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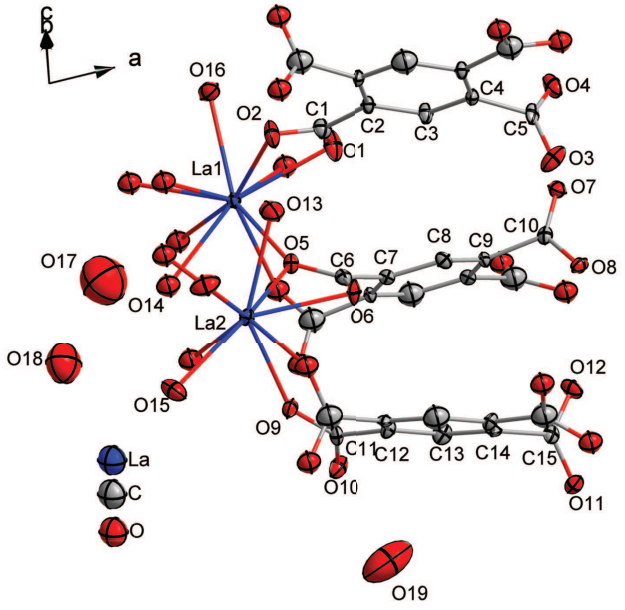
Crystal structure of poly[octaaqua-tris(benzene-1,2,4,5-tetracarboxylato)tetralanthanum(III)] hexahydrate, $C_{30}H_{34}La_4O_{38}$



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Abstract

$C_{30}H_{34}La_4O_{38}$, triclinic, $P\bar{1}$ (no. 2), $a = 9.2405(1)$ Å, $b = 10.6952(1)$ Å, $c = 10.8307(1)$ Å, $\alpha = 86.239(1)°$, $\beta = 84.014(1)°$, $\gamma = 81.367(1)°$, $V = 1051.13(2)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.033$, $wR_{ref}(F^2) = 0.072$, $T = 296$ (2) K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless, prism, size 0.03×0.05×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	41.10 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, ω scans
$2\theta_{max}$:	56.59°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22001, 5208
$N(param)_{refined}$:	325
Programs:	SHELX [8]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	1.0388	−0.4064	0.8034	0.018
H(8)	2*i*	1.0712	−0.6388	0.6638	0.016
H(13)	2*i*	1.0116	−0.8461	0.3410	0.020
H(18A)	2*i*	0.1116	−0.8796	0.8632	0.018
H(18B)	2*i*	−0.0484	−0.8378	0.7665	0.018
H(16A)	2*i*	0.5610	−0.0787	0.8152	0.015
H(16B)	2*i*	0.3772	−0.1039	0.8426	0.015
H(14A)	2*i*	0.3367	−0.8457	0.9191	0.015
H(14B)	2*i*	0.4576	−0.8761	1.0002	0.015
H(15A)	2*i*	0.2366	−0.8842	0.7190	0.015
H(15B)	2*i*	0.3273	−0.8732	0.5812	0.015
H(13A)	2*i*	0.6943	−0.7360	0.9312	0.015
H(13B)	2*i*	0.5489	−0.6480	0.9739	0.015
H(19A)	2*i*	0.8410	−0.8636	0.1306	0.015
H(19B)	2*i*	0.8787	−0.9230	0.0039	0.015
H(17A)	2*i*	0.2055	−0.9495	1.1245	0.049
H(17B)	2*i*	0.1363	−0.8520	1.0303	0.037

Source of material

A mixture of benzene-1,2,4,5-tetracarboxylic dianhydride ($C_{10}H_2O_6$) (0.1 mmol), $La(NO_3)_3$ (0.05 mmol), methanol (5 mL) and water (5 mL) whose pH value was adjusted to 5 by KOH, was placed in a 30-mL Teflon-lined stainless steel autoclave. Then the autoclave was sealed, heated to 160°C under autogenous pressure for 48 h. After slowly cooled to room temperature at a rate of 5°C/h, a few colorless prismatic crystals were recovered by filtration. They were washed by distilled water, and air dried.

