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A two-dimensional holmium(III) coordination polymer: poly[amino-tris(μ-4-aminobenzoato)holmium(III)]

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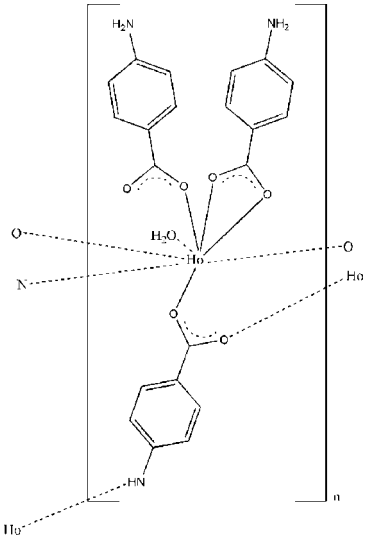
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.021; wR factor = 0.052; data-to-parameter ratio = 12.4.

In the title compound, $[Ho(C_7H_6NO_2)_3(H_2O)]_n$, a two-dimensional coordination polymer, the eight-coordinate Ho^{III} ions are bridged by two carboxylate groups from two 4-aminobenzoate ligands, forming a centrosymmetric dinuclear block. These blocks are further connected by 4-aminobenzoate ligands, yielding a two-dimensional network; a three-dimensional supramolecular structure is then formed *via* hydrogen bonds.

Related literature

For related literature, see: Chen *et al.* (2006); Chu *et al.* (2001); Kepert & Rosseinsky (1998); Reineke *et al.* (1999).



Experimental

Crystal data

$[Ho(C_7H_6NO_2)_3(H_2O)]$
M_r = 591.33
Monoclinic, $P2_1/n$
a = 9.772 (1) Å
b = 22.761 (3) Å
c = 9.832 (1) Å
β = 100.02 (1)°
V = 2153.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.72 mm^{-1}
T = 294 (2) K
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.413, T_{max} = 0.512
4521 measured reflections
4001 independent reflections
2954 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.052
S = 1.03
4001
322 parameters
10 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O6^{i}	0.861 (10)	2.046 (12)	2.902 (3)	172 (3)
N1—H1B···N2^{ii}	0.850 (10)	2.510 (11)	3.360 (5)	177 (3)
N2—H2A···O7^{iii}	0.849 (10)	2.43 (3)	3.139 (5)	142 (3)
N3—H3A···O5^{iv}	0.858 (10)	2.23 (2)	2.974 (4)	145 (3)
O7—H7B···O2	0.816 (10)	2.000 (18)	2.773 (3)	158 (4)
O7—H7A···N3^{v}	0.817 (10)	2.079 (16)	2.875 (4)	165 (5)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2003); 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: GW2020).

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supplementary materials

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A two-dimensional holmium(III) coordination polymer: poly[aminotris(μ-4-aminobenzoato)holmium(III)]

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Comment

During the last two decades, lanthanide complexes have received much attention because of their interesting photophysical properties which have potential applications in the luminescent probes for chemical or biological macromolecules and the active center for luminescent materials(Chu *et al.*, 2001; Kepert & Rosseinsky, 1998; Reineke *et al.*, 1999; Chen *et al.*, 2006;). We have chosen 4-aminobenzonitrile as ligand and investigated its reaction with Ho_2O_3 under hydrothermal synthesis, and report herein the X-ray crystal structure of the novel terbium coordination polymer, $[Ho(4\text{-aminobenzoate})_3(H_2O)]_n$, (I).

In (I) (Fig. 1), the Ho^{III} center is eight-coordinated by six O atoms from five different 4-aminobenzoate ligands, one N atoms from a 4-aminobenzoate ligand and one O atoms from a coordinated water molecule, thus the Ho^{III} ion has a distorted bicapped trigonal prism. In the coordination polyhedron of Ho^{III} ion, the Ho—O distance of carboxyl is in the range 2.337 (2)–2.482 (2) Å, and the mean Ho—O bond length is 2.428 (2) Å. The Ho—O bond length involving the coordination water is 2.546 (2) Å, and the Ho—N distance is 2.691 (3) Å. In (I), each 4-aminobenzoate ligand adopts an O,*O*-bidentate bridging mode using a carboxylate group. Adjacent Ho^{III} centers are doubly bridged by the ligands, forming a big eight-membered ring, while the other two Ho atoms has a 16-membered ring bridged by the carboxylate groups and amino groups (Fig. 2).

