

Poly[tetraquadi- μ_4 -oxalato-lutetium(III)-potassium]

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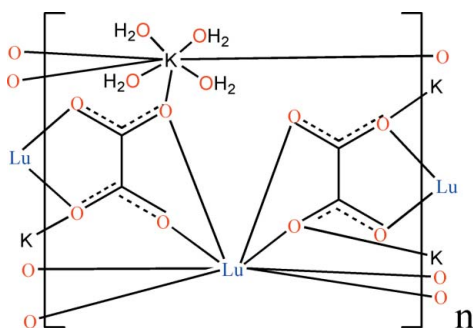
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.014; wR factor = 0.035; data-to-parameter ratio = 16.0.

In the title compound, $[\text{KLu}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_4]_n$, the Lu^{III} ion lies on a site of $\bar{4}$ symmetry in a dodecahedron defined by eight O atoms from four oxalate ligands. The K atom lies on another site of the same symmetry and is coordinated by four oxalate O atoms and four O water atoms. The mid-point of the C—C bond of the oxalate group lies on an inversion center. In the packing structure, each oxalate ligand links two Lu(III) and two K atoms, forming a three-dimensional open framework with channels running along $[001]$. Intermolecular O—H...O hydrogen bonds occur.

Related literature

For background to oxalate anions as bridging ligands in high dimensional frameworks and for a similar structure, see: Camara *et al.* (2003); Zhang *et al.* (2009).



Experimental

Crystal data

$[\text{KLu}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_4]$
 $M_r = 462.17$
Tetragonal, $I4_1/a$
 $a = 11.3337$ (16) Å
 $c = 8.9121$ (18) Å
 $V = 1144.8$ (3) Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 9.05$ mm⁻¹
 $T = 293$ K
 $0.08 \times 0.08 \times 0.06$ mm

Data collection

Rigaku R-Axis RAPID diffractometer
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\text{min}} = 0.546$, $T_{\text{max}} = 0.604$

5421 measured reflections
655 independent reflections
594 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.014$
 $wR(F^2) = 0.035$
 $S = 1.13$
655 reflections

41 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.64$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O3}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.85	2.06	2.836 (4)	151
$\text{O3}-\text{H1}\cdots\text{O3}^{\text{ii}}$	0.85	2.06	2.891 (3)	166

Symmetry codes: (i) $-x + 1, -y + \frac{1}{2}, z$; (ii) $y + \frac{1}{4}, -x + \frac{1}{4}, z + \frac{1}{4}$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: NG5233).

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supporting information

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Poly[tetraquadi- μ_4 -oxalato-lutetium(III)potassium]**Feng-Ming Zhang, Tao-Zhu Sun, Guang-Feng Hou, Peng-Fei Yan and Guang-Ming Li****S1. Comment**

Lanthanide complexes with spectroscopic and magnetic properties are currently of considerable interest; the oxalate ligand can serve as bridging ligand in high dimensional frameworks (Camara *et al.*, 2003; Zhang *et al.*, 2009). In this paper, we present here 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, $[\text{LuK}(\text{C}_2\text{O}_4)_2(\text{H}_2\text{O})_4]_n$, the eight-coordinated lutetium(III) ion lies on a 4-fold inverse axis in a distorted dodecahedron defined by eight oxygen atoms from four oxalate ligands, and while the eight-coordinated potassium is also locate on a 4-fold inverseaxis in a distorted dodecahedron defined by four oxygen atoms from oxalate ligands and four oxygen atoms from water molecules (Fig. 1, Table 1).

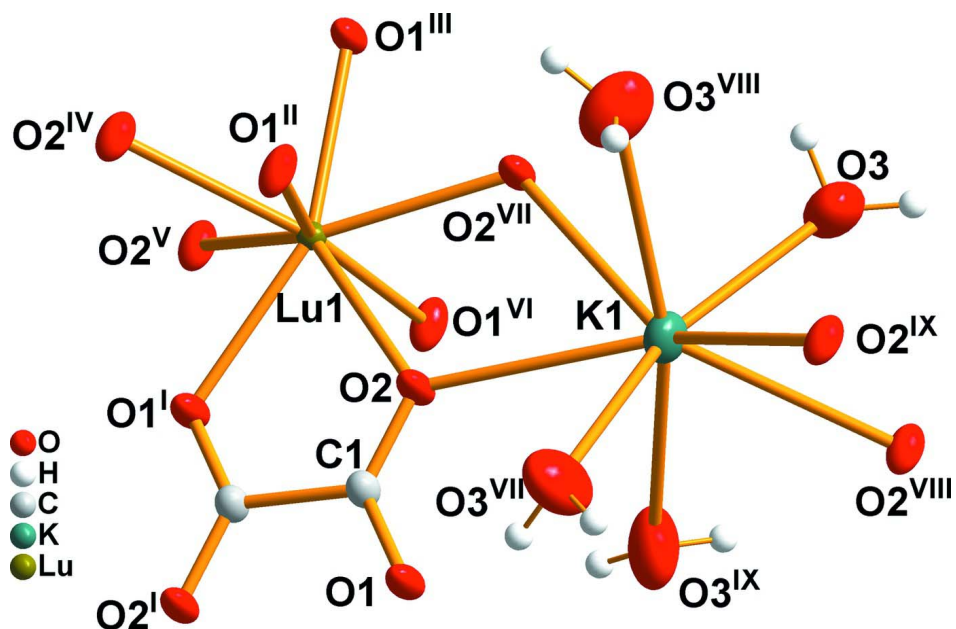
In the packing structure, each oxalate ligand links two Lu(III) and two K atoms to form a three-dimensional open framework with channels running along [001] (Fig. 2).

