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Poly[hexaaquabis(μ_3-naphthalene-2,6-dicarboxylato)(μ_2-naphthalene-2,6-dicarboxylato)diholmium(III)]

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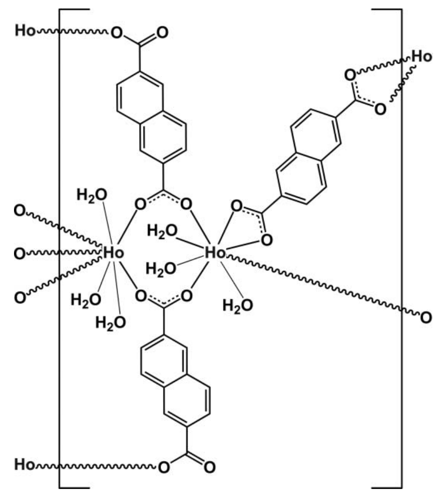
Key indicators: single-crystal X-ray study; T = 180 K; mean σ(C–C) = 0.008 Å; R factor = 0.043; wR factor = 0.079; data-to-parameter ratio = 14.7.

The crystal structure of the title compound, $[Ho_2(C_{12}H_6O_4)_3(H_2O)_6]_n$, contains binuclear centrosymmetric $\{Ho_2O_2(CO_2)_4(H_2O)_6\}$ cores interconnected *via* the naphthalene-2,6-dicarboxylate (NDC^{2-}) bridging ligands into a two-dimensional neutral plane net, $\infty^2[Ho_2(NDC)_3(H_2O)_6]$, exhibiting a typical (4,4)-topology. Interactions between adjacent layers are assured by a series of C—H$\cdots\pi$ contacts and a number of strong and highly directional O—H$\cdots$O hydrogen bonds involving the coordinated water molecules and neighbouring coordinated carboxylate groups. One NDC^{2-} bridging ligand has its centroid located at a crystallographic centre of inversion.

Related literature

For related structures see: Zheng, Sun *et al.* (2004); Zheng, Wang *et al.* (2004); Paz & Klinowski (2003); Min & Lee (2002); Wang *et al.* (2002). For related literature, see: Cunha-Silva, Mafra *et al.* (2007); Cunha-Silva, Shi *et al.* (2007); Shi *et al.* (2007); Mafra *et al.* (2006); Shi *et al.* (2006); Paz, Rocha, Klinowski *et al.* (2005); Almeida Paz, Shi, Mafra *et al.* (2005); Almeida Paz, Shi, Trindade *et al.* (2005); Shi *et al.* (2005); Paz & Klinowski (2004); Almeida Paz *et al.* (2002*a*,*b*,*c*); Allen (2002); Allen & Motherwell (2002); Altomare *et al.* (1994); Deluzet *et al.* (2003).



Experimental

Crystal data

$[Ho_2(C_{12}H_6O_4)_3(H_2O)_6]$
M_r = 540.23
Triclinic, $P\overline{1}$
a = 7.8856 (3) Å
b = 9.6537 (5) Å
c = 12.5438 (6) Å
α = 75.191 (2)°
β = 74.224 (2)°
γ = 75.352 (2)°
V = 870.98 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.60 mm^{-1}
T = 180 (2) K
0.10 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.730, T_{max} = 0.796
11608 measured reflections
3987 independent reflections
3135 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.079
S = 1.00
3987 reflections
271 parameters
9 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.51 e Å^{-3}
$\Delta\rho_{min}$ = −1.50 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ho1—O1	2.267 (4)	Ho1—O6	2.450 (4)
Ho1—O3^{i}	2.252 (3)	Ho1—O1W	2.370 (4)
Ho1—O4^{ii}	2.279 (4)	Ho1—O2W	2.461 (4)
Ho1—O5	2.389 (3)	Ho1—O3W	2.366 (4)
O1—Ho1—O4^{ii}	144.69 (15)	O4^{ii}—Ho1—O1W	140.01 (13)
O1—Ho1—O5	98.23 (13)	O4^{ii}—Ho1—O2W	72.11 (13)
O1—Ho1—O6	77.54 (13)	O4^{ii}—Ho1—O3W	78.31 (14)
O1—Ho1—O1W	72.71 (14)	O5—Ho1—O6	54.22 (13)
O1—Ho1—O2W	142.57 (14)	O5—Ho1—O2W	76.91 (13)
O1—Ho1—O3W	76.58 (14)	O6—Ho1—O2W	123.35 (12)
O3^{i}—Ho1—O1	101.02 (13)	O1W—Ho1—O5	78.18 (13)
O3^{i}—Ho1—O4^{ii}	96.82 (14)	O1W—Ho1—O6	118.31 (14)
O3^{i}—Ho1—O5	147.95 (14)	O1W—Ho1—O2W	69.97 (13)
O3^{i}—Ho1—O6	155.80 (14)	O3W—Ho1—O5	133.18 (14)
O3^{i}—Ho1—O1W	83.38 (14)	O3W—Ho1—O6	79.62 (14)
O3^{i}—Ho1—O2W	72.20 (13)	O3W—Ho1—O2W	133.39 (14)
O3^{i}—Ho1—O3W	76.60 (14)	O3W—Ho1—O1W	139.08 (13)
O4^{ii}—Ho1—O5	81.43 (13)		
O4^{ii}—Ho1—O6	73.82 (13)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H1$A\cdots$O2^{iii}	0.95 (4)	1.82 (2)	2.725 (5)	157 (5)
O1W—H1$B\cdots$O5^{iii}	0.95 (4)	1.95 (3)	2.818 (5)	150 (4)
O2W—H2$A\cdots$O2^{iv}	0.95 (4)	1.98 (4)	2.782 (5)	140 (5)
O2W—H2$B\cdots$O2^{iii}	0.95 (4)	2.14 (4)	2.901 (6)	136 (4)
O3W—H3$A\cdots$O6^{v}	0.95 (4)	1.78 (4)	2.704 (5)	165 (5)
O3W—H3$B\cdots$O2W^{vi}	0.95 (4)	2.26 (4)	3.181 (6)	165 (4)
O3W—H3$B\cdots$O4^{i}	0.95 (4)	2.53 (5)	3.145 (6)	123 (4)

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Bruker, 2001); molecular graphics: *DIAMOND* (Brandenburg, 2006); 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: HJ2008).