Experimental

A mixture of Ho_2O_3 (0.25 mmol), 4-aminobenzonitrile (2.0 mmol), H_2O (10 ml, 0.55 mmol) and two drops of acetic acid with the pH value of about 3.0, was heated in a 25 ml capacity Teflon-lined reaction vessel at 160 ° for 5 days, the reaction mixture was cooled to room temperature over a period of 48 h. The product was collected by filtration, washed with H_2O and air-dried, colorless crystals suitable for X-ray analysis were obtained. Analysis calculated (%): C, 42.62; H, 3.38; N, 7.10%; Found: C, 43.05; H, 3.32%; N, 7.17%.

Refinement

H atoms bonded to C atoms were placed geometrically and treated as riding, (C—H distances are 0.93 Å), with U_{iso}(H) = 1.2U_{eq}(C). The water H atoms found from Fourier difference maps were refined with restraints for O—H distances (0.816–0.817 Å) and U_{iso}(H) fixed at 0.05.

Figures

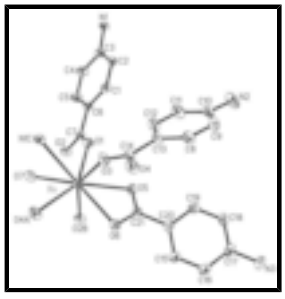


Fig. 1. The *ORTEP* drawing of the title compound (I). Displacement ellipsoids are drawn at 30% probability level. The symmetry codes are: A: −*x* + 1,-*y* + 1,-*z* + 2 B: −*x* + 1,-*y* + 1,-*z* + 1 C: −*x*,-*y* + 1,-*z* + 1

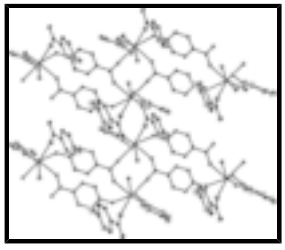


Fig. 2. Projection showing the two-dimensional layer structure of the compound (I).

poly[aminotris(μ-4-aminobenzoato)holmium(III)]

Crystal data

$[Ho(C_7H_6NO_2)_3(H_2O)]$	F_{000} = 1160
M_r = 591.33	D_x = 1.824 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.772 (1) Å	Cell parameters from 30 reflections
b = 22.761 (3) Å	θ = 4.9–13.5º
c = 9.832 (1) Å	μ = 3.72 mm^{-1}
β = 100.02 (1)º	T = 294 (2) K
V = 2153.5 (4) Å^3	Block, colorless
Z = 4	0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4001 independent reflections
Radiation source: fine-focus sealed tube	2954 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.014
T = 294(2) K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = 0→11
T_{min} = 0.413, T_{max} = 0.512	k = 0→27
4521 measured reflections	l = −11→11

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.021$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.052$	$w = 1/[\sigma^2(F_o^2) + (0.027P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
4005 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
322 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}
10 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
Ho	0.481566 (14)	0.501123 (7)	0.758702 (13)	0.01914 (7)
O1	0.3005 (2)	0.52755 (10)	0.5758 (2)	0.0273 (5)
O2	0.3446 (2)	0.48881 (9)	0.3815 (2)	0.0288 (6)
O3	0.3698 (3)	0.54166 (10)	0.9282 (2)	0.0334 (6)
O4	0.3944 (3)	0.58076 (11)	1.1353 (2)	0.0371 (6)
O5	0.5125 (2)	0.60754 (10)	0.7181 (2)	0.0288 (5)
O6	0.6608 (2)	0.56728 (9)	0.8857 (2)	0.0272 (5)
N1	−0.2838 (3)	0.57271 (13)	0.1850 (3)	0.0245 (6)
N2	0.1092 (4)	0.79410 (17)	0.7699 (5)	0.0550 (10)
N3	0.8320 (4)	0.83857 (14)	0.9754 (3)	0.0373 (8)
C1	0.0375 (3)	0.56663 (14)	0.4452 (3)	0.0253 (7)
H1	0.0687	0.5791	0.5354	0.030*
C2	−0.0937 (3)	0.58147 (13)	0.3808 (3)	0.0258 (7)
H2	−0.1493	0.6049	0.4263	0.031*
C3	−0.1444 (3)	0.56162 (14)	0.2473 (3)	0.0242 (7)
C4	−0.0586 (3)	0.52806 (16)	0.1795 (3)	0.0317 (8)

