Electronic Supporting information for:

Synthesis and Characterisation of a New Anion Exchangeable Layered Hydroxyiodide

Faye Y. Southworth,[a] Claire Wilson,[b] Simon J. Coles [b] and Andrew M. Fogg*[a]

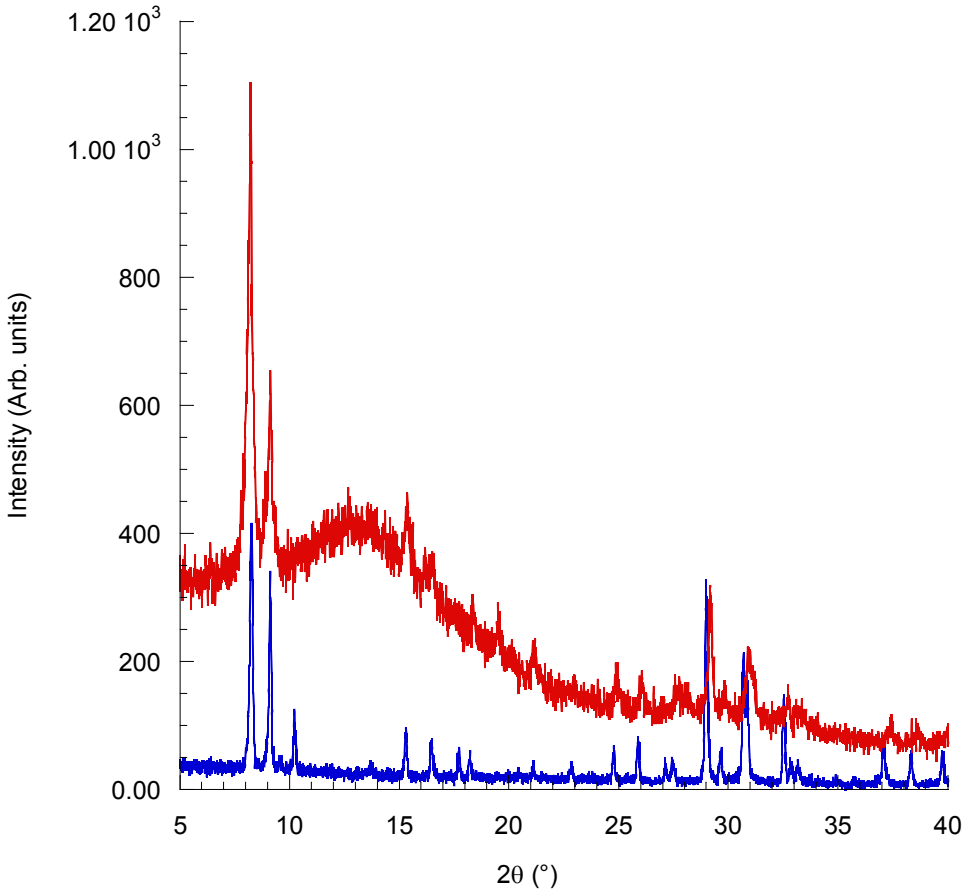

Figure S1 Comparison of the powder X-ray diffraction patterns of (a) $Lu_3O(OH)_9I.2H_2O$ (red) and (b) $Yb_3O(OH)_9Cl.2H_2O$ (blue)

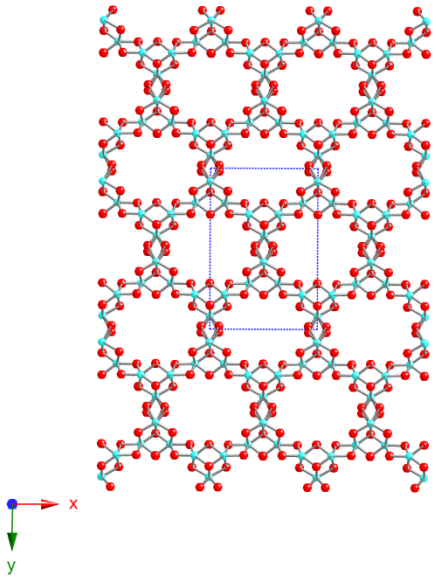

Figure S2 Crystal structure of $Yb_3O(OH)_9Cl.2H_2O$.

ncs | UK National Crystallography Service

Table 1. Crystal data and structure refinement details.

