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## Structure Reports

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# Poly[[hexaaquabis[ $\mu_2$ -2,5-dihydroxy-1,4-benzoquinato(2-)]diholmium(III)] octadecahydrate]

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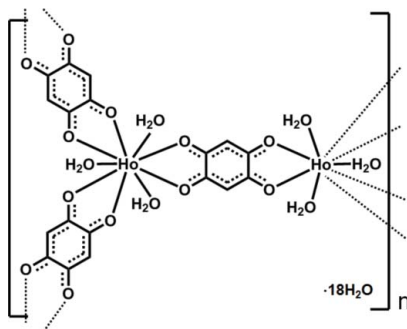
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Key indicators: single-crystal X-ray study;  $T = 90$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.023;  $wR$  factor = 0.063; data-to-parameter ratio = 18.5.

In the polymeric title compound,  $\{[\text{Ho}_2(\text{C}_6\text{H}_2\text{O}_4)_3(\text{H}_2\text{O})_6] \cdot 18\text{H}_2\text{O}\}_n$ , the  $\text{Ho}^{\text{III}}$  ion is nine-coordinated by six O atoms derived from three bidentate 2,5-dihydroxy-1,4-benzoquinone (DHBQ $^{2-}$ ) ligands and three O atoms from three water molecules. The  $\text{Ho}^{\text{III}}$  ions are connected *via* three ligands, resulting in the formation of a two-dimensional honeycomb layer parallel to the *ab* plane. The layer is racemic in which  $\Delta$ - and  $\Lambda$ -coordination geometries around  $\text{Ho}^{\text{III}}$  ions are alternately arranged. The asymmetric unit comprises a third of a  $\text{Ho}^{\text{III}}$  ion, located on a threefold axis, one-half of a DHBQ $^{2-}$  ion, located on a centre of inversion, one coordinated water molecule and three uncoordinated water molecules.

## Related literature

For general background, see: Kitagawa & Kawata (2002); Nakabayashi & Ohkoshi (2009); Ohkoshi *et al.* (2001). For details of the synthesis, see: Weider *et al.* (1985). For related structures, see: Robl & Sheldrick (1988); Weiss *et al.* (1986).



## Experimental

### Crystal data

 $[\text{Ho}_2(\text{C}_6\text{H}_2\text{O}_4)_3(\text{H}_2\text{O})_6] \cdot 18\text{H}_2\text{O}$   
 $M_r = 1176.46$ 

 Trigonal,  $R\bar{3}$   
 $a = 14.1407$  (3) Å

 $c = 18.0629$  (5) Å  
 $V = 3127.93$  (12) Å $^3$   
 $Z = 3$   
 Mo  $K\alpha$  radiation

 $\mu = 3.88$  mm $^{-1}$   
 $T = 90$  K  
 $0.10 \times 0.10 \times 0.04$  mm

### Data collection

 Rigaku R-Axis RAPID  
 diffractometer  
 Absorption correction: multi-scan  
 (ABSCOR; Higashi, 1995)  
 $T_{\text{min}} = 0.704$ ,  $T_{\text{max}} = 0.856$ 

 11262 measured reflections  
 1594 independent reflections  
 1525 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.025$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.023$   
 $wR(F^2) = 0.063$   
 $S = 1.23$   
 1594 reflections

 86 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.65$  e Å $^{-3}$   
 $\Delta\rho_{\text{min}} = -0.30$  e Å $^{-3}$ 

**Table 1**  
Selected bond lengths (Å).

Ho1—O1	2.371 (2)	Ho1—O3	2.385 (3)
Ho1—O2	2.463 (2)		

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *pyMOL* (DeLano, 2007); software used to prepare material for publication: *CrystalStructure*.

We are thankful for a Grant-in-Aid for Young Scientists (S) from JSPS, the Global COE Program, "Chemistry Innovation through Cooperation of Science and Engineering" from MEXT Japan, the Photon Frontier Network Program from MEXT, the Izumi Science and Technology Foundation and Asahi Glass Foundation. We also thank the Cryogenic Research Center and the Center for Nano Lithography & Analysis, The University of Tokyo, supported by MEXT Japan. This work has been approved by the Photon Factory Program Advisory Committee (Proposal 2009 G678).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2691).