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supporting information

Acta Cryst. (2008). E**64**, m140–m141 [https://doi.org/10.1107/S1600536807064483]

Poly[hexaaquabis(μ_3-naphthalene-2,6-dicarboxylato)(μ_2-naphthalene-2,6-dicarboxylato)diholmium(III)]

Filipe A. Almeida Paz and Jacek Klinowski

S1. Comment

In less than twenty years, the field of Crystal Engineering involving the synthesis and characterization of multi-dimensional metal-organic frameworks (also known as coordination polymers) has grown immensely to become one of the most active research areas in inorganic chemistry. These worldwide efforts are motivated by the new and often striking structural features obtained by varying the metal centres and the bridging organic ligands, and by the prospect of making materials with direct industrial applications. Following our efforts in the hydrothermal synthesis and structural characterization of highly crystalline materials of this kind, (Cunha-Silva, Mafra *et al.*, 2007; Cunha-Silva, Shi *et al.*, 2007; Shi *et al.*, 2007; Mafra *et al.*, 2006; Shi *et al.*, 2006; Paz, Rocha, Klinowski *et al.*, 2005; Almeida Paz, Shi, Mafra *et al.*, 2005; Almeida Paz, Shi, Trindade *et al.*, 2005; Shi *et al.*, 2005; Paz & Klinowski, 2004; Paz & Klinowski, 2003; Almeida Paz *et al.* 2002*a*, 2002*b*, 2002*c*), we report here the low temperature crystal structure at 180 (2) K of a two-dimensional lanthanide-organic framework containing residues of naphthalene-2,6-dicarboxylic acid (H_2NDC), $[Ho_2(NDC)_3(H_2O)_6]$, which is analogous to that reported by Deluzet *et al.* (2003) but containing instead Er^{3+}, $[Er_2(NDC)_3(H_2O)_6]$. A search in the literature and in the Cambridge Structural Database (CSD, Version 5.28 with three updates - August 2007; Allen, 2002; Allen & Motherwell, 2002) produced only a handful of reports in which lanthanide centres are coordinated to $H_{2-x}NDC^{-x}$ residues (Zheng, Sun *et al.*, 2004; Zheng, Wang *et al.*, 2004; Paz & Klinowski, 2003; Wang *et al.*, 2002; Min & Lee, 2002).

The structure of the title compound, I, contains a single crystallographically independent metallic centre, Ho1, coordinated to three water molecules (O1W, O2W and O3W) and four NDC^{2-} bridging ligands (Figure 1a), with a $\{HoO_8\}$ coordination geometry resembling a highly distorted dodecahedron (Figure 1 b). The Ho—O bond lengths were found in the 2.252 (3)–2.461 (4) Å range,in good agreement with those of related materials as revealed by a search in the CSD. The three crystallographically independent carboxylate groups coordinate to the Ho^{3+} centres in distinct coordination fashions as shown in Figure 1a. Notably, the C8 carboxylate group is coordinated *via* a typical *syn,syn*-μ_2-bridging coordination fashion leading to the formation of binuclear centrosymmetric anionic $[Ho_2(NDC)_6(H_2O)_6]^{6-}$ unit (Figure 1) with the Ho(1)···Ho(1)^{vi} intermetallic distance being of 5.0172 (4) Å [symmetry code: (vi) *3 - x*, *-y*, *-z*]. While the C1 carboxylate group is coordinated *via* a *syn*-unidentate coordination fashion, the C13 carboxylate is instead bound to Ho1 *via* a typical *syn,syn*-chelate bidentate mode with a bite angle of 54.22 (13)°.

$\{Ho_2O_2(CO_2)_4(H_2O)_6\}$ cores are interconnected *via* the bridging NDC^{2-}ligands into an inclined two-dimensional plane net (Figure 2). By taking the centre of gravity of each binuclear centrosymmetric anionic $[Ho_2(NDC)_6(H_2O)_6]^{6-}$ unit as a node of the network, the resulting $\infty^2[Ho_2(NDC)_3(H_2O)_6]$ plane net has a typical (4,4) topology with the inter-nodal distances being of 12.8742 (6) Å and 16.3127 (6) Å. As shown in Figures 3a and 3 b, individual $\infty^2[Ho_2(NDC)_3(H_2O)_6]$ plane nets close pack in a parallel fashion (not along a principal axis of the unit cell) to produce the crystal structure.

Along the [010] crystallographic direction the packing occurs in an orderly ABAB··· fashion (Figure 3 b). Connections between adjacent layers are mainly assured by strong and highly directional O—H···.O hydrogen bonds involving the O2W and O3W coordinated water molecules from one layer and the coordinated carboxylate groups from the neighbouring layer (Figure 4 and Table in the main paper summarizing the geometrical parameters of the hydrogen bonding interactions). Moreover, these connections are reinforced by weak C—H···*π* interactions between coordinated NDC^{2-} residues belonging to adjacent layers (not shown). It is important to stress that, within each $\infty^2[Ho_2(NDC)_3(H_2O)_6]$ layer, O1W is also engaged in strong O—H···.O hydrogen bonds which reinforce the connections between neighbouring binuclear units (Figure 4).