Identification code	**2012ncs0239**
Empirical formula	$H_{7.50}I_{0.50}Lu_2O_{6.50}$
Formula weight	524.95
Temperature	100(2) K
Wavelength	0.6889 Å
Crystal system	Orthorhombic
Space group	*Pbcm*
Unit cell dimensions	a = 9.399(7) Å α = 90°
	b = 12.413(9) Å β = 90°
	c = 13.5909(10) Å γ = 90°
Volume	1585.6(17) Å^3
Z	8
Density (calculated)	4.398 Mg / m^3
Absorption coefficient	24.440 mm^{-1}
F(000)	1824
Crystal	colourless rod
Crystal size	0.06 x 0.01 x 0.01 mm^3
θ range for data collection	2.635 − 26.643°
Index ranges	$-12 \le h \le 11$, $-16 \le k \le 14$, $-17 \le l \le 11$
Reflections collected	8292
Independent reflections	1898 [R_{int} = 0.1397]
Completeness to θ = 27.500°	91.0 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1898 / 0 / 61
Goodness-of-fit on F^2	0.988
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0833, *wR2* = 0.2135
R indices (all data)	*R1* = 0.1148, *wR2* = 0.2367
Extinction coefficient	n/a
Largest diff. peak and hole	9.746 and −5.078 e Å^{-3}

Diffractometer: Beamline I19 situated on an undulator insertion device with a combination of double crystal monochromator, vertical and horizontal focussing mirrors and a series of beam slits (primary white beam and either side of the focussing mirrors). The experimental hutch (EH1) is equipped with a Crystal Logic 4-circle kappa geometry goniometer with a Rigaku Saturn 724+ CCD detector and an Oxford Cryosystems Cryostream plus cryostat (80-500K). For conventional service crystallography the beamline operates at a typical energy of 18 keV (Zr K absorption edge) and a Rigaku ACTOR robotic sample changing system is available. **Cell determination and data collection**: *CrystalClear-SM Expert 2.0 r5* (Rigaku, 2010). **Data reduction, cell refinement and absorption correction**: *CrystalClear-SM Expert 2.0 r5* (Rigaku, 2010). **Structure solution**: *SUPERFLIP* (Palatinus, L. & Chapuis, G. (2007). J. Appl. Cryst. 40, 786-790). **Structure refinement**: *SHELXL2013* (Sheldrick, G.M. (2012) within OLEX2. **Graphics:** *OLEX2* (Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). J. Appl. Cryst. 42, 339-341).

Special details: Oxygen atoms were refined with isotropic adps. Through charge considerations and geometry, O1 is an oxide (O2-), O2 to O6 OH- and O7 and O8 H2O. Hydrogen atoms were not located or included explicitly in the model but are included in the unit cell contents and any values derived from them. Iodide anions are refined as 0.5 chemical occupancy. The single crystal diffraction data for Lu4O(OH)9I.3H2O were collected from a small needle-shaped crystal (0.06 x 0.01 x 0.01mm) on the beamline I19, Diamond Light Source. The sample suffered from radiation decay meaning that part of the data collected could not be used. Although the data completeness is still acceptable it is likely that this has affected the overall data quality resulting in the relatively high crystallographic R-factors (R1 = 0.083, Rint = 0.14).

Figure 1. View showing asymmetric unit contents of 2012ncs0239 and atom labelling schem. ADps drawn at 50%

probability level.