S2. Experimental

The title compound was obtained as a byproduct caused by the decomposition of 1,3,5-triazine-2,4,6-tricarboxylate ligand. $\text{Lu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (14.07 mg, 0.03 mmol) and the 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 40% yield.

S3. Refinement

Water H atoms were initially located in a differece Fourier map, but they were treated as riding on their parent atoms with $\text{O}-\text{H} = 0.85 \text{ \AA}$, and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level for non-H atoms, Symmetry codes: (I) $1 - x, 1 - y, -z$; (II) $1.25 - y, -1/4 + x, -1/4 + z$; (III) $x, -1/2 + y, -z$; (IV) $0.25 + Y, 0.75 - X, -0.25 - Z$; (V) $0.75 - y, -1/4 + x, -0.25 - z$; (VI) $-1/4 + y, 0.75 - x, -1/4 + z$; (VII) $1 - x, 0.5 - y, z$; (VIII) $0.75 - y, -1/4 + x, 0.75 - z$; (IX) $1/4 + y, 0.75 - x, 0.75 - z$.

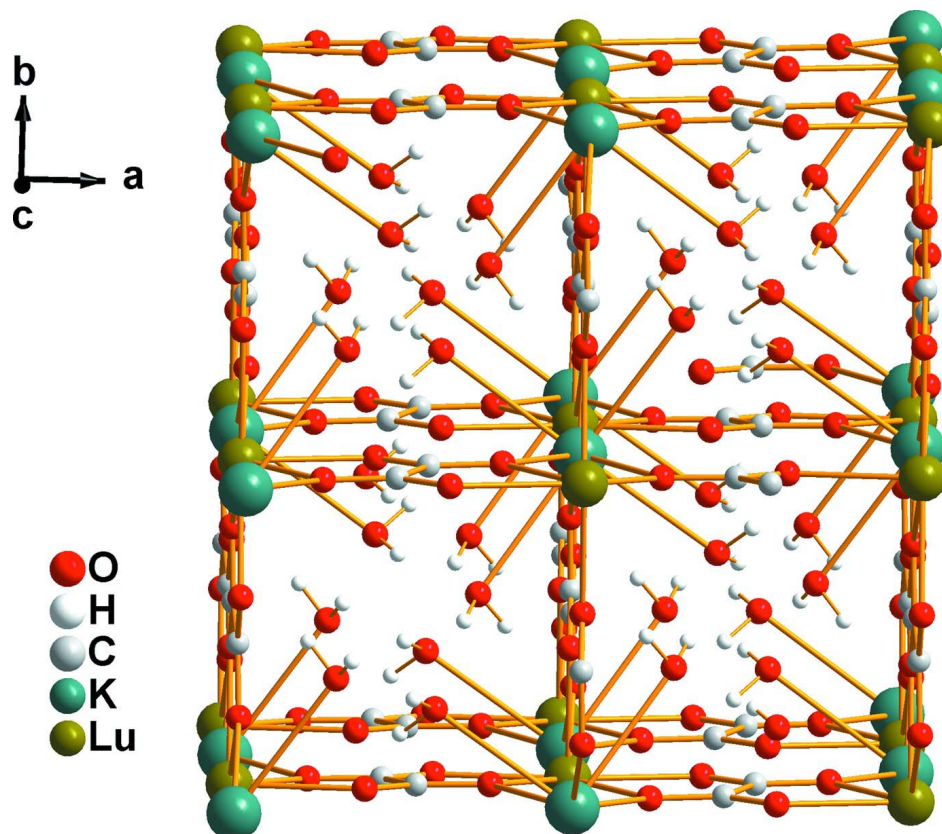


Figure 2

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

Poly[tetraaquadimutetium(III)potassium]

