# metal-organic papers

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#### Key indicators

Single-crystal X-ray study T = 295 KMean  $\sigma(\text{C-C}) = 0.006 \text{ Å}$  R factor = 0.020 wR factor = 0.049 Data-to-parameter ratio = 15.6

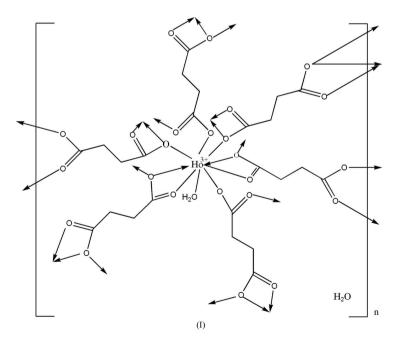
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

# Poly[[diaqua- $\mu_6$ -succinato-di- $\mu_5$ -succinato-diholmium(III)] monohydrate]

The Ho<sup>III</sup> center in the title coordination polymer,  $\{[Ho_2(C_4H_4O_4)_3(H_2O)_2]\cdot H_2O\}_n$ , is nine-coordinated in a tricapped trigonal prism by eight O atoms, derived from six carboxylate groups and a water molecule. One of the independent succinate anions is located about a crystallographic center of inversion and the uncoordinated water molecule lies on a twofold axis. The crystal structure comprises edge-shared HoO<sub>9</sub> polyhedra linked by succinate bridges, forming a three-dimensional network structure.

#### Comment

An area of contemporary interest is the study of coordination frameworks with suitable rigid multidentate ligands. Amongst these, those having long chain dicarboxylates present interesting behavior owing to their conformational flexibility and coordination diversity. Lanthanide ions exhibit high affinity for oxygen and diverse coordination modes. In an attempt to further understand the formation of lanthanide-organic framework materials, we present here the hydrothermal synthesis and crystal structure of a new Ln–succinate complex, (I).

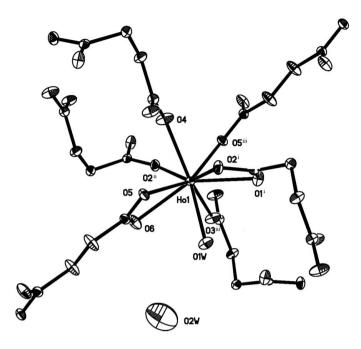


Complex (I) represents the first reported succinate coordination polymer of holmium(III) but it is isostructural with the known *Ln*-succinate complexes: Ln = Y and La (Josefina *et al.*, 2004), Pr (Serpaggi & Ferey, 1999), Sm (Seguatni *et al.*, 2004), Gd (Zhou *et al.*, 2005) and Tb (Cui *et al.*, 2005) analogs.

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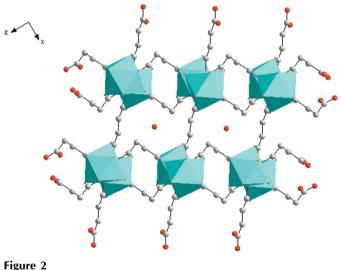
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#### Figure 1

The coordination environment of the Ho<sup>III</sup> ion in (I), showing the atom labeling and displacement ellipsoids drawn at the 50% probability level. H atoms have been removed for clarity. [Symmetry codes: (i) x, 1 - y,  $z = \frac{1}{2}$ ; (ii)  $\frac{1}{2} - x$ ,  $\frac{1}{2} - y$ , 1 - z; (iii)  $\frac{1}{2} - x$ ,  $y = \frac{1}{2}$ ,  $\frac{1}{2} - z$ .]



View of the packing in (I).

The asymmetric unit in (I) comprises a Ho atom, one and a half succinate anions, a coordinated water molecule and half an uncoordinated water molecule. One of the independent succinate anions, containing atoms O5 and O6, is situated about a crystallographic center of inversion; the uncoordinated water molecule is located on a twofold axis. The Ho<sup>III</sup> center (Fig. 1) is nine-coordinated within a tricapped trigonalprismatic geometry defined by eight O atoms, derived from six carboxylate anions, and a water molecule. The Ho-O bond distances lie in the range 2.276 (2)–2.496 (3) Å (Table 1). Each succinate anion binds a total of five or six Ho atoms. Each carboxylate group of the centrosymmetric anion chelates one Ho center and at the same time uses one O atom. O5, to bridge a symmetry-related Ho atom. A similar coordination mode is found for the carboxylate group containing atoms O1 and O2 of the second succinate anion, whereas the other end is bidentate bridging.

