Crystal structure of diaquabis(μ_2-oxalato)-(μ_6-adipato)disamarium(III), $Sm_2(H_2O)_2(C_2O_4)_2(C_6H_8O_4)$

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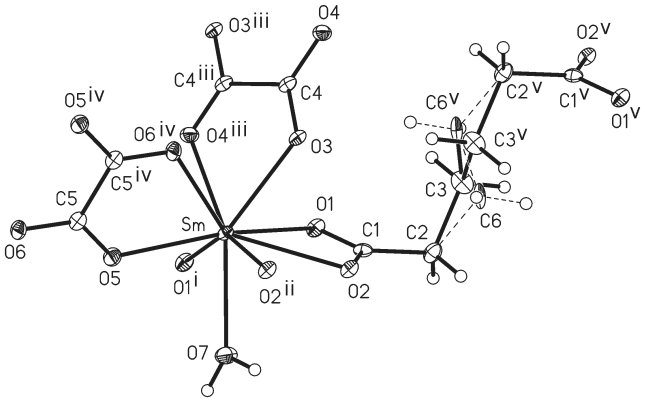

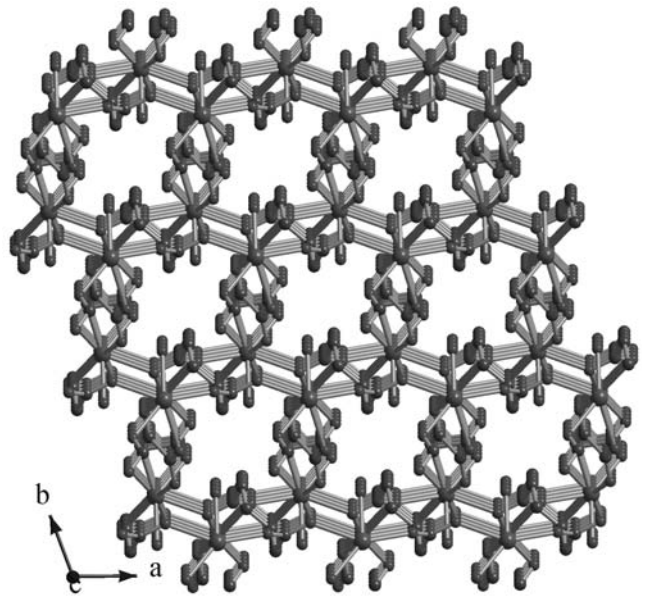

Abstract

$C_5H_6O_7Sm$, triclinic, $P\bar{1}$ (no. 2), a = 6.855(1) Å, b = 7.006(1) Å, c = 9.042(1) Å, α = 104.455(2)°, β = 108.541(2)°, γ = 104.139(2)°, V = 373.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.069, T = 295 K.

Source of material

A mixture of $SmCl_3 \cdot 6H_2O$ (1.00 mmol, 0.36 g), oxalic acid (0.50 mmol, 0.05 g), adipic acid (0.50 mmol, 0.07 g), NaOH (2.00 mmol, 0.08 g) and H_2O (10.0 ml) was heated in a 23 ml Teflon-lined stainless steel reactor at 443 K for 48 h. A small amount of colorless column-like crystals was filtered and washed with water and acetone.

Discussion

Being isostructural with $Gd_2(H_2O)_2(C_2O_4)_2(C_6H_8O_4)$ and $La_2(H_2O)_2(C_2O_4)_2(C_6H_8O_4)$ [1,2], the asymmetric unit of the title compound consists of one Sm(III) cation, a half of adipate anion, two halfs of oxalate anions and one aqua ligand (figure, top). The Sm atom is coordinated by nine oxygen atoms, of which four oxygen are from three adipate anions, four from two oxalate anions and one from a water molecule, forming a SmO_9 polyhedron with a distorted tricapped trigonal-prismatic shape. The bond lengthes of Sm—$O_{adipate}$ vary in the range of 2.459(3) - 2.603(4) Å (average 2.493 Å), which is nearly identical to the values of 2.444(5) - 2.622(5) Å (average 2.490 Å) observed in $Sm_2(C_6H_8O_4)_3(H_2O)_{5.5}$ [3]. The bond lengthes of Sm—$O_{oxalate}$ are 2.389(4) Å and 2.545(4) Å, which are usual for lanthanide oxalates complexes [4]. In the title complex, the adipate anions are located on a centre of symmetry and atom C3 is positionally disordered (C3 and C6 sites; occupancies 0.64/0.36). The adipate ligands act in a η^2,μ_3-η^2,μ_3-chelating-bridging octadentate coordination modes and link the SmO_9 polyhedra into layers parallel to (010), in which the adjacent Sm···Sm distances are 4.251(7) Å and 4.287(4) Å. In the title complex, two symmetry indepedent oxalate ions are also located on centers of inversion and act as double bidentate (tetradentate) ligands in a zigzag chain along [001]. Through the oxalate and adipate ligand bridging interactions, the Sm atoms build up a three-dimensional open framework with the channels propagating in [001] (figure, bottom). The aqua ligands donate hydrogen atoms to oxalate oxygen atoms O3 and O6 to form hydrogen bonds (d(O···O) = 2.808 Å, 2.877 Å).