H4	−0.0911	0.5148	0.0903	0.038*
C5	0.0748 (4)	0.51421 (14)	0.2436 (3)	0.0306 (8)
H5	0.1318	0.4920	0.1971	0.037*
C6	0.1244 (3)	0.53352 (14)	0.3786 (3)	0.0219 (7)
C7	0.2652 (3)	0.51582 (12)	0.4497 (3)	0.0217 (7)
C8	0.2766 (4)	0.68773 (15)	1.0271 (4)	0.0343 (8)
H8	0.3135	0.6867	1.1209	0.041*
C9	0.2192 (4)	0.73920 (16)	0.9689 (4)	0.0401 (9)
H9	0.2185	0.7725	1.0237	0.048*
C10	0.1626 (4)	0.74188 (15)	0.8301 (4)	0.0370 (9)
C11	0.1606 (4)	0.69082 (16)	0.7522 (4)	0.0381 (9)
H11	0.1184	0.6912	0.6599	0.046*
C12	0.2196 (4)	0.64017 (15)	0.8089 (3)	0.0339 (8)
H12	0.2193	0.6069	0.7538	0.041*
C13	0.2802 (3)	0.63734 (14)	0.9478 (3)	0.0259 (7)
C14	0.3513 (3)	0.58321 (14)	1.0068 (3)	0.0254 (7)
C15	0.8018 (4)	0.67744 (15)	0.9384 (3)	0.0319 (8)
H15	0.8548	0.6442	0.9657	0.038*
C16	0.8547 (4)	0.73237 (15)	0.9764 (4)	0.0348 (8)
H16	0.9433	0.7358	1.0286	0.042*
C17	0.7770 (4)	0.78271 (14)	0.9375 (3)	0.0303 (8)
C18	0.6457 (4)	0.77666 (15)	0.8593 (4)	0.0384 (9)
H18	0.5922	0.8098	0.8326	0.046*
C19	0.5935 (4)	0.72156 (15)	0.8207 (4)	0.0342 (8)
H19	0.5053	0.7182	0.7676	0.041*
C20	0.6701 (4)	0.67137 (14)	0.8595 (3)	0.0269 (7)
C21	0.6117 (3)	0.61238 (14)	0.8182 (3)	0.0257 (7)
O7	0.4542 (3)	0.41368 (11)	0.5947 (2)	0.0293 (5)
H1A	−0.292 (4)	0.5723 (15)	0.0963 (11)	0.035 (10)*
H1B	−0.311 (3)	0.6058 (8)	0.210 (3)	0.034 (10)*
H2A	0.067 (3)	0.8143 (14)	0.822 (3)	0.042 (12)*
H2B	0.062 (4)	0.792 (2)	0.689 (2)	0.12 (3)*
H3A	0.888 (3)	0.8377 (15)	1.0532 (19)	0.035 (11)*
H3B	0.768 (3)	0.8636 (14)	0.981 (3)	0.054 (14)*
H7A	0.520 (3)	0.3920 (16)	0.590 (5)	0.075 (17)*
H7B	0.427 (4)	0.4282 (16)	0.519 (2)	0.051 (13)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ho	0.01833 (9)	0.01936 (9)	0.01852 (9)	0.00174 (9)	−0.00016 (5)	−0.00054 (8)
O1	0.0259 (12)	0.0290 (12)	0.0234 (12)	0.0024 (10)	−0.0058 (10)	−0.0016 (10)
O2	0.0213 (11)	0.0363 (15)	0.0289 (11)	0.0033 (10)	0.0044 (10)	−0.0005 (10)
O3	0.0408 (15)	0.0235 (12)	0.0386 (14)	0.0086 (11)	0.0142 (12)	0.0004 (11)
O4	0.0403 (15)	0.0374 (15)	0.0291 (13)	0.0108 (12)	−0.0063 (12)	0.0032 (11)
O5	0.0308 (13)	0.0235 (12)	0.0274 (12)	−0.0008 (10)	−0.0082 (11)	−0.0003 (10)
O6	0.0312 (13)	0.0218 (12)	0.0255 (12)	0.0006 (10)	−0.0039 (10)	0.0026 (10)
N1	0.0188 (14)	0.0298 (16)	0.0235 (15)	0.0026 (12)	−0.0002 (12)	−0.0014 (13)

