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Tetra-μ-but-2-enoato-bis[diaqua(but-2-enoato)yttrium] di-μ-but-2-enoato-bis-[diaquabis(but-2-enoato)yttrium] 4.6-hydrate

The title compound, $[Y_2(C_4H_5O_2)_6(H_2O)_4]_2 \cdot 4.6H_2O$, has two independent dimeric units, displaying different bridging patterns between metal ions which lead to dissimilar Y···Y distances. The compound is isostructural with an already reported Gd complex.

Comment

The title compound, (I) (Fig. 1), is isostructural with the Gd homolog described by Rizzi *et al.* (2003), where the reader is referred for additional details. It consists of two centrosymmetric dimers with the metal ions linked by different carboxylate bridging motifs, but with similar YO_9 cores (Fig. 2).

$4.6 \, H_2O$

(I)

The Y1 dimer, quadruply bridged, shows Y1—O distances spanning the range 2.285 (2)–2.571 (2) Å (Table 1) with an
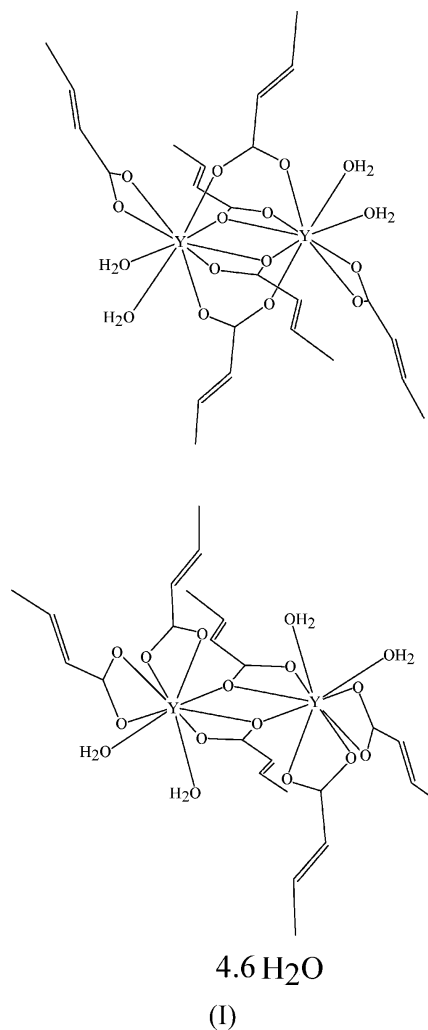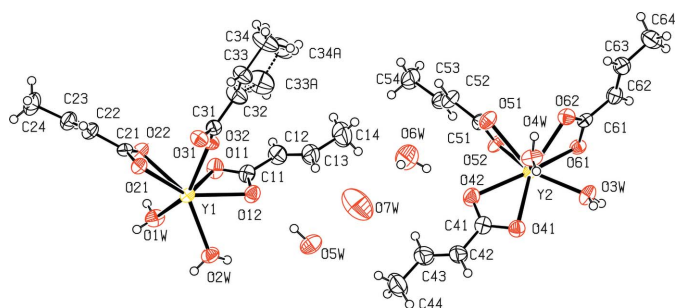
Figure 1
The structure of the asymmetric unit of (I) with the ellipsoids drawn at the 30% probability level and H atoms drawn as spheres of arbitrary radii. The disordered tail of one of the butenoate anions is shown as dashed lines.
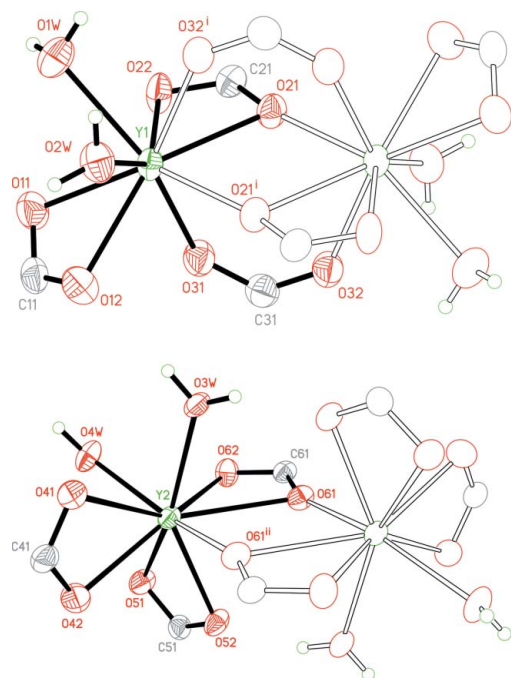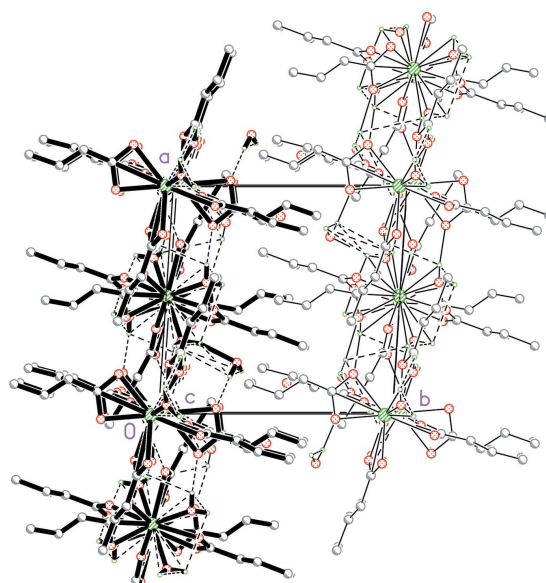

