.*, 2005, 2006; Rafizadeh *et al.*, 2005; Song *et al.*, 2005), Eu^{III} (Brayshaw *et al.*, 2005), Gd^{III} (Hao & Yu, 2007*a*) and Tb^{III} (Hao & Yu, 2007*b*).



Experimental

Crystal data

$[Ho(C_7H_3NO_4)(C_7H_4NO_4)(H_2O)_2]\cdot 4H_2O$
M_r = 604.24
Monoclinic, $P2_1/c$
a = 14.1227 (5) Å
b = 11.2565 (4) Å
c = 13.0342 (5) Å
β = 101.892 (1)°
V = 2027.60 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.98 mm^{-1}
T = 293 (2) K
0.33 × 0.30 × 0.26 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.354, T_{max} = 0.424
(expected range = 0.296–0.355)
7113 measured reflections
3546 independent reflections
3213 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.122
S = 1.00
3546 reflections
317 parameters
18 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −1.07 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O9—H1W···O8^{i}	0.82 (5)	2.10 (4)	2.882 (6)	160 (12)
O9—H2W···O7^{ii}	0.81 (8)	2.13 (9)	2.924 (6)	166 (9)
O10—H3W···O12^{iii}	0.81 (7)	2.85 (10)	3.361 (8)	123 (10)
O10—H4W···O11	0.82 (9)	2.68 (13)	2.813 (11)	90 (9)
O11—H6W···O3^{iii}	0.82 (9)	2.23 (9)	2.638 (8)	111 (8)
O12—H7W···O9	0.81 (6)	2.16 (7)	2.782 (6)	133 (10)
O12—H8W···O14^{iv}	0.81 (8)	1.95 (9)	2.720 (6)	158 (11)
O13—H9W···O3^{v}	0.82 (7)	2.41 (10)	2.724 (6)	104 (8)
O13—H10W···O5^{vi}	0.82 (7)	2.06 (7)	2.725 (5)	139 (10)
O14—H11W···O9^{vi}	0.815 (11)	2.10 (3)	2.899 (6)	168 (10)
O2—H2···O11	0.82 (1)	1.70 (1)	2.491 (9)	160 (1)

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

  doi:10.1107/S1600536807041657

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2104).

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supplementary materials

Acta Cryst. (2007). E**63**, m2438-m2439 [**doi:10.1107/S1600536807041657**]

catena-Poly[[[diaqua(6-carboxypyridine-2-carboxylato)holmium(III)]-μ-pyridine-2,6-dicarboxylato] tetrahydrate]

Z. Li, S. Wang, Q. Zhang and X. Yu

Comment

Complexes containing carboxyl acids have been the interest of chemists these years due to their potential applications, such as catalysis, optics, information storage, medicine, molecular electrochemistry, biochemistry and biological pharmaceutics (Li *et al.* (1993); Gao *et al.* (2004); Go *et al.* (2004)) Thus far, N-containing aromatic carboxyl acid has been widely used in dye intermediate, organic synthesis, sensitization material, functional pigment, adipiodone and acetrizoic acid (An *et al.* (2000). Pyridine carboxylic acid is also a good ligand in coordination chemistry due to its strong coordination ability and versatile coordination modes, so much attention has been paid to it in these decades (Baroni *et al.* (1996); Hundal *et al.* (2002)). Herein, we report the new complex, *catena*-Poly[[[diaqua(6-carboxypyridine-2-carboxylato)Holmium(III)]- µ-pyridine-2,6-dicarboxylato] tetrahydrate].

In the title compound, Ho^{III} is of nona-coordination, chelated by two independent 2, 6-pyridine dicarboxylate, and further coordinated by two water molecules (Fig. 1). The unit is linked by one carboxylate oxygen of neighboring 2, 6-pyridine dicarboxylate forming infinite chains (Fig. 2). Extensive hydrogen bonding (Table 2) *via* hydrogen bonds between carboxylate oxygen atoms of 2,6-pyridinedicarboxylate and lattice water molecules or coordinated aqua ligands gives rise to three dimensional network (Fig. 3).