S2. Experimental

Starting materials were purchased from commercial sources and were used as received without further purification: holmium(III) chloride hexahydrate ($HoCl_3.6H_2O$, 99.9%, Aldrich), naphthalene-2,6-dicarboxylic acid (H_2NDC, 99%, Aldrich) and triethylamine (TEA, 99%, Avocado).

To a solution of $HoCl_3.6H_2O$ (1.054 g, 2.778 mmol) in distilled water (6.88 g), naphthalene-2,6-dicarboxylic acid (0.100 g, 0.463 mmol) and triethylamine (0.097 g, 0.959 mmol) were added and the mixture was stirred thoroughly for 5 minutes at ambient temperature. The suspension, with a molar composition of 6.01 Ho^{3+}: 1.00 H_2NDC: 2.07 TEA: 137 H_2O, was transferred to a Parr teflon-lined stainless steel vessel (*ca* 21 cm^3) and placed for 8 h at 145 °C in a preheated oven. Before opening, the reaction vessel was allowed to cool slowly to ambient temperature at a rate of 10 ° per hour over a period of 14 h. The isolated crystalline material was mainly composed of crystals of the title compound which were preserved in a portion of the mother liquor before being manually selected under a polarized microscope for subsequent crystal mounting on a glass fibre.

A small amount of colourless plate-like crystals, which could not be physically separated from the title compound, were also investigated and revealed to be isostructural with the frameworks reported by Zheng, Sun *et al.* (2004). The crystal data for this material will be the subject of a separate communication.

S3. Refinement

A slightly smeared-out electron density was found surrounding the carbon atoms of one bridging naphthalene-2,6-di-carboxylate ligand. However, the quality of the data set did not allow a sensible modelling of this disorder over, at least, two istinct crystallographic positions. C3, C4, C9 and C10 atoms were instead refined using anisotropic displacement parameters which define a typical prolate thermal motion for these atoms.

H atoms associated with the water molecules were clearly visible in difference Fourier maps and were included in the final structural model with the O—H and H···H restrained to 0.95 (1) and 1.55 (1) Å, respectively, in order to ensure a chemically reasonable geometry for these moieties. These H atoms were allowed to ride on their parent O atoms with U_{iso} fixed at $1.5\times U_{eq}(O)$. H atoms bound to carbon were instead placed at idealized positions and allowed to ride on their parent atoms with U_{iso} fixed at $1.2\times U_{eq}(C)$. All C—H distances are of 0.95 Å.

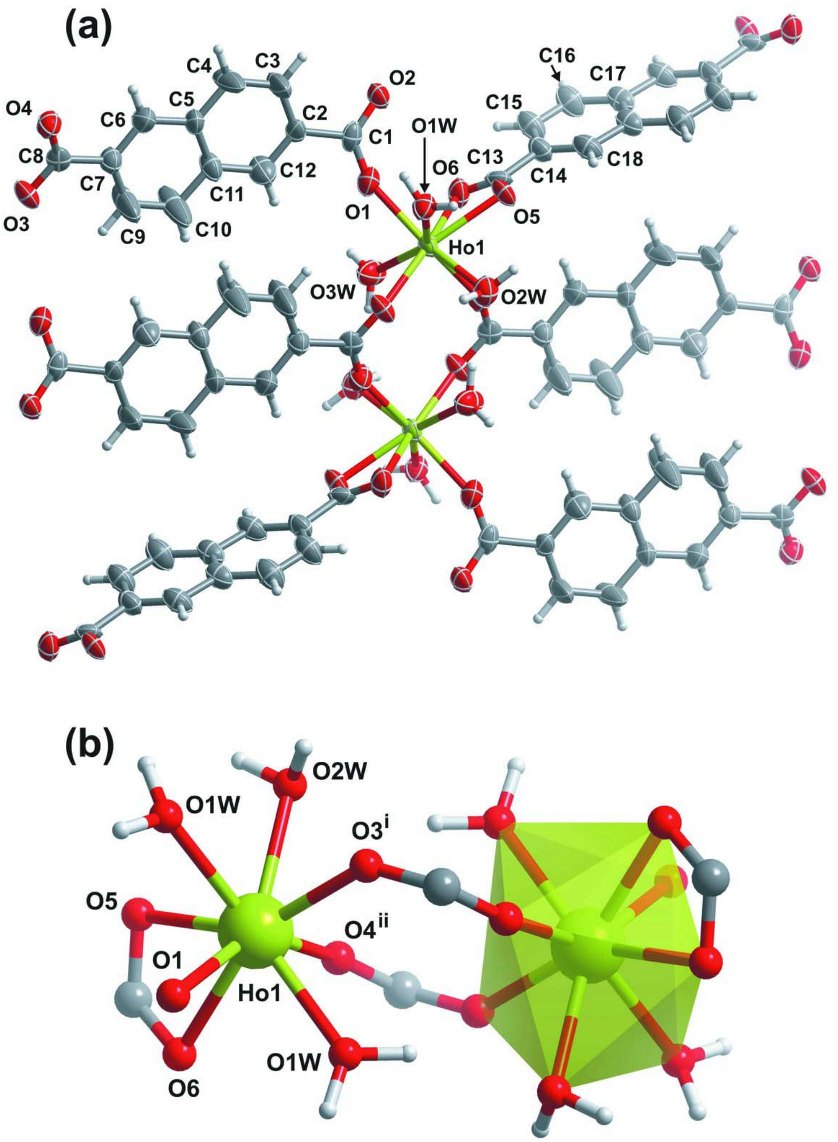

Figure 1

(*a*) Schematic representation of the binuclear centrosymmetric anionic $[Ho_2(NDC)_6(H_2O)_6]^{6-}$ unit showing the labelling scheme for all non-H atoms composing the asymmetric unit. Displacement ellipsoids are drawn at the 80% probability level and H atoms are represented as small spheres with arbitrary radii. (*b*) Magnification of the $\{Ho_2O_2(CO_2)_4(H_2O)_6\}$ core of the binuclear unit, emphasizing the highly distorted $\{HoO_8\}$ dodecahedral coordination environment for the Ho^{3+} centres. For selected bond lengths and angles of the $\{HoO_8\}$ coordination polyhedron see the Table summarizing the geometrical details. Symmetry codes used to generate equivalent atoms: (i) *2 - x*, *-y*, *1 - z*; (ii) *1 + x*, *y*, *-1 + z*.

