

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Poly[di- μ_4 -benzene-1,4-dicarboxylato- μ_6 -succinato-diholmium(III)]

Qin He* and Bao-Jun Huang

Department of Chemistry, Xuchang University, Xuchang 461000, People's Republic of China

Correspondence e-mail: qhe.xcu@163.com

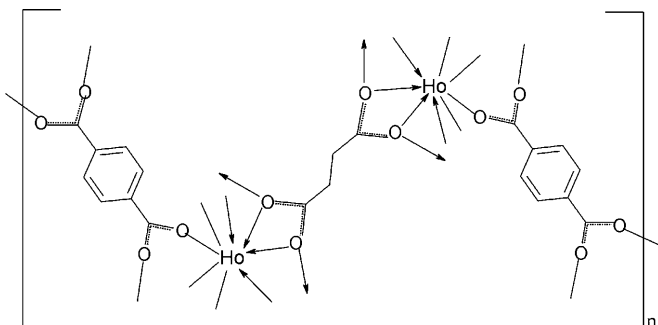
Received 25 October 2007; accepted 14 December 2007

Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.015; wR factor = 0.042; data-to-parameter ratio = 15.6.

The title compound, $[\text{Ho}_2(\text{C}_4\text{H}_4\text{O}_4)(\text{C}_8\text{H}_4\text{O}_4)_2]_n$, was synthesized hydrothermally. The Ho atom is coordinated by four O atoms from four benzene-1,4-dicarboxylate (BDC) anions and four O atoms from three succinate anions, in a distorted square-antiprismatic coordination geometry. The antiprisms are bridged by the benzene-1,4-dicarboxylate and succinate anions, into a three-dimensional coordination network. The succinate anions are located on centres of inversion.

Related literature

For related literature, see: Li & Wang (2005); Li *et al.* (2006); Wang & Li (2005); He *et al.* (2006).



Experimental

Crystal data

$[\text{Ho}_2(\text{C}_4\text{H}_4\text{O}_4)(\text{C}_8\text{H}_4\text{O}_4)_2]$
 $M_r = 387.08$
 Orthorhombic, $Pbca$
 $a = 13.8147$ (3) Å
 $b = 6.7850$ (2) Å
 $c = 21.7103$ (5) Å

$V = 2034.97$ (9) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 7.79$ mm⁻¹
 $T = 291$ (2) K
 $0.18 \times 0.15 \times 0.05$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.245$, $T_{\max} = 0.682$

11598 measured reflections
 2411 independent reflections
 2140 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.015$
 $wR(F^2) = 0.042$
 $S = 1.06$
 2411 reflections

155 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.94$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ho—O1	2.2899 (17)	Ho—O5	2.4753 (16)
Ho—O2 ⁱ	2.2320 (17)	Ho—O5 ^{iv}	2.4015 (18)
Ho—O3 ⁱⁱ	2.3208 (16)	Ho—O6 ^v	2.4633 (17)
Ho—O4 ⁱⁱⁱ	2.3013 (16)	Ho—O6	2.5230 (16)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2068).

References

- Bruker (1998). *SMART* (Version 5.16), *SAINT* (Version 6.01) and *SHELXTL* (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
 He, Q., Zi, J.-F. & Zhang, F.-J. (2006). *Acta Cryst.* **E62**, m997–m998.
 Li, Z.-F. & Wang, C.-X. (2005). *Acta Cryst.* **E61**, m2689–m2690.
 Li, Z.-F., Wang, C.-X., Li, Y., Cai, D.-J. & Xiao, Y.-J. (2006). *Acta Cryst.* **E62**, m251–m252.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Wang, C.-X. & Li, Z.-F. (2005). *Acta Cryst.* **E61**, m2212–m2213.

supplementary materials

Acta Cryst. (2008). E64, m237 [doi:10.1107/S1600536807066950]

Poly[di- μ_4 -benzene-1,4-dicarboxylato- μ_6 -succinato-diholmium(III)]

Q. He and B.-J. Huang

Comment

The title compound, (I), is isostructural with its $[M_2(C_8H_4O_4)_2(C_4H_4O_4)]_n$ [$M = \text{Gd}$ (Wang & Li, 2005), Dy ((Li & Wang, 2005), Nd (Li *et al.*, 2006) and Er (He *et al.*, 2006)] analogues. The Ho^{3+} ion is located at the center of a distorted square antiprism and is coordinated by four oxygen atoms from four benzene-1,4-dicarboxylate anions and four oxygen atoms from three succinate anions (Fig. 1). The Ho—O bond distances ranging from 2.2851 (2) to 2.5764 (16) Å.