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## supporting information

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## Poly[[hexaaquatriss[ $\mu_2$ -2,5-dihydroxy-1,4-benzoquinato(2-)]diholmium(III)] octadecahydrate]

Koji Nakabayashi and Shin-ichi Ohkoshi

### S1. Comment

Lanthanide complexes have attracted attention as magnetic and luminescent materials due to the properties of the  $4f$  orbitals in lanthanide ions. Although the magnetism of mononuclear lanthanide complexes are well understood, studies on polynuclear lanthanide complexes are much less advanced (Ohkoshi *et al.*, 2001). The dimensionality of complexes and coordination geometry around lanthanide ions are key factors to control their magnetic properties. From this viewpoint, constructing polynuclear lanthanide complexes with various topologies are interesting. In our previous work, we reported the 3-D monometallic lanthanide metal assembly,  $\text{Na}_5[\{\text{Ho}(\text{THB}^+)_2\} \cdot 7\text{H}_2\text{O}]_n$  (THB = 1,2,4,5-tetrahydroxybenzene) (Nakabayashi *et al.*, 2009). In this work, we synthesized a 2-D honeycomb network composed of holmium ions ( $\text{Ho}^{\text{III}}$ ) and 2,5-dihydroxy-1,4-benzoquinone,  $[\{\text{Ho}_2(\text{DHBQ}^{2-})_3(\text{H}_2\text{O})_6\} \cdot 18\text{H}_2\text{O}]_n$  (Kitagawa *et al.*, 2002; Robl *et al.*, 1988).

The asymmetric unit comprises a third of a  $\text{Ho}^{\text{III}}$  ion, being located on a three-fold axis, one-half of a  $\text{DHBQ}^{2-}$  ion, being disposed about a centre of inversion, one coordinated water molecule, and three zeolitic water molecules.

The C—O distances of 1.276 (4) Å (C1—O1) and 1.274 (3) Å (C2—O2) in this compound agree with those of the  $\text{DHBQ}^{2-}$  ligands which were previously reported, *e.g.* 1.276 (6) Å (Weiss *et al.* 1986). In the coordination geometry, a  $\text{Ho}^{\text{III}}$  ion is coordinated to six O atoms from three bidentate  $\text{DHBQ}^{2-}$  ligands and three O atoms from three water molecules (Fig. 1 and Table 1). The  $\text{Ho}^{\text{III}}$  ion is connected *via* three ligands, which results in a two-dimensional honeycomb layer with a diameter of 16.6 Å. The layer has a racemic structure in which  $\Delta$ - and  $\Lambda$ -coordination geometries around  $\text{Ho}^{\text{III}}$  ions are alternately arranged (Fig. 2). The zeolitic water molecules occupy regions between the layers.

The product of the molar magnetic susceptibility ( $\chi_M$ ) and temperature ( $T$ ),  $\chi_M T$ , values at room temperature was 13.6  $\text{cm}^3 \text{K mol}^{-1}$ . This value nearly corresponds to the expected value of 13.9  $\text{cm}^3 \text{K mol}^{-1}$  due to  $\text{Ho}^{\text{III}}$  ions ( $J = 8$ ,  $L = 6$ ,  $S = 2$ , and  $g = 5/4$ ).

All known compounds of this type have two specific structures, honeycomb or racemic, and show paramagnetism. Mixing lanthanide ions, the chiral lanthanide assemblies with  $\Delta$ - or  $\Lambda$ -coordination geometries are targeted for synthesis, which should show a magneto-chiral dichroism. A study to clarify this hypotheses, work is currently under way.