N2	0.062 (3)	0.038 (2)	0.066 (3)	0.0252 (19)	0.013 (2)	0.0180 (19)
N3	0.041 (2)	0.0280 (17)	0.042 (2)	−0.0095 (15)	0.0051 (16)	−0.0054 (14)
C1	0.0233 (17)	0.0300 (18)	0.0205 (15)	−0.0028 (14)	−0.0013 (13)	−0.0046 (14)
C2	0.0230 (17)	0.0264 (17)	0.0276 (17)	0.0012 (14)	0.0036 (14)	−0.0059 (14)
C3	0.0189 (16)	0.0293 (17)	0.0231 (16)	0.0003 (14)	0.0002 (13)	0.0047 (13)
C4	0.0234 (18)	0.048 (2)	0.0216 (16)	0.0016 (17)	−0.0027 (14)	−0.0081 (16)
C5	0.0239 (17)	0.044 (2)	0.0245 (17)	0.0034 (15)	0.0051 (13)	−0.0073 (14)
C6	0.0180 (16)	0.0244 (17)	0.0220 (16)	0.0001 (13)	−0.0001 (13)	0.0035 (13)
C7	0.0189 (16)	0.0184 (17)	0.0272 (16)	−0.0038 (12)	0.0024 (14)	0.0018 (12)
C8	0.032 (2)	0.036 (2)	0.0332 (19)	0.0071 (17)	0.0021 (16)	−0.0032 (16)
C9	0.045 (2)	0.0256 (19)	0.049 (2)	0.0056 (17)	0.0083 (19)	−0.0072 (17)
C10	0.034 (2)	0.0284 (19)	0.051 (2)	0.0120 (16)	0.0146 (18)	0.0098 (17)
C11	0.044 (2)	0.042 (2)	0.0256 (18)	0.0127 (18)	−0.0008 (16)	0.0069 (16)
C12	0.041 (2)	0.0314 (19)	0.0285 (18)	0.0109 (17)	0.0032 (16)	−0.0027 (15)
C13	0.0273 (18)	0.0241 (17)	0.0261 (17)	0.0083 (14)	0.0046 (14)	0.0022 (14)
C14	0.0210 (17)	0.0245 (17)	0.0312 (19)	0.0039 (14)	0.0061 (14)	0.0043 (14)
C15	0.0280 (19)	0.0264 (18)	0.039 (2)	−0.0015 (15)	−0.0017 (16)	0.0009 (16)
C16	0.0270 (19)	0.036 (2)	0.039 (2)	−0.0074 (16)	−0.0015 (16)	−0.0034 (17)
C17	0.037 (2)	0.0258 (18)	0.0297 (18)	−0.0115 (15)	0.0109 (16)	−0.0017 (14)
C18	0.043 (2)	0.0220 (18)	0.047 (2)	0.0039 (16)	0.0012 (19)	−0.0002 (16)
C19	0.030 (2)	0.030 (2)	0.038 (2)	0.0000 (15)	−0.0069 (16)	−0.0002 (16)
C20	0.0326 (19)	0.0226 (17)	0.0241 (17)	−0.0065 (14)	0.0013 (15)	−0.0010 (13)
C21	0.0265 (18)	0.0251 (18)	0.0253 (17)	−0.0017 (14)	0.0038 (15)	−0.0057 (14)
O7	0.0325 (15)	0.0290 (14)	0.0265 (13)	0.0058 (11)	0.0053 (11)	0.0007 (11)

Geometric parameters (Å, °)

