metal-organic compounds

Poly[tetraaquadi-μ_4-oxalato-potassium-ytterbium(III)]

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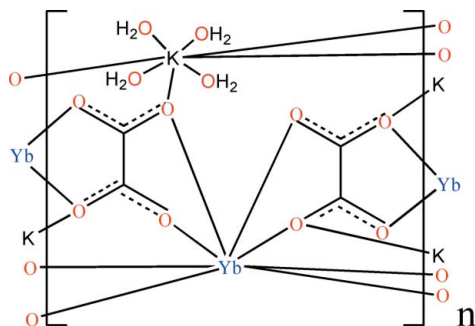Correspondence e-mail: gmli_2000@163.com

In the title compound, $[\text{KYb}(C_2O_4)_2(H_2O)_4]_n$, the Yb^{III} ion lies on a site of $\bar{4}$ symmetry in a dodecahedral environment defined by eight O atoms from four oxalate ligands. The K atom lies on a different $\bar{4}$ axis and is coordinated by four O atoms from four oxalate ligands and four water O atoms. The oxalate ligand has an inversion center at the mid-point of the C—C bond. The metal ions are linked by the oxalate ligands into a three-dimensional framework. O—H···O hydrogen bonding is present in the crystal structure.

Related literature

For related structures, see: Camara et al. (2003); Zhang et al. (2009).

Experimental

Crystal data

$[\text{KYb}(C_2O_4)_2(H_2O)_4]$	c = 8.9142 (18) Å
M_r = 460.24	V = 1148.4 (3) Å^3
Tetragonal, $I4_1/a$	Z = 4
a = 11.3502 (16) Å	Mo $K\alpha$ radiation

μ = 8.57 mm^{-1}	$0.08 \times 0.07 \times 0.07$ mm
T = 293 K	

Data collection

Rigaku R-AXIS RAPID diffractometer	5407 measured reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	648 independent reflections
T_{min} = 0.562, T_{max} = 0.606	585 reflections with $I > 2\sigma(I)$
	R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017	3 restraints
$wR(F^2)$ = 0.039	H-atom parameters constrained
S = 0.94	$\Delta\rho_{max}$ = 0.55 e Å^{-3}
648 reflections	$\Delta\rho_{min}$ = -0.43 e Å^{-3}
41 parameters	

Table 1
Selected bond lengths (Å).

K1—O1	2.8402 (19)	Yb1—O1	2.3629 (19)
K1—O3	2.871 (3)	Yb1—O2^i	2.304 (2)

Symmetry code: (i) $-x, -y+1, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O3^{ii}	0.85	2.08	2.899 (3)	163
O3—H2···O2^{iii}	0.85	2.06	2.837 (3)	152

Symmetry codes: (ii) $-y+\frac{5}{4}, x+\frac{3}{4}, z-\frac{1}{4}$; (iii) $-y+\frac{3}{4}, x+\frac{3}{4}, -z+\frac{3}{4}$.

Data collection: RAPID-AUTO (Rigaku, 1998); cell refinement: RAPID-AUTO; data reduction: CrystalStructure (Rigaku/MSC, 2002); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg, 1999); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2482).

References

Brandenburg, K. (1999). DIAMOND. Crystal Impact GbR, Bonn, Germany.
Camara, M., Daiguebonne, C., Boubekeur, K., Roisnel, T., Gérault, Y., Baux, C., Dret, F. L. & Guillou, O. (2003). Compt. Rend. Chim. 6, 405–415.
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Zhang, X.-J., Xing, Y.-H., Wang, C.-G., Han, J., Li, J., Ge, M.-F., Zeng, X.-Q. & Niu, S.-Y. (2009). Inorg. Chim. Acta, 362, 1058–1064.

supplementary materials

Poly[tetraaquadi-*μ*₄-oxalato-potassiumytterbium(III)]

F.-M. Zhang, G.-F. Hou, P.-F. Yan and G.-M. Li

Comment

Lanthanide complexes with spectroscopic and magnetic properties are currently of considerable interest. Oxalate ligand can serve as bridging ligand in high dimensional frameworks (Camara *et al.*, 2003; Zhang *et al.*, 2009). In this paper, we present the synthesis and crystal structure of the title compound.