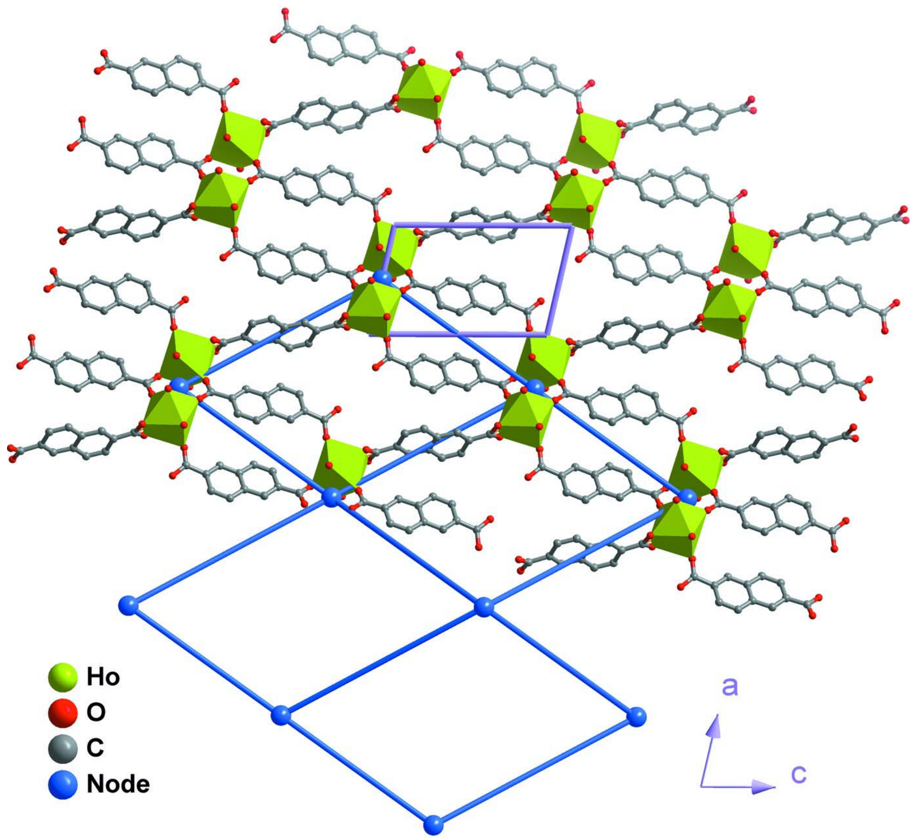

Figure 2

Mixed polyhedral and ball-and-stick schematic representation of the two-dimensional (4,4) plane net formed by the self-assembly of the binuclear centrosymmetric anionic $[Ho_2(NDC)_6(H_2O)_6]^{6-}$ units depicted in Fig. 1. The centres of gravity of the binuclear units were taken as the nodes of the network (blue spheres). Inter-nodal distances: 12.8742 (6) Å and 16.3127 (6) Å. Hydrogen atoms have been omitted for clarity.

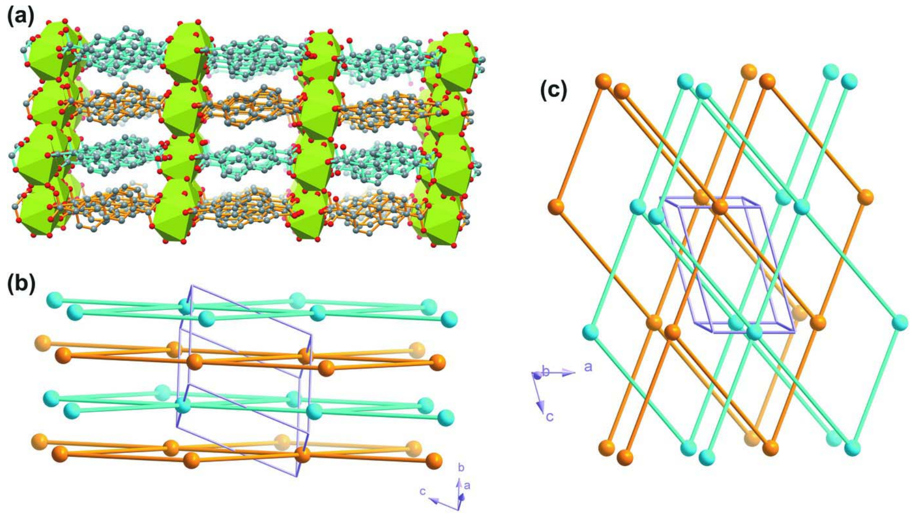

Figure 3

(*a*) Mixed polyhedral and ball-and-stick and (*b*) and (*c*) topological representations of the crystal packing of the title compound. Hydrogen atoms have been omitted for clarity and adjacent (4,4) plane nets are represented with alternating colours.