Ho—O3	2.337 (2)	C3—C4	1.388 (4)
Ho—O4^{i}	2.364 (2)	C4—C5	1.382 (5)
Ho—O1	2.371 (2)	C4—H4	0.9300
Ho—O2^{ii}	2.379 (2)	C5—C6	1.403 (4)
Ho—O6	2.478 (2)	C5—H5	0.9300
Ho—O5	2.482 (2)	C6—C7	1.487 (4)
Ho—O7	2.546 (2)	C8—C9	1.380 (5)
Ho—N1^{iii}	2.691 (3)	C8—C13	1.391 (5)
O1—C7	1.257 (4)	C8—H8	0.9300
O2—C7	1.270 (4)	C9—C10	1.382 (5)
O2—Ho^{ii}	2.379 (2)	C9—H9	0.9300
O3—C14	1.254 (4)	C10—C11	1.390 (5)
O4—C14	1.263 (4)	C11—C12	1.364 (5)
O4—Ho^{i}	2.364 (2)	C11—H11	0.9300
O5—C21	1.262 (4)	C12—C13	1.393 (4)
O6—C21	1.270 (4)	C12—H12	0.9300
N1—C3	1.417 (4)	C13—C14	1.482 (4)
N1—Ho^{iii}	2.691 (3)	C15—C16	1.379 (4)
N1—H1A	0.861 (10)	C15—C20	1.390 (5)
N1—H1B	0.850 (10)	C15—H15	0.9300
N2—C10	1.389 (5)	C16—C17	1.392 (5)

N2—H2A	0.849 (10)	C16—H16	0.9300
N2—H2B	0.851 (10)	C17—C18	1.384 (5)
N3—C17	1.405 (4)	C18—C19	1.382 (5)
N3—H3A	0.858 (10)	C18—H18	0.9300
N3—H3B	0.856 (10)	C19—C20	1.383 (5)
C1—C2	1.371 (4)	C19—H19	0.9300
C1—C6	1.383 (4)	C20—C21	1.488 (4)
C1—H1	0.9300	O7—H7A	0.817 (10)
C2—C3	1.395 (4)	O7—H7B	0.816 (10)
C2—H2	0.9300		
O3—Ho—O4^{i}	105.32 (8)	C2—C3—N1	120.5 (3)
O3—Ho—O1	93.52 (8)	C5—C4—C3	120.5 (3)
O4^{i}—Ho—O1	142.40 (8)	C5—C4—H4	119.8
O3—Ho—O2^{ii}	147.74 (8)	C3—C4—H4	119.8
O4^{i}—Ho—O2^{ii}	88.18 (8)	C4—C5—C6	120.2 (3)
O1—Ho—O2^{ii}	92.87 (8)	C4—C5—H5	119.9
O3—Ho—O6	77.16 (8)	C6—C5—H5	119.9
O4^{i}—Ho—O6	89.61 (8)	C1—C6—C5	118.7 (3)
O1—Ho—O6	126.70 (7)	C1—C6—C7	121.3 (3)
O2^{ii}—Ho—O6	73.72 (7)	C5—C6—C7	120.0 (3)
O3—Ho—O5	79.32 (8)	O1—C7—O2	122.4 (3)
O4^{i}—Ho—O5	140.61 (8)	O1—C7—C6	118.7 (3)
O1—Ho—O5	74.09 (7)	O2—C7—C6	118.9 (3)
O2^{ii}—Ho—O5	72.15 (8)	C9—C8—C13	121.0 (3)
O6—Ho—O5	52.62 (7)	C9—C8—H8	119.5
O3—Ho—O7	138.60 (8)	C13—C8—H8	119.5
O4^{i}—Ho—O7	69.67 (8)	C8—C9—C10	120.8 (3)
O1—Ho—O7	74.66 (8)	C8—C9—H9	119.6
O2^{ii}—Ho—O7	73.43 (8)	C10—C9—H9	119.6
O6—Ho—O7	141.40 (8)	C9—C10—N2	121.0 (4)
O5—Ho—O7	131.53 (7)	C9—C10—C11	118.2 (3)
O3—Ho—N1^{iii}	69.98 (8)	N2—C10—C11	120.8 (4)
O4^{i}—Ho—N1^{iii}	75.45 (9)	C12—C11—C10	121.1 (3)
O1—Ho—N1^{iii}	81.16 (8)	C12—C11—H11	119.4
O2^{ii}—Ho—N1^{iii}	142.27 (8)	C10—C11—H11	119.4
O6—Ho—N1^{iii}	138.27 (8)	C11—C12—C13	121.2 (3)
O5—Ho—N1^{iii}	138.98 (8)	C11—C12—H12	119.4
O7—Ho—N1^{iii}	69.05 (8)	C13—C12—H12	119.4
O3—Ho—C21	75.52 (9)	C8—C13—C12	117.6 (3)
O4^{i}—Ho—C21	115.69 (9)	C8—C13—C14	121.5 (3)
O1—Ho—C21	100.27 (9)	C12—C13—C14	120.8 (3)
O2^{ii}—Ho—C21	72.24 (8)	O3—C14—O4	121.7 (3)
O6—Ho—C21	26.44 (8)	O3—C14—C13	119.6 (3)
O5—Ho—C21	26.24 (8)	O4—C14—C13	118.7 (3)
O7—Ho—C21	144.97 (9)	C16—C15—C20	120.6 (3)