The title compound was obtained as a byproduct by the decomposition of 1,3,5-triazine-2,4,6-tricarboxylate ligand. In the title compound, $[YbK(C_2O_4)_2(H_2O)_4]_n$, the eight-coordinated Yb^{III} ion lies on a $\overline{4}$ site symmetry in a distorted dodecahedral geometry defined by eight O atoms from four oxalate ligands. The eight-coordinated K ion is also locate on another site of $\overline{4}$ symmetry in a distorted dodecahedral geometry defined by four O atoms from oxalate ligands and four O atoms from water molecules (Fig. 1, Table 1).

In the crystal, each oxalate ligand links two Yb and two K atoms, forming a three-dimensional framework (Fig. 2). O—H···O hydrogen bonds are present (Table 2).

Experimental

The title compound was obtained as a byproduct caused by the decomposition of 1,3,5-triazine-2,4,6-tricarboxylate ligand. $Yb(NO_3)_3.6H_2O$ (14.01 mg, 0.03 mmol) and potassium salt of 1,3,5-triazine-2,4,6-tricarboxylate (9.8 mg, 0.03 mmol) were dissolved in 15 ml water. After stirring at room temperature for 0.5 h, the solution was allowed to stand for about one week. Colorless block crystals were obtained in 36% yield.

Refinement

Water H atoms were initially located in a differece Fourier map and were treated as riding atoms, with O—H = 0.85 Å and with $U_{iso}(H) = 1.5U_{eq}(O)$.

Figures

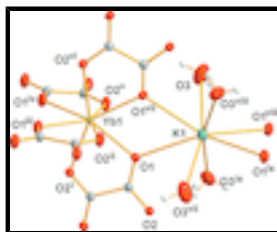Fig. 1. The asymmetric unit of the title compound, showing displacement ellipsoids at the 50% probability level. [Symmetry codes: (i) -*x*, 1-*y*, -*z*; (ii) 1/4-*y*, 3/4+*x*, -1/4+*z*; (iii) -3/4+*y*, 3/4-*x*, -1/4-*z*; (iv) 3/4-*y*, 3/4+*x*, -1/4-*z*; (v) -1/4+*y*, 3/4-*x*, -1/4+*z*; (vi) *x*, 1/2+*y*, *z*; (vii) -*x*, 3/2-*y*, *z*; (viii) 3/4-*y*, 3/4+*x*, 3/4-*z*; (ix) -3/4+*y*, 3/4-*x*, 3/4-*z*.]
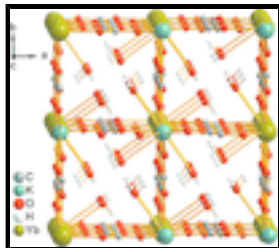
Fig. 2. A packing view along [001], showing the three-dimensional framework.

Poly[tetraaquadi-μ₄-oxalato-potassiumytterbium(III)]

Crystal data

[KYb(C$_2$O$_4$)$_2$(H$_2$O)$_4$]	D_x = 2.662 Mg m^{-3}
M_r = 460.24	Mo $K\alpha$ radiation, λ = 0.71073 Å
Tetragonal, $I4_1/a$	Cell parameters from 4696 reflections
Hall symbol: -I 4ad	θ = 3.6–27.4°
a = 11.3502 (16) Å	μ = 8.57 mm^{-1}
c = 8.9142 (18) Å	T = 293 K
V = 1148.4 (3) Å^3	Block, colorless
Z = 4	0.08 × 0.07 × 0.07 mm
$F(000)$ = 868	

Data collection

Rigaku R-AXIS RAPID diffractometer	648 independent reflections
Radiation source: fine-focus sealed tube	585 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.051
ω scan	θ_{max} = 27.4°, θ_{min} = 3.6°
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$h = -14 \rightarrow 14$
T_{min} = 0.562, T_{max} = 0.606	$k = -13 \rightarrow 14$
5407 measured reflections	$l = -10 \rightarrow 11$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.017	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.039	H-atom parameters constrained
S = 0.94	$w = 1/[\sigma^2(F_o^2) + (0.P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
648 reflections	$(\Delta/\sigma)_{max} < 0.001$
41 parameters	$\Delta\rho_{max}$ = 0.55 e Å^{-3}
3 restraints	$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
C1	0.0038 (3)	0.5244 (3)	0.0802 (3)	0.0159 (6)
K1	0.0000	0.7500	0.3750	0.0296 (4)
O1	0.0084 (2)	0.63272 (19)	0.0937 (2)	0.0209 (5)
O2	0.0045 (2)	0.44765 (18)	0.1836 (2)	0.0224 (5)
O3	0.2057 (3)	0.8934 (3)	0.3322 (3)	0.0509 (8)
H1	0.2363	0.9125	0.2486	0.076*
H2	0.2520	0.8473	0.3787	0.076*
Yb1	0.0000	0.7500	−0.1250	0.01065 (10)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0188 (16)	0.0154 (15)	0.0136 (12)	0.0004 (12)	−0.0010 (11)	0.0004 (11)
K1	0.0345 (6)	0.0345 (6)	0.0198 (7)	0.000	0.000	0.000
O1	0.0346 (14)	0.0125 (11)	0.0156 (9)	−0.0003 (10)	−0.0013 (8)	−0.0017 (8)
O2	0.0399 (14)	0.0122 (11)	0.0150 (9)	−0.0010 (10)	−0.0009 (9)	−0.0006 (8)
O3	0.0374 (17)	0.075 (2)	0.0402 (13)	0.0106 (16)	−0.0038 (12)	0.0195 (15)
Yb1	0.01004 (12)	0.01004 (12)	0.01186 (15)	0.000	0.000	0.000