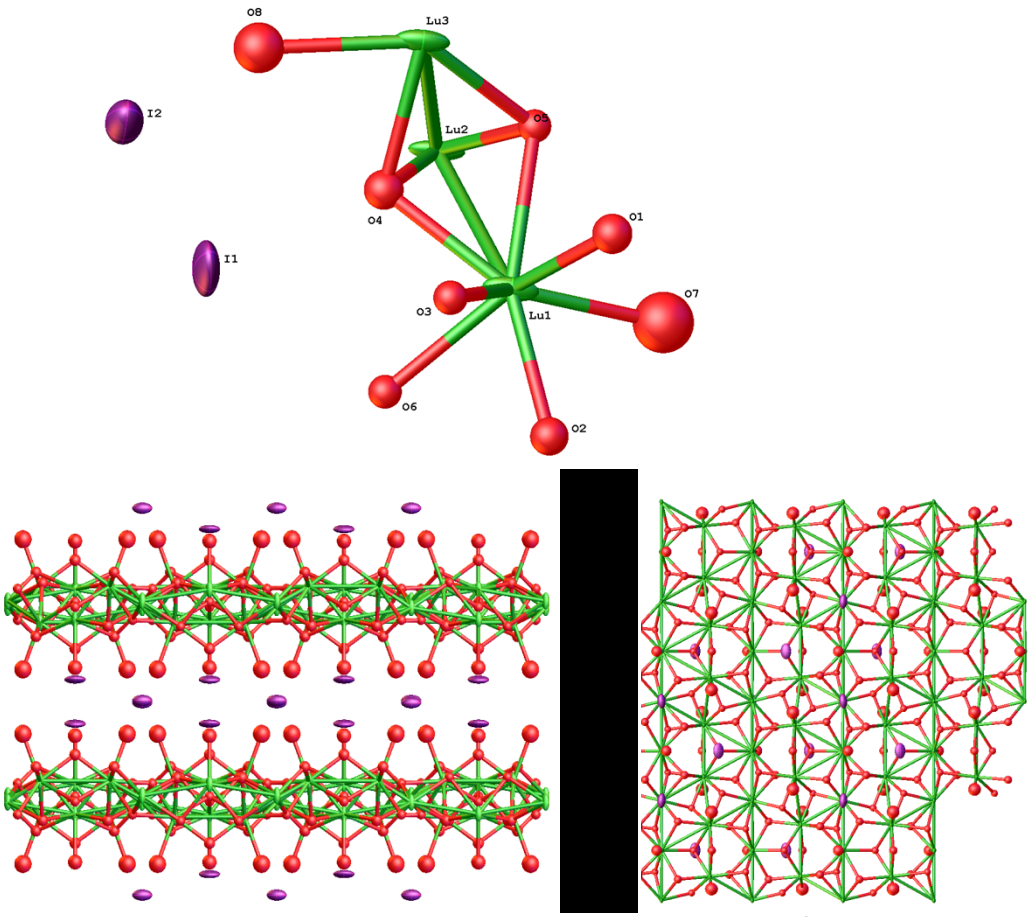

Table 2. Atomic coordinates [× 10^4], equivalent isotropic displacement parameters [Å^2 × 10^3] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
Lu1	5926(1)	164(1)	1193(1)	32(1)	1
Lu2	4823(2)	2500	0	31(1)	1
Lu3	4230(2)	2516(1)	2500	30(1)	1
O1	7270(30)	269(19)	2500	35(6)	1
O2	6650(20)	−1592(13)	1046(13)	32(4)	1
O3	4870(30)	−733(17)	2500	25(5)	1
O4	3900(20)	1232(14)	1288(12)	34(4)	1
O5	6127(17)	2147(12)	1420(12)	24(3)	1
O6	4198(17)	−841(12)	366(12)	24(3)	1
O7	8340(40)	260(20)	560(20)	81(8)	1
O8	1750(40)	2240(20)	2500	55(8)	1
I2	58(6)	2500	0	57(2)	0.5
I1	1132(6)	−643(5)	2500	65(2)	0.5

Table 3. Bond lengths [Å] and angles [°].