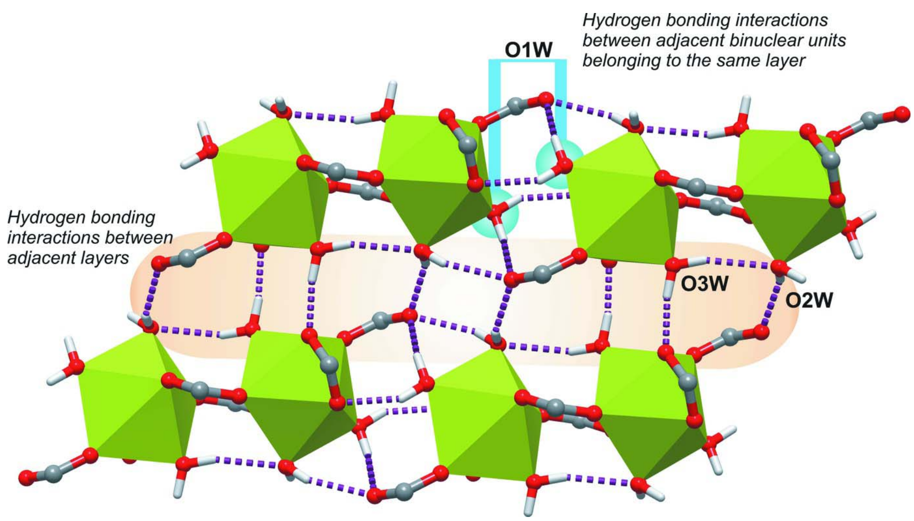

Figure 4

O—H···O hydrogen bonding interactions connecting adjacent binuclear units within the two-dimensional layer (*via* O1W) and between adjacent layers (*via* O2W and O3W). Hydrogen bonds are represented as dashed purple lines. For geometrical details on the represented hydrogen bonding interactions see dedicated Table in the main paper.

poly[hexaaquabis(μ_3-naphthalene-2,6-dicarboxylato)(μ_2-naphthalene-2,6- dicarboxylato)diholmium(III)]

Crystal data

$[Ho_2(C_{12}H_6O_4)_3(H_2O)_6]$	c = 12.5438 (6) Å
M_r = 540.23	α = 75.191 (2)°
Triclinic, $P\overline{1}$	β = 74.224 (2)°
Hall symbol: -P 1	γ = 75.352 (2)°
a = 7.8856 (3) Å	V = 870.98 (7) Å^3
b = 9.6537 (5) Å	Z = 2

$F(000) = 524$
$D_x = 2.060$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14333 reflections
$\theta = 1.0–27.5°$
$\mu = 4.60$ mm^{-1}
$T = 180$ K
Block, white
0.10 × 0.05 × 0.05 mm

Data collection

Nonius Kappa CCD diffractometer
Radiation source: fine-focus sealed tube
Thin slice ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.730$, $T_{max} = 0.796$
11608 measured reflections
3987 independent reflections
3135 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.5°$
$h = -10 \rightarrow 9$
$k = -9 \rightarrow 12$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.079$
$S = 1.00$
3987 reflections
271 parameters
9 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
$w = 1/[\sigma^2(F_o^2) + (0.0301P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.51$ e Å^{-3}
$\Delta\rho_{min} = -1.50$ e Å^{-3}

Special details

Experimental. See dedicated section in the main paper

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Ho1	1.23306 (3)	0.20677 (3)	−0.001555 (19)	0.01786 (10)
O1W	1.1573 (5)	0.4195 (4)	0.0764 (3)	0.0232 (9)
H1A	1.207 (6)	0.504 (3)	0.039 (4)	0.035*
H1B	1.039 (3)	0.450 (5)	0.118 (4)	0.035*
O2W	1.4902 (5)	0.3308 (4)	−0.0586 (3)	0.0259 (10)
H2A	1.506 (8)	0.340 (5)	0.011 (2)	0.039*
H2B	1.466 (8)	0.425 (2)	−0.105 (3)	0.039*
O3W	1.2069 (5)	−0.0416 (4)	0.0556 (3)	0.0301 (10)
H3A	1.104 (4)	−0.083 (5)	0.073 (5)	0.045*
H3B	1.311 (4)	−0.116 (4)	0.057 (5)	0.045*