The succinate anions are located on centres of inversion and acts as a bis-chelating ligands for each two symmetry related Ho atoms. Each of the four oxygen atom are additionally connected by Ho atoms into layers which are parallel to the (001) plane. These layers are connected *via* the benzene-1,4-dicarboxylate anions into a three-dimensional coordination network.

Experimental

A mixture of $\text{HoCl}_3 \cdot 6\text{H}_2\text{O}$ (2.00 mmol, 0.74 g), benzene-1,4-dicarboxylic acid (1.0 mmol, 0.16 g), succinic acid (1.0 mmol, 0.10 g), NaOH (6.0 ml, 1 mol/L) and H_2O (20.0 ml) was heated in a 35 ml stainless steel reactor with a Teflon liner at 453 K for 48 h. The column-like crystals were filtered and washed with ethanol. Yield: 30% based on Ho.

Refinement

H atoms were included at calculated positions and treated as riding atoms, with C—H distances of 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

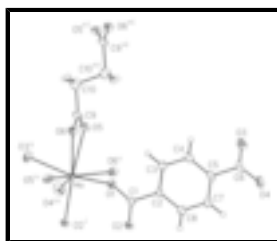


Fig. 1. The coordination environment of the Ho atom, with the atom-numbering scheme, showing displacement ellipsoids drawn at the 50% probability level. Symmetry codes: (i) $2 - x, 2 - y, 1 - z$; (ii) $x, 3/2 - y, z + 1/2$; (iii) $3/2 - x, 2 - y, z + 1/2$; (iv) $3/2 - x, y + 1/2, z$; (v) $3/2 - x, y - 1/2, z$; (vi) $1 - x, 2 - y, 1 - z$.

Poly[di- μ_4 -benzene-1,4-dicarboxylato- μ_6 -succinato-diholmium(III)]

Crystal data

$[\text{Ho}_2(\text{C}_4\text{H}_4\text{O}_4)(\text{C}_8\text{H}_4\text{O}_4)_2]$

$F_{000} = 1448$

$M_r = 387.08$

$D_x = 2.527 \text{ Mg m}^{-3}$

supplementary materials

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 13.8147$ (3) Å

$b = 6.7850$ (2) Å

$c = 21.7103$ (5) Å

$V = 2034.97$ (9) Å³

$Z = 8$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 254 reflections

$\theta = 2.1$ – 27.1°

$\mu = 7.79$ mm⁻¹

$T = 291$ (2) K

Column, orange

$0.18 \times 0.15 \times 0.05$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.245$, $T_{\max} = 0.682$

11598 measured reflections

2411 independent reflections

2140 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\text{max}} = 28.0^\circ$

$\theta_{\text{min}} = 1.9^\circ$

$h = -17 \rightarrow 16$

$k = -7 \rightarrow 8$

$l = -28 \rightarrow 25$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.015$

$wR(F^2) = 0.042$

$S = 1.06$

2411 reflections

155 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0238P)^2 + 0.923P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta\rho_{\text{max}} = 0.94$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.60$ e Å⁻³

Extinction correction: SHELXTL (Bruker, 1998),

$$F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.00060 (5)

