

Lutetium ultraphosphate

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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{P}-\text{O}) = 0.002$ Å; R factor = 0.019; wR factor = 0.051; data-to-parameter ratio = 15.9.

The structure of the title compound, $\text{LuP}_5\text{O}_{14}$, comprises puckered eight-membered PO_4 rings linked by the lutetium cations in a complex way, forming a three-dimensional framework. Each eight-membered phosphate ring shares a bridging tetrahedron with each of four adjacent tetrahedra, to form layers of PO_4 tetrahedra. These layers are $c/2$ in thickness and parallel to the ab plane. Each Lu ion is contained in one such layer, forming bonds to six O atoms in that layer and also to one O atom belonging to a tetrahedron in each of the layers lying above and below it. The LuO_8 polyhedra are isolated from one another, since they share no common atoms. The Lu ions lie on twofold axes (special position 4e) and the shortest Lu...Lu distance is 5.703 (1) Å.

Related literature

For related literature, see: Durif (1971); Hong (1974); Hong & Pierce (1974). For the classification of ultraphosphates, see: Bagieu-Beucher & Tranqui (1970).

Experimental

Crystal data

$\text{LuP}_5\text{O}_{14}$
 $M_r = 553.82$

Monoclinic, $C2/c$
 $a = 12.8128$ (14) Å

$b = 12.6821$ (13) Å
 $c = 12.3330$ (13) Å
 $\beta = 91.295$ (3)°
 $V = 2003.5$ (4) Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 10.74$ mm⁻¹
 $T = 298$ (2) K
 $0.20 \times 0.19 \times 0.18$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.121$, $T_{\text{max}} = 0.145$
10422 measured reflections

2912 independent reflections
2734 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
2 standard reflections every 150 reflections
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.050$
 $S = 1.08$
2912 reflections

183 parameters
 $\Delta\rho_{\text{max}} = 1.34$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.98$ e Å⁻³

Data collection: *CAD-4 EXPRESS* (Duisenberg, 1992; Enraf-Nonius, 1994; Macíček & Yordanov, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2001); 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: BR2072).

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supplementary materials

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Comment

The structure of $\text{LuP}_5\text{O}_{14}$ is a type (II) rare-earth ultraphosphates as classified by Bagieu-Beucher & Tranqui (1970), since it crystallizes in the monoclinic system with space group $C2/c$. In this structure, the lutetium ion is surrounded by eight oxygen atoms that form distorted polyhedra. Each of the oxygen atoms in the LuO_8 polyhedra are shared exclusively with PO_4 tetrahedra to form a three-dimensional framework, which delimits interesting tunnels (Fig.1). The structure is built up from (PO_4) tetrahedra (Fig.2) which are cross-linked by bridging O atoms, but these do not form helical ribbons, as in the $\text{NdP}_5\text{O}_{14}$ structure type (I) (Hong, 1974), and $\text{HoP}_5\text{O}_{14}$ structure type (III) (Durif, 1971). The anion is being constructed from a succession of eight-membered rings interconnected through the ternary tetrahedra in a complex way (Fig.3a). The members of an individual ring are shown with yellow color. Each ring shares a bridging tetrahedron with each of four adjacent tetrahedra to form layers of PO_4 tetrahedra, as illustrated in Fig.3b. These layers are about $c/2$ in thickness and parallel to the a - b plane. Each Lu ion is contained in one such layer, forming bonds to six oxygen in that layer and also to one oxygen belonging to a tetrahedron in each of the layers lying above and below it. The LuO_8 polyhedra are isolated from one another, since they share no common atoms. The shortest Lu-Lu distances are 5.703. The $\text{LuP}_5\text{O}_{14}$ ultraphosphate is isostructural with $\text{YbP}_5\text{O}_{14}$ (Hong & Pierce, 1974).

Experimental

Single crystal of $\text{LuP}_5\text{O}_{14}$ was prepared by flux method. At room temperature, 0.5 g of Lu_2O_3 were slowly added to 10 ml of phosphoric acid H_3PO_4 (85%). The mixture was then slowly heated to 673 K and kept at this temperature for seven days. colorless, crystals were separated from the excess phosphoric acid by washing the product in boiling water.