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
La(1)	2*i*	0.52199(3)	−0.32292(2)	0.68603(2)	0.0091(1)	0.0124(1)	0.0109(2)	−0.00084(9)	−0.0016(1)	0.0001(1)
La(2)	2*i*	0.53072(3)	−0.76016(2)	0.71951(2)	0.0111(1)	0.0123(1)	0.0127(2)	−0.0017(1)	0.0003(1)	0.0000(1)
C(1)	2*i*	0.7558(5)	−0.3848(4)	0.8638(4)	0.016(2)	0.018(2)	0.015(3)	−0.001(2)	−0.005(2)	0.002(2)
C(2)	2*i*	0.8802(4)	−0.4406(4)	0.9383(4)	0.010(2)	0.012(2)	0.013(2)	0.000(2)	−0.003(2)	0.001(2)
C(3)	2*i*	1.0229(5)	−0.4444(4)	0.8824(4)	0.011(2)	0.021(2)	0.012(2)	−0.001(2)	−0.002(2)	0.004(2)
C(4)	2*i*	1.1425(4)	−0.5041(4)	0.9425(4)	0.008(2)	0.014(2)	0.013(2)	0.001(2)	−0.001(2)	−0.002(2)
C(5)	2*i*	1.2905(5)	−0.5110(5)	0.8658(4)	0.009(2)	0.026(3)	0.009(2)	0.003(2)	−0.003(2)	0.004(2)
C(6)	2*i*	0.7819(5)	−0.6209(4)	0.6513(4)	0.012(2)	0.015(2)	0.011(2)	−0.003(2)	−0.002(2)	0.001(2)
C(7)	2*i*	0.8973(5)	−0.5603(4)	0.5733(4)	0.011(2)	0.016(2)	0.014(2)	−0.002(2)	−0.001(2)	−0.005(2)
C(8)	2*i*	1.0429(5)	−0.5827(4)	0.5985(4)	0.014(2)	0.013(2)	0.013(2)	−0.002(2)	−0.003(2)	0.001(2)
C(9)	2*i*	1.1467(4)	−0.5215(4)	0.5265(4)	0.008(2)	0.014(2)	0.017(2)	0.001(2)	−0.004(2)	−0.005(2)
C(10)	2*i*	1.3026(5)	−0.5327(4)	0.5596(4)	0.011(2)	0.016(2)	0.009(2)	−0.001(2)	−0.000(2)	0.003(2)
C(11)	2*i*	0.7358(4)	−0.8492(4)	0.4215(4)	0.008(2)	0.014(2)	0.018(3)	−0.003(2)	−0.001(2)	0.001(2)
C(12)	2*i*	0.8717(5)	−0.9274(4)	0.4650(4)	0.011(2)	0.012(2)	0.015(2)	0.002(2)	−0.000(2)	−0.001(2)
C(13)	2*i*	1.0070(5)	−0.9084(4)	0.4047(4)	0.019(2)	0.015(2)	0.015(3)	0.001(2)	−0.002(2)	0.004(2)
C(14)	2*i*	1.1366(5)	−0.9814(4)	0.4381(4)	0.012(2)	0.016(2)	0.008(2)	0.001(2)	−0.002(2)	−0.005(2)
C(15)	2*i*	1.2812(4)	−0.9534(4)	0.3700(4)	0.011(2)	0.015(2)	0.010(2)	0.002(2)	−0.003(2)	0.004(2)
O(1)	2*i*	0.7807(4)	−0.3222(4)	0.7646(3)	0.017(2)	0.042(2)	0.023(2)	−0.004(2)	−0.006(2)	0.017(2)
O(2)	2*i*	0.6281(3)	−0.4090(3)	0.9007(3)	0.011(2)	0.038(2)	0.023(2)	−0.004(2)	−0.004(1)	0.008(2)
O(3)	2*i*	1.3350(4)	−0.6147(3)	0.8172(3)	0.031(2)	0.025(2)	0.024(2)	0.009(2)	0.007(2)	−0.001(2)
O(4)	2*i*	1.3474(4)	−0.4123(3)	0.8482(3)	0.019(2)	0.039(2)	0.017(2)	−0.015(2)	−0.000(1)	0.004(2)
O(5)	2*i*	0.6533(3)	−0.5565(3)	0.6695(3)	0.011(1)	0.017(2)	0.017(2)	−0.001(1)	−0.001(1)	0.000(1)
O(6)	2*i*	0.8078(3)	−0.7324(3)	0.6927(3)	0.014(2)	0.017(2)	0.028(2)	−0.001(1)	−0.001(1)	0.006(2)
O(7)	2*i*	1.3418(3)	−0.4387(3)	0.5976(3)	0.018(2)	0.019(2)	0.015(2)	−0.004(1)	−0.003(1)	−0.002(1)
O(8)	2*i*	1.3831(3)	−0.6409(3)	0.5453(3)	0.016(2)	0.014(2)	0.014(2)	0.004(1)	−0.002(1)	−0.003(1)
O(9)	2*i*	0.6239(3)	−0.8224(3)	0.4989(3)	0.012(2)	0.020(2)	0.019(2)	0.002(1)	0.001(1)	−0.002(1)
O(10)	2*i*	0.7358(3)	−0.8131(3)	0.3100(3)	0.020(2)	0.026(2)	0.014(2)	0.007(1)	−0.001(1)	0.002(1)
O(11)	2*i*	1.3296(3)	−1.0140(3)	0.2733(3)	0.017(2)	0.016(2)	0.019(2)	−0.002(1)	0.003(1)	−0.002(1)
O(12)	2*i*	1.3410(3)	−0.8695(3)	0.4092(3)	0.022(2)	0.020(2)	0.021(2)	−0.012(1)	−0.002(1)	0.001(1)
O(13)	2*i*	0.6122(4)	−0.6847(4)	0.9182(3)	0.030(2)	0.064(3)	0.019(2)	−0.017(2)	0.002(2)	−0.011(2)
O(14)	2*i*	0.4301(4)	−0.8752(4)	0.9180(3)	0.046(2)	0.030(2)	0.020(2)	−0.005(2)	0.003(2)	−0.001(2)
O(15)	2*i*	0.3337(4)	−0.8845(3)	0.6688(3)	0.031(2)	0.034(2)	0.023(2)	−0.016(2)	−0.008(2)	0.001(2)
O(16)	2*i*	0.4802(4)	−0.1419(3)	0.8407(3)	0.024(2)	0.024(2)	0.025(2)	−0.007(2)	0.006(2)	−0.007(2)
O(17)	2*i*	0.1742(9)	−0.9334(8)	1.0421(8)	0.126(3)	0.132(3)	0.125(3)	−0.022(2)	−0.016(2)	−0.002(2)
O(18)	2*i*	0.0607(6)	−0.8665(6)	0.7938(5)	0.069(2)	0.082(2)	0.076(2)	−0.005(2)	−0.012(2)	0.004(2)
O(19)	2*i*	0.8490(8)	−0.8348(6)	0.0494(6)	0.139(6)	0.087(5)	0.071(5)	0.023(4)	0.018(4)	−0.016(4)