Lu1—O1	2.184(16)
Lu1—O6^{i}	2.282(16)
Lu1—O2	2.293(16)
Lu1—O4	2.320(18)
Lu1—O3	2.320(14)
Lu1—O6	2.336(15)
Lu1—O7	2.43(3)
Lu1—O5	2.488(15)
Lu1—Lu2	3.480(2)
Lu1—Lu1^{ii}	3.553(2)
Lu1—Lu1^{i}	3.702(2)
Lu1—Lu3^{iii}	3.740(2)
Lu2—O2^{i}	2.283(18)
Lu2—O2^{iv}	2.283(18)
Lu2—O6^{iv}	2.310(15)
Lu2—O6^{i}	2.310(15)
Lu2—O5	2.328(17)
Lu2—O5^{v}	2.328(17)
Lu2—O4^{v}	2.507(17)
Lu2—O4	2.507(17)
Lu2—Lu3^{v}	3.4431(5)
Lu2—Lu3	3.4431(5)
Lu2—Lu1^{v}	3.480(2)
Lu3—O4	2.312(17)
Lu3—O4^{ii}	2.313(17)
Lu3—O3^{iv}	2.33(2)
Lu3—O5	2.354(16)
Lu3—O5^{ii}	2.354(16)
Lu3—O8	2.36(4)
Lu3—O2^{vi}	2.412(17)
Lu3—O2^{iv}	2.412(17)
Lu3—Lu2^{ii}	3.4431(5)
Lu3—Lu1^{vi}	3.740(2)
Lu3—Lu1^{iv}	3.740(2)
O1—Lu1^{ii}	2.185(16)
O2—Lu2^{i}	2.283(18)
O2—Lu3^{iii}	2.412(17)
O3—Lu1^{ii}	2.320(14)
O3—Lu3^{iii}	2.33(2)
O6—Lu1^{i}	2.282(16)
O6—Lu2^{i}	2.310(15)
O1—Lu1—O6^{i}	139.7(7)
O1—Lu1—O2	87.4(8)

O6^{i}—Lu1—O2	106.5(6)
O1—Lu1—O4	113.2(7)
O6^{i}—Lu1—O4	78.4(6)
O2—Lu1—O4	142.4(6)
O1—Lu1—O3	69.8(8)
O6^{i}—Lu1—O3	150.0(6)
O2—Lu1—O3	74.8(7)
O4—Lu1—O3	83.2(7)
O1—Lu1—O6	145.6(7)
O6^{i}—Lu1—O6	73.4(6)
O2—Lu1—O6	70.0(6)
O4—Lu1—O6	76.3(6)
O3—Lu1—O6	79.3(6)
O1—Lu1—O7	75.1(9)
O6^{i}—Lu1—O7	72.7(8)
O2—Lu1—O7	74.7(8)
O4—Lu1—O7	139.3(7)
O3—Lu1—O7	133.9(9)
O6—Lu1—O7	120.4(8)
O1—Lu1—O5	78.2(7)
O6^{i}—Lu1—O5	75.8(5)
O2—Lu1—O5	158.2(6)
O4—Lu1—O5	59.3(6)
O3—Lu1—O5	114.3(6)
O6—Lu1—O5	129.9(5)
O7—Lu1—O5	85.7(7)
O1—Lu1—Lu2	120.1(6)
O6^{i}—Lu1—Lu2	41.0(4)
O2—Lu1—Lu2	147.2(4)
O4—Lu1—Lu2	46.0(4)
O3—Lu1—Lu2	129.0(6)
O6—Lu1—Lu2	90.8(4)
O7—Lu1—Lu2	94.3(6)
O5—Lu1—Lu2	41.9(4)
O1—Lu1—Lu1^{ii}	35.6(6)
O6^{i}—Lu1—Lu1^{ii}	158.2(4)
O2—Lu1—Lu1^{ii}	95.0(4)
O4—Lu1—Lu1^{ii}	86.8(4)
O3—Lu1—Lu1^{ii}	40.0(4)
O6—Lu1—Lu1^{ii}	118.7(4)
O7—Lu1—Lu1^{ii}	110.6(7)
O5—Lu1—Lu1^{ii}	82.9(4)
Lu2—Lu1—Lu1^{ii}	117.76(2)
O1—Lu1—Lu1^{i}	172.2(6)
O6^{i}—Lu1—Lu1^{i}	37.2(4)
O2—Lu1—Lu1^{i}	87.7(4)