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ho	0.830676 (8)	1.016049 (15)	0.554834 (5)	0.01275 (6)
O1	0.86282 (15)	1.0368 (2)	0.45156 (8)	0.0198 (4)
O2	1.01131 (12)	0.9282 (3)	0.43269 (7)	0.0206 (3)
C1	0.92600 (17)	0.9674 (3)	0.41630 (11)	0.0148 (4)
C2	0.89870 (17)	0.9259 (3)	0.35070 (10)	0.0177 (5)
C3	0.96527 (18)	0.8440 (4)	0.31009 (11)	0.0257 (5)
H3A	1.0274	0.8143	0.3237	0.031*
C4	0.93977 (19)	0.8064 (4)	0.24966 (11)	0.0276 (6)
H4A	0.9846	0.7510	0.2228	0.033*
C5	0.84715 (17)	0.8514 (3)	0.22885 (11)	0.0208 (5)
C6	0.81879 (18)	0.8035 (3)	0.16377 (11)	0.0213 (5)
C7	0.78017 (19)	0.9332 (4)	0.26926 (11)	0.0259 (5)
H7A	0.7181	0.9634	0.2555	0.031*
C8	0.8056 (2)	0.9698 (4)	0.32984 (13)	0.0244 (5)
H8A	0.7605	1.0240	0.3568	0.029*
O3	0.87624 (13)	0.6998 (3)	0.13253 (8)	0.0284 (4)
O4	0.73920 (13)	0.8685 (3)	0.14403 (7)	0.0265 (4)
O5	0.66712 (11)	0.8691 (3)	0.54719 (8)	0.0229 (4)
O6	0.67647 (11)	1.1742 (2)	0.51694 (8)	0.0205 (4)
C9	0.6257 (2)	1.0274 (3)	0.53160 (14)	0.0240 (6)
C10	0.5157 (2)	1.0403 (4)	0.53093 (15)	0.0313 (6)
H10A	0.4883	0.9627	0.5641	0.038*
H10B	0.4948	1.1760	0.5356	0.038*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ho	0.01198 (8)	0.01651 (7)	0.00977 (8)	-0.00044 (3)	0.00010 (4)	0.00131 (3)
O1	0.0203 (10)	0.0274 (9)	0.0118 (9)	0.0012 (7)	0.0002 (7)	-0.0009 (6)
O2	0.0158 (9)	0.0311 (8)	0.0149 (8)	-0.0020 (7)	-0.0033 (7)	-0.0017 (7)
C1	0.0166 (12)	0.0170 (10)	0.0108 (11)	-0.0019 (8)	0.0003 (9)	0.0010 (8)
C2	0.0190 (12)	0.0229 (11)	0.0112 (11)	0.0006 (9)	-0.0022 (9)	-0.0021 (8)
C3	0.0203 (13)	0.0393 (14)	0.0175 (13)	0.0050 (10)	-0.0054 (10)	-0.0067 (10)
C4	0.0250 (14)	0.0385 (14)	0.0193 (13)	0.0056 (11)	-0.0008 (10)	-0.0087 (11)
C5	0.0278 (13)	0.0224 (11)	0.0123 (12)	0.0005 (9)	-0.0051 (10)	-0.0036 (9)
C6	0.0302 (14)	0.0206 (11)	0.0131 (12)	-0.0036 (9)	-0.0049 (10)	-0.0017 (9)
C7	0.0228 (13)	0.0364 (13)	0.0186 (13)	0.0066 (11)	-0.0097 (11)	-0.0037 (11)
C8	0.0210 (13)	0.0356 (13)	0.0167 (13)	0.0063 (10)	-0.0022 (11)	-0.0065 (10)
O3	0.0338 (10)	0.0312 (9)	0.0203 (10)	0.0021 (8)	-0.0054 (8)	-0.0088 (7)
O4	0.0264 (10)	0.0378 (10)	0.0154 (8)	0.0011 (8)	-0.0072 (7)	0.0012 (7)
O5	0.0173 (9)	0.0174 (8)	0.0341 (11)	0.0002 (6)	-0.0071 (7)	0.0010 (7)
O6	0.0189 (9)	0.0188 (8)	0.0238 (10)	-0.0010 (6)	-0.0022 (7)	0.0019 (7)

supplementary materials

C9	0.0197 (14)	0.0212 (12)	0.0310 (16)	-0.0020 (9)	-0.0089 (12)	0.0025 (10)
C10	0.0376 (18)	0.0236 (12)	0.0326 (17)	0.0037 (11)	-0.0005 (14)	-0.0019 (11)

Geometric parameters (Å, °)

