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Lutetium(III) oxotellurate(IV), $Lu_2Te_4O_{11}$

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Te–O) = 0.007 Å
R factor = 0.034
wR factor = 0.074
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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Rare-earth(III) oxotellurates(IV) with the composition $M_2Te_4O_{11}$ are known for yttrium and all lanthanides except promethium and lutetium from single-crystal X-ray structure determinations. Single crystals of the last missing non-radioactive isostructural compound, $Lu_2Te_4O_{11}$, can now be obtained by modifying the common method of synthesis from the binary oxides (Lu_2O_3 and TeO_2; 1:4 molar ratio) using torch-sealed non-evacuated silica ampoules as reaction containers. The structure contains layers of edge-sharing [LuO_8] polyhedra connected by oxotellurate(IV) chains. These consist of $[TeO_3]^{2-}$ and $[Te_2O_5]^{2-}$ anions (with ψ^1-tetrahedral oxygen coordination for all central Te^{4+} cations) linked by strong secondary Te—O interactions.

Comment

$Lu_2Te_4O_{11}$ is isostructural to all other known rare-earth(III) oxotellurates(IV) of the type $M_2Te_4O_{11}$ (M = Y, La–Nd and Sm–Yb; Castro *et al.*, 1990; Weber *et al.*, 2001; Ijjaali *et al.*, 2003; Höss *et al.*, 2004; Meier & Schleid, 2004; Shen & Mao, 2004). The structure contains only one lutetium site, where Lu^{3+} is coordinated by eight O atoms in the shape of a distorted trigonal dodecahedron (Fig. 1). The [LuO_8] polyhedra [Lu—O = 2.189 (7)–2.480 (7) Å] form reticulated layers parallel to the (001) plane by sharing three edges each. The two tellurium sites are each coordinated by four (better: 3 + 1) O atoms. Three of them bond covalently [Te—O = 1.841 (7)–2.023 (4) Å] to each Te^{4+} cation and form ψ^1-tetrahedra together with the lone pair. The fourth O atom belongs to the primary coordination sphere of the other Te^{4+} cation [Te1—O4 = 2.567 (7) Å and Te2—O1 = 2.312 (7) Å], so that the

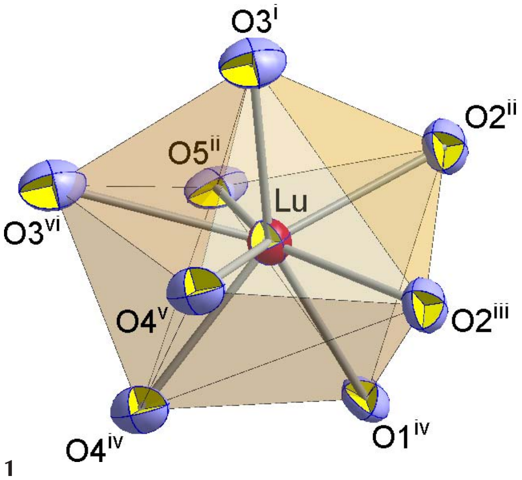

Figure 1
The distorted trigonal dodecahedral coordination of the Lu^{3+} cation. Displacement ellipsoids are drawn at the 90% probability level. Symmetry codes are as in Table 1.

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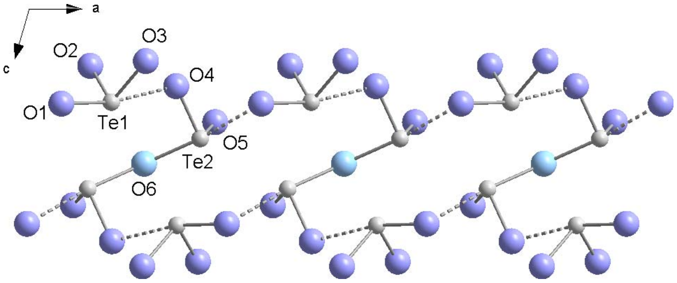