O4—Lu1—Lu1^{i}	74.1(4)
O3—Lu1—Lu1^{i}	114.6(5)
O6—Lu1—Lu1^{i}	36.2(4)
O7—Lu1—Lu1^{i}	97.8(7)
O5—Lu1—Lu1^{i}	104.7(4)
Lu2—Lu1—Lu1^{i}	62.85(4)
Lu1^{ii}—Lu1—Lu1^{i}	151.14(4)
O1—Lu1—Lu3^{iii}	71.9(6)
O6^{i}—Lu1—Lu3^{iii}	139.7(4)
O2—Lu1—Lu3^{iii}	38.5(4)
O4—Lu1—Lu3^{iii}	116.3(4)
O3—Lu1—Lu3^{iii}	36.7(5)
O6—Lu1—Lu3^{iii}	74.4(4)
O7—Lu1—Lu3^{iii}	104.2(6)
O5—Lu1—Lu3^{iii}	144.5(4)
Lu2—Lu1—Lu3^{iii}	160.41(6)
Lu1^{ii}—Lu1—Lu3^{iii}	61.64(2)
Lu1^{i}—Lu1—Lu3^{iii}	107.52(4)
O2^{i}—Lu2—O2^{iv}	105.3(9)
O2^{i}—Lu2—O6^{iv}	144.7(6)
O2^{iv}—Lu2—O6^{iv}	70.6(6)
O2^{i}—Lu2—O6^{i}	70.6(6)
O2^{iv}—Lu2—O6^{i}	144.7(6)
O6^{iv}—Lu2—O6^{i}	133.0(8)
O2^{i}—Lu2—O5	138.0(6)
O2^{iv}—Lu2—O5	84.0(6)
O6^{iv}—Lu2—O5	77.2(5)
O6^{i}—Lu2—O5	78.5(5)
O2^{i}—Lu2—O5^{v}	84.0(6)
O2^{iv}—Lu2—O5^{v}	138.0(6)
O6^{iv}—Lu2—O5^{v}	78.5(5)
O6^{i}—Lu2—O5^{v}	77.2(5)
O5—Lu2—O5^{v}	116.5(8)
O2^{i}—Lu2—O4^{v}	70.5(6)
O2^{iv}—Lu2—O4^{v}	85.2(6)
O6^{iv}—Lu2—O4^{v}	74.2(6)
O6^{i}—Lu2—O4^{v}	123.1(6)
O5—Lu2—O4^{v}	151.4(5)
O5^{v}—Lu2—O4^{v}	59.0(6)
O2^{i}—Lu2—O4	85.2(6)
O2^{iv}—Lu2—O4	70.5(6)
O6^{iv}—Lu2—O4	123.1(6)
O6^{i}—Lu2—O4	74.2(6)
O5—Lu2—O4	59.0(6)
O5^{v}—Lu2—O4	151.4(5)
O4^{v}—Lu2—O4	139.7(9)