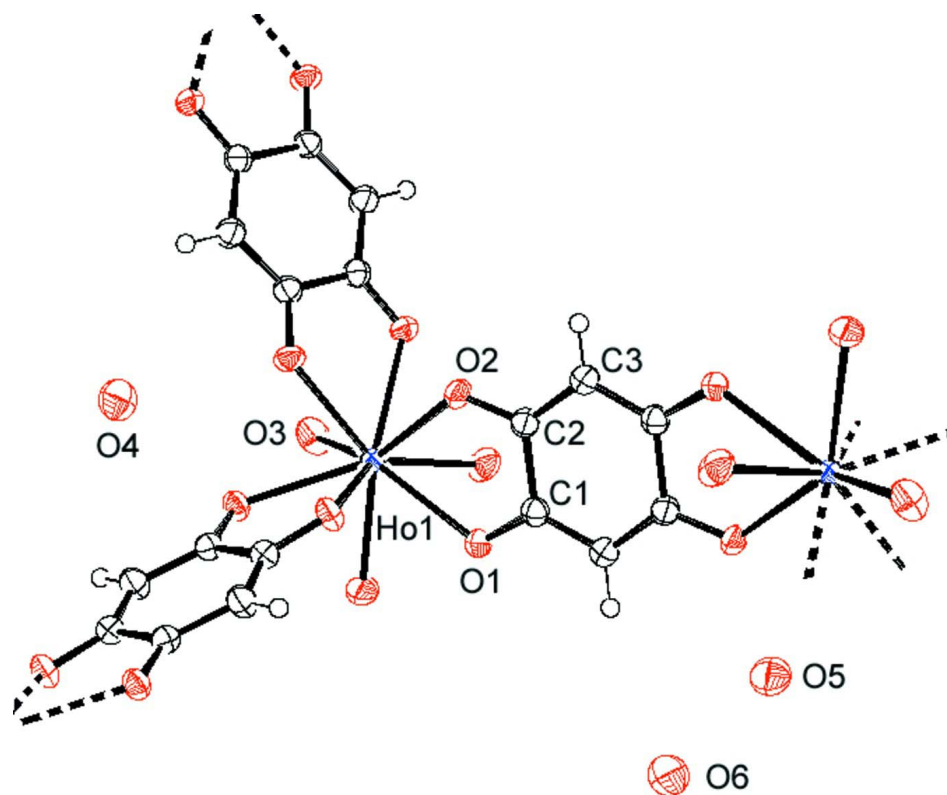
### S2. Experimental

Under air, aqueous solutions of 0.1 *M*  $\text{Ho}(\text{NO}_3)_3$  and 0.4 *M* 1,2,4,5-tetrahydroxybenzene (THB) (Weider *et al.*, 1985) mixed. THB was gradually oxidized to 2,5-dihydroxy-1,4-benzoquinone (DHBQ) in the mixed solution, and slow complexation of  $\text{Ho}(\text{NO}_3)_3$  and DHBQ) produced red crystals of the title polymer in 24% yield within a week. The obtained polycrystalline compound was dried under air. Elemental analysis indicated the formula was  $[\text{Ho}_2(\text{C}_6\text{H}_2\text{O}_4)_3(\text{H}_2\text{O})_6] \cdot 17\text{H}_2\text{O}$ ,  $\text{C}_{18}\text{H}_{52}\text{O}_{35}\text{Ho}_2$ , calcd. Ho, 28.18%; C, 18.47%; H, 4.49%, found. Ho, 28.29%; C, 18.23%; H, 4.64%. There is a slight difference of zeolitic water molecules between the elemental analysis and the crystallographic

formulation because zeolitic water molecules are easy to be lost from the crystals and their number depends on the drying processes.

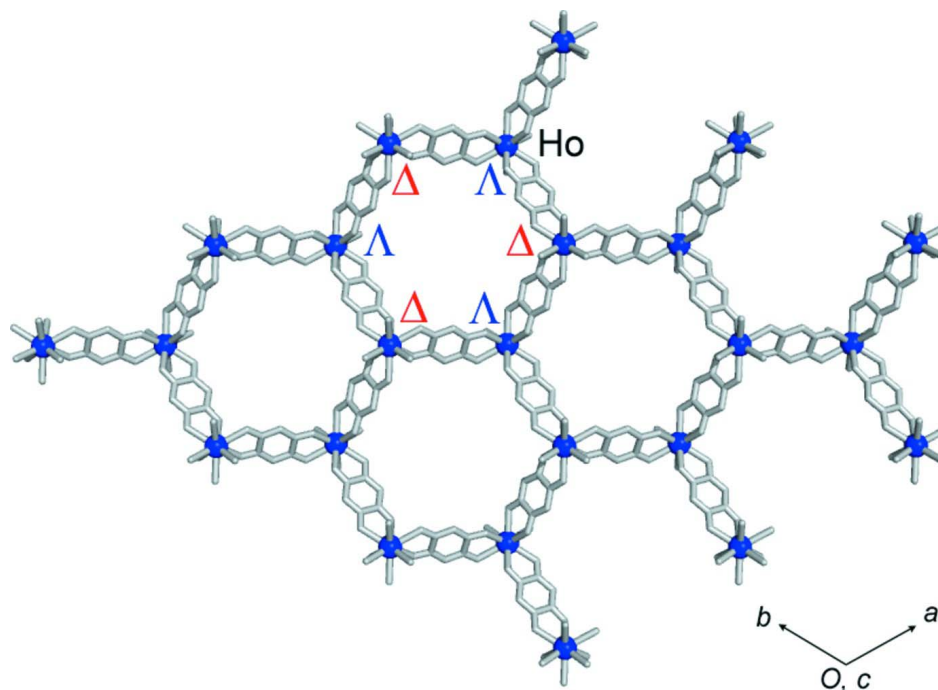
### S3. Refinement

The H atoms were placed in their calculated positions, with C—H = 0.95 Å, and refined using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ .



