Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Lutetium(III) oxide iodide

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Received 22 October 2007; accepted 24 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Lu–O) = 0.001 Å; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 14.0.

Single crystals of lutetium oxide iodide, LuOI, were obtained as a by-product of the reaction of lutetium metal, rhenium powder and lutetium triiodide, LuI_3, in a sealed tantalum container. LuOI crystallizes in the tetragonal PbFCl-type of structure (matlockite), where Lu, O and I are situated on positions with 4*mm*, $\overline{4}m2$ and 4*mm* symmetry, respectively.

Related literature

For a previous powder study of LuOI, see: Batsanov *et al.* (1983); Meyer (1993). Syntheses of lanthanide compounds have been compiled by Meyer (1991).

Experimental

Crystal data

LuOI
M_r = 317.87
Tetragonal, $P4/nmm$
a = 3.8585 (7) Å
c = 9.189 (2) Å
V = 136.81 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 47.02 mm^{-1}
T = 293 (2) K
0.20 × 0.10 × 0.05 mm

Data collection

Stoe IPDSI diffractometer
Absorption correction: numerical [*X-RED* (Stoe & Cie, 2001) and *X-SHAPE* (Stoe & Cie, 1999)]
T_{min} = 0.008, T_{max} = 0.092
1249 measured reflections
126 independent reflections
121 reflections with $I > 2\sigma(I)$
R_{int} = 0.093

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.094
S = 1.20
126 reflections
9 parameters
$\Delta\rho_{max}$ = 2.82 e Å^{-3}
$\Delta\rho_{min}$ = −3.05 e Å^{-3}

Table 1
Selected bond lengths (Å).

Lu—O	2.2048 (5)	Lu—Lu^{i}	3.4641 (10)
Lu—I	3.3138 (10)		

Symmetry code: (i) $-x, -y+2, -z+1$.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); 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*.

This work was supported by the Deutsche Forschungsgemeinschaft (DFG), SFB 608 (Complex transition metal compounds with spin and charge degrees of freedom and disorder).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2159).

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supplementary materials

Acta Cryst. (2007). E**63**, i193 [**doi:10.1107/S160053680705283X**]

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Comment

In conproportionation reactions of rare-earth halides with their respective metals (frequently with the addition of a transition metal), the oxide halides *REOX* (*RE* = rare earth metal, *X* = halogen) often appear as a few single crystals as by-products. Except for impurities from the reaction containers, *e.g.* tantalum, this may be due to impure anhydrous rare-earth trihalides MX_3.

LuOI was obtained in a reaction of lutetium metal, rhenium powder and nominally pure lutetium triiodide, LuI_3, in a tantalum container at 1223 K. It crystallizes with the tetragonal PbFCl type of structure, in which Lu^{3+} is surrounded by four oxygen and four iodine atoms in a distorted square antiprismatic coordination with Lu—O distances of 2.2048 (5) Å and Lu—I distances of 3.3138 (10) Å. An additional iodide ion is capping one of the square faces at a distance of 4.0152 (18) Å (Figs. 1, 2). The cell parameters obtained from the present single-crystal study show no significant differences to those of a previous powder study (*a* = 3.850, *c* = 9.179 Å; Batsanov *et al.*, 1983; Meyer, 1993).

Experimental

Light orange, transparent plates of LuOI were obtained as a by-product (*ca* 30%) from the reaction of lutetium powder (0.066 g, 0.37 mmol, Smart Elements, 99.99%), rhenium powder (0.030 g, 0.16 mmol, Merck, 99.9%) and LuI_3 (0.150 g, 0.4 mmol). Most of LuI_3 and apparently all rhenium powder remained unreacted. LuI_3 was prepared by the direct reaction of Lu chips (Chempur, 99.9%) with I_2 (Acros, 95%) in a sealed silica tube at 503 K and subsequent purification of the product by high-vacuum sublimation. The reaction was carried out in a He-arc welded tantalum container within a silica jacket at 1223 K for 17 d. Due to their moisture and air sensitivity, reagents and products were handled in an argon-filled glove box (*M*. Braun, Garching, Germany).

Refinement

For the present refinement, origin choice 2 for space group *P*4/*nmm* was chosen. The highest peak in the final difference Fourier map is 0.90 Å from atom Lu and the deepest hole is 0.81 Å from the same atom.