O1	0.9506 (5)	0.2157 (4)	0.1072 (3)	0.0280 (10)
O2	0.6931 (5)	0.3656 (4)	0.0793 (3)	0.0241 (9)
O3	0.6021 (5)	−0.1174 (4)	0.8691 (3)	0.0269 (10)
O4	0.4373 (5)	0.1075 (4)	0.8570 (3)	0.0251 (9)
O5	1.1644 (5)	0.3945 (4)	−0.1584 (3)	0.0236 (9)
O6	1.0619 (5)	0.1934 (5)	−0.1337 (3)	0.0264 (10)
C1	0.7893 (8)	0.2736 (6)	0.1411 (4)	0.0199 (13)
C2	0.7080 (7)	0.2329 (7)	0.2662 (4)	0.0228 (14)
C3	0.5563 (10)	0.3242 (9)	0.3151 (5)	0.064 (3)
H3	0.4986	0.4070	0.2690	0.076*
C4	0.4889 (11)	0.2952 (10)	0.4304 (5)	0.087 (4)
H4	0.3852	0.3585	0.4629	0.105*
C5	0.5711 (8)	0.1739 (7)	0.5001 (4)	0.0257 (14)
C6	0.5125 (8)	0.1470 (7)	0.6203 (4)	0.0317 (16)
H6	0.4139	0.2127	0.6549	0.038*
C7	0.5957 (8)	0.0288 (6)	0.6862 (4)	0.0219 (13)
C8	0.5390 (7)	0.0055 (6)	0.8131 (4)	0.0183 (12)
C9	0.7352 (12)	−0.0676 (9)	0.6357 (5)	0.068 (3)
H9	0.7895	−0.1530	0.6811	0.081*
C10	0.7972 (13)	−0.0426 (9)	0.5211 (5)	0.084 (4)
H10	0.8956	−0.1102	0.4886	0.101*
C11	0.7190 (8)	0.0806 (6)	0.4501 (4)	0.0250 (14)
C12	0.7875 (9)	0.1138 (7)	0.3314 (4)	0.0318 (15)
H12	0.8908	0.0512	0.2975	0.038*
C13	1.0862 (7)	0.3182 (7)	−0.1918 (4)	0.0217 (13)
C14	1.0289 (8)	0.3725 (7)	−0.3018 (4)	0.0242 (14)
C15	0.9115 (8)	0.3065 (7)	−0.3296 (4)	0.0286 (15)
H15	0.8619	0.2299	−0.2759	0.034*
C16	0.8682 (9)	0.3509 (7)	−0.4318 (4)	0.0307 (15)
H16	0.7879	0.3056	−0.4492	0.037*
C17	0.9428 (8)	0.4657 (7)	−0.5143 (4)	0.0241 (13)
C18	1.0982 (8)	0.4868 (6)	−0.3770 (4)	0.0264 (14)
H18	1.1732	0.5338	−0.3562	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ho1	0.01942 (16)	0.02085 (16)	0.01278 (13)	−0.00448 (11)	−0.00501 (9)	−0.00036 (9)
O1W	0.024 (2)	0.023 (2)	0.0200 (19)	−0.0050 (18)	−0.0003 (16)	−0.0053 (16)
O2W	0.027 (2)	0.030 (3)	0.023 (2)	−0.012 (2)	−0.0058 (18)	−0.0037 (17)
O3W	0.022 (2)	0.024 (2)	0.042 (2)	−0.0073 (19)	−0.006 (2)	−0.0011 (19)
O1	0.026 (2)	0.034 (3)	0.0193 (19)	−0.008 (2)	0.0008 (17)	−0.0019 (17)
O2	0.024 (2)	0.029 (2)	0.0183 (18)	−0.0072 (19)	−0.0085 (17)	0.0027 (17)
O3	0.034 (2)	0.024 (2)	0.0217 (19)	−0.004 (2)	−0.0144 (18)	0.0037 (17)
O4	0.027 (2)	0.026 (2)	0.0197 (19)	−0.004 (2)	−0.0015 (17)	−0.0053 (17)
O5	0.032 (2)	0.023 (2)	0.0164 (18)	−0.0026 (19)	−0.0130 (17)	0.0011 (16)
O6	0.026 (2)	0.037 (3)	0.0165 (18)	−0.013 (2)	−0.0058 (16)	0.0015 (17)
C1	0.023 (3)	0.026 (3)	0.016 (3)	−0.015 (3)	−0.002 (2)	−0.004 (2)

C2	0.016 (3)	0.035 (4)	0.015 (3)	−0.007 (3)	−0.005 (2)	0.002 (2)
C3	0.044 (5)	0.080 (6)	0.022 (3)	0.029 (4)	0.001 (3)	0.019 (3)
C4	0.055 (5)	0.111 (8)	0.026 (4)	0.061 (5)	0.008 (3)	0.013 (4)
C5	0.021 (3)	0.034 (4)	0.015 (3)	−0.004 (3)	−0.002 (2)	0.004 (2)
C6	0.023 (3)	0.042 (4)	0.021 (3)	0.000 (3)	−0.002 (2)	0.000 (3)
C7	0.025 (3)	0.023 (3)	0.016 (3)	−0.004 (3)	−0.003 (2)	−0.003 (2)
C8	0.017 (3)	0.026 (4)	0.018 (3)	−0.011 (3)	−0.009 (2)	−0.003 (2)
C9	0.088 (6)	0.055 (5)	0.019 (3)	0.033 (5)	−0.001 (4)	0.006 (3)
C10	0.113 (8)	0.058 (6)	0.022 (3)	0.059 (5)	0.003 (4)	0.001 (3)
C11	0.035 (4)	0.023 (3)	0.015 (3)	−0.004 (3)	−0.007 (2)	−0.002 (2)
C12	0.039 (4)	0.027 (4)	0.024 (3)	0.003 (3)	−0.004 (3)	−0.008 (3)
C13	0.013 (3)	0.027 (4)	0.024 (3)	0.002 (3)	−0.009 (2)	−0.004 (3)
C14	0.026 (3)	0.027 (4)	0.018 (3)	0.000 (3)	−0.010 (2)	−0.001 (2)
C15	0.039 (4)	0.026 (4)	0.023 (3)	−0.003 (3)	−0.015 (3)	−0.003 (2)
C16	0.042 (4)	0.028 (4)	0.027 (3)	−0.007 (3)	−0.016 (3)	−0.004 (3)
C17	0.030 (4)	0.024 (4)	0.019 (3)	0.002 (3)	−0.010 (2)	−0.008 (2)
C18	0.032 (4)	0.024 (4)	0.026 (3)	0.000 (3)	−0.015 (3)	−0.006 (3)

Geometric parameters (Å, º)

Ho1—O1	2.267 (4)	C3—H3	0.9500
Ho1—O3^{i}	2.252 (3)	C4—C5	1.399 (8)
Ho1—O4^{ii}	2.279 (4)	C4—H4	0.9500
Ho1—O5	2.389 (3)	C5—C11	1.387 (8)
Ho1—O6	2.450 (4)	C5—C6	1.428 (7)
Ho1—O1W	2.370 (4)	C6—C7	1.359 (7)
Ho1—O2W	2.461 (4)	C6—H6	0.9500
Ho1—O3W	2.366 (4)	C7—C9	1.372 (9)
Ho1—C13	2.784 (5)	C7—C8	1.504 (7)
O1W—H1A	0.95 (4)	C9—C10	1.365 (8)
O1W—H1B	0.95 (4)	C9—H9	0.9500
O2W—H2A	0.95 (4)	C10—C11	1.405 (8)
O2W—H2B	0.95 (4)	C10—H10	0.9500
O3W—H3A	0.95 (4)	C11—C12	1.420 (7)
O3W—H3B	0.95 (4)	C12—H12	0.9500
O1—C1	1.256 (6)	C13—C14	1.494 (7)
O2—C1	1.262 (6)	C14—C18	1.378 (8)
O3—C8	1.270 (6)	C14—C15	1.404 (8)
O3—Ho1^{i}	2.252 (3)	C15—C16	1.352 (7)
O4—C8	1.247 (7)	C15—H15	0.9500
O4—Ho1^{iii}	2.279 (4)	C16—C17	1.434 (8)
O5—C13	1.275 (7)	C16—H16	0.9500
O6—C13	1.268 (7)	C17—C17^{iv}	1.407 (12)
C1—C2	1.514 (7)	C17—C18^{iv}	1.421 (7)
C2—C12	1.349 (7)	C18—C17^{iv}	1.421 (7)
C2—C3	1.389 (9)	C18—H18	0.9500
C3—C4	1.379 (8)		