Experimental

A mixture of Holmium oxide (0.1 mmol, 0.38 g), pyridine-2,6-dicarboxylic acid (0.2 mmol, 0.33 g), H_2O (16 ml) in a 25 ml Teflon-lined stainless steel autoclave was kept at 473 K for three days. Colorless crystals were obtained after cooling to room temperature with a yield of 6%. Anal. Calc. for $C_{14}H_{19}HoN_2O_{14}$: C 28.43, H 3.21, N 4.74%; Found: C 28.38, H 3.23, N 4.71%.

Refinement

The H atoms of the water molecule were located from difference density maps and were refined with distance restraints of d(H—H) = 1.38 (2) Å and d(O—H) = 0.82 (2) Å. All other H atoms were placed in calculated positions with a C—H bond distance of 0.93%A and U_{iso}(H) = 1.2U_{eq} of the respective carrier atom.

Figures

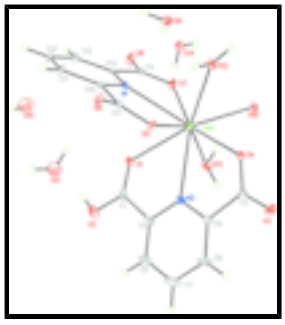


Fig. 1. The coordination of the Ho atom in the title structure, drawn with 30% probability displacement ellipsoids. Atoms labeled with i at the symmetry positions (x,-y + 1/2,z + 1/2).

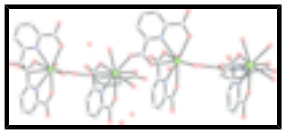


Fig. 2. Chains of the title compound along [010] direction. The balls represent holmium (green), C (gray), N (blue) and O (red)·For clarity all H atoms have been omitted.

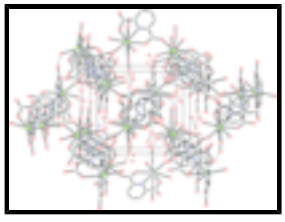


Fig. 3. A view of the packing structure of the title compound. The balls represent holmium (green), C (gray), N (blue) and O (red).

catena-Poly[[[diaqua(6-carboxypyridine-2-carboxylato)holmium(III)]-\ μ-pyridine-2,6-dicarboxylato] tetrahydrate]

Crystal data

$[Ho(C_7H_3NO_4)(C_7H_4NO_4)(H_2O)_2]\cdot 4H_2O$	F_{000} = 1184
M_r = 604.24	D_x = 1.979 Mg m^{-3}
Monoclinic, $P2(1)/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3546 reflections
a = 14.1227 (5) Å	θ = 2.3–25.0º
b = 11.2565 (4) Å	μ = 3.98 mm^{-1}
c = 13.0342 (5) Å	T = 293 (2) K
β = 101.8920 (10)º	Block, colorless
V = 2027.60 (13) Å^3	0.33 × 0.30 × 0.26 mm
Z = 4	

Data collection

Bruker APEX II CCD area-detector diffractometer	3546 independent reflections
Radiation source: fine-focus sealed tube	3213 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.3º

Absorption correction: multi-scan (SADABS; Bruker, 2001)

T_{min} = 0.354, T_{max} = 0.424

7113 measured reflections

h = −16→16

k = −13→13

l = −7→15

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.038

$wR(F^2)$ = 0.122

S = 1.00

3546 reflections

317 parameters

18 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

$w = 1/[\sigma^2(F_o^2) + (0.098P)^2 + 5.4784P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 1.28 e Å^{-3}

$\Delta\rho_{min}$ = −1.07 e Å^{-3}

Extinction correction: none

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.4422 (5)	0.4997 (5)	0.6323 (5)	0.0266 (13)
C2	0.4323 (4)	0.0766 (5)	0.6274 (5)	0.0218 (12)
C3	0.2257 (4)	0.3472 (4)	0.3809 (4)	0.0139 (10)
C4	0.1196 (4)	0.5249 (5)	0.6731 (4)	0.0167 (11)
C5	0.5032 (5)	0.3901 (6)	0.6334 (5)	0.0279 (13)
C6	0.6008 (5)	0.3890 (8)	0.6347 (7)	0.0314 (8)
H6	0.6346	0.4600	0.6351	0.058*
C7	0.6479 (7)	0.2832 (8)	0.6354 (10)	0.0366 (11)
H7	0.7144	0.2811	0.6389	0.076*
C8	0.5951 (6)	0.1783 (8)	0.6309 (8)	0.0378 (11)
H8	0.6251	0.1050	0.6285	0.060*
C9	0.4969 (5)	0.1852 (6)	0.6301 (5)	0.0245 (13)