Figures

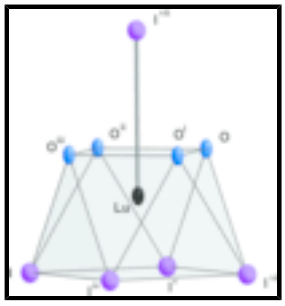

Fig. 1. : The surrounding of Lu^{3+} in LuOI with displacement ellipsoids drawn at the 75% probability level. [Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, y+1, z$; (iii) $-x-1, -y+1, -z+1$; (iv) $x-1, y-1, z$; (v) $x-1, y, z$; (vi) $x, y-1, z$; (vii) $-x, -y+2, -z+1$.]

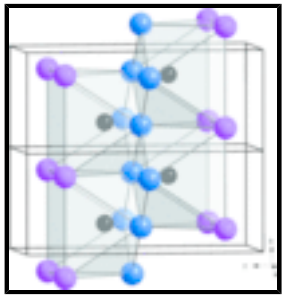

Fig. 2. : Part of the crystal structure of LuOI, viewed approximately along the *a* axis. Lu atoms are represented as black, O as blue and I as pink spheres.

Lutetium(III) oxide iodide

Crystal data

LuOI	$Z = 2$
$M_r = 317.87$	$F_{000} = 264$
Tetragonal, *P*4/*nmm*	$D_x = 7.717$ Mg m^{-3}
Hall symbol: -P 4a 2a	Mo *K*α radiation λ = 0.71073 Å
$a = 3.8585$ (7) Å	Cell parameters from 1205 reflections
$b = 3.8585$ (7) Å	θ = 1.9–28.2°
$c = 9.189$ (2) Å	μ = 47.02 mm^{-1}
α = 90°	$T = 293$ (2) K
β = 90°	Plate, orange
γ = 90°	0.20 × 0.10 × 0.05 mm
$V = 136.81$ (5) Å^3	

Data collection

Stoe IPDS-I diffractometer	126 independent reflections
Radiation source: fine-focus sealed tube	121 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.093$
$T = 293(2)$ K	$\theta_{max} = 27.8°$
φ scans	$\theta_{min} = 4.4°$
Absorption correction: numerical [X-RED (Stoe & Cie, 2001) and X-SHAPE (Stoe & Cie, 1999)]	$h = -4 \rightarrow 5$

T_{min} = 0.008, T_{max} = 0.092
1249 measured reflections
$k = -5 \rightarrow 5$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.094$
$S = 1.20$
126 reflections
9 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.82$ e Å^{-3}
$\Delta\rho_{min} = -3.05$ e Å^{-3}
Extinction correction: none

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Lu	−0.2500	0.7500	0.38386 (8)	0.0147 (5)
I	0.2500	1.2500	0.17918 (15)	0.0235 (5)
O	−0.2500	0.2500	0.5000	0.015 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Lu	0.0104 (5)	0.0104 (5)	0.0232 (6)	0.000	0.000	0.000
I	0.0222 (6)	0.0222 (6)	0.0260 (8)	0.000	0.000	0.000
O	0.010 (4)	0.010 (4)	0.024 (5)	0.000	0.000	0.000

Geometric parameters (Å, °)

Lu—O^{i}	2.2048 (5)	Lu—Lu^{i}	3.4641 (10)
Lu—O^{ii}	2.2048 (5)	Lu—Lu^{viii}	3.4641 (10)

