Poly[[tetraaquadi-μ_4-succinato-μ_5-succinato-diytterbium(III)] hexahydrate]

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.024
wR factor = 0.065
Data-to-parameter ratio = 27.5

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

The title compound, $\{[Yb_2(C_4H_4O_4)_3(H_2O)_4]\cdot6H_2O\}_n$, is a two-dimensional open-framework built up from centrosymmetric binuclear units developed along [100] through the spacers of four extended ligands, and interconnected along [101] by two twisted ligands, forming a layered coordination polymer in the (010) plane embedding six uncoordinated water molecules. The water-coordinated Yb^{III} atom completes a nine-coordination mode *via* seven O atoms from four carboxylate ligands, in a monocapped square antiprism. One independent succinate ligand, located on an inversion center, is in a bidentate coordination mode, exhibiting an *anti* conformation. The second independent succinate ligand, in a *gauche* conformation, is bidentate and bridging–chelating, involving one triply ligating O atom displaying the longest Yb—O bond [2.598 (2) Å]. In addition to interlayer hydrogen bonds, intralayer hydrogen bonds involve, as acceptors, carboxylate O atoms which are in bidentate mode.

Comment

α,ω-Dicarboxylic acids give flexible ambidentate and templating ligands leading to coordination polymers having a wide breadth of applications (Kitagawa *et al.*, 2004; Rao *et al.*, 2004). With lanthanide cations, known for their oxygen affinity and their great range of coordination numbers and modes, the resulting self-assembly networks have enhanced the field of supramolecular chemistry from both a theoretical and practical point of view (Bradshaw *et al.*, 2005; Mellot-Drazniels *et al.*, 2002). In an attempt to understand the significance of specific interactions in hybrid materials based on rare-earth elements (Benmerad, 2000; Rahahlia, 2003), we have synthesized, under hydrothermal conditions, and determined the first crystal structure of an ytterbium succinate.

The title compound, (I), is isostructural with the erbium analog (Sun *et al.*, 2004), but its chemical formula is different from the ytterbium succinate obtained in powder form (Sevost'yanov & Dvornikova, 1972). Except for Pm and Tm, but in addition to Y and Sc, the single-crystal structures of all other rare-earth succinates have been obtained, reflecting the increased activity in the field of metal–organic frameworks involving lanthanides (Cui *et al.*, 2005; Fleck, 2002; He *et al.*, 2006; Nika *et al.*, 2005; Perles *et al.*, 2004; Seguatni *et al.*, 2004; Serpaggi *et al.*, 1999; Wang *et al.*, 2006; Yu *et al.*, 2006; Zhou *et al.*, 2005).

The asymmetric unit of (I) contains one Yb^{III} atom, one and a half succinate anions, two coordinated and three uncoordinated water molecules. If the longer Yb—O5^{ii} [symmetry code: (i) $1 + x, y, z$] distance of 2.589 (2) Å is considered to be a bond, then two Yb^{III} atoms are linked through two $\mu_{1,1}$– and two $\mu_{1,3}$-oxo-bridges, forming the basic centrosymmetric

binuclear unit, with a Yb···Yb distance of 4.081 (2) Å (Fig. 1). Hence, the Yb^{III} atom is nine-coordinated by seven O atoms from succinate ligands and two O atoms from aqua ligands. The coordination polyhedron is a slightly distorted mono-capped square antiprism, with atom O1 in the capping position. An extended succinate ligand, located on an inversion center, is tetradentate, in an *anti* conformation, with a C1—C2—C2^i—C1^i torsion angle [symmetry code: (i) $-x$, $1-y$, $2-z$] of 180° and connects two symmetry-related Yb^{III} atoms. A twisted succinate ligand is pentadentate, in a *syn* conformation with a C3—C4—C5—C6 torsion angle of −61.5 (3)°, connecting three Yb^{III} atoms. The Yb—O$_{carboxylate}$ bond lengths (Table 1) are consistent with expected values (Wu *et al.*, 2003), while the longer Yb—O5^{ii} distance involves the triply ligating O atom of the bridging–chelating ligand. Within the carboxylate ligands, all geometric parameters have normal bond distances (Zheng & Sun, 2003).
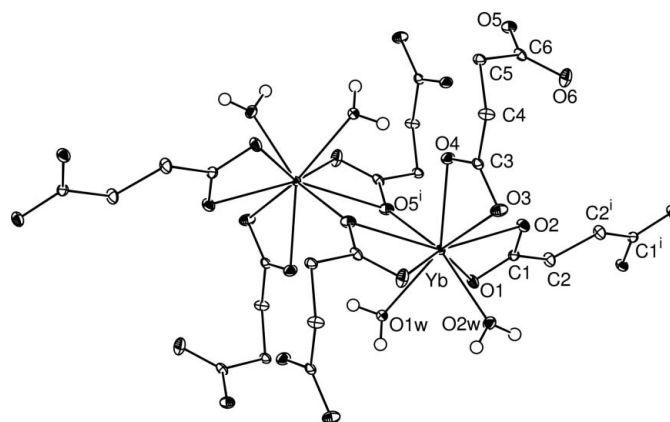