C10	0.1738 (3)	0.4516 (4)	0.4171 (4)	0.0121 (10)
C11	0.1380 (4)	0.5471 (5)	0.3507 (4)	0.0174 (11)
H11	0.1413	0.5467	0.2801	0.021*
C12	0.0980 (4)	0.6409 (5)	0.3942 (4)	0.0223 (12)
H12	0.0750	0.7063	0.3530	0.027*
C13	0.0916 (4)	0.6390 (5)	0.4995 (4)	0.0206 (11)
H13	0.0648	0.7026	0.5292	0.025*
C14	0.1266 (3)	0.5392 (4)	0.5595 (4)	0.0112 (9)
Ho1	0.269861 (18)	0.29481 (2)	0.648479 (18)	0.01835 (15)
H1W	0.020 (8)	0.259 (4)	0.338 (7)	0.080*
H2W	0.066 (7)	0.163 (8)	0.305 (5)	0.080*
H3W	0.216 (8)	0.960 (5)	0.619 (6)	0.080*
H4W	0.220 (9)	0.900 (8)	0.528 (5)	0.080*
H5W	0.330 (5)	0.729 (8)	0.608 (8)	0.080*
H6W	0.378 (7)	0.820 (8)	0.569 (6)	0.080*
H7W	0.135 (8)	0.192 (8)	0.505 (3)	0.080*
H8W	0.088 (6)	0.131 (7)	0.572 (7)	0.080*
H9W	0.418 (3)	0.315 (9)	0.852 (7)	0.080*
H10W	0.326 (5)	0.342 (10)	0.863 (6)	0.080*
H11W	0.025 (6)	0.4863 (18)	0.891 (8)	0.080*
H12W	0.083 (3)	0.584 (7)	0.926 (8)	0.080*
N1	0.1688 (3)	0.4484 (3)	0.5189 (3)	0.0102 (8)
N2	0.4520 (4)	0.2880 (4)	0.6325 (4)	0.0196 (11)
O1	0.3567 (3)	0.4920 (3)	0.6354 (3)	0.0243 (9)
O2	0.4875 (4)	0.6000 (4)	0.6264 (5)	0.0454 (13)
H2	0.4537	0.6554	0.6384	0.068*
O3	0.4692 (3)	−0.0218 (4)	0.6230 (4)	0.0385 (12)
O4	0.3442 (3)	0.0968 (3)	0.6298 (3)	0.0215 (8)
O5	0.2737 (3)	0.2818 (3)	0.4533 (3)	0.0158 (8)
O6	0.2188 (3)	0.3335 (3)	0.2838 (3)	0.0182 (8)
O7	0.1549 (3)	0.4294 (3)	0.7180 (3)	0.0209 (8)
O8	0.0792 (3)	0.6042 (4)	0.7147 (3)	0.0291 (10)
O9	0.0416 (3)	0.1919 (4)	0.3505 (3)	0.0238 (9)
O10	0.2016 (6)	0.9006 (6)	0.5839 (7)	0.083 (2)
O11	0.3770 (6)	0.7738 (6)	0.6176 (8)	0.086 (3)
O12	0.1248 (3)	0.1839 (4)	0.5634 (3)	0.0250 (9)
O13	0.3662 (4)	0.3461 (5)	0.8260 (3)	0.0356 (11)
O14	0.0295 (3)	0.5577 (4)	0.9011 (4)	0.0317 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.035 (4)	0.017 (3)	0.026 (3)	−0.008 (2)	0.001 (3)	0.002 (2)
C2	0.013 (3)	0.028 (3)	0.024 (3)	−0.001 (2)	0.001 (2)	0.000 (2)
C3	0.016 (3)	0.014 (2)	0.013 (2)	0.003 (2)	0.0043 (19)	0.000 (2)
C4	0.016 (3)	0.018 (3)	0.017 (3)	0.001 (2)	0.004 (2)	−0.001 (2)
C5	0.026 (3)	0.028 (3)	0.030 (3)	−0.013 (3)	0.006 (2)	0.002 (3)
C6	0.0177 (15)	0.0427 (19)	0.0322 (19)	0.002 (2)	−0.0009 (13)	0.000 (2)