Refinement

The highest peak and the deepest hole are located 0.79 Å and 0.54 Å, respectively, from Lu1 and Lu2.

Figures

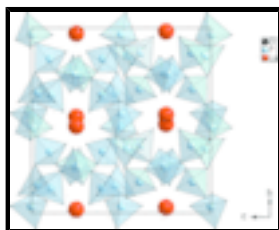


Fig. 1. The structural arrangement of $\text{LuP}_5\text{O}_{14}$ along the a axis, showing tunnels in which the Lu ions are located.



Fig. 2. Projection of the ultraphosphate with anisotropic displacement parameters drawn at the 50% probability level. [Symmetry codes: (i) $x, y+1, z$; (ii) $-x+5/2, y+1/2, -z+3/2$; (iii) $x+1/2, y+1/2, z$; (iv) $x, -y+2, z-1/2$; (v) $x-1/2, -y+3/2, z-1/2$.

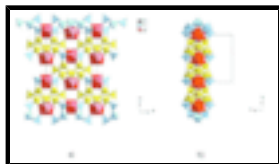


Fig. 3. a) Projection of one layer showing LuO_8 polyhedra and linkage of PO_4 tetrahedra forming rings (yellow) in $\text{LuP}_5\text{O}_{14}$. b) Projection of one layer along the b axis with $c/2$ thickness.

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$M_r = 553.82$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 12.8128\ (14)\ \text{\AA}$

$b = 12.6821\ (13)\ \text{\AA}$

$c = 12.3330\ (13)\ \text{\AA}$

$\beta = 91.295\ (3)^\circ$

$V = 2003.5\ (4)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 2064$

$D_x = 3.672\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 7043 reflections

$\theta = 2.3\text{--}30.0^\circ$

$\mu = 10.74\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Prism, colorless

$0.20 \times 0.19 \times 0.18\ \text{mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer

Monochromator: graphite

$T = 298\ (2)\ \text{K}$

$\omega/2\theta$ scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.121, T_{\max} = 0.145$

10422 measured reflections

2912 independent reflections

2734 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 30.0^\circ$

$\theta_{\min} = 2.3^\circ$

$h = -17 \rightarrow 18$

$k = -17 \rightarrow 17$

$l = -17 \rightarrow 17$

2 standard reflections

every 150 reflections

intensity decay: 2%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.019$

$wR(F^2) = 0.050$

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.028P)^2 + 3.0307P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 1.34\ \text{e \AA}^{-3}$

$S = 1.08$

$$\Delta\rho_{\min} = -0.98 \text{ e } \text{\AA}^{-3}$$

2912 reflections

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

183 parameters

Extinction coefficient: 0.00637 (11)

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Lu1	1.0000	1.019590 (11)	0.7500	0.00946 (6)
Lu2	1.0000	0.469251 (11)	0.7500	0.00947 (6)
P1	1.18263 (5)	0.63687 (5)	0.89095 (5)	0.01020 (13)
P2	0.97504 (5)	0.65029 (5)	0.96672 (5)	0.01037 (13)
P3	0.85012 (5)	0.83192 (5)	0.89783 (5)	0.01042 (13)
P4	0.85469 (5)	0.96507 (5)	0.50182 (6)	0.01080 (14)
P5	1.17542 (6)	1.24856 (5)	0.76032 (6)	0.01017 (14)
O1	1.15332 (15)	0.54741 (15)	0.82204 (16)	0.0131 (4)
O2	1.24259 (17)	0.72754 (16)	0.83475 (17)	0.0131 (4)
O3	1.09081 (14)	0.69824 (15)	0.94396 (15)	0.0120 (3)
O4	1.24970 (13)	0.60463 (17)	0.99342 (13)	0.0121 (4)
O5	0.97503 (15)	0.59905 (15)	1.07328 (15)	0.0137 (4)
O6	0.93961 (15)	0.59620 (15)	0.86627 (15)	0.0132 (4)
O7	0.91291 (17)	0.75979 (14)	0.97911 (18)	0.0114 (4)
O8	0.90954 (15)	0.87184 (15)	0.80717 (15)	0.0132 (4)
O9	0.75333 (18)	0.76371 (15)	0.86473 (18)	0.0128 (4)
O10	0.80606 (15)	0.91824 (14)	0.97493 (15)	0.0123 (4)
O11	0.88893 (16)	0.96091 (15)	0.61582 (16)	0.0139 (4)
O12	0.92416 (15)	1.06579 (15)	0.91286 (15)	0.0135 (4)
O13	1.12590 (16)	0.34943 (15)	0.73083 (16)	0.0142 (4)
O14	1.11367 (16)	1.15411 (15)	0.78473 (16)	0.0147 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Lu1	0.00912 (9)	0.00934 (9)	0.00992 (9)	0.000	-0.00025 (6)	0.000