Figure 2
Coordination polyhedra for each independent dimer [top, Y1 dimer; bottom, Y2 dimer; symmetry codes: (i) $1 - x, 2 - y, 1 - z$; (ii) $-x, -y, -z$], showing the differences in the inner dimer bridging modes. For clarity, only the carboxylate groups of the butenoate anions are shown.

average value of 2.406 (9) Å. The Y1···Y1^i distance is 3.8789 (15) Å [symmetry code: (i) $1 - x, 2 - y, 1 - z$]. In the Y2 dimer, doubly bridged, the Y2—O bond lengths span the range 2.314 (2)–2.631 (2) Å (Table 1) with a mean value of 2.412 (10) Å. The looser link between cations results in a longer Y2···Y2^{ii} [symmetry code: (ii) $-x, -y, -z$] distance of 4.1123 (16) Å.

The shortest non-bridged metal···metal distance between different dimers [Y1···Y2^{iii}; symmetry code: (iii): $x + 1, y + 1, z$] is 5.992 (1) Å. The two similar four-membered coordination loops in both polyhedra (Y1—O21—Y1^i—O21^i; Y2—O61—Y2^{ii}—O61^{ii}) are almost perpendicular to each other, their mean planes subtending a dihedral angle of 88.9 (1)°.

The dimers are linked together through a dense hydrogen-bonding network (Table 2) defining tight two-dimensional

Figure 3
Packing view of (1) projected down the c axis, showing (sideways) two two-dimensional structures with interdigitated butenoate units in between.

structures parallel to (010), with metal atoms and hydrophilic parts at the centre, and the hydrophobic chains pointing outwards and interdigitating when these planes stack up along b (Fig. 3). Interplanar linkage is achieved through weak interactions involving disordered parts of the structure, viz. hydrogen bonds involving the partially occupied solvent water molecule O7W or the π–π contact between the centro-symmetrically related double bonds C22=C23···C22^{vi}=C23^{vi} [symmetry code (vi): $1 - x, 1 - y, 1 - z$], with a mid-point-to-mid-point distance of 3.75 (1) Å.

Experimental

The complex was obtained by mixing a hot aqueous solution (30 ml) of crotonic acid (6 mmol) with an aqueous solution (20 ml) of Y$_2$O$_3$ (1 mmol). The resulting mixture was refluxed for 24 h, filtered while hot and then concentrated to 25 ml. The filtrate was left at room temperature. On standing, suitable crystals for single-crystal X-ray diffraction appeared and were used without further processing.