C7	0.026 (2)	0.050 (3)	0.033 (3)	0.005 (2)	0.0015 (19)	−0.001 (2)
C8	0.031 (2)	0.043 (2)	0.038 (3)	0.003 (2)	0.0025 (19)	0.003 (2)
C9	0.015 (3)	0.028 (3)	0.030 (3)	−0.003 (2)	0.002 (2)	0.001 (2)
C10	0.013 (2)	0.013 (2)	0.009 (2)	−0.0024 (19)	0.0009 (18)	−0.0014 (18)
C11	0.025 (3)	0.017 (3)	0.009 (2)	0.007 (2)	0.001 (2)	0.0026 (19)
C12	0.032 (3)	0.018 (3)	0.014 (3)	0.009 (2)	−0.002 (2)	0.005 (2)
C13	0.027 (3)	0.016 (3)	0.020 (3)	0.007 (2)	0.007 (2)	0.001 (2)
C14	0.013 (2)	0.009 (2)	0.012 (2)	0.0019 (18)	0.0023 (18)	−0.0009 (18)
Ho1	0.0194 (2)	0.0194 (2)	0.0157 (2)	0.00111 (8)	0.00213 (13)	0.00128 (8)
N1	0.011 (2)	0.0103 (19)	0.0092 (19)	0.0014 (15)	0.0026 (15)	0.0016 (15)
N2	0.016 (3)	0.026 (3)	0.016 (2)	0.0007 (18)	0.001 (2)	−0.0005 (17)
O1	0.024 (2)	0.0156 (19)	0.031 (2)	−0.0079 (16)	0.0020 (17)	0.0028 (16)
O2	0.047 (3)	0.026 (2)	0.062 (3)	−0.015 (2)	0.009 (3)	0.004 (2)
O3	0.028 (2)	0.019 (2)	0.065 (3)	0.0107 (19)	0.002 (2)	−0.002 (2)
O4	0.020 (2)	0.0129 (18)	0.031 (2)	0.0005 (15)	0.0038 (16)	0.0016 (15)
O5	0.020 (2)	0.0185 (18)	0.0081 (17)	0.0112 (14)	0.0008 (15)	0.0013 (13)
O6	0.026 (2)	0.0183 (18)	0.0084 (18)	0.0066 (16)	−0.0004 (15)	−0.0025 (15)
O7	0.033 (2)	0.0191 (19)	0.0123 (17)	0.0125 (16)	0.0085 (16)	0.0044 (14)
O8	0.049 (3)	0.020 (2)	0.023 (2)	0.0165 (19)	0.0192 (19)	0.0009 (16)
O9	0.024 (2)	0.028 (2)	0.019 (2)	0.0023 (16)	0.0023 (18)	−0.0009 (16)
O10	0.065 (5)	0.035 (3)	0.132 (7)	−0.013 (3)	−0.017 (5)	0.001 (4)
O11	0.066 (5)	0.035 (3)	0.152 (8)	−0.004 (3)	0.013 (5)	0.012 (4)
O12	0.028 (2)	0.035 (2)	0.0105 (19)	−0.0128 (18)	0.0000 (17)	0.0046 (16)
O13	0.037 (3)	0.054 (3)	0.014 (2)	−0.029 (2)	−0.0008 (18)	0.000 (2)
O14	0.040 (3)	0.030 (2)	0.030 (2)	0.011 (2)	0.019 (2)	−0.0005 (19)

Geometric parameters (Å, °)