Figure 2
Double chains built up by oxotellurate(IV) units, $[TeO_3]^{2-}$ and $[Te_2O_5]^{2-}$, in the direction of the *a* axis.

alternating oxotellurate units build up zigzag chains in the direction of the *a* axis (Fig. 2). These chains are located above and below the reticulated layers of condensed $[LuO_8]$ polyhedra with the Te atoms lying within the meshes (Fig. 3). Through Te2—O6—Te2 bridges [Te2—O6 = 2.023 (4) Å and Te2—O6—Te2 = 138.9 (5)°], the tellurate units, $[Te_2O_5]^{2-}$, connect these layers and build up a three-dimensional structure (Fig. 4). Between the layers, there is still enough space left to accommodate the lone pairs of the Te^{4+} cations. The motifs of mutual adjunction (Hoppe, 1980) and the coordination numbers (CN) are shown in Table 2. With the strong secondary Te—O interactions disregarded, the structure of $Lu_2Te_4O_{11}$ could be formulated as $Lu_2[TeO_3]_2[Te_2O_5]$.

Experimental

Single crystals of $Lu_2Te_4O_{11}$ were grown in torch-sealed but non-evacuated silica ampoules by fusing a mixture of Lu_2O_3 and TeO_2 in a 1:4 molar ratio at 1123 K for 10 d. CsCl was used as a flux to improve single-crystal growth. The ampoules were cooled to 923 K over two days and finally to room temperature. The common method of synthesis (Weber *et al.*, 2001) using evacuated silica ampoules always leads to a two-phase product consisting of the formal dismutation compounds $Lu_2Te_3O_9$ (Meier & Schleid, 2002) and $Lu_2Te_5O_{13}$ (Meier & Schleid, 2005). The slightly higher pressure (~4 bar at 1123 K) in the sealed air-containing ampoules seems to promote the formation of $Lu_2Te_4O_{11}$, where Lu^{3+} has an unusually large coordination number, with eight O atoms at more or less equal distances.

Crystal data

$Lu_2Te_4O_{11}$
$M_r = 1036.34$
Monoclinic, $C2/c$
$a = 12.2953$ (8) Å
$b = 5.0596$ (3) Å
$c = 15.9134$ (9) Å
$\beta = 106.202$ (7)°
$V = 950.64$ (10) Å^3
$Z = 4$
$D_x = 7.241$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3912 reflections
$\theta = 1.0$–27.5°
$\mu = 32.74$ mm^{-1}
$T = 293$ (2) K
Spheroid, colourless
0.04 × 0.03 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 1998)
$T_{min} = 0.320$, $T_{max} = 0.539$
10819 measured reflections
1108 independent reflections
791 reflections with $I > 2\sigma(I)$
$R_{int} = 0.096$
$\theta_{max} = 27.6$°
$h = -16 \rightarrow 16$
$k = -6 \rightarrow 6$
$l = -20 \rightarrow 20$

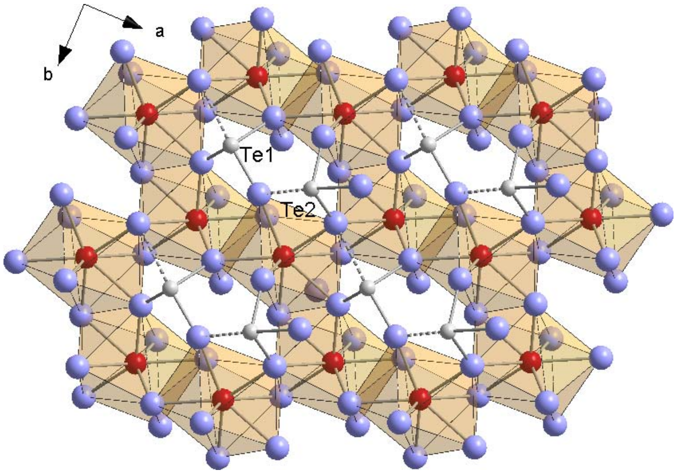

Figure 3
Reticulated layers of triple edge-shared $[LuO_8]$ polyhedra parallel to the (001) plane. The Te^{4+} cations lie above and below the meshes.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.074$
$S = 1.00$
1108 reflections
79 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0272P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.86$ e Å^{-3}
$\Delta\rho_{min} = -1.38$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00360 (11)

Table 1
Selected geometric parameters (Å, °).