Crystal data

[Y$_2$(C$_4$H$_5$O$_2$)$_6$(H$_2$O)$_4$]$_2$·4.6H$_2$O	$V = 1711.9$ (11) Å^3
$M_r = 1603.60$	$Z = 1$
Triclinic, $P\bar{1}$	$D_x = 1.556$ Mg m^{-3}
$a = 10.431$ (4) Å	Mo $K\alpha$ radiation
$b = 10.519$ (4) Å	$\mu = 3.45$ mm^{-1}
$c = 15.802$ (6) Å	$T = 298$ (2) K
$\alpha = 89.382$ (6)°	Block, colourless
$\beta = 81.627$ (6)°	$0.22 \times 0.19 \times 0.17$ mm
$\gamma = 86.338$ (6)°	

Data collection

Bruker SMART CCD area-detector diffractometer	14298 measured reflections
φ and ω scans	8173 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	5632 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.035$
$T_{min} = 0.48$, $T_{max} = 0.56$	$\theta_{max} = 27.9°$

Refinement

Refinement on F^2		H atoms treated by a mixture of	
$R[F^2 > 2\sigma(F^2)] = 0.043$		independent and constrained	
$wR(F^2) = 0.112$		refinement	
$S = 0.97$		$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2]$	
8173 reflections		where $P = (F_o^2 + 2F_c^2)/3$	
465 parameters		$(\Delta/\sigma)_{max} = 0.001$	
		$\Delta\rho_{max} = 0.65$ e Å^{-3}	
		$\Delta\rho_{min} = -0.36$ e Å^{-3}	

Table 1
Selected bond lengths (Å).

Y1—O21^i	2.285 (2)	Y2—O61^{ii}	2.314 (2)
Y1—O31	2.314 (2)	Y2—O4W	2.322 (2)
Y1—O32^i	2.332 (3)	Y2—O3W	2.339 (2)
Y1—O2W	2.390 (3)	Y2—O52	2.365 (2)
Y1—O11	2.415 (2)	Y2—O42	2.407 (3)
Y1—O1W	2.444 (3)	Y2—O62	2.408 (3)
Y1—O12	2.451 (3)	Y2—O41	2.458 (2)
Y1—O22	2.452 (3)	Y2—O51	2.462 (3)
Y1—O21	2.571 (2)	Y2—O61	2.631 (2)

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

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1W—H1WA$\cdots$O41^{iii}	0.85 (3)	1.88 (3)	2.722 (4)	171 (3)
O2W—H2WA$\cdots$O51^{iv}	0.85 (2)	1.90 (3)	2.731 (4)	164 (3)
O2W—H2WB$\cdots$O6W^{iv}	0.85 (2)	2.09 (3)	2.861 (6)	151 (3)
O3W—H3WA$\cdots$O11^v	0.85 (3)	1.90 (3)	2.738 (3)	173 (4)
O3W—H3WB$\cdots$O52^{ii}	0.85 (3)	1.90 (3)	2.703 (3)	158 (3)
O4W—H4WA$\cdots$O22^v	0.85 (3)	1.90 (3)	2.721 (3)	164 (3)
O4W—H4WB$\cdots$O5W^{vi}	0.85 (3)	2.05 (3)	2.844 (4)	158 (3)
O5W—H5WA$\cdots$O62^{iv}	0.85 (2)	2.10 (3)	2.820 (4)	141 (2)
O5W—H5WB$\cdots$O12	0.85 (2)	2.06 (3)	2.891 (4)	165 (4)
O6W—H6WA$\cdots$O42	0.85 (3)	2.03 (3)	2.880 (5)	176 (6)
O6W—H6WB$\cdots$O7W	0.85 (3)	2.22 (5)	2.800 (16)	126 (6)

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

H atoms attached to carbon were placed at idealized positions [C—H = 0.96 (CH$_3$) and 0.93 Å (CH)] and allowed to ride. Those of water molecules (except for the partially occupied O7W) were found in difference maps and refined with restrained O—H distances [0.85 (2) Å] . All H atoms were assigned an isotropic displacement parameter $U_{iso}(H) = xU_{eq}$(parent atom), with x = 1.5 for methyl groups and x = 1.2 otherwise. One of the solvent water sites (O7W) appeared as partially occupied, and its site occupancy factor, initially refined, was finally fixed at 0.30; the isostructural Gd compound does not have this third solvent water molecule, probably becuase lower data quality impairs resolution. Atoms C33 and C34 of one of the butenoate units attached to Y1 are disordered over two sites with occupancy factors of 0.75/0.25. Their occupancy factors were allowed to vary in the early stages of refinement, and were fixed in the final stages at their convergence values of 0.75/0.25.

Data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-NT* (Bruker, 2001); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-NT* (Bruker, 2001); software used to prepare material for publication: *SHELXTL-NT* and *PLATON* (Spek, 2003).

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