O1—Ho1—O4^{ii}	144.69 (15)	C12—C2—C1	120.6 (5)
O1—Ho1—O5	98.23 (13)	C3—C2—C1	119.5 (5)
O1—Ho1—O6	77.54 (13)	C4—C3—C2	120.2 (6)
O1—Ho1—O1W	72.71 (14)	C4—C3—H3	119.9
O1—Ho1—O2W	142.57 (14)	C2—C3—H3	119.9
O1—Ho1—O3W	76.58 (14)	C3—C4—C5	121.0 (7)
O3^{i}—Ho1—O1	101.02 (13)	C3—C4—H4	119.5
O3^{i}—Ho1—O4^{ii}	96.82 (14)	C5—C4—H4	119.5
O3^{i}—Ho1—O5	147.95 (14)	C11—C5—C4	118.4 (5)
O3^{i}—Ho1—O6	155.80 (14)	C11—C5—C6	119.3 (5)
O3^{i}—Ho1—O1W	83.38 (14)	C4—C5—C6	122.2 (6)
O3^{i}—Ho1—O2W	72.20 (13)	C7—C6—C5	121.0 (6)
O3^{i}—Ho1—O3W	76.60 (14)	C7—C6—H6	119.5
O4^{ii}—Ho1—O5	81.43 (13)	C5—C6—H6	119.5
O4^{ii}—Ho1—O6	73.82 (13)	C6—C7—C9	119.2 (5)
O4^{ii}—Ho1—O1W	140.01 (13)	C6—C7—C8	120.6 (5)
O4^{ii}—Ho1—O2W	72.11 (13)	C9—C7—C8	120.2 (5)
O4^{ii}—Ho1—O3W	78.31 (14)	O4—C8—O3	123.9 (5)
O5—Ho1—O6	54.22 (13)	O4—C8—C7	118.8 (5)
O5—Ho1—O2W	76.91 (13)	O3—C8—C7	117.3 (5)
O6—Ho1—O2W	123.35 (12)	C10—C9—C7	121.1 (6)
O1W—Ho1—O5	78.18 (13)	C10—C9—H9	119.5
O1W—Ho1—O6	118.31 (14)	C7—C9—H9	119.5
O1W—Ho1—O2W	69.97 (13)	C9—C10—C11	121.6 (7)
O3W—Ho1—O5	133.18 (14)	C9—C10—H10	119.2
O3W—Ho1—O6	79.62 (14)	C11—C10—H10	119.2
O3W—Ho1—O2W	133.39 (14)	C5—C11—C10	117.6 (5)
O3W—Ho1—O1W	139.08 (13)	C5—C11—C12	119.6 (5)
O3^{i}—Ho1—C13	170.11 (14)	C10—C11—C12	122.7 (6)
O1—Ho1—C13	88.87 (14)	C2—C12—C11	120.9 (6)
O4^{ii}—Ho1—C13	74.78 (15)	C2—C12—H12	119.6
O3W—Ho1—C13	106.20 (17)	C11—C12—H12	119.6
O1W—Ho1—C13	99.61 (15)	O6—C13—O5	120.3 (5)
O5—Ho1—C13	27.19 (15)	O6—C13—C14	119.5 (5)
O6—Ho1—C13	27.09 (15)	O5—C13—C14	120.2 (5)
O2W—Ho1—C13	99.84 (15)	O6—C13—Ho1	61.6 (3)
Ho1—O1W—H1A	122 (3)	O5—C13—Ho1	58.9 (2)
Ho1—O1W—H1B	121 (3)	C14—C13—Ho1	173.3 (4)
H1A—O1W—H1B	108 (4)	C18—C14—C15	120.6 (5)
Ho1—O2W—H2A	103 (3)	C18—C14—C13	118.3 (5)
Ho1—O2W—H2B	113 (4)	C15—C14—C13	121.1 (5)
H2A—O2W—H2B	110 (4)	C16—C15—C14	120.8 (6)
Ho1—O3W—H3A	129 (3)	C16—C15—H15	119.6
Ho1—O3W—H3B	120 (3)	C14—C15—H15	119.6
H3A—O3W—H3B	110 (4)	C15—C16—C17	120.4 (6)
C1—O1—Ho1	155.6 (4)	C15—C16—H16	119.8
C8—O3—Ho1^{i}	138.9 (4)	C17—C16—H16	119.8
C8—O4—Ho1^{iii}	154.8 (3)	C17^{iv}—C17—C18^{iv}	119.4 (6)

C13—O5—Ho1	93.9 (3)
C13—O6—Ho1	91.3 (3)
O1—C1—O2	124.8 (5)
O1—C1—C2	117.0 (5)
O2—C1—C2	118.2 (5)
C12—C2—C3	119.8 (5)
C17^{iv}—C17—C16	118.9 (6)
C18^{iv}—C17—C16	121.7 (6)
C14—C18—C17^{iv}	119.8 (6)
C14—C18—H18	120.1
C17^{iv}—C18—H18	120.1