**Figure 1**

A part of the title polymeric compound, thermal ellipsoids are shown at the 50% probability level. Blue, gray, and red represent Ho, C and O atoms, respectively. The labeled atoms indicate all independent atoms.

**Figure 2**

A layer with a honeycomb structure. Blue spheres and gray sticks represent Ho atoms and other atoms (C and O), respectively.

**Poly[[hexaaquatrakis( $\mu_2$ -2,5-dihydroxy-1,4-benzoquinonato(2-))]diholmium(III)] octadecahydrate]**

*Crystal data*

$[\text{Ho}_2(\text{C}_6\text{H}_2\text{O}_4)_3(\text{H}_2\text{O})_6] \cdot 18\text{H}_2\text{O}$

$M_r = 1176.46$

Trigonal,  $R\bar{3}$

Hall symbol:  $-R\ 3$

$a = 14.1407(3)\ \text{\AA}$

$c = 18.0629(5)\ \text{\AA}$

$V = 3127.93(12)\ \text{\AA}^3$

$Z = 3$

$F(000) = 1608.00$

$D_x = 1.796\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71075\ \text{\AA}$

Cell parameters from 9640 reflections

$\theta = 3.4\text{--}27.5^\circ$

$\mu = 3.88\ \text{mm}^{-1}$

$T = 90\ \text{K}$

Platelet, red

$0.10 \times 0.10 \times 0.04\ \text{mm}$

*Data collection*

Rigaku R-Axis RAPID

diffractometer

Detector resolution:  $10.00\ \text{pixels mm}^{-1}$

$\omega$  scans

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.704$ ,  $T_{\max} = 0.856$

11262 measured reflections

1594 independent reflections

1525 reflections with  $F^2 > 2\sigma(F^2)$

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

$\theta_{\max} = 27.5^\circ$

$h = -18 \rightarrow 18$

$k = -18 \rightarrow 18$

$l = -23 \rightarrow 23$

*Refinement*

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.023$

$wR(F^2) = 0.063$

$S = 1.23$

1594 reflections

86 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 19.995P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$   
 $\Delta\rho_{\max} = 0.65 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$

### Special details

#### Geometry. loop\_ Bond lengths and angles

Ho1 - Distance Angles O1\_\$1 2.3715 (0.0023) O1 2.3715 (0.0024) 78.04 (0.09) O1\_\$2 2.3715 (0.0023) 78.05 (0.09) 78.05 (0.09) O3\_\$2 2.3851 (0.0025) 138.54 (0.09) 85.02 (0.10) 134.88 (0.09) O3\_\$1 2.3851 (0.0025) 134.88 (0.09) 138.54 (0.09) 85.02 (0.10) 80.68 (0.11) O3 2.3852 (0.0025) 85.02 (0.10) 134.88 (0.09) 138.54 (0.09) 80.68 (0.11) 80.68 (0.11) O2\_\$1 2.4626 (0.0024) 65.01 (0.08) 134.82 (0.08) 69.90 (0.08) 140.07 (0.09) 69.92 (0.09) 68.65 (0.09) O2 2.4626 (0.0023) 69.90 (0.08) 65.01 (0.08) 134.82 (0.08) 68.65 (0.09) 140.07 (0.09) 69.92 (0.08) 119.91 (0.01) O2\_\$2 2.4626 (0.0023) 134.82 (0.08) 69.90 (0.08) 65.01 (0.08) 69.92 (0.08) 68.65 (0.09) 140.07 (0.09) 119.91 (0.01) Ho1 - O1\_\$1 O1 O1\_\$2 O3\_\$2 O3\_\$1 O3 O2\_\$1 O1 - Distance Angles C1 1.2764 (0.0041) Ho1 2.3715 (0.0023) 123.38 (0.21) O1 - C1 O2 - Distance Angles C2 1.2739 (0.0041) Ho1 2.4626 (0.0023) 119.92 (0.21) O2 - C2 O3 - Distance Angles Ho1 2.3852 (0.0025) O3 - C1 - Distance Angles O1 1.2764 (0.0041) C3 1.3846 (0.0049) 125.21 (0.32) C2 1.5290 (0.0046) 114.26 (0.29) 120.50 (0.30) C1 - O1 C3 C3 - Distance Angles C1 1.3846 (0.0049) C2\_\$3 1.3982 (0.0048) 119.67 (0.32) H3 0.9500 120.16 120.16 C3 - C1 C2\_\$3 C2 - Distance Angles O2 1.2739 (0.0041) C3\_\$3 1.3982 (0.0048) 124.89 (0.31) C1 1.5290 (0.0046) 115.30 (0.29) 119.80 (0.30) C2 - O2 C3\_\$3