$O2^{i}$—Lu2—$Lu3^{v}$	44.3(4)
$O2^{iv}$—Lu2—$Lu3^{v}$	121.3(4)
$O6^{iv}$—Lu2—$Lu3^{v}$	106.4(4)
$O6^{i}$—Lu2—$Lu3^{v}$	81.2(4)
O5—Lu2—$Lu3^{v}$	154.5(4)
$O5^{v}$—Lu2—$Lu3^{v}$	42.9(4)
$O4^{v}$—Lu2—$Lu3^{v}$	42.2(4)
O4—Lu2—$Lu3^{v}$	129.0(4)
$O2^{i}$—Lu2—Lu3	121.3(4)
$O2^{iv}$—Lu2—Lu3	44.3(4)
$O6^{iv}$—Lu2—Lu3	81.2(4)
$O6^{i}$—Lu2—Lu3	106.4(4)
O5—Lu2—Lu3	42.9(4)
$O5^{v}$—Lu2—Lu3	154.5(4)
$O4^{v}$—Lu2—Lu3	129.0(4)
O4—Lu2—Lu3	42.2(4)
$Lu3^{v}$—Lu2—Lu3	161.38(9)
$O2^{i}$—Lu2—$Lu1^{v}$	107.6(4)
$O2^{iv}$—Lu2—$Lu1^{v}$	93.4(4)
$O6^{iv}$—Lu2—$Lu1^{v}$	40.4(4)
$O6^{i}$—Lu2—$Lu1^{v}$	121.6(4)
O5—Lu2—$Lu1^{v}$	112.7(4)
$O5^{v}$—Lu2—$Lu1^{v}$	45.6(4)
$O4^{v}$—Lu2—$Lu1^{v}$	41.8(4)
O4—Lu2—$Lu1^{v}$	162.0(4)
$Lu3^{v}$—Lu2—$Lu1^{v}$	65.99(3)
Lu3—Lu2—$Lu1^{v}$	120.21(3)
$O2^{i}$—Lu2—Lu1	93.4(4)
$O2^{iv}$—Lu2—Lu1	107.6(4)
$O6^{iv}$—Lu2—Lu1	121.6(4)
$O6^{i}$—Lu2—Lu1	40.4(4)
O5—Lu2—Lu1	45.6(4)
$O5^{v}$—Lu2—Lu1	112.7(4)
$O4^{v}$—Lu2—Lu1	162.0(4)
O4—Lu2—Lu1	41.8(4)
$Lu3^{v}$—Lu2—Lu1	120.21(3)
Lu3—Lu2—Lu1	65.99(3)
$Lu1^{v}$—Lu2—Lu1	145.34(8)
O4—Lu3—$O4^{ii}$	90.9(9)
O4—Lu3—$O3^{iv}$	133.6(4)
$O4^{ii}$—Lu3—$O3^{iv}$	133.6(4)
O4—Lu3—O5	61.5(6)
$O4^{ii}$—Lu3—O5	114.2(6)
$O3^{iv}$—Lu3—O5	84.6(6)
O4—Lu3—$O5^{ii}$	114.2(6)
$O4^{ii}$—Lu3—$O5^{ii}$	61.5(6)

O3^{iv}—Lu3—O5^{ii}	84.6(6)
O5—Lu3—O5^{ii}	77.1(8)
O4—Lu3—O8	76.6(7)
O4^{ii}—Lu3—O8	76.6(7)
O3^{iv}—Lu3—O8	119.8(9)
O5—Lu3—O8	136.1(5)
O5^{ii}—Lu3—O8	136.1(5)
O4—Lu3—O2^{vi}	148.7(6)
O4^{ii}—Lu3—O2^{vi}	71.8(6)
O3^{iv}—Lu3—O2^{vi}	72.4(5)
O5—Lu3—O2^{vi}	149.3(6)
O5^{ii}—Lu3—O2^{vi}	80.7(6)
O8—Lu3—O2^{vi}	74.2(6)
O4—Lu3—O2^{iv}	71.8(6)
O4^{ii}—Lu3—O2^{iv}	148.7(6)
O3^{iv}—Lu3—O2^{iv}	72.4(5)
O5—Lu3—O2^{iv}	80.7(6)
O5^{ii}—Lu3—O2^{iv}	149.3(6)
O8—Lu3—O2^{iv}	74.2(6)
O2^{vi}—Lu3—O2^{iv}	110.0(8)
O4—Lu3—Lu2^{ii}	136.1(4)
O4^{ii}—Lu3—Lu2^{ii}	46.7(4)
O3^{iv}—Lu3—Lu2^{ii}	86.93(9)
O5—Lu3—Lu2^{ii}	119.4(4)
O5^{ii}—Lu3—Lu2^{ii}	42.4(4)
O8—Lu3—Lu2^{ii}	99.16(5)
O2^{vi}—Lu3—Lu2^{ii}	41.4(4)
O2^{iv}—Lu3—Lu2^{ii}	150.1(4)
O4—Lu3—Lu2	46.7(4)
O4^{ii}—Lu3—Lu2	136.1(4)
O3^{iv}—Lu3—Lu2	86.93(9)
O5—Lu3—Lu2	42.4(4)
O5^{ii}—Lu3—Lu2	119.4(4)
O8—Lu3—Lu2	99.16(5)
O2^{vi}—Lu3—Lu2	150.1(4)
O2^{iv}—Lu3—Lu2	41.4(4)
Lu2^{ii}—Lu3—Lu2	161.37(9)
O4—Lu3—Lu1^{vi}	159.9(4)
O4^{ii}—Lu3—Lu1^{vi}	105.2(4)
O3^{iv}—Lu3—Lu1^{vi}	36.4(4)
O5—Lu3—Lu1^{vi}	119.8(4)
O5^{ii}—Lu3—Lu1^{vi}	84.5(4)
O8—Lu3—Lu1^{vi}	95.2(6)
O2^{vi}—Lu3—Lu1^{vi}	36.3(4)
O2^{iv}—Lu3—Lu1^{vi}	88.4(4)
Lu2^{ii}—Lu3—Lu1^{vi}	62.77(3)