N1^{iii}—Ho—C21	145.48 (9)	C16—C15—H15	119.7
C7—O1—Ho	137.4 (2)	C20—C15—H15	119.7
C7—O2—Ho^{ii}	145.5 (2)	C15—C16—C17	120.7 (3)
C14—O3—Ho	150.8 (2)	C15—C16—H16	119.6
C14—O4—Ho^{i}	122.2 (2)	C17—C16—H16	119.6
C21—O5—Ho	93.32 (19)	C18—C17—C16	118.7 (3)
C21—O6—Ho	93.30 (18)	C18—C17—N3	120.9 (3)
C3—N1—Ho^{iii}	118.1 (2)	C16—C17—N3	120.4 (3)
C3—N1—H1A	111 (2)	C19—C18—C17	120.4 (3)
Ho^{iii}—N1—H1A	105 (2)	C19—C18—H18	119.8
C3—N1—H1B	111 (2)	C17—C18—H18	119.8
Ho^{iii}—N1—H1B	103 (2)	C18—C19—C20	121.2 (3)
H1A—N1—H1B	109 (3)	C18—C19—H19	119.4
C10—N2—H2A	113 (3)	C20—C19—H19	119.4
C10—N2—H2B	117 (4)	C19—C20—C15	118.5 (3)
H2A—N2—H2B	111.4 (18)	C19—C20—C21	120.4 (3)
C17—N3—H3A	112 (2)	C15—C20—C21	121.1 (3)
C17—N3—H3B	112 (3)	O5—C21—O6	120.5 (3)
H3A—N3—H3B	108.6 (16)	O5—C21—C20	119.9 (3)
C2—C1—C6	121.2 (3)	O6—C21—C20	119.6 (3)
C2—C1—H1	119.4	O5—C21—Ho	60.44 (16)
C6—C1—H1	119.4	O6—C21—Ho	60.26 (16)
C1—C2—C3	120.4 (3)	C20—C21—Ho	174.2 (2)
C1—C2—H2	119.8	Ho—O7—H7A	120 (3)
C3—C2—H2	119.8	Ho—O7—H7B	104 (3)
C4—C3—C2	119.0 (3)	H7A—O7—H7B	110 (4)
C4—C3—N1	120.4 (3)		
O3—Ho—O1—C7	−167.7 (3)	C13—C8—C9—C10	0.5 (6)
O4^{i}—Ho—O1—C7	−46.8 (4)	C8—C9—C10—N2	−177.6 (4)
O2^{ii}—Ho—O1—C7	44.0 (3)	C8—C9—C10—C11	2.3 (6)
O6—Ho—O1—C7	115.8 (3)	C9—C10—C11—C12	−3.5 (6)
O5—Ho—O1—C7	114.5 (3)	N2—C10—C11—C12	176.4 (4)
O7—Ho—O1—C7	−28.0 (3)	C10—C11—C12—C13	1.9 (6)
N1^{iii}—Ho—O1—C7	−98.6 (3)	C9—C8—C13—C12	−2.2 (6)
C21—Ho—O1—C7	116.4 (3)	C9—C8—C13—C14	175.2 (3)
O4^{i}—Ho—O3—C14	116.5 (5)	C11—C12—C13—C8	1.0 (6)
O1—Ho—O3—C14	−96.4 (5)	C11—C12—C13—C14	−176.4 (3)
O2^{ii}—Ho—O3—C14	4.7 (5)	Ho—O3—C14—O4	−107.2 (5)
O6—Ho—O3—C14	30.5 (5)	Ho—O3—C14—C13	71.2 (6)
O5—Ho—O3—C14	−23.3 (4)	Ho^{i}—O4—C14—O3	1.3 (4)
O7—Ho—O3—C14	−167.0 (4)	Ho^{i}—O4—C14—C13	−177.1 (2)
N1^{iii}—Ho—O3—C14	−175.6 (5)	C8—C13—C14—O3	−169.8 (3)
C21—Ho—O3—C14	3.3 (4)	C12—C13—C14—O3	7.5 (5)
O3—Ho—O5—C21	78.9 (2)	C8—C13—C14—O4	8.7 (5)
O4^{i}—Ho—O5—C21	−22.3 (2)	C12—C13—C14—O4	−174.1 (3)
O1—Ho—O5—C21	175.7 (2)	C20—C15—C16—C17	0.4 (5)