**Refinement.** Refinement was performed using all reflections. The weighted  $R$ -factor ( $wR$ ) and goodness of fit ( $S$ ) are based on  $F^2$ .  $R$ -factor (gt) are based on  $F$ . The threshold expression of  $F^2 > 2.0 \sigma(F^2)$  is used only for calculating  $R$ -factor (gt).

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Ho1	0.0000	0.0000	0.754297 (13)	0.01802 (8)
O1	-0.1198 (2)	-0.12391 (19)	0.66415 (13)	0.0270 (5)
O2	-0.00678 (19)	-0.17736 (19)	0.75023 (13)	0.0252 (4)
O3	0.1248 (2)	-0.0026 (2)	0.84201 (15)	0.0366 (6)
O4	0.1390 (2)	-0.1541 (2)	0.93023 (18)	0.0481 (7)
O5	0.2672 (2)	0.1817 (2)	0.92192 (19)	0.0508 (7)
O6	-0.1882 (2)	-0.0598 (2)	0.54192 (17)	0.0474 (7)
C1	-0.1492 (2)	-0.2252 (2)	0.66312 (19)	0.0246 (6)
C3	-0.2332 (2)	-0.3039 (2)	0.6206 (2)	0.0283 (7)
C2	-0.0802 (2)	-0.2555 (2)	0.71218 (18)	0.0237 (6)
H3	-0.2770	-0.2850	0.5912	0.034*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ho1	0.01630 (11)	0.01630 (11)	0.02146 (14)	0.00815 (5)	0.0000	0.0000
O1	0.0310 (13)	0.0178 (11)	0.0320 (12)	0.0121 (10)	-0.0067 (9)	-0.0020 (9)
O2	0.0240 (11)	0.0194 (11)	0.0316 (12)	0.0103 (9)	-0.0047 (9)	-0.0032 (9)
O3	0.0363 (14)	0.0309 (14)	0.0405 (14)	0.0154 (12)	-0.0164 (11)	-0.0020 (11)
O4	0.0455 (18)	0.0513 (18)	0.0552 (18)	0.0299 (15)	-0.0062 (14)	0.0051 (15)
O5	0.0490 (18)	0.0453 (18)	0.0558 (19)	0.0218 (15)	-0.0121 (15)	-0.0078 (14)
O6	0.0528 (19)	0.0470 (18)	0.0418 (16)	0.0244 (15)	-0.0105 (13)	0.0015 (13)
C1	0.0241 (16)	0.0227 (15)	0.0271 (15)	0.0116 (13)	0.0016 (12)	0.0014 (12)

C3	0.0274 (17)	0.0235 (17)	0.0334 (17)	0.0122 (14)	-0.0049 (13)	0.0001 (13)
C2	0.0228 (15)	0.0236 (16)	0.0241 (15)	0.0111 (13)	0.0019 (12)	0.0005 (12)