C1—O1	1.219 (8)	C13—C14	1.399 (7)
C1—O2	1.307 (8)	C13—H13	0.9300
C1—C5	1.503 (9)	C14—N1	1.345 (6)
C2—O3	1.231 (7)	Ho1—O12	2.460 (4)
C2—O4	1.271 (7)	Ho1—O4	2.497 (4)
C2—C9	1.521 (8)	Ho1—O6^{i}	2.499 (4)
C3—O6	1.259 (6)	Ho1—O13	2.499 (4)
C3—O5	1.276 (6)	Ho1—O7	2.522 (4)
C3—C10	1.511 (7)	Ho1—O1	2.559 (4)
C4—O8	1.241 (7)	Ho1—O5	2.559 (4)
C4—O7	1.275 (6)	Ho1—N2	2.625 (5)
C4—C14	1.514 (7)	Ho1—N1	2.623 (4)
C5—N2	1.357 (7)	O2—H2	0.8200
C5—C6	1.376 (10)	O6—Ho1^{ii}	2.499 (4)
C6—C7	1.364 (12)	O9—H1W	0.82 (5)
C6—H6	0.9300	O9—H2W	0.81 (8)
C7—C8	1.391 (12)	O10—H3W	0.81 (7)
C7—H7	0.9300	O10—H4W	0.82 (9)
C8—C9	1.387 (10)	O11—H5W	0.82 (8)
C8—H8	0.9300	O11—H6W	0.82 (9)
C9—N2	1.323 (8)	O12—H7W	0.81 (6)

C10—N1	1.345 (6)	O12—H8W	0.81 (8)
C10—C11	1.407 (7)	O13—H9W	0.82 (7)
C11—C12	1.374 (8)	O13—H10W	0.82 (7)
C11—H11	0.9300	O14—H11W	0.815 (11)
C12—C13	1.395 (8)	O14—H12W	0.81 (7)
C12—H12	0.9300		
O1—C1—O2	124.3 (6)	$O6^{i}$—Ho1—O7	78.04 (12)
O1—C1—C5	120.7 (5)	O13—Ho1—O7	78.27 (16)
O2—C1—C5	115.0 (6)	O12—Ho1—O1	140.95 (13)
O3—C2—O4	126.0 (6)	O4—Ho1—O1	123.42 (14)
O3—C2—C9	117.8 (5)	$O6^{i}$—Ho1—O1	139.51 (13)
O4—C2—C9	116.2 (5)	O13—Ho1—O1	71.59 (14)
O6—C3—O5	126.4 (5)	O7—Ho1—O1	81.70 (13)
O6—C3—C10	117.8 (4)	O12—Ho1—O5	73.07 (13)
O5—C3—C10	115.8 (4)	O4—Ho1—O5	75.91 (12)
O8—C4—O7	125.3 (5)	$O6^{i}$—Ho1—O5	137.46 (12)
O8—C4—C14	118.6 (5)	O13—Ho1—O5	144.57 (14)
O7—C4—C14	116.1 (4)	O7—Ho1—O5	122.56 (11)
N2—C5—C6	121.5 (7)	O1—Ho1—O5	82.82 (13)
N2—C5—C1	113.1 (5)	O12—Ho1—N2	133.50 (15)
C6—C5—C1	125.4 (6)	O4—Ho1—N2	61.78 (13)
C7—C6—C5	119.6 (7)	$O6^{i}$—Ho1—N2	117.99 (14)
C7—C6—H6	120.2	O13—Ho1—N2	74.01 (16)
C5—C6—H6	120.2	O7—Ho1—N2	139.50 (14)
C6—C7—C8	119.0 (8)	O1—Ho1—N2	61.90 (13)
C6—C7—H7	120.5	O5—Ho1—N2	72.42 (14)
C8—C7—H7	120.5	O12—Ho1—N1	75.65 (13)
C9—C8—C7	118.6 (8)	O4—Ho1—N1	135.17 (13)
C9—C8—H8	120.7	$O6^{i}$—Ho1—N1	129.61 (13)
C7—C8—H8	120.7	O13—Ho1—N1	124.34 (16)
N2—C9—C8	122.2 (6)	O7—Ho1—N1	61.97 (11)
N2—C9—C2	114.5 (5)	O1—Ho1—N1	65.81 (13)
C8—C9—C2	123.3 (6)	O5—Ho1—N1	61.19 (11)
N1—C10—C11	122.9 (4)	N2—Ho1—N1	112.35 (13)
N1—C10—C3	114.5 (4)	C14—N1—C10	119.0 (4)
C11—C10—C3	122.6 (4)	C14—N1—Ho1	118.3 (3)
C12—C11—C10	117.3 (5)	C10—N1—Ho1	121.4 (3)
C12—C11—H11	121.3	C9—N2—C5	119.0 (6)
C10—C11—H11	121.3	C9—N2—Ho1	120.7 (4)
C11—C12—C13	120.7 (5)	C5—N2—Ho1	120.2 (4)
C11—C12—H12	119.6	C1—O1—Ho1	123.7 (4)
C13—C12—H12	119.7	C1—O2—H2	109.5
C12—C13—C14	118.4 (5)	C2—O4—Ho1	126.6 (4)
C12—C13—H13	120.8	C3—O5—Ho1	125.9 (3)
C14—C13—H13	120.8	C3—O6—$Ho1^{ii}$	143.8 (3)
N1—C14—C13	121.7 (4)	C4—O7—Ho1	124.2 (3)
N1—C14—C4	115.3 (4)	H1W—O9—H2W	115 (9)

