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The crystal structure of poly[triaqua-bis(μ_3 -2,5-dihydroxyterephthalato- $\kappa^4 O, O': O'': O'''$)-(μ_4 -oxalato- $\kappa^4 O, O': O'', O'''$)cerium(III)], $C_9H_{10}CeO_{11}$

Table 1: Data collection and handling.

Crystal:	Block, light pink
Size:	0.18 × 0.11 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.66 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX, φ and ω -scans
$\theta_{\max}$, completeness:	26°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3454, 2440, 0.040
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2158
$N(\text{param})_{\text{refined}}$:	223
Programs:	Bruker programs [1], SHELX [2, 3], DIAMOND [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ce	0.20420(4)	0.22812(3)	0.61969(2)	0.01414(7)
O1	0.1677(5)	0.4292(4)	0.4069(3)	0.0262(8)
O2	0.0915(4)	0.6697(4)	0.2791(3)	0.0224(7)
O3	0.3195(5)	0.1400(3)	0.0870(3)	0.0239(7)
O4	0.1843(5)	0.7626(4)	0.0235(3)	0.0301(8)
O5	0.3346(5)	0.2172(4)	−0.1623(3)	0.0293(8)
O6	0.2945(5)	0.4591(4)	−0.2940(3)	0.0292(8)
O7	0.2453(4)	0.0484(3)	0.4842(3)	0.0213(7)
O8	0.1077(4)	−0.1450(4)	0.4439(3)	0.0281(8)
O9	0.4438(5)	−0.0009(4)	0.7025(4)	0.0312(9)
O10	0.5377(6)	0.3029(5)	0.5097(5)	0.0370(10)
O11	0.0440(7)	0.0008(5)	0.8083(5)	0.0450(12)
C1	0.1543(6)	0.5323(5)	0.2948(4)	0.0168(9)
C2	0.2108(6)	0.4928(5)	0.1703(4)	0.0161(9)
C3	0.2176(6)	0.6083(5)	0.0414(4)	0.0168(9)
C4	0.2478(6)	0.3389(5)	0.1817(4)	0.0188(10)
H4	0.246735	0.262881	0.266246	0.023*
C5	0.2861(6)	0.2963(5)	0.0695(4)	0.0177(9)
C6	0.2541(6)	0.5651(5)	−0.0705(4)	0.0190(10)
H6	0.256250	0.641202	−0.155075	0.023*
C7	0.2878(6)	0.4103(5)	−0.0591(4)	0.0167(9)
C8	0.3103(6)	0.3625(5)	−0.1797(4)	0.0197(10)
C9	0.1015(6)	−0.0287(5)	0.4802(4)	0.0182(10)
H3	0.335(7)	0.146(6)	−0.011(5)	0.050(16)*
H4A	0.165(9)	0.776(7)	0.113(5)	0.08(2)*
H11A	−0.053(7)	−0.028(6)	0.825(5)	0.026(17)*
H10A	0.647(8)	0.242(7)	0.540(5)	0.050(17)*
H9B	0.516(8)	−0.027(6)	0.648(5)	0.047(18)*
H9A	0.501(9)	−0.016(8)	0.782(6)	0.08(2)*
H11B	0.104(8)	−0.058(7)	0.855(5)	0.043(19)*
H10B	0.559(11)	0.356(8)	0.453(6)	0.08(3)*

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Abstract

$C_9H_{10}CeO_{11}$, triclinic, $P\bar{1}$ (no. 2), $a = 6.9100(14)$ Å, $b = 9.1700(18)$ Å, $c = 10.720(2)$ Å, $\alpha = 69.53(3)^\circ$, $\beta = 83.56(3)^\circ$, $\gamma = 87.01(3)^\circ$, $V = 632.3(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0265$, $wR_{\text{ref}}(F^2) = 0.0487$, $T = 298(2)$ K.

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The crystal structure is shown in the figure (ⁱ = x, y, 1 + z; ⁱⁱ = −x, 1 − y, 1 − z). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Source of materials

A mixture of Ce₂(C₂O₄)₃ (54.4 mg, 0.1 mmol), 2,5-dihydroxyterephthalic acid (DHTA) (39.6 mg, 0.2 mmol) in 1:2 molar ratio was sealed in a 30 mL Teflon-lined stainless steel containing deionized H₂O (20 mL) and methanol (2 mL), heated at 150 °C for 96 h and cooled down to room temperature. Pink block crystals were isolated and washed with deionized water.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Metal-organic frameworks (MOFs) have received attention during the last decades not only due to their architectures and topologies but also for their promising applications [5–9]. Metal-organic frameworks are built up by lanthanide metals and poly-functional organic ligands forming 1-D chain, 2-D sheet or 3-D networks [10–13]. In this study we employed cerium ions, with large radii and high coordination numbers, *exo*-multidentate ligands like 2,5-dihydroxyterephthalate and oxalate as the third ligand to hydrothermally synthesize metal-organic framework compounds with interesting 2-D structures [13].

The local geometry around Ce is a nine-coordinated tricapped trigonal prism made up of two oxygen (O7 and O8ⁱⁱ) from the same carboxylate group attached on an oxalato ligand, four oxygen (O1, O2ⁱⁱⁱ, O5ⁱ, O6ⁱ) from the 2,5-dihydroxyterephthalato ligand, three oxygen (O9, O10, O11) from three water molecules.

In the [Ce(OA)_{0.5}(DHTA)(H₂O)₃]_n, the oxalic acid ligands chelate two different cerium ions. The 2,5-dihydroxyterephthalates act as tetradentate linker to join three different cerium ions, in which two oxygen atoms (O5 and O6) from a carboxylate group link one Ce³⁺ in chelating mode, and the O2 link the other Ce³⁺ ions, the O1 also link the other one Ce³⁺ ions. The Ce³⁺ ions are linked by oxalato ligands into double metal centers and further joined into 2-D layers.

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Note: All authors contributed equally to this work.

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