*Geometric parameters (Å, °)*

Ho1—O1	2.371 (2)	Ho1—O3 <sup>ii</sup>	2.385 (2)
Ho1—O1 <sup>i</sup>	2.371 (2)	O1—C1	1.276 (4)
Ho1—O1 <sup>ii</sup>	2.371 (2)	O2—C2	1.274 (3)
Ho1—O2	2.463 (2)	C1—C3	1.385 (4)
Ho1—O2 <sup>i</sup>	2.4625 (19)	C1—C2	1.529 (6)
Ho1—O2 <sup>ii</sup>	2.463 (3)	C3—C2 <sup>iii</sup>	1.398 (5)
Ho1—O3	2.385 (3)	C3—H3	0.950
Ho1—O3 <sup>i</sup>	2.385 (3)		
Ho1…C1	3.253 (3)	O6…C1	3.444 (5)
Ho1…C1 <sup>i</sup>	3.253 (3)	O6…C1 <sup>ii</sup>	3.369 (4)
Ho1…C1 <sup>ii</sup>	3.253 (4)	O6…C3	3.485 (5)
Ho1…C2	3.289 (3)	O6…C3 <sup>xi</sup>	3.326 (5)
Ho1…C2 <sup>i</sup>	3.289 (2)	C1…Ho1	3.253 (3)
Ho1…C2 <sup>ii</sup>	3.289 (4)	C1…O1 <sup>i</sup>	3.592 (3)
O1…O1 <sup>i</sup>	2.986 (3)	C1…O2	2.372 (4)
O1…O1 <sup>ii</sup>	2.986 (3)	C1…O3 <sup>ii</sup>	3.481 (4)
O1…O2	2.599 (4)	C1…O6	3.444 (5)
O1…O2 <sup>ii</sup>	2.770 (4)	C1…O6 <sup>i</sup>	3.369 (4)
O1…O3 <sup>ii</sup>	3.214 (3)	C1…C1 <sup>iii</sup>	2.847 (5)
O1…O4 <sup>iv</sup>	3.464 (4)	C1…C3 <sup>iii</sup>	2.533 (6)
O1…O6	2.740 (4)	C1…C2 <sup>iii</sup>	2.406 (4)
O1…O6 <sup>i</sup>	3.390 (4)	C3…O1	2.363 (3)
O1…C1 <sup>ii</sup>	3.592 (5)	C3…O2 <sup>iii</sup>	2.370 (4)
O1…C3	2.363 (3)	C3…O6	3.485 (5)
O1…C2	2.360 (5)	C3…O6 <sup>xii</sup>	3.326 (4)
O1…C2 <sup>ii</sup>	3.458 (5)	C3…C1 <sup>iii</sup>	2.533 (6)
O2…O1	2.599 (4)	C3…C3 <sup>iii</sup>	2.927 (6)
O2…O1 <sup>i</sup>	2.770 (3)	C3…C2	2.531 (5)
O2…O3	2.778 (3)	C2…Ho1	3.289 (3)
O2…O3 <sup>ii</sup>	2.734 (4)	C2…O1	2.360 (5)
O2…O4 <sup>v</sup>	2.868 (5)	C2…O1 <sup>i</sup>	3.458 (3)
O2…O5 <sup>ii</sup>	3.325 (4)	C2…O3 <sup>ii</sup>	3.254 (5)
O2…C1	2.372 (4)	C2…O4 <sup>v</sup>	3.499 (5)
O2…C3 <sup>iii</sup>	2.370 (4)	C2…C1 <sup>iii</sup>	2.406 (4)
O3…O1 <sup>i</sup>	3.214 (3)	C2…C3	2.531 (5)
O3…O2	2.778 (3)	C2…C2 <sup>iii</sup>	2.855 (4)
O3…O2 <sup>i</sup>	2.734 (3)	O1…H3	2.608
O3…O3 <sup>i</sup>	3.088 (5)	O2…H3 <sup>iii</sup>	2.614
O3…O3 <sup>ii</sup>	3.088 (4)	O4…H3 <sup>ix</sup>	3.259
O3…O4	2.757 (5)	O4…H3 <sup>vi</sup>	3.484
O3…O5	2.772 (3)	O5…H3 <sup>ix</sup>	3.135
O3…C1 <sup>i</sup>	3.481 (4)	O6…H3	2.918