Crystal data

[KLu(C₂O₄)₂(H₂O)₄]

$M_r = 462.17$

Tetragonal, $I4_1/a$

Hall symbol: -I 4ad

$a = 11.3337 (16) \text{ \AA}$

$c = 8.9121 (18) \text{ \AA}$

$V = 1144.8 (3) \text{ \AA}^3$

$Z = 4$

$F(000) = 872$

$D_x = 2.682 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 4696 reflections

$\theta = 3.1\text{--}27.5^\circ$

$\mu = 9.05 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colorless

$0.08 \times 0.08 \times 0.06 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scan

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.546$, $T_{\max} = 0.604$

5421 measured reflections

655 independent reflections

594 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.6^\circ$

$h = -13 \rightarrow 14$

$k = -14 \rightarrow 14$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.014$	H-atom parameters constrained
$wR(F^2) = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.0077P)^2 + 3.796P]$
$S = 1.13$	where $P = (F_o^2 + 2F_c^2)/3$
655 reflections	$(\Delta/\sigma)_{\max} = 0.001$
41 parameters	$\Delta\rho_{\max} = 0.64 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O3	0.3564 (3)	0.0435 (3)	0.4179 (4)	0.0588 (9)
H2	0.3981	-0.0060	0.3698	0.088*
H1	0.3356	0.0106	0.4995	0.088*
O2	0.5074 (2)	0.36714 (18)	0.0938 (2)	0.0216 (5)
O1	0.5037 (2)	0.55246 (18)	0.1842 (2)	0.0229 (5)
C1	0.5028 (3)	0.4761 (3)	0.0803 (3)	0.0167 (6)
K1	0.5000	0.2500	0.3750	0.0300 (3)
Lu1	0.5000	0.2500	-0.1250	0.01106 (8)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.081 (2)	0.0483 (19)	0.0472 (19)	0.0081 (17)	0.0202 (18)	-0.0078 (15)
O2	0.0371 (13)	0.0112 (9)	0.0164 (11)	-0.0010 (8)	-0.0012 (9)	0.0000 (8)
O1	0.0411 (13)	0.0127 (10)	0.0149 (10)	0.0009 (9)	-0.0012 (10)	-0.0011 (8)
C1	0.0198 (14)	0.0159 (14)	0.0143 (14)	-0.0008 (11)	-0.0012 (11)	0.0010 (11)
K1	0.0351 (5)	0.0351 (5)	0.0199 (7)	0.000	0.000	0.000
Lu1	0.01045 (9)	0.01045 (9)	0.01229 (13)	0.000	0.000	0.000

Geometric parameters (\AA , $^\circ$)

O3—K1	2.876 (3)	K1—O2 ^{iv}	2.837 (2)
O3—H2	0.8499	K1—O3 ⁱⁱ	2.876 (3)
O3—H1	0.8500	K1—O3 ⁱⁱⁱ	2.876 (3)
O2—C1	1.242 (4)	K1—O3 ^{iv}	2.876 (3)