Figure 1
Part of the crystal structure of (I), showing the labeling scheme and 50% probability displacement ellipsoids. [Symmetry code: (i) $1+x$, y, z.]
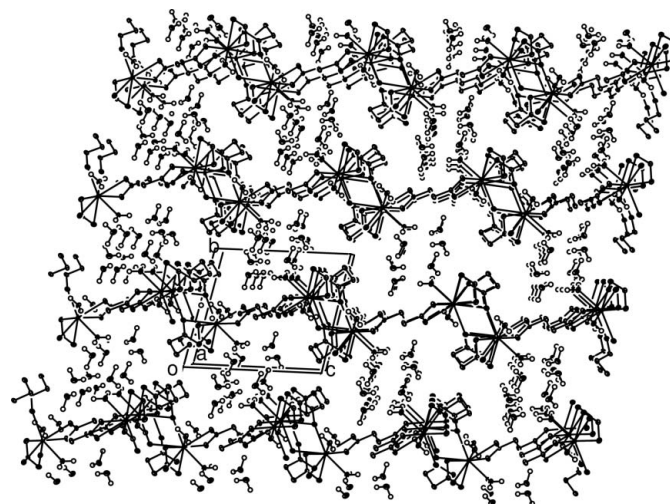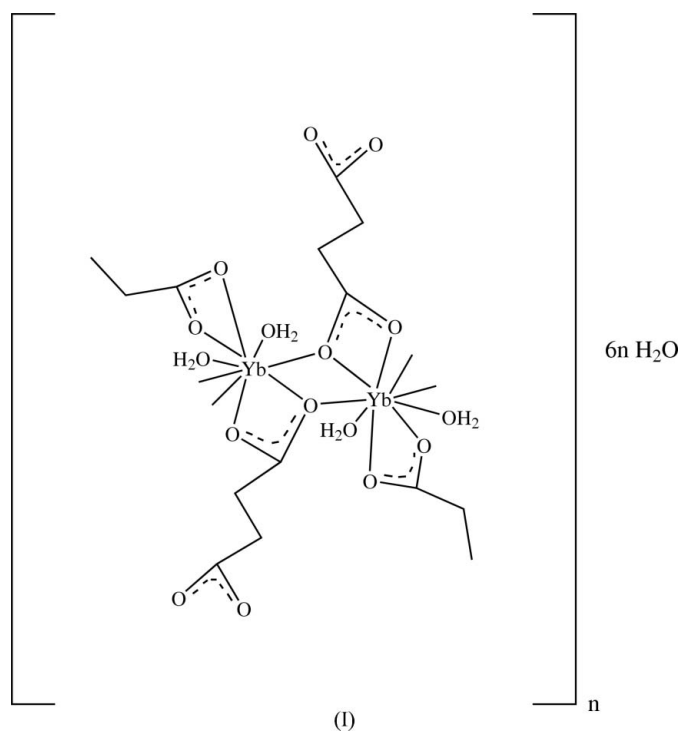

(I)

6n H$_2$O

Figure 2
Packing diagram of (I), viewed along the *a* axis.

extended network is constructed from binuclear entities, or a cage assembly.