O3...C2 <sup>i</sup>	3.254 (3)	O6...H3 <sup>xi</sup>	3.032
O4...O1 <sup>vi</sup>	3.464 (5)	C1...H3	2.035
O4...O2 <sup>v</sup>	2.868 (5)	C1...H3 <sup>iii</sup>	3.400
O4...O3	2.757 (5)	C2...H3	3.396
O4...O5 <sup>ii</sup>	2.753 (4)	C2...H3 <sup>iii</sup>	2.048
O4...O5 <sup>vii</sup>	2.802 (4)	H3...O1	2.608
O4...C2 <sup>v</sup>	3.499 (5)	H3...O2 <sup>iii</sup>	2.614
O5...O2 <sup>i</sup>	3.325 (4)	H3...O4 <sup>x</sup>	3.259
O5...O3	2.772 (3)	H3...O4 <sup>iv</sup>	3.484
O5...O4 <sup>i</sup>	2.753 (6)	H3...O5 <sup>x</sup>	3.135
O5...O4 <sup>viii</sup>	2.802 (4)	H3...O6	2.918
O5...O6 <sup>ix</sup>	2.728 (4)	H3...O6 <sup>xii</sup>	3.032
O6...O1	2.740 (4)	H3...C1	2.035
O6...O1 <sup>ii</sup>	3.390 (3)	H3...C1 <sup>iii</sup>	3.400
O6...O5 <sup>x</sup>	2.728 (4)	H3...C2	3.396
O6...O6 <sup>xi</sup>	2.801 (4)	H3...C2 <sup>iii</sup>	2.048
O6...O6 <sup>xii</sup>	2.801 (5)		
O1—Ho1—O1 <sup>i</sup>	78.04 (8)	O2—Ho1—O3 <sup>i</sup>	140.07 (9)
O1—Ho1—O1 <sup>ii</sup>	78.04 (8)	O2—Ho1—O3 <sup>ii</sup>	68.65 (10)
O1—Ho1—O2	65.01 (9)	O2 <sup>i</sup> —Ho1—O2 <sup>ii</sup>	119.91 (9)
O1—Ho1—O2 <sup>i</sup>	134.81 (7)	O2 <sup>i</sup> —Ho1—O3	68.65 (8)
O1—Ho1—O2 <sup>ii</sup>	69.90 (9)	O2 <sup>i</sup> —Ho1—O3 <sup>i</sup>	69.92 (8)
O1—Ho1—O3	134.88 (10)	O2 <sup>i</sup> —Ho1—O3 <sup>ii</sup>	140.07 (8)
O1—Ho1—O3 <sup>i</sup>	138.54 (11)	O2 <sup>ii</sup> —Ho1—O3	140.07 (9)
O1—Ho1—O3 <sup>ii</sup>	85.02 (8)	O2 <sup>ii</sup> —Ho1—O3 <sup>i</sup>	68.65 (9)
O1 <sup>i</sup> —Ho1—O1 <sup>ii</sup>	78.04 (10)	O2 <sup>ii</sup> —Ho1—O3 <sup>ii</sup>	69.92 (10)
O1 <sup>i</sup> —Ho1—O2	69.90 (8)	O3—Ho1—O3 <sup>i</sup>	80.68 (11)
O1 <sup>i</sup> —Ho1—O2 <sup>i</sup>	65.01 (8)	O3—Ho1—O3 <sup>ii</sup>	80.68 (9)
O1 <sup>i</sup> —Ho1—O2 <sup>ii</sup>	134.81 (8)	O3 <sup>i</sup> —Ho1—O3 <sup>ii</sup>	80.68 (9)
O1 <sup>i</sup> —Ho1—O3	85.02 (10)	Ho1—O1—C1	123.4 (2)
O1 <sup>i</sup> —Ho1—O3 <sup>i</sup>	134.88 (7)	Ho1—O2—C2	119.9 (2)
O1 <sup>i</sup> —Ho1—O3 <sup>ii</sup>	138.54 (10)	O1—C1—C3	125.2 (4)
O1 <sup>ii</sup> —Ho1—O2	134.81 (8)	O1—C1—C2	114.3 (2)
O1 <sup>ii</sup> —Ho1—O2 <sup>i</sup>	69.90 (8)	C3—C1—C2	120.5 (3)
O1 <sup>ii</sup> —Ho1—O2 <sup>ii</sup>	65.01 (8)	C1—C3—C2 <sup>iii</sup>	119.7 (4)
O1 <sup>ii</sup> —Ho1—O3	138.54 (7)	O2—C2—C1	115.3 (3)
O1 <sup>ii</sup> —Ho1—O3 <sup>i</sup>	85.02 (9)	O2—C2—C3 <sup>iii</sup>	124.9 (3)
O1 <sup>ii</sup> —Ho1—O3 <sup>ii</sup>	134.88 (11)	C1—C2—C3 <sup>iii</sup>	119.8 (2)
O2—Ho1—O2 <sup>i</sup>	119.91 (9)	C1—C3—H3	120.2
O2—Ho1—O2 <sup>ii</sup>	119.91 (7)	C2 <sup>iii</sup> —C3—H3	120.2
O2—Ho1—O3	69.92 (9)		
O1—Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	166.8 (3)	O3—Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	-125.1 (2)
O1 <sup>i</sup> —Ho1—O1—C1	86.7 (2)	O3 <sup>i</sup> —Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	-55.3 (2)
O1—Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	86.7 (2)	O3 <sup>ii</sup> —Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	16.3 (2)
O1 <sup>ii</sup> —Ho1—O1—C1	166.8 (2)	O2—Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	34.3 (2)
O1—Ho1—O2—C2	-10.8 (2)	O2 <sup>i</sup> —Ho1—O2—C2	-139.8 (2)