C13—C14—C4	123.0 (4)	H3W—O10—H4W	115 (9)
O12—Ho1—O4	80.27 (15)	H5W—O11—H6W	114 (10)
O12—Ho1—O6^{i}	71.96 (12)	Ho1—O12—H7W	94 (7)
O4—Ho1—O6^{i}	75.01 (13)	Ho1—O12—H8W	144 (6)
O12—Ho1—O13	141.17 (13)	H7W—O12—H8W	117 (9)
O4—Ho1—O13	97.86 (16)	Ho1—O13—H9W	123 (8)
O6^{i}—Ho1—O13	70.18 (13)	Ho1—O13—H10W	103 (7)
O12—Ho1—O7	85.92 (15)	H9W—O13—H10W	114 (8)
O4—Ho1—O7	152.41 (12)	H11W—O14—H12W	117 (8)

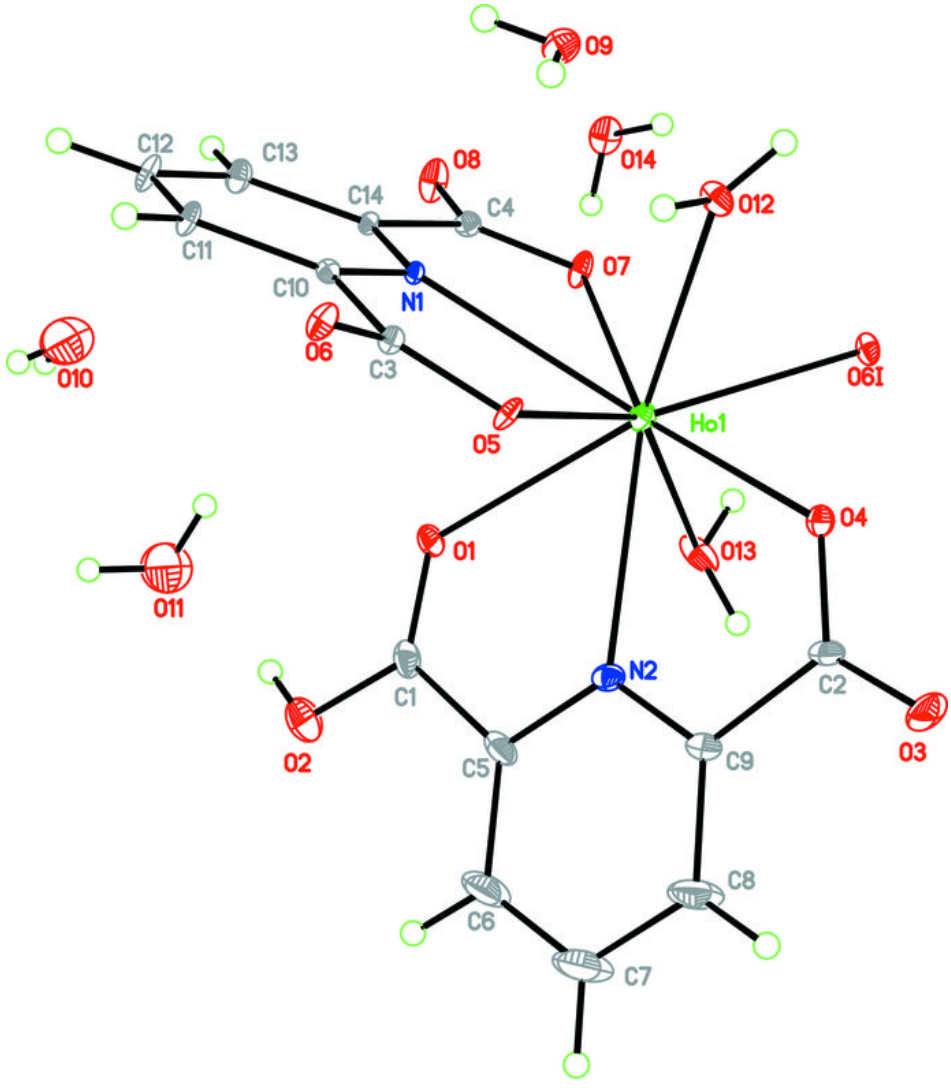
Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H1W···O8^{iii}	0.82 (5)	2.10 (4)	2.882 (6)	160 (12)
O9—H2W···O7^{ii}	0.81 (8)	2.13 (9)	2.924 (6)	166 (9)
O10—H3W···O12^{iv}	0.81 (7)	2.85 (10)	3.361 (8)	123 (10)
O10—H4W···O11	0.82 (9)	2.68 (13)	2.813 (11)	90 (9)
O11—H6W···O3^{iv}	0.82 (9)	2.23 (9)	2.638 (8)	111 (8)