Discussion

Coordination polymers (CPs) has gained great recognition in recent years as an important interface between synthetic chemistry and material science not only because of their fascinating topology and intriguing architectures, but also due to their potential applications in magnetism, luminescence, ion exchange, electronic apparatus, catalysis *etc.* [1–5]. The most effective and facile approach for constructing new CPs is the judicious choice of various organic linkers such as benzene carboxylates and N-donor ligands. As part of this research, benzene-1,2,4,5-tetracarboxylate (btec) can be used as a ligand to form various supramolecular architectures with its four rigid carboxyl groups. In order to enrich this family of compounds [6], we used the hydrothermal method to synthesise the title compound, a new lanthanum(III) based complex, $La_4(C_{10}H_2O_8)_3(H_2O)_8{\cdot}6H_2O$.

Structure solution revealed that there are two La^{3+} cations, three half $btec^{4-}$ anions, four coordinated water molecules and three lattice water moleculas in the asymmetric unit of title structure. The La1 is 10-coordinated by nine carboxylate oxygen atoms from seven $btec^{4-}$ ligands, and one oxygen atoms from an aqua ligand. Unlike La1, La2 is 9-coordinated by six carboxylate oxygen atoms from five $btec^{4-}$ ligands, and three oxygen atoms from aqua ligands. The La–O distances range from 2.405 (3) to 2.787 (3) Å, which are common values compared with those reported for other La(III) compounds [6, 7]. La1 and La2 polyhedra are connected via corner-sharing O atoms into an one-dimensional (1D) infinite

chains which are further interconnected by $btec^{4-}$ ligands to form the 3D framework.

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