Ho—O1	2.2899 (17)	C4—C5	1.391 (3)
Ho—O2 ⁱ	2.2320 (17)	C4—H4A	0.9300
Ho—O3 ⁱⁱ	2.3208 (16)	C5—C7	1.391 (3)
Ho—O4 ⁱⁱⁱ	2.3013 (16)	C5—C6	1.502 (3)
Ho—O5	2.4753 (16)	C6—O3	1.259 (3)
Ho—O5 ^{iv}	2.4015 (18)	C6—O4	1.260 (3)
Ho—O6 ^v	2.4633 (17)	C7—C8	1.384 (4)
Ho—O6	2.5230 (16)	C7—H7A	0.9300
O1—C1	1.253 (3)	C8—H8A	0.9300
O2—C1	1.259 (3)	O5—C9	1.263 (3)
C1—C2	1.500 (3)	O6—C9	1.259 (3)
C2—C3	1.390 (3)	C9—C10	1.523 (4)
C2—C8	1.395 (4)	C10—C10 ^{vi}	1.514 (6)
C3—C4	1.382 (3)	C10—H10A	0.9700
C3—H3A	0.9300	C10—H10B	0.9700
O2 ⁱ —Ho—O1	85.34 (7)	C3—C2—C8	119.3 (2)
O2 ⁱ —Ho—O4 ⁱⁱⁱ	104.50 (6)	C3—C2—C1	120.7 (2)
O1—Ho—O4 ⁱⁱⁱ	152.17 (6)	C8—C2—C1	120.0 (2)
O2 ⁱ —Ho—O3 ⁱⁱ	75.73 (6)	C4—C3—C2	120.5 (2)
O1—Ho—O3 ⁱⁱ	134.25 (7)	C4—C3—H3A	119.8
O4 ⁱⁱⁱ —Ho—O3 ⁱⁱ	73.56 (7)	C2—C3—H3A	119.8
O2 ⁱ —Ho—O5 ^{iv}	80.03 (6)	C3—C4—C5	120.2 (2)
O1—Ho—O5 ^{iv}	82.44 (6)	C3—C4—H4A	119.9
O4 ⁱⁱⁱ —Ho—O5 ^{iv}	73.97 (6)	C5—C4—H4A	119.9
O3 ⁱⁱ —Ho—O5 ^{iv}	132.55 (6)	C7—C5—C4	119.7 (2)
O2 ⁱ —Ho—O6 ^v	103.85 (6)	C7—C5—C6	120.4 (2)
O1—Ho—O6 ^v	74.86 (6)	C4—C5—C6	119.9 (2)
O4 ⁱⁱⁱ —Ho—O6 ^v	125.79 (6)	O3—C6—O4	124.2 (2)
O3 ⁱⁱ —Ho—O6 ^v	70.08 (6)	O3—C6—C5	117.6 (2)
O5 ^{iv} —Ho—O6 ^v	156.47 (6)	O4—C6—C5	118.2 (2)
O2 ⁱ —Ho—O5	165.74 (6)	C8—C7—C5	120.1 (2)
O1—Ho—O5	97.82 (7)	C8—C7—H7A	119.9
O4 ⁱⁱⁱ —Ho—O5	79.09 (6)	C5—C7—H7A	119.9
O3 ⁱⁱ —Ho—O5	92.42 (6)	C7—C8—C2	120.3 (2)
O5 ^{iv} —Ho—O5	114.12 (5)	C7—C8—H8A	119.9
O6 ^v —Ho—O5	64.00 (6)	C2—C8—H8A	119.9
O2 ⁱ —Ho—O6	142.50 (6)	C6—O3—Ho ^{vii}	124.97 (15)
O1—Ho—O6	79.54 (6)	C6—O4—Ho ^{viii}	140.49 (16)

O4 ⁱⁱⁱ —Ho—O6	77.02 (6)	C9—O5—Ho ^v	150.76 (16)
O3 ⁱⁱ —Ho—O6	137.25 (6)	C9—O5—Ho	95.09 (15)
O5 ^{iv} —Ho—O6	64.15 (5)	Ho ^v —O5—Ho	112.68 (6)
O6 ^v —Ho—O6	104.93 (5)	C9—O6—Ho ^{iv}	129.67 (17)
O5—Ho—O6	51.58 (6)	C9—O6—Ho	92.94 (15)
O2 ⁱ —Ho—C9	168.32 (6)	Ho ^{iv} —O6—Ho	108.99 (6)
O1—Ho—C9	91.01 (8)	O6—C9—O5	119.2 (2)
O4 ⁱⁱⁱ —Ho—C9	74.08 (8)	O6—C9—C10	120.5 (2)
O3 ⁱⁱ —Ho—C9	114.28 (7)	O5—C9—C10	120.2 (2)
O5 ^{iv} —Ho—C9	88.50 (6)	O6—C9—Ho	61.15 (13)
O6 ^v —Ho—C9	85.82 (6)	O5—C9—Ho	58.99 (13)
O5—Ho—C9	25.92 (6)	C10—C9—Ho	170.3 (2)
O6—Ho—C9	25.91 (6)	C10 ^{vi} —C10—C9	105.9 (3)
C1—O1—Ho	135.25 (16)	C10 ^{vi} —C10—H10A	110.6
C1—O2—Ho ⁱ	156.02 (15)	C9—C10—H10A	110.6
O1—C1—O2	124.0 (2)	C10 ^{vi} —C10—H10B	110.6
O1—C1—C2	118.4 (2)	C9—C10—H10B	110.6
O2—C1—C2	117.6 (2)	H10A—C10—H10B	108.7

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

Fig. 1