O2^{ii}—Ho—O5—C21	−85.9 (2)	C15—C16—C17—C18	−0.3 (5)
O6—Ho—O5—C21	−3.06 (18)	C15—C16—C17—N3	−179.3 (3)
O7—Ho—O5—C21	−132.63 (19)	C16—C17—C18—C19	−0.1 (5)
N1^{iii}—Ho—O5—C21	120.5 (2)	N3—C17—C18—C19	178.9 (3)
O3—Ho—O6—C21	−83.24 (19)	C17—C18—C19—C20	0.4 (6)
O4^{i}—Ho—O6—C21	170.95 (19)	C18—C19—C20—C15	−0.3 (5)
O1—Ho—O6—C21	1.5 (2)	C18—C19—C20—C21	179.4 (3)
O2^{ii}—Ho—O6—C21	82.74 (19)	C16—C15—C20—C19	−0.1 (5)
O5—Ho—O6—C21	3.04 (18)	C16—C15—C20—C21	−179.8 (3)
O7—Ho—O6—C21	115.4 (2)	Ho—O5—C21—O6	5.5 (3)
N1^{iii}—Ho—O6—C21	−121.70 (19)	Ho—O5—C21—C20	−173.3 (3)
C6—C1—C2—C3	2.2 (5)	Ho—O6—C21—O5	−5.5 (3)
C1—C2—C3—C4	−1.8 (5)	Ho—O6—C21—C20	173.3 (3)
C1—C2—C3—N1	174.7 (3)	C19—C20—C21—O5	20.0 (5)
Ho^{iii}—N1—C3—C4	92.5 (3)	C15—C20—C21—O5	−160.3 (3)
Ho^{iii}—N1—C3—C2	−83.9 (3)	C19—C20—C21—O6	−158.8 (3)
C2—C3—C4—C5	0.4 (5)	C15—C20—C21—O6	20.8 (5)
N1—C3—C4—C5	−176.1 (3)	O3—Ho—C21—O5	−95.2 (2)
C3—C4—C5—C6	0.6 (5)	O4^{i}—Ho—C21—O5	164.48 (17)
C2—C1—C6—C5	−1.2 (5)	O1—Ho—C21—O5	−4.2 (2)
C2—C1—C6—C7	−178.1 (3)	O2^{ii}—Ho—C21—O5	85.51 (19)
C4—C5—C6—C1	−0.2 (5)	O6—Ho—C21—O5	174.5 (3)
C4—C5—C6—C7	176.7 (3)	O7—Ho—C21—O5	73.7 (2)
Ho—O1—C7—O2	−11.6 (5)	N1^{iii}—Ho—C21—O5	−93.5 (2)
Ho—O1—C7—C6	167.5 (2)	O3—Ho—C21—O6	90.23 (19)
Ho^{ii}—O2—C7—O1	−91.0 (4)	O4^{i}—Ho—C21—O6	−10.1 (2)
Ho^{ii}—O2—C7—C6	89.9 (4)	O1—Ho—C21—O6	−178.77 (18)
C1—C6—C7—O1	5.5 (4)	O2^{ii}—Ho—C21—O6	−89.03 (19)
C5—C6—C7—O1	−171.3 (3)	O5—Ho—C21—O6	−174.5 (3)
C1—C6—C7—O2	−175.3 (3)	O7—Ho—C21—O6	−100.9 (2)
C5—C6—C7—O2	7.8 (4)	N1^{iii}—Ho—C21—O6	91.9 (2)