O3^{i}—Ho1—O1—C1	131.2 (9)
O4^{ii}—Ho1—O1—C1	−109.8 (9)
O3W—Ho1—O1—C1	−155.6 (9)
O1W—Ho1—O1—C1	51.8 (9)
O5—Ho1—O1—C1	−23.0 (9)
O6—Ho1—O1—C1	−73.4 (9)
O2W—Ho1—O1—C1	56.2 (9)
C13—Ho1—O1—C1	−48.6 (9)
O3^{i}—Ho1—O5—C13	162.5 (3)
O1—Ho1—O5—C13	−71.0 (3)
O4^{ii}—Ho1—O5—C13	73.3 (3)
O3W—Ho1—O5—C13	8.3 (4)
O1W—Ho1—O5—C13	−141.3 (3)
O6—Ho1—O5—C13	−2.9 (3)
O2W—Ho1—O5—C13	146.8 (3)
O3^{i}—Ho1—O6—C13	−158.0 (3)
O1—Ho1—O6—C13	112.8 (3)
O4^{ii}—Ho1—O6—C13	−88.1 (3)
O3W—Ho1—O6—C13	−168.8 (3)
O1W—Ho1—O6—C13	50.5 (3)
O5—Ho1—O6—C13	2.9 (3)
O2W—Ho1—O6—C13	−33.1 (4)
Ho1—O1—C1—O2	32.9 (12)
Ho1—O1—C1—C2	−144.8 (7)
O1—C1—C2—C12	−16.9 (8)
O2—C1—C2—C12	165.2 (5)
O1—C1—C2—C3	159.2 (6)
O2—C1—C2—C3	−18.7 (9)
C12—C2—C3—C4	1.3 (13)
C1—C2—C3—C4	−174.8 (8)
C2—C3—C4—C5	−0.1 (15)
C3—C4—C5—C11	−2.0 (14)
C3—C4—C5—C6	175.3 (8)
C11—C5—C6—C7	−2.3 (10)
C4—C5—C6—C7	−179.5 (8)
C5—C6—C7—C9	−1.6 (10)
C5—C6—C7—C8	176.9 (5)
Ho1^{iii}—O4—C8—O3	−23.0 (12)
Ho1^{iii}—O4—C8—C7	158.7 (6)
Ho1^{i}—O3—C8—O4	−57.1 (8)
Ho1^{i}—O3—C8—C7	121.2 (5)
C6—C7—C8—O3	169.4 (5)
C9—C7—C8—O3	−12.2 (9)
C6—C7—C9—C10	3.3 (13)
C8—C7—C9—C10	−175.1 (8)
C7—C9—C10—C11	−1.3 (16)
C4—C5—C11—C10	−178.5 (9)
C6—C5—C11—C10	4.2 (10)
C4—C5—C11—C12	2.8 (10)
C6—C5—C11—C12	−174.5 (6)
C9—C10—C11—C5	−2.5 (14)
C9—C10—C11—C12	176.2 (9)
C3—C2—C12—C11	−0.5 (10)
C1—C2—C12—C11	175.6 (6)
C5—C11—C12—C2	−1.6 (10)
C10—C11—C12—C2	179.7 (7)
Ho1—O6—C13—O5	−5.1 (5)
Ho1—O6—C13—C14	172.4 (4)
Ho1—O5—C13—O6	5.3 (5)
Ho1—O5—C13—C14	−172.3 (4)
O1—Ho1—C13—O6	−64.2 (3)
O4^{ii}—Ho1—C13—O6	84.1 (3)
O3W—Ho1—C13—O6	11.5 (3)
O1W—Ho1—C13—O6	−136.5 (3)
O5—Ho1—C13—O6	−174.8 (5)
O2W—Ho1—C13—O6	152.4 (3)
O1—Ho1—C13—O5	110.6 (3)
O4^{ii}—Ho1—C13—O5	−101.0 (3)
O3W—Ho1—C13—O5	−173.7 (3)
O1W—Ho1—C13—O5	38.4 (3)
O6—Ho1—C13—O5	174.8 (5)
O2W—Ho1—C13—O5	−32.8 (3)
O6—C13—C14—C18	−162.7 (5)
O5—C13—C14—C18	14.9 (8)
O6—C13—C14—C15	15.6 (8)
O5—C13—C14—C15	−166.9 (5)
C18—C14—C15—C16	2.0 (9)
C13—C14—C15—C16	−176.2 (5)
C14—C15—C16—C17	0.4 (9)
C15—C16—C17—C17^{iv}	−1.9 (11)
C15—C16—C17—C18^{iv}	179.1 (6)
C15—C14—C18—C17^{iv}	−2.9 (9)

C6—C7—C8—O4	−12.2 (8)	C13—C14—C18—C17^{iv}	175.4 (5)
C9—C7—C8—O4	166.2 (7)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*A*···O2^{v}	0.95 (4)	1.82 (2)	2.725 (5)	157 (5)
O1*W*—H1*B*···O5^{v}	0.95 (4)	1.95 (3)	2.818 (5)	150 (4)
O2*W*—H2*A*···O2^{vi}	0.95 (4)	1.98 (4)	2.782 (5)	140 (5)
O2*W*—H2*B*···O2^{v}	0.95 (4)	2.14 (4)	2.901 (6)	136 (4)
O3*W*—H3*A*···O6^{vii}	0.95 (4)	1.78 (4)	2.704 (5)	165 (5)
O3*W*—H3*B*···O2*W*^{viii}	0.95 (4)	2.26 (4)	3.181 (6)	165 (4)
O3*W*—H3*B*···O4^{i}	0.95 (4)	2.53 (5)	3.145 (6)	123 (4)

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