Lu—O^{iii}	2.2048 (5)	Lu—Lu^{iii}	3.4641 (10)
Lu—O	2.2048 (5)	I—Lu^{ix}	3.3138 (10)
Lu—I^{iv}	3.3138 (10)	I—Lu^{ii}	3.3138 (10)
Lu—I	3.3138 (10)	I—Lu^{x}	3.3138 (10)
Lu—I^{v}	3.3138 (10)	O—Lu^{i}	2.2048 (5)
Lu—I^{vi}	3.3138 (10)	O—Lu^{vi}	2.2048 (5)
Lu—Lu^{vii}	3.4641 (10)	O—Lu^{iii}	2.2048 (5)
O^{i}—Lu—O^{ii}	76.449 (15)	O—Lu—Lu^{i}	38.225 (8)
O^{i}—Lu—O^{iii}	122.10 (3)	I^{iv}—Lu—Lu^{i}	110.470 (13)
O^{ii}—Lu—O^{iii}	76.449 (15)	I—Lu—Lu^{i}	110.470 (13)
O^{i}—Lu—O	76.449 (15)	I^{v}—Lu—Lu^{i}	176.54 (4)
O^{ii}—Lu—O	122.10 (3)	I^{vi}—Lu—Lu^{i}	72.62 (3)
O^{iii}—Lu—O	76.449 (15)	Lu^{vii}—Lu—Lu^{i}	67.69 (2)
O^{i}—Lu—I^{iv}	141.643 (6)	O^{i}—Lu—Lu^{viii}	100.90 (3)
O^{ii}—Lu—I^{iv}	141.643 (6)	O^{ii}—Lu—Lu^{viii}	38.225 (8)
O^{iii}—Lu—I^{iv}	76.426 (19)	O^{iii}—Lu—Lu^{viii}	38.225 (8)
O—Lu—I^{iv}	76.426 (19)	O—Lu—Lu^{viii}	100.90 (3)
O^{i}—Lu—I	76.426 (19)	I^{iv}—Lu—Lu^{viii}	110.470 (13)
O^{ii}—Lu—I	76.426 (19)	I—Lu—Lu^{viii}	110.470 (13)
O^{iii}—Lu—I	141.643 (6)	I^{v}—Lu—Lu^{viii}	72.62 (3)
O—Lu—I	141.643 (6)	I^{vi}—Lu—Lu^{viii}	176.54 (4)
I^{iv}—Lu—I	110.84 (5)	Lu^{vii}—Lu—Lu^{viii}	67.69 (2)
O^{i}—Lu—I^{v}	141.643 (6)	Lu^{i}—Lu—Lu^{viii}	103.93 (4)
O^{ii}—Lu—I^{v}	76.426 (19)	O^{i}—Lu—Lu^{iii}	100.90 (3)
O^{iii}—Lu—I^{v}	76.426 (19)	O^{ii}—Lu—Lu^{iii}	100.90 (3)
O—Lu—I^{v}	141.643 (6)	O^{iii}—Lu—Lu^{iii}	38.225 (8)
I^{iv}—Lu—I^{v}	71.21 (2)	O—Lu—Lu^{iii}	38.225 (8)
I—Lu—I^{v}	71.21 (2)	I^{iv}—Lu—Lu^{iii}	72.62 (3)
O^{i}—Lu—I^{vi}	76.426 (19)	I—Lu—Lu^{iii}	176.54 (4)
O^{ii}—Lu—I^{vi}	141.643 (6)	I^{v}—Lu—Lu^{iii}	110.470 (13)
O^{iii}—Lu—I^{vi}	141.643 (6)	I^{vi}—Lu—Lu^{iii}	110.470 (13)
O—Lu—I^{vi}	76.426 (19)	Lu^{vii}—Lu—Lu^{iii}	103.93 (4)
I^{iv}—Lu—I^{vi}	71.21 (2)	Lu^{i}—Lu—Lu^{iii}	67.69 (2)
I—Lu—I^{vi}	71.21 (2)	Lu^{viii}—Lu—Lu^{iii}	67.69 (2)
I^{v}—Lu—I^{vi}	110.84 (5)	Lu^{ix}—I—Lu^{ii}	71.21 (2)
O^{i}—Lu—Lu^{vii}	38.225 (8)	Lu^{ix}—I—Lu^{x}	71.21 (2)
O^{ii}—Lu—Lu^{vii}	38.225 (7)	Lu^{ii}—I—Lu^{x}	110.84 (5)
O^{iii}—Lu—Lu^{vii}	100.90 (3)	Lu^{ix}—I—Lu	110.84 (5)
O—Lu—Lu^{vii}	100.90 (3)	Lu^{ii}—I—Lu	71.21 (2)
I^{iv}—Lu—Lu^{vii}	176.54 (4)	Lu^{x}—I—Lu	71.21 (2)