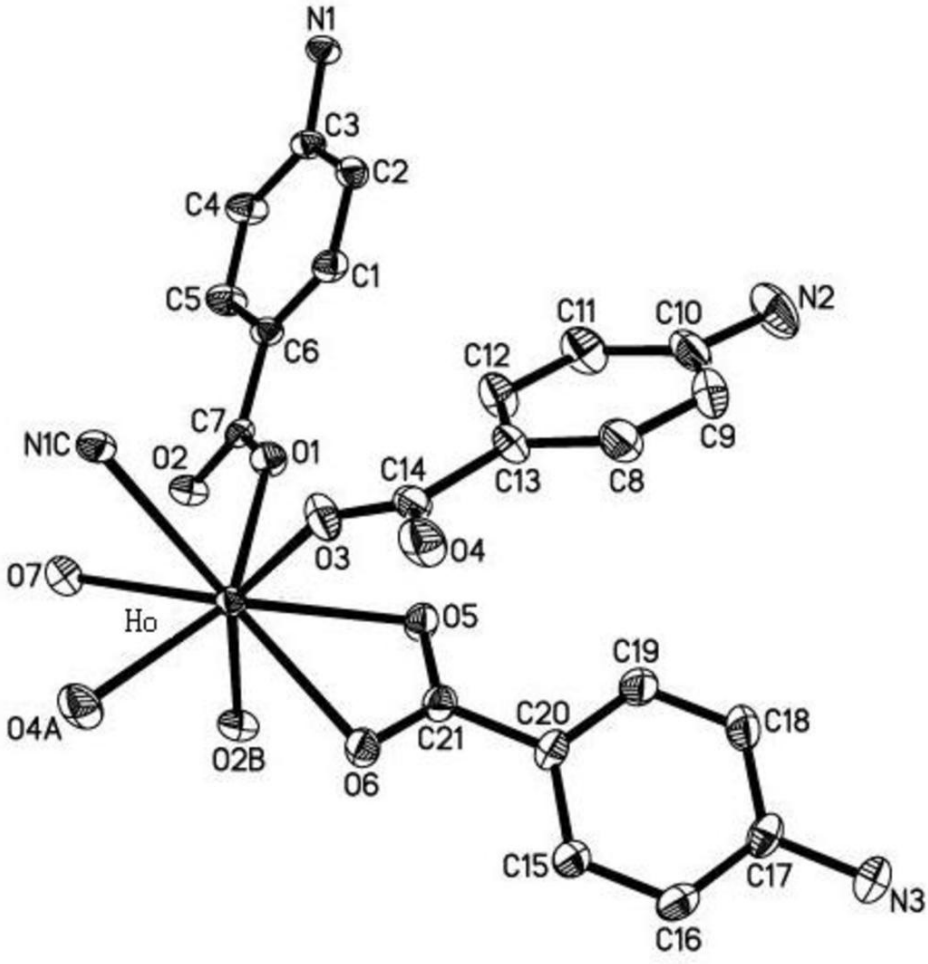
Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O6^{iv}	0.861 (10)	2.046 (12)	2.902 (3)	172 (3)
N1—H1B···N2^{v}	0.850 (10)	2.510 (11)	3.360 (5)	177 (3)
N2—H2A···O7^{vi}	0.849 (10)	2.43 (3)	3.139 (5)	142 (3)
N3—H3A···O5^{vii}	0.858 (10)	2.23 (2)	2.974 (4)	145 (3)
O7—H7B···O2	0.816 (10)	2.000 (18)	2.773 (3)	158 (4)
O7—H7A···N3^{viii}	0.817 (10)	2.079 (16)	2.875 (4)	165 (5)

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

Fig. 1


N1
C3
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C9
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N1C
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C14
C13
C8
O3
O4
O7
Ho
O5
C19
O4A
O2B
C21
C20
C18
O6
C15
C17
N3
C16

Fig. 2