O12—H7W···O9	0.81 (6)	2.16 (7)	2.782 (6)	133 (10)
O12—H8W···O14^{v}	0.81 (8)	1.95 (9)	2.720 (6)	158 (11)
O13—H9W···O3^{vi}	0.82 (7)	2.41 (10)	2.724 (6)	104 (8)
O13—H10W···O5^{i}	0.82 (7)	2.06 (7)	2.725 (5)	139 (10)
O14—H11W···O9^{i}	0.815 (11)	2.10 (3)	2.899 (6)	168 (10)
O2—H2···O11	0.82 (1)	1.70 (1)	2.491 (9)	160 (1)

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

Fig. 1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O8
O14
C14
C4
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O7
C10
N1
O6
C3
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Ho1
O5
O4
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O11
N2
C1
C2
O2
O3
C5
C9
C8
C6
C7

Fig. 2

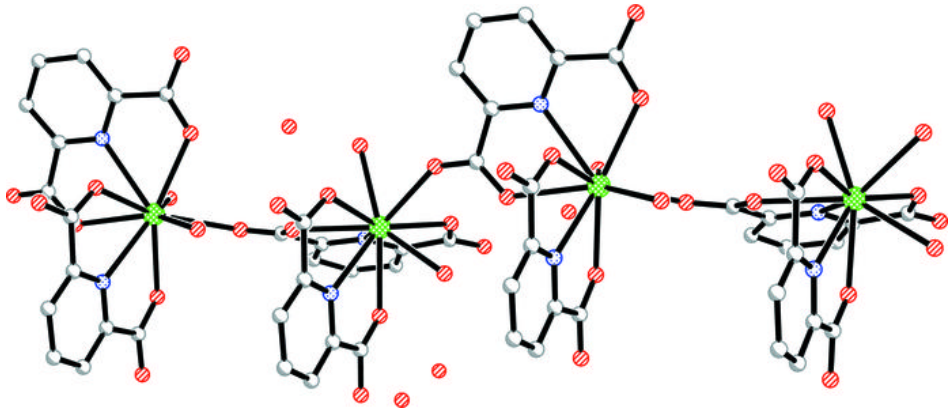

Fig. 3