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

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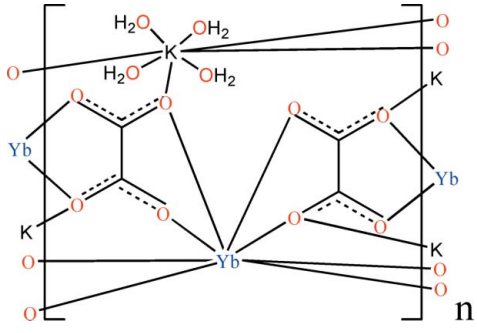
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.017; wR factor = 0.039; data-to-parameter ratio = 15.8.

In the title compound, $[KYb(C_2O_4)_2(H_2O)_4]_n$, the Yb^{III} ion lies on a site of $\overline{4}$ symmetry in a dodecahedral environment defined by eight O atoms from four oxalate ligands. The K atom lies on a different $\overline{4}$ 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

$[KYb(C_2O_4)_2(H_2O)_4]$
M_r = 460.24
Tetragonal, $I4_1/a$
a = 11.3502 (16) Å
c = 8.9142 (18) Å
V = 1148.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.57 mm^{-1}
T = 293 K
0.08 × 0.07 × 0.07 mm

Data collection

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

Refinement

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

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*.

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

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supporting information

Acta Cryst. (2011). E**67**, m1719 [https://doi.org/10.1107/S1600536811046022]

Poly[tetraaquadi-μ_4-oxalato-potassiumytterbium(III)]

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S1. 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 $\bar{4}$ 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 $\bar{4}$ 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).

S2. 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.

S3. 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 $U_{iso}(H) = 1.5U_{eq}(O)$.

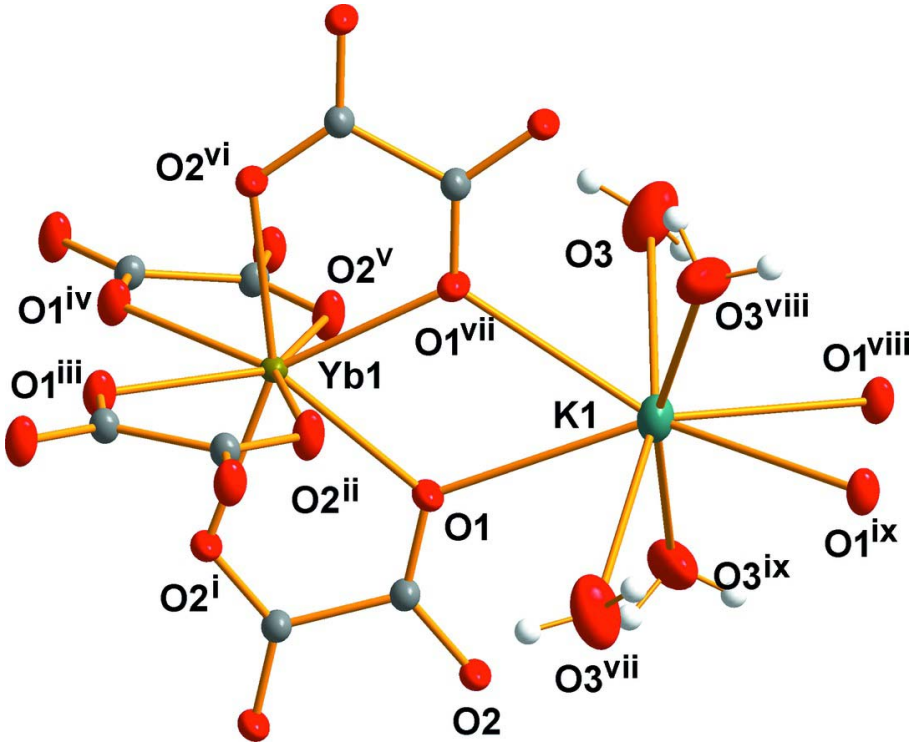

Figure 1

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*.]

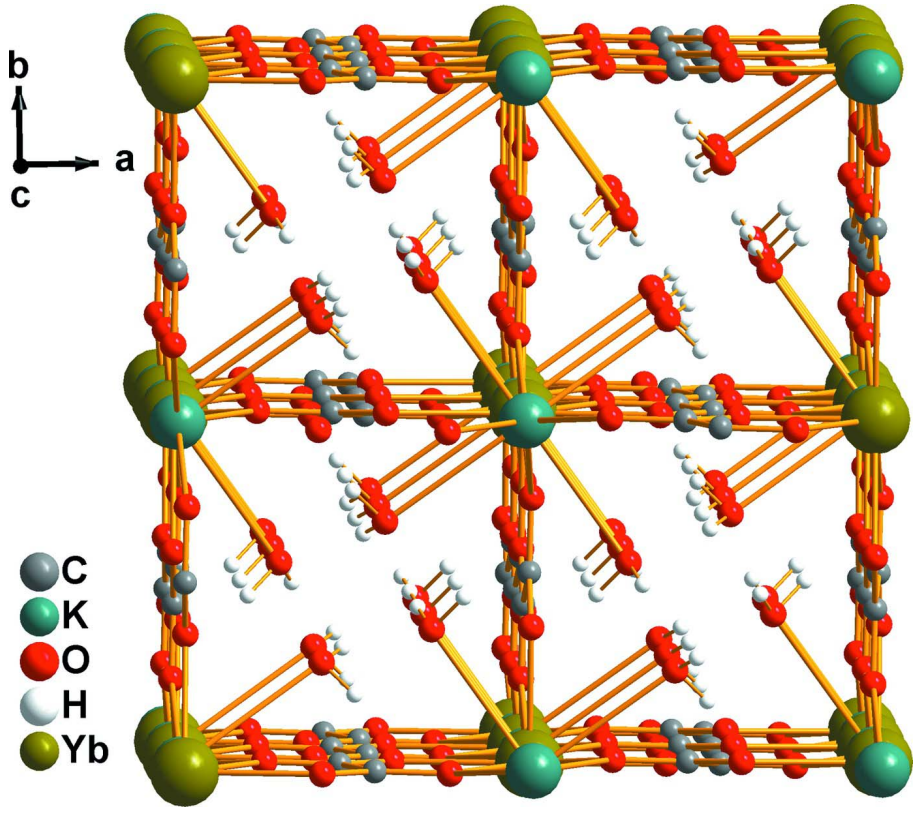

Figure 2

A packing view along [001], showing the three-dimensional framework.

Poly[tetraaquadi-μ_4-oxalato-potassiumytterbium(III)]

Crystal data

$[KYb(C_2O_4)_2(H_2O)_4]$
$M_r = 460.24$
Tetragonal, $I4_1/a$
Hall symbol: -I 4ad
$a = 11.3502\ (16)$ Å
$c = 8.9142\ (18)$ Å
$V = 1148.4\ (3)$ Å^3
$Z = 4$
$F(000) = 868$
$D_x = 2.662$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4696 reflections
$\theta = 3.6–27.4°$
$\mu = 8.57$ mm^{-1}
$T = 293$ K
Block, colorless
0.08 × 0.07 × 0.07 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.562$, $T_{max} = 0.606$
5407 measured reflections
648 independent reflections
585 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.6°$
$h = -14\rightarrow14$
$k = -13\rightarrow14$
$l = -10\rightarrow11$

Refinement

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	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 (Å²)

	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, º)

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, º)

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) $-y+3/4$, $x+3/4$, $-z+3/4$.