Lu2—Lu3—Lu1^{vi}	118.69(4)
O4—Lu3—Lu1^{iv}	105.2(4)
O4^{ii}—Lu3—Lu1^{iv}	159.9(4)
O3^{iv}—Lu3—Lu1^{iv}	36.4(4)
O5—Lu3—Lu1^{iv}	84.5(4)
O5^{ii}—Lu3—Lu1^{iv}	119.8(4)
O8—Lu3—Lu1^{iv}	95.2(6)
O2^{vi}—Lu3—Lu1^{iv}	88.4(4)
O2^{iv}—Lu3—Lu1^{iv}	36.3(4)
Lu2^{ii}—Lu3—Lu1^{iv}	118.69(4)
Lu2—Lu3—Lu1^{iv}	62.77(3)
Lu1^{vi}—Lu3—Lu1^{iv}	56.73(5)
Lu1—O1—Lu1^{ii}	108.8(12)
Lu2^{i}—O2—Lu1	110.0(7)
Lu2^{i}—O2—Lu3^{iii}	94.3(6)
Lu1—O2—Lu3^{iii}	105.3(7)
Lu1—O3—Lu1^{ii}	99.9(9)
Lu1—O3—Lu3^{iii}	106.9(7)
Lu1^{ii}—O3—Lu3^{iii}	106.9(7)
Lu3—O4—Lu1	109.0(7)
Lu3—O4—Lu2	91.1(6)
Lu1—O4—Lu2	92.2(6)
Lu2—O5—Lu3	94.7(6)
Lu2—O5—Lu1	92.5(5)
Lu3—O5—Lu1	102.2(6)
Lu1^{i}—O6—Lu2^{i}	98.5(6)
Lu1^{i}—O6—Lu1	106.6(6)
Lu2^{i}—O6—Lu1	107.6(6)

Symmetry transformations used to generate equivalent atoms:
(i) −x+1,−y,−z (ii) x,y,−z+1/2 (iii) −x+1,y−1/2,z
(iv) −x+1,y+1/2,z (v) x,−y+1/2,−z (vi) −x+1,y+1/2,−z+1/2

Table 4. Anisotropic displacement parameters [Å^2× 10^3]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + \cdots + 2\,h\,k\,a^*\,b^*\,U^{12}]$.

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Lu1	70(1)	10(1)	16(1)	2(1)	−1(1)	−2(1)
Lu2	70(1)	7(1)	15(1)	−1(1)	0	0
Lu3	65(1)	9(1)	16(1)	0	0	−1(1)
I2	31(3)	33(3)	107(6)	−5(3)	0	0
I1	16(3)	55(3)	124(7)	0	0	−8(2)