Geometric parameters (Å, °)

C1—O1	1.236 (4)	Yb1—O1	2.3629 (19)
C1—O2	1.269 (4)	Yb1—O2^i	2.304 (2)
C1—C1^i	1.536 (5)	O3—H1	0.8500
K1—O1	2.8402 (19)	O3—H2	0.8499
K1—O3	2.871 (3)		
O1—C1—O2	127.7 (3)	O2^i—Yb1—O1^v	137.29 (7)
O1—C1—C1^i	116.9 (3)	O2^{ii}—Yb1—O1^{vi}	137.29 (7)
O2—C1—C1^i	115.4 (3)	O2^{iii}—Yb1—O1^{vi}	82.21 (8)
O1—K1—O3	96.95 (7)	O2^{iv}—Yb1—O1^{vi}	68.87 (7)
C1—O1—Yb1	118.53 (17)	O2^i—Yb1—O1^{vi}	76.19 (8)
C1—O1—K1	123.44 (17)	O1^v—Yb1—O1^{vi}	132.92 (6)
Yb1—O1—K1	117.59 (8)	O2^{ii}—Yb1—O1	76.19 (8)
C1—O2—Yb1^i	120.27 (18)	O2^{iii}—Yb1—O1	137.29 (7)
K1—O3—H1	126.4	O2^{iv}—Yb1—O1	82.21 (8)
K1—O3—H2	95.1	O2^i—Yb1—O1	68.87 (7)
H1—O3—H2	109.3	O1^v—Yb1—O1	68.77 (10)
O2^{ii}—Yb1—O2^{iii}	92.95 (2)	O1^{vi}—Yb1—O1	132.92 (6)
O2^{ii}—Yb1—O2^{iv}	153.79 (9)	O2^{ii}—Yb1—O1^{vii}	68.87 (7)
O2^{iii}—Yb1—O2^{iv}	92.95 (2)	O2^{iii}—Yb1—O1^{vii}	76.19 (8)
O2^{ii}—Yb1—O2^i	92.95 (2)	O2^{iv}—Yb1—O1^{vii}	137.29 (7)

O2^{iii}—Yb1—O2^{i}	153.79 (9)	O2^{i}—Yb1—O1^{vii}	82.21 (8)
O2^{iv}—Yb1—O2^{i}	92.95 (2)	O1^{v}—Yb1—O1^{vii}	132.92 (6)
O2^{ii}—Yb1—O1^{v}	82.21 (8)	O1^{vi}—Yb1—O1^{vii}	68.77 (10)
O2^{iii}—Yb1—O1^{v}	68.87 (7)	O1—Yb1—O1^{vii}	132.92 (6)
O2^{iv}—Yb1—O1^{v}	76.19 (8)		

Symmetry codes: (i) −x, −y+1, −z; (ii) y−1/4, −x+3/4, z−1/4; (iii) x, y+1/2, −z; (iv) −y+1/4, x+3/4, z−1/4; (v) −x, −y+3/2, z; (vi) y−3/4, −x+3/4, −z−1/4; (vii) −y+3/4, x+3/4, −z−1/4.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H1⋯O3^{viii}	0.85	2.08	2.899 (3)	163
O3—H2⋯O2^{ix}	0.85	2.06	2.837 (3)	152

Symmetry codes: (viii) −y+5/4, x+3/4, z−1/4; (ix) −y+3/4, x+3/4, −z+3/4.

Fig. 1

Fig. 2