O2—Lu1	2.361 (2)	Lu1—O1 ^v	2.300 (2)
O2—K1	2.837 (2)	Lu1—O1 ^{vi}	2.300 (2)
O1—C1	1.267 (4)	Lu1—O1 ^{vii}	2.300 (2)
O1—Lu1 ⁱ	2.300 (2)	Lu1—O1 ⁱ	2.300 (2)
C1—C1 ⁱ	1.531 (6)	Lu1—O2 ^{viii}	2.361 (2)
K1—O2 ⁱⁱ	2.837 (2)	Lu1—O2 ⁱⁱ	2.361 (2)
K1—O2 ⁱⁱⁱ	2.837 (2)	Lu1—O2 ^{ix}	2.361 (2)
K1—O3—H2	98.9	O2 ^{iv} —K1—O3 ^{iv}	120.99 (8)
K1—O3—H1	128.9	O2—K1—O3 ^{iv}	97.09 (9)
H2—O3—H1	107.2	O3—K1—O3 ^{iv}	91.015 (18)
C1—O2—Lu1	118.51 (19)	O3 ⁱⁱ —K1—O3 ^{iv}	91.015 (18)
C1—O2—K1	123.38 (18)	O3 ⁱⁱⁱ —K1—O3 ^{iv}	164.71 (13)
Lu1—O2—K1	117.74 (8)	O1 ^v —Lu1—O1 ^{vi}	93.01 (2)
C1—O1—Lu1 ⁱ	119.80 (19)	O1 ^v —Lu1—O1 ^{vii}	93.01 (2)
O2—C1—O1	127.4 (3)	O1 ^{vi} —Lu1—O1 ^{vii}	153.50 (11)
O2—C1—C1 ⁱ	116.4 (3)	O1 ^v —Lu1—O1 ⁱ	153.50 (11)
O1—C1—C1 ⁱ	116.2 (3)	O1 ^{vi} —Lu1—O1 ⁱ	93.01 (2)
O2 ⁱⁱ —K1—O2 ⁱⁱⁱ	141.27 (5)	O1 ^{vii} —Lu1—O1 ⁱ	93.01 (2)
O2 ⁱⁱ —K1—O2 ^{iv}	141.27 (6)	O1 ^v —Lu1—O2 ^{viii}	81.67 (8)
O2 ⁱⁱⁱ —K1—O2 ^{iv}	55.93 (8)	O1 ^{vi} —Lu1—O2 ^{viii}	69.06 (7)
O2 ⁱⁱ —K1—O2	55.93 (8)	O1 ^{vii} —Lu1—O2 ^{viii}	137.39 (7)
O2 ⁱⁱⁱ —K1—O2	141.27 (6)	O1 ⁱ —Lu1—O2 ^{viii}	76.48 (8)
O2 ^{iv} —K1—O2	141.27 (5)	O1 ^v —Lu1—O2	137.39 (7)
O2 ⁱⁱ —K1—O3	73.75 (8)	O1 ^{vi} —Lu1—O2	81.67 (8)
O2 ⁱⁱⁱ —K1—O3	97.09 (9)	O1 ^{vii} —Lu1—O2	76.48 (8)
O2 ^{iv} —K1—O3	68.99 (8)	O1 ⁱ —Lu1—O2	69.06 (7)
O2—K1—O3	120.99 (8)	O2 ^{viii} —Lu1—O2	133.04 (6)
O2 ⁱⁱ —K1—O3 ⁱⁱ	120.99 (8)	O1 ^v —Lu1—O2 ⁱⁱ	69.06 (7)
O2 ⁱⁱⁱ —K1—O3 ⁱⁱ	68.99 (8)	O1 ^{vi} —Lu1—O2 ⁱⁱ	76.48 (8)
O2 ^{iv} —K1—O3 ⁱⁱ	97.09 (9)	O1 ^{vii} —Lu1—O2 ⁱⁱ	81.67 (8)
O2—K1—O3 ⁱⁱ	73.75 (8)	O1 ⁱ —Lu1—O2 ⁱⁱ	137.39 (7)
O3—K1—O3 ⁱⁱ	164.71 (13)	O2 ^{viii} —Lu1—O2 ⁱⁱ	133.04 (6)
O2 ⁱⁱ —K1—O3 ⁱⁱⁱ	97.09 (9)	O2—Lu1—O2 ⁱⁱ	68.60 (10)
O2 ⁱⁱⁱ —K1—O3 ⁱⁱⁱ	120.99 (8)	O1 ^v —Lu1—O2 ^{ix}	76.48 (8)
O2 ^{iv} —K1—O3 ⁱⁱⁱ	73.75 (8)	O1 ^{vi} —Lu1—O2 ^{ix}	137.39 (7)
O2—K1—O3 ⁱⁱⁱ	68.99 (8)	O1 ^{vii} —Lu1—O2 ^{ix}	69.06 (7)
O3—K1—O3 ⁱⁱⁱ	91.015 (18)	O1 ⁱ —Lu1—O2 ^{ix}	81.67 (8)
O3 ⁱⁱ —K1—O3 ⁱⁱⁱ	91.015 (18)	O2 ^{viii} —Lu1—O2 ^{ix}	68.60 (10)
O2 ⁱⁱ —K1—O3 ^{iv}	68.99 (8)	O2—Lu1—O2 ^{ix}	133.04 (6)
O2 ⁱⁱⁱ —K1—O3 ^{iv}	73.75 (8)	O2 ⁱⁱ —Lu1—O2 ^{ix}	133.04 (6)

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, -y+1/2, z$; (iii) $y+1/4, -x+3/4, -z+3/4$; (iv) $-y+3/4, x-1/4, -z+3/4$; (v) $x, y-1/2, -z$; (vi) $y-1/4, -x+3/4, z-1/4$; (vii) $-y+5/4, x-1/4, z-1/4$; (viii) $-y+3/4, x-1/4, -z-1/4$; (ix) $y+1/4, -x+3/4, -z-1/4$.

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H2 \cdots O1 ⁱⁱ	0.85	2.06	2.836 (4)	151

O3—H1...O3 ^x	0.85	2.06	2.891 (3)	166
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Symmetry codes: (ii) $-x+1, -y+1/2, z$; (x) $y+1/4, -x+1/4, z+1/4$.