Crystal structure of aqua(benzene-1,3,5-tricarboxylato)-lutetium(III), $Lu(H_2O)(C_9H_3O_6)$

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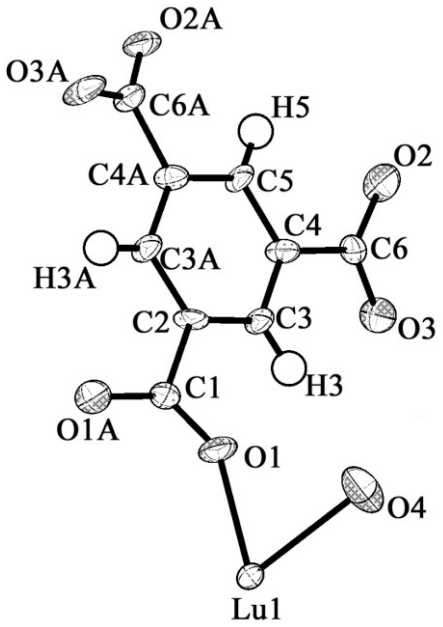

Abstract

$C_9H_5LuO_7$, tetragonal, $P4_122$ (no. 91), a = 10.225(1) Å, c = 14.369(3) Å, V = 1502.1 Å^3, Z = 8, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.125, T = 293 K.

Source of material

A clear solution of $Lu(NO_3)_3 \cdot nH_2O$ (40 mg, 0.10 mmol), 1,3,5-benzenetricarboxylic acid (H_3BTC, 20 mg, 0.10 mmol), DMF (10 ml) and H_2O (4 ml) was left at 85 °C for 10 hours to give colorless rod-shaped crystals. They were washed with 10 mL methanol three times and dried in air (yield 65 %, based on H_3BTC). The samples were stable in air and insoluble in common organic solvents such as acetone, methanol, ethanol, dichloromethane and DMF.

Experimental details

The Flack parameter is –0.08(5). Water H atoms have not been included in the refinement.

Discussion

In the title crystal structure, each rare earth metal Lu^{3+} is coordinated by six oxygen atoms from six ligands and one terminal coordinated water molecule, and each BTC ligand coordinates six Lu^{3+} with dimonodentate mode. Lu^{3+} connect with each other through carboxylates to form one-dimensional chiral lines along [001] direction, which can be defined as rod SBUs (second building units). Each rod SBU connects to others through BTC ligands along [100] and [010] directions to further construct three-dimensional frameworks. There are one-dimensional circular channels (approximately 6 × 6 Å^2) in the frameworks along the [001] direction, after the terminal coordinated water molecules have been removed.

Table 1. Data collection and handling.

Crystal:	colorless rod, size 0.3 × 0.3 × 0.8 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	65.87 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ/ω
$2\theta_{max}$:	56.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9550, 1787
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1630
$N(param)_{refined}$:	80
Programs:	SHELXS-97, SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	8*d*	1.079	0.4415	0.129	0.027
H(5)	4*a*	1	0.1000	0	0.026

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Lu(1)	4*c*	1.14819(3)	2–x	⅛	0.0174(2)	U_{11}	0.0116(2)	–0.0016(2)	0.0001(2)	U_{13}
C(1)	4*a*	1	0.613(1)	0	0.030(7)	0.015(6)	0.028(7)	0	–0.006(6)	0
C(2)	4*a*	1	0.463(1)	0	0.041(8)	0.008(6)	0.024(7)	0	–0.008(6)	0
C(3)	8*d*	1.047(1)	0.3959(9)	0.0779(7)	0.034(5)	0.018(4)	0.015(4)	0.004(4)	–0.007(4)	–0.001(3)
C(4)	8*d*	1.044(1)	0.259(1)	0.0781(6)	0.037(6)	0.016(5)	0.018(4)	0.000(4)	–0.002(4)	0.002(4)
C(5)	4*a*	1	0.191(1)	0	0.034(8)	0.017(6)	0.015(6)	0	–0.009(6)	0
C(6)	8*d*	1.089(1)	0.181(1)	0.1613(7)	0.024(5)	0.024(5)	0.021(4)	0.006(4)	–0.005(4)	0.002(4)
O(1)	8*d*	1.0732(9)	0.6665(7)	0.0581(6)	0.057(6)	0.019(4)	0.035(4)	–0.003(4)	–0.016(4)	–0.006(3)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	8*d*	1.1139(9)	0.0642(8)	0.1498(6)	0.063(6)	0.020(4)	0.040(5)	–0.002(4)	–0.020(4)	0.008(3)
O(3)	8*d*	1.0959(9)	0.7601(8)	0.2621(5)	0.052(5)	0.038(4)	0.014(3)	–0.012(4)	0.009(3)	0.006(3)
O(4)	4*c*	1.3110(8)	2–x	⅛	0.036(4)	U_{11}	0.08(1)	0.008(5)	–0.002(6)	U_{13}

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References

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