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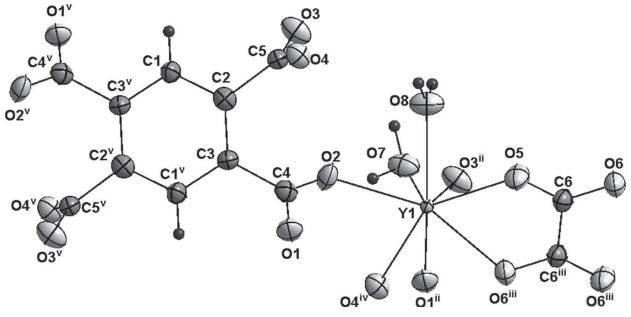
Crystal structure of poly[tetraaqua-(μ_4-oxalato-$\kappa^4O,O':O'',O'''$)-(μ_8-benzene-1,2,4,5-tetracarboxylato-$\kappa^8O^1:O^2:O^3:O^4:O^5:O^6:O^7:O^8$) yttrium(III)], $C_6H_5O_8Y$



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Abstract

$C_6H_5O_8Y$, triclinic, $P\bar{1}$ (no. 2), $a = 6.2090(17)$ Å, $b = 8.573(4)$ Å, $c = 8.863(2)$ Å, $\alpha = 111.018(6)°$, $\beta = 109.850(4)°$, $\gamma = 91.866(6)°$, $V = 407.8(2)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0435$, $wR_{ref}(F^2) = 0.1332$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of benzenetetracarboxylic acid (H_4btec, 0.10 g), oxalic acid ($H_2C_2O_4$, 0.05 g), $Y(NO_3)_3{\cdot}6H_2O$ (0.05 g), KOH (0.15 g) and deionized water (10 mL) was placed in a 30-mL Teflon-lined stainless steel autoclave. Then the autoclave was sealed, heated to 180 °C under autogenous pressure for 48 h. After slowly cooling to room temperature at a rate of 5 °C/h, a few colorless block shaped crystals were recovered by filtration, washed by distilled water, and air dried.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.15 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	7.18 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω-scans
θ_{max}, completeness:	28.2°, >92% (up to 25.2°, 99%)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	2579, 1846, 0.021
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1594
$N(param)_{refined}$:	137
Programs:	Bruker programs [11], SHELX [12]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
Y1	−1.18996(9)	0.35228(7)	−0.81616(7)	0.0106(2)
C1	−0.3644(13)	0.9862(9)	−0.5977(9)	0.0263(14)
H1	−0.274567	0.977428	−0.664676	0.032*
C2	−0.5079(12)	0.8424(9)	−0.6264(9)	0.0242(14)
C3	−0.6474(12)	0.8567(9)	−0.5293(9)	0.0239(14)
C4	−0.8150(12)	0.7052(9)	−0.5612(9)	0.0250(14)
C5	−0.5006(11)	0.6737(8)	−0.7612(8)	0.0218(13)
C6	−1.3928(13)	−0.0381(9)	−1.0120(9)	0.0269(15)
O1	−0.8143(10)	0.6751(7)	−0.4332(7)	0.0357(13)
O2	−0.9474(9)	0.6188(7)	−0.7160(7)	0.0343(12)
O3	−0.5788(10)	0.6528(8)	−0.9171(7)	0.0386(13)
O4	−0.4014(9)	0.5687(7)	−0.7024(7)	0.0317(12)
O5	−1.2015(9)	0.0589(7)	−0.9381(8)	0.0381(13)
O6	−1.4317(10)	−0.1955(7)	−1.1041(7)	0.0360(13)
O7	−0.8120(9)	0.3090(8)	−0.6656(7)	0.0390(13)
O8	−0.9711(10)	0.3167(8)	−0.9924(7)	0.0396(14)
H8A	−1.030843	0.328902	−1.102652	0.047*
H8B	−0.815473	0.295872	−0.942332	0.047*
H7B	−0.714603	0.374022	−0.543962	0.047*
H7A	−0.682813	0.311102	−0.697523	0.047*

Experimental details

H atoms were located in difference Fourier maps, and then refined with a riding model, with $U_{iso}(H) = 1.2U_{eq}(O)$.

Discussion

Coordination polymers have caught much attention in the field of crystal engineering due to the fascinating structures and potential applications in fluorescence, magnetism especially those of rare earth elements [1, 2]. It is well known

that the organic ligand is a key factor that affects the crystal structures as well as physical properties. Among various ligands, 1,2,4,5-benzenetetracarboxylate has been widely used in constructing metal coordination polymers due to its multiconnectivity, i.e. from two to eight carboxylate oxygen atoms which can bridge metal ions to create chains, layers, and 3D networks [3–6]. On the other hand, the oxalate anion is also an attractive small auxiliary ligand, which may construct many structures with H_4btec liand and metal ions [7, 8]. Hou et al. [9] reported a series of compounds associating with Ln^{3+} ions, $btec^{4-}$ ligands and $C_2O_4^{2-}$ ligands for Ln = Eu, Tb, Dy and Ho. In this study, we provide a isostructural compound $C_6H_5O_8Y$ by a self-assembly process through simple mixing of the starting reagents.

Single crystal X-ray diffraction (SC-XRD) analysis reveals that the title compound exhibits a three–dimensional framework in the triclinic space group $P\bar{1}$. There is one unique Y^{3+} ion, a half $btec^{4-}$ ligand, half $C_2O_4^{2-}$ ligand, and two coordinated aqua in the asymmetric unit. Each Y^{3+} is surrounded by eight O atoms. In this group, four O atoms come from carboxylate groups from four different $btec^{4-}$ ligands, two O atoms belongs to a two carboxylate groups of one $C_2O_4^{2-}$ ligand, and two O atoms are coordinated water molecules. The Y—O bond distances fall in the range of 2.296(5)–2.429(5) Å, which is the common value comparing with other Y^{3+} compounds [10]. Moreover, the rationality of eight Y—O bonds can be evaluated by bond valance sum (BVS) calculation. The calculated BVS for Y^{3+} is 3.190, very close to 3. The four carboxylate groups of btec ligand are completely deprotonated, and each carboxylate connects with two Y^{3+} ions via μ_2-mode. For each $C_2O_4^{2-}$ ligand, it connects to two Y^{3+} ions via μ_2-bridging mode.

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