O2—Ho1—O1—C1	13.5 (2)	O2—Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	-139.8 (2)
O1—Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	-49.0 (3)	O2 <sup>ii</sup> —Ho1—O2—C2	34.3 (2)
O2 <sup>i</sup> —Ho1—O1—C1	121.7 (2)	O3—Ho1—O2—C2	171.3 (2)
O1—Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	-96.6 (2)	O3 <sup>i</sup> —Ho1—O2—C2	126.2 (2)
O2 <sup>ii</sup> —Ho1—O1—C1	-125.7 (2)	O3 <sup>ii</sup> —Ho1—O2—C2	83.8 (2)
O3—Ho1—O1—C1	16.3 (3)	O2 <sup>i</sup> —Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	34.3 (2)
O3 <sup>i</sup> —Ho1—O1—C1	-125.1 (2)	O2 <sup>ii</sup> —Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	-139.8 (2)
O3 <sup>ii</sup> —Ho1—O1—C1	-55.3 (2)	O3—Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	83.8 (2)
O1 <sup>i</sup> —Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	166.8 (2)	O3 <sup>i</sup> —Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	171.3 (2)
O1 <sup>ii</sup> —Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	86.7 (3)	O3 <sup>ii</sup> —Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	126.2 (2)
O1 <sup>i</sup> —Ho1—O2—C2	-96.6 (2)	O3—Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	126.2 (2)
O2—Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	-125.7 (3)	O3 <sup>i</sup> —Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	83.8 (2)
O1 <sup>i</sup> —Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	-10.8 (2)	O3 <sup>ii</sup> —Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	171.3 (2)
O2 <sup>i</sup> —Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	13.5 (3)	Ho1—O1—C1—C3	167.6 (2)
O1 <sup>i</sup> —Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	-49.0 (2)	Ho1—O1—C1—C2	-14.4 (4)
O2 <sup>ii</sup> —Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	121.7 (3)	Ho1—O2—C2—C1	8.1 (3)
O3—Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	-55.3 (3)	Ho1—O2—C2—C3 <sup>iii</sup>	-171.9 (2)
O3 <sup>i</sup> —Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	16.3 (3)	O1—C1—C3—C2 <sup>iii</sup>	176.2 (3)
O3 <sup>ii</sup> —Ho1—O1 <sup>i</sup> —C1 <sup>i</sup>	-125.1 (3)	O1—C1—C2—O2	3.4 (4)
O1 <sup>ii</sup> —Ho1—O2—C2	-49.0 (2)	O1—C1—C2—C3 <sup>iii</sup>	-176.5 (3)
O2—Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	121.7 (2)	C3—C1—C2—O2	-178.4 (3)
O1 <sup>ii</sup> —Ho1—O2 <sup>i</sup> —C2 <sup>i</sup>	-96.6 (2)	C3—C1—C2—C3 <sup>iii</sup>	1.7 (5)
O2 <sup>i</sup> —Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	-125.7 (2)	C2—C1—C3—C2 <sup>iii</sup>	-1.7 (5)
O1 <sup>ii</sup> —Ho1—O2 <sup>ii</sup> —C2 <sup>ii</sup>	-10.8 (2)	C1—C3—C2 <sup>iii</sup> —O2 <sup>iii</sup>	-178.5 (3)
O2 <sup>ii</sup> —Ho1—O1 <sup>ii</sup> —C1 <sup>ii</sup>	13.5 (2)	C1—C3—C2 <sup>iii</sup> —C1 <sup>iii</sup>	1.6 (5)

Symmetry codes: (i)  $-y, x-y, z$ ; (ii)  $-x+y, -x, z$ ; (iii)  $-x-1/3, -y-2/3, -z+4/3$ ; (iv)  $x-y-2/3, x-1/3, -z+5/3$ ; (v)  $-x+1/3, -y-1/3, -z+5/3$ ; (vi)  $y+1/3, -x+y-1/3, -z+5/3$ ; (vii)  $y, -x+y, -z+2$ ; (viii)  $x-y, x, -z+2$ ; (ix)  $x+2/3, y+1/3, z+1/3$ ; (x)  $x-2/3, y-1/3, z-1/3$ ; (xi)  $y, -x+y, -z+1$ ; (xii)  $x-y, x, -z+1$ .