Lu—O3^{i}	2.189 (7)	Te1—O3	1.871 (7)
Lu—O5^{ii}	2.218 (7)	Te1—O2	1.885 (7)
Lu—O2^{iii}	2.243 (7)	Te1—O1	1.892 (7)
Lu—O2^{ii}	2.327 (7)	Te1—O4^{vii}	2.567 (7)
Lu—O4^{iv}	2.365 (7)	Te2—O5^{iv}	1.841 (7)
Lu—O1^{iv}	2.416 (7)	Te2—O4^{iv}	1.912 (7)
Lu—O4^{v}	2.471 (7)	Te2—O6	2.023 (4)
Lu—O3^{vi}	2.480 (7)	Te2—O1^{iv}	2.312 (7)
Te2^{vi}—O6—Te2	138.9 (5)		

Symmetry codes: (i) $x, -y, z - \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, -y + 1, z - \frac{1}{2}$; (iv) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (v) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (vi) $-x, y, -z + \frac{1}{2}$; (vii) $x - \frac{1}{2}, y + \frac{1}{2}, z$.

Table 2
Motifs of mutual adjunction (Hoppe, 1980) and coordination numbers (CN).

	O1	O2	O3	O4	O5	O6	CN
Lu	1/1	2/2	2/2	2/2	1/1	0/0	8
Te1	1/1	1/1	1/1	0 + 1/0 + 1	0/0	0/0	3 + 1
Te2	0 + 1/0 + 1	0/0	0/0	1/1	1/1	1/2	3 + 1
CN	2 + 1	3	3	3 + 1	2	2	–

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *SHELXL97*.

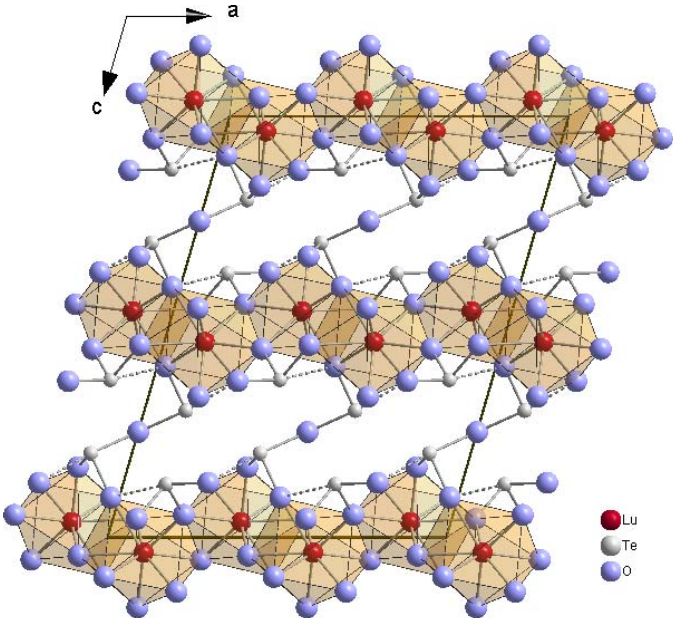

Figure 4
Crystal structure of $Lu_2Te_4O_{11}$ viewed along the *b* axis. The oxotellurate(IV) double chains connect the reticulated layers of condensed $[LuO_8]$ polyhedra through Te2—O6—Te2 bridges.

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