The crystal structure comprises edge-sharing  $HoO_8(OH_2)$ polyhedra forming chains along the *b*-axis direction by sharing one edge with each neigboring polyhedron; The Ho...Ho distance within chains is 4.002 (3) Å. These chains are in turn linked via succinate bridges, forming a three-dimensional framework as represented in Fig. 2.

### **Experimental**

A mixture of holmium(III) nitrate hexahydrate (0.441 g, 1.0 mmol), succinic acid (0.118 g, 1.0 mmol) and NaOH (0.052 g, 1.3 mmol) in water (15 ml) was introduced into a Parr Teflon-lined stainless steel vessel (25 ml), after which the vessel was sealed and heated to 433 K. The temperature was maintained for 72 h, and then the mixture was slowly cooled to room temperature at a rate of 10 K h<sup>-1</sup>. Yellow X-ray diffraction quality crystals were isolated (yield 26%, based on Ho).

#### Crystal data

$[Ho_2(C_4H_4O_4)_3(H_2O)_2] \cdot H_2O$	Z = 4
$M_r = 732.12$	$D_x = 2.712 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 19.852 (4)  Å	$\mu = 8.84 \text{ mm}^{-1}$
b = 7.6723 (15)  Å	T = 295 (2) K
c = 13.800 (3)  Å	Prism, yellow
$\beta = 121.46 \ (3)^{\circ}$	$0.67 \times 0.23 \times 0.14 \text{ mm}$
V = 1792.9 (6) Å <sup>3</sup>	

#### Data collection

Rigaku R-AXIS RAPID IP areadetector diffractometer oscillation scans Absorption correction: multi-scan (Jacobson, 1998)  $T_{\min} = 0.066, \ T_{\max} = 0.373$ (expected range = 0.051-0.290)

#### Refinement

Refinement on  $F^2$  $w = 1/[\sigma^2(F_0^2) + (0.0139P)^2]$  $R[F^2 > 2\sigma(F^2)] = 0.020$  $wR(F^2) = 0.049$ where  $P = (F_0^2 + 2F_c^2)/3$ S = 1.05 $(\Delta/\sigma)_{\rm max} = 0.001$  $\Delta \rho_{\rm max} = 0.92 \ {\rm e} \ {\rm \AA}^{-3}$ 2058 reflections  $\Delta \rho_{\rm min} = -1.59 \text{ e} \text{ Å}^{-3}$ 132 parameters H-atom parameters constrained

# Table 1

Selected bond lengths (Å).

Symmetry codes: $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}.$	(i) $x, -y + 1, z$	$-\frac{1}{2}$ ; (ii)	$-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1;$	(iii)
Ho1-O3 <sup>iii</sup>	2.276 (2)			
Ho1-O2 <sup>ii</sup>	2.385 (3)	Ho1-O6	2.44	42 (3)
Ho1-O2 <sup>i</sup>	2.496 (3)	Ho1-O5	2.38	33 (3)
Ho1-O1 <sup>i</sup>	2.448 (3)	Ho1-O5	2.42	23 (2)
Ho1 - O1W	2.429 (3)	Ho1-O4	2.39	92 (3)

8553 measured reflections

 $R_{\rm int} = 0.036$  $\theta_{\rm max} = 27.5^{\circ}$ 

+15.8528P]

2058 independent reflections

2011 reflections with  $I > 2\sigma(I)$ 

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

$D - H \cdots A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$O1W-H1A\cdots O4^{iv}$	0.85	2.06	2.857 (4)	155
$O1W-H1B\cdots O1^{iii}$	0.85	1.86	2.709 (4)	172
$O2W-H2C\cdots O1W$	0.85	2.18	2.930 (4)	147

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

All H atoms belonging to coordinated and solvent water molecules were located in a difference Fourier map and were allowed to ride on the attached O atoms, with O-H = 0.85 Å and  $U_{iso}(H) = 1.5U_{eq}(O)$ . Other H atoms were placed in calculated positions, with C-H = 0.97 Å, and refined as riding, with  $U_{iso}(H) = 1.2U_{eq}(C)$ . The deepest residual electron density hole is located 0.87 Å from atom Ho1.

Data collection: *RAPID-AUTO* (Rigaku ,2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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