The connection of the one-edge-sharing bi-antiprisms in the [100] and [101] directions, through the ligands as spacers, and *via* interlayer hydrogen bonds, leads to the formation of channels surrounded by methylene groups and hydrogen-bonded water molecules (Fig. 2). Interlayer hydrogen bonds are observed, involving uncoordinated water molecules (O3*W*, O4*W* and O5*W*) as acceptors. There are strong interactions between two H atoms bonded to O4*W* and O3*W* respectively. Moreover, these uncoordinated water molecules, as well as the aqua ligands (O1*W* and O2*W*), act as donors in strong interlayer and intralayer bonds respectively (Table 2). This study confirms that coordination polymers comprising the infinite typical metal–O–metal subfeature distinguishing rare earth and alkaline earth dicarboxylates cannot exist when an

Experimental

Yb$_2$O$_3$ (0.394 g, 1 mmol) was dissolved upon heating in 5 ml concentrated HCl. The resulting residue, added to succinic acid (0.236 g, 2 mmol) in 15 ml deionized water, was introduced into a 23 ml Teflon-lined stainless steel vessel. The vessel was sealed, heated to 433 K for 3 d and then cooled to room temperature; colorless crystals were separated by filtration.

Crystal data

[Yb$_2$(C$_4$H$_4$O$_4$)$_3$(H$_2$O)$_4$]·6H$_2$O	$V = 607.7$ (6) Å^3
$M_r = 437.23$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 2.389$ Mg m^{-3}
$a = 6.457$ (5) Å	Mo $K\alpha$ radiation
$b = 9.741$ (5) Å	$\mu = 7.75$ mm^{-1}
$c = 10.384$ (5) Å	$T = 100$ (2) K
$\alpha = 79.959$ (5)°	Prism, colorless
$\beta = 89.571$ (5)°	$0.46 \times 0.17 \times 0.05$ mm
$\gamma = 71.133$ (5)°	

Data collection

Nonius KappaCCD diffractometer	29166 measured reflections
ω scans	5330 independent reflections
Absorption correction: numerical	5008 reflections with $I > 2\sigma(I)$
($ABSORB$; DeTitta, 1988)	$R_{int} = 0.059$
$T_{min} = 0.184$, $T_{max} = 0.690$	$\theta_{max} = 35.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.024$	$+ 1.5702P]$
$wR(F^2) = 0.065$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.002$
5330 reflections	$\Delta\rho_{max} = 2.23$ e Å^{-3}
194 parameters	$\Delta\rho_{min} = -3.31$ e Å^{-3}
H-atom parameters constrained	Extinction correction: $SHELXL97$
	Extinction coefficient: 0.0182 (9)

Table 1
Selected geometric parameters (Å, °).

O1—Yb1	2.375 (2)	O1W—Yb1	2.322 (3)
O2—Yb1	2.435 (2)	O2W—Yb1	2.283 (2)
O3—Yb1	2.479 (2)	Yb1—O5^{i}	2.300 (2)
O4—Yb1	2.350 (2)	Yb1—O6^{ii}	2.372 (2)
C3—C4—C5—C6	−61.5 (3)	O1—C1—Yb1—O2	179.6 (3)
O3—C3—Yb1—O5^{i}	−174.58 (14)		

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H11$\cdots$O2^{ii}	0.79	1.997	2.768 (4)	165
O1W—H12$\cdots$O4^{iii}	0.80	1.861	2.654 (3)	170
O2W—H21$\cdots$O3W	0.81	1.915	2.722 (4)	172
O2W—H22$\cdots$O4W	0.82	1.857	2.671 (4)	171
O3W—H31$\cdots$O4W^{iv}	0.95	1.883	2.801 (3)	161
O3W—H32$\cdots$O3^{ii}	0.94	2.171	3.017 (4)	149
O3W—H32$\cdots$O2^{ii}	0.94	2.341	3.035 (4)	130
O4W—H42$\cdots$O1^{v}	0.91	1.846	2.735 (3)	164
O4W—H41$\cdots$O5W	0.83	1.914	2.723 (4)	163
O5W—H51$\cdots$O3^{vi}	0.80	2.191	2.952 (4)	157
O5W—H52$\cdots$O3W^{vii}	0.76	2.121	2.880 (4)	176

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

Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.5U_{eq}(O)$. Methylene H atoms were placed in geometrically idealized positions, with C—H = 0.97 Å, and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak in the final difference Fourier map is located 0.73 Å from H42 and the deepest hole 0.73 Å from atom Yb1.

Data collection: *KappaCCD Software* (Nonius, 1999); cell refinement: *DIRAX/LSQ* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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