Table 1. Data collection and handling.

Crystal:	colorless column, size 0.071 × 0.077 × 0.125 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	78.74 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEXII CCD, φ/ω
$2\theta_{max}$:	50.48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1923, 1327
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1239
$N(param)_{refined}$:	128
Programs:	SHELXS-97, SHELXL-97, SHELXTL [5]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(3A)	2*i*	0.64	0.4851	0.6595	1.1243	0.031
H(3B)	2*i*	0.64	0.2807	0.4939	0.9661	0.031
H(6A)	2*i*	0.36	0.6850	0.5527	0.9603	0.028
H(6B)	2*i*	0.36	0.6729	0.6825	1.1244	0.028
H(2A)	2*i*		0.4038	0.8306	0.9372	0.025
H(2B)	2*i*		0.6226	0.7949	0.9425	0.025
H(7A)	2*i*		0.4118	0.7402	0.3727	0.037
H(7B)	2*i*		0.2310	0.7785	0.2920	0.037

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(3)	2*i*	0.64(2)	0.436(2)	0.576(2)	1.008(1)	0.030(8)	0.032(7)	0.026(6)	0.019(5)	0.017(5)	0.013(5)
C(6)	2*i*	0.36	0.587(3)	0.595(3)	1.008(2)	0.02(1)	0.04(1)	0.010(8)	0.009(8)	–0.001(7)	0.004(7)
C(1)	2*i*		0.3618(9)	0.6101(8)	0.7252(7)	0.021(3)	0.011(3)	0.019(3)	0.008(2)	0.011(3)	0.007(2)
C(2)	2*i*		0.467(1)	0.7240(9)	0.9090(7)	0.019(3)	0.020(3)	0.019(3)	0.003(2)	0.010(3)	0.001(2)
C(4)	2*i*		0.1221(8)	0.0197(8)	0.5559(7)	0.016(3)	0.011(2)	0.016(3)	0.003(2)	0.010(2)	0.000(2)
C(5)	2*i*		–0.0441(9)	0.0760(9)	–0.0383(7)	0.014(3)	0.018(3)	0.017(3)	0.005(2)	0.009(2)	0.003(2)
Sm	2*i*		0.15510(4)	0.34703(4)	0.36093(3)	0.0133(2)	0.0101(2)	0.0139(2)	0.0025(1)	0.0068(1)	0.0020(1)
O(1)	2*i*		0.1535(6)	0.5228(6)	0.6503(5)	0.016(2)	0.016(2)	0.020(2)	0.006(2)	0.008(2)	0.002(2)
O(2)	2*i*		0.4770(6)	0.5950(6)	0.6404(5)	0.015(2)	0.015(2)	0.017(2)	0.003(2)	0.010(2)	0.002(2)
O(3)	2*i*		0.2668(6)	0.1526(6)	0.5391(5)	0.014(2)	0.012(2)	0.019(2)	0.001(2)	0.005(2)	0.003(2)
O(4)	2*i*		0.1540(6)	–0.0834(6)	0.6485(5)	0.018(2)	0.018(2)	0.023(2)	0.004(2)	0.008(2)	0.010(2)
O(5)	2*i*		–0.0133(7)	0.2505(6)	0.0648(5)	0.025(2)	0.016(2)	0.017(2)	0.008(2)	0.008(2)	0.003(2)
O(6)	2*i*		–0.1331(7)	0.0143(6)	–0.1935(5)	0.026(2)	0.017(2)	0.012(2)	0.007(2)	0.005(2)	0.001(2)
O(7)	2*i*		0.2720(7)	0.6902(6)	0.3326(6)	0.020(2)	0.020(2)	0.034(3)	0.006(2)	0.009(2)	0.013(2)

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