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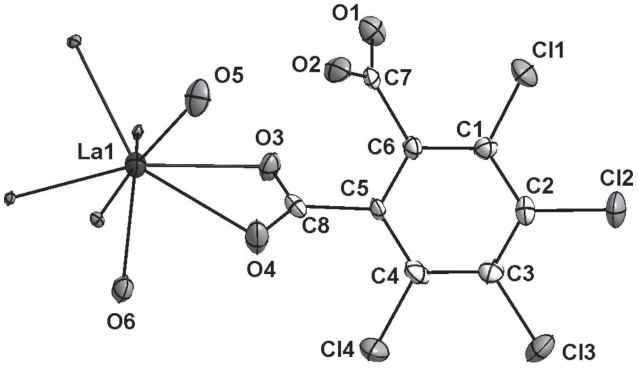
Crystal structure of *poly*-[diaqua-[bis(μ$_2$-hydroxy)-bis(μ$_4$-3,4,5,6-tetrachlorophthalato-κ^{3}O,O′:O′;κ^{2}O″:O‴)dilanthanum(III)], $C_8H_3Cl_4LaO_6$



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Abstract

$C_8H_3Cl_4LaO_6$, monoclinic, $P2_1/n$ (no. 14), $a = 6.6874(10)$ Å, $b = 6.0956(9)$ Å, $c = 30.281(4)$ Å, $\beta = 91.094(3)°$, $V = 1234.1(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.143$, $T = 296(2)$ K.

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A part of the title 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

Colorless prism-shaped single crystals of the title compound were initially obtained using hydrothermal method. A mixture of 2 mmol 3,4,5,6-tetrachlorophthalic acid, 2 mmol of $La(Ac)_3$ and 4.0 mmol KOH was sealed in a 30 mL Teflon-lined bomb at 180 °C for 7 days and then cooled to room temperature. A few crystals suitable for X-ray diffraction analysis were obtained.

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.20 × 0.05 × 0.05 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	43.4 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, ω-scans
$2\theta_{max}$, completeness:	56.8°, >97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7933, 3014, 0.081
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1926
$N(param)_{refined}$:	172
Programs:	Bruker programs [5], SHELX [6]

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

Atom	x	y	z	U_{iso}*/U_{eq}
La1	0.23121(8)	0.21435(8)	0.00479(2)	0.01912(17)
Cl1	1.1296(4)	0.5628(4)	0.14107(8)	0.0337(6)
Cl2	1.1677(4)	0.2442(5)	0.22141(9)	0.0435(7)
Cl3	0.8639(4)	−0.1394(5)	0.23045(8)	0.0395(7)
Cl4	0.5247(4)	−0.1971(4)	0.16043(8)	0.0363(6)
C1	0.9758(13)	0.3391(15)	0.1454(3)	0.0196(19)
C2	0.9864(13)	0.2002(14)	0.1820(3)	0.0205(19)
C3	0.8507(13)	0.0333(14)	0.1857(3)	0.022(2)
C4	0.6990(13)	0.0043(15)	0.1541(3)	0.023(2)
C5	0.6955(12)	0.1340(14)	0.1164(3)	0.0151(17)
C6	0.8345(12)	0.2998(14)	0.1123(3)	0.0164(17)
C7	0.8333(12)	0.4488(14)	0.0723(3)	0.0174(18)
C8	0.5308(12)	0.1181(14)	0.0807(3)	0.0163(18)
O1	0.9517(9)	0.4087(10)	0.0414(2)	0.0251(15)
O2	0.7135(9)	0.6037(10)	0.0706(2)	0.0260(15)
O3	0.5820(9)	0.0603(10)	0.04201(19)	0.0234(14)
O4	0.3586(10)	0.1723(12)	0.0889(2)	0.0334(17)
H5B	0.4244	0.5898	0.0561	0.040*
H5A	0.2755	0.6970	0.0369	0.040*
H6	0.0159	−0.1128	0.0627	0.040*
O5	0.3535(10)	0.5944(11)	0.0308(2)	0.0310(16)
O6	0.0674(9)	−0.1014(10)	0.03728(19)	0.0228(14)

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Experimental details

Hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq} of the parent atom.

Discussion

The self-assembly of coordination polymers (CPs) is an intriguing area, which is yielding new generations of supramolecular architectures [1, 2]. Coordination by transition or rare-earth metals with multifunctional ligands is one of the main design principles. The 3,4,5,6-tetrachlorophthalate ligand is a remarkably versatile building block for the construction of supramolecular architectures due to its two rigid carboxyl groups and various coordination modes in the self-assembly reactions. By such coordination, a variety of supramolecular scaffolds are generated to control the arrangement of periodic channels and the formation of 1-, 2-, 3-D networks [3, 4].

The title structure contains one La(III) atom, one water molecule, one hydroxy ligand and one 3,4,5,6-tetrachlorophthalate in an asymmetric unit. The 3,4,5,6-tetrachlorophthalate ligand is deprotonated and acts as μ_4-bridging. Considering the linking environment of the La(III) atoms, it is surrounded by nine O atoms, six from 3,4,5,6-tetrachlorophthalate ligands, one from coordinated water molecule, and two from hydroxy ligands. In an overview, the title compound features a two-dimensional structure, in which the layers are stacking along the *c*-axis.

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