I—Lu—Lu^{vii}	72.62 (3)	Lu^{i}—O—Lu^{vi}	103.551 (15)
I^{v}—Lu—Lu^{vii}	110.470 (13)	Lu^{i}—O—Lu^{iii}	122.10 (3)
I^{vi}—Lu—Lu^{vii}	110.470 (13)	Lu^{vi}—O—Lu^{iii}	103.551 (15)
O^{i}—Lu—Lu^{i}	38.225 (8)	Lu^{i}—O—Lu	103.551 (15)
O^{ii}—Lu—Lu^{i}	100.90 (3)	Lu^{vi}—O—Lu	122.10 (3)
O^{iii}—Lu—Lu^{i}	100.90 (3)	Lu^{iii}—O—Lu	103.551 (15)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) *x*, *y*+1, *z*; (iii) −*x*−1, −*y*+1, −*z*+1; (iv) *x*−1, *y*−1, *z*; (v) *x*−1, *y*, *z*; (vi) *x*, *y*−1, *z*; (vii) −*x*, −*y*+2, −*z*+1; (viii) −*x*−1, −*y*+2, −*z*+1; (ix) *x*+1, *y*+1, *z*; (x) *x*+1, *y*, *z*.

Fig. 1

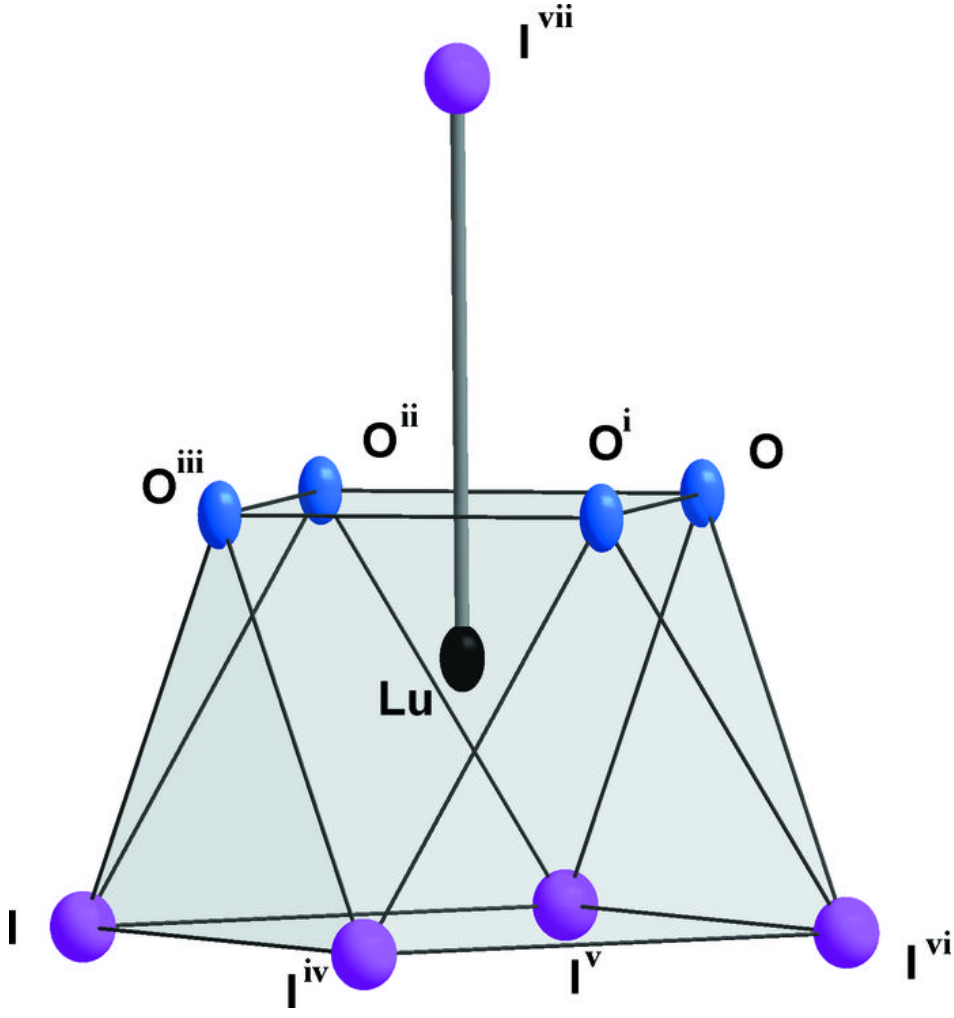

Fig. 2