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Lu2	0.00918 (9)	0.00909 (8)	0.01011 (9)	0.000	-0.00038 (6)	0.000
P1	0.0100 (3)	0.0094 (3)	0.0112 (3)	0.0001 (2)	-0.0002 (2)	0.0002 (2)
P2	0.0100 (3)	0.0099 (3)	0.0112 (3)	0.0004 (2)	0.0001 (2)	0.0002 (2)
P3	0.0103 (3)	0.0097 (3)	0.0112 (3)	-0.0001 (2)	-0.0002 (2)	0.0000 (2)
P4	0.0108 (3)	0.0097 (3)	0.0119 (3)	-0.0006 (2)	-0.0003 (2)	0.0001 (2)
P5	0.0094 (3)	0.0099 (3)	0.0112 (3)	-0.0002 (2)	-0.0004 (3)	-0.0006 (2)
O1	0.0125 (9)	0.0125 (8)	0.0143 (9)	-0.0011 (7)	0.0008 (7)	-0.0012 (7)
O2	0.0142 (10)	0.0108 (8)	0.0144 (9)	-0.0013 (7)	0.0030 (7)	0.0002 (7)
O3	0.0099 (8)	0.0113 (8)	0.0149 (9)	0.0002 (7)	0.0013 (7)	-0.0004 (7)
O4	0.0115 (10)	0.0129 (9)	0.0118 (9)	0.0024 (6)	-0.0014 (7)	-0.0003 (6)
O5	0.0148 (9)	0.0127 (8)	0.0135 (9)	0.0004 (7)	-0.0001 (7)	0.0015 (7)
O6	0.0132 (9)	0.0135 (8)	0.0129 (9)	0.0018 (7)	-0.0009 (7)	-0.0010 (7)
O7	0.0107 (10)	0.0119 (8)	0.0116 (9)	0.0017 (6)	-0.0008 (7)	-0.0007 (6)
O8	0.0153 (9)	0.0122 (8)	0.0122 (8)	-0.0023 (7)	0.0012 (7)	-0.0001 (7)
O9	0.0119 (10)	0.0136 (8)	0.0129 (10)	-0.0016 (7)	-0.0012 (8)	0.0000 (7)
O10	0.0123 (9)	0.0098 (8)	0.0150 (9)	0.0007 (7)	0.0013 (7)	-0.0012 (7)
O11	0.0150 (10)	0.0142 (8)	0.0125 (9)	-0.0020 (7)	-0.0014 (7)	0.0002 (7)
O12	0.0143 (9)	0.0129 (8)	0.0135 (9)	-0.0007 (7)	0.0013 (7)	0.0002 (7)
O13	0.0148 (9)	0.0137 (8)	0.0142 (9)	0.0028 (7)	-0.0001 (7)	0.0002 (7)
O14	0.0156 (9)	0.0131 (8)	0.0151 (9)	-0.0044 (7)	-0.0009 (7)	0.0002 (7)

Geometric parameters (Å, °)

Lu1—O14	2.2775 (19)	P2—O7	1.6097 (19)
Lu1—O14 ⁱ	2.2775 (19)	P2—O3	1.633 (2)
Lu1—O11	2.283 (2)	P3—O8	1.458 (2)
Lu1—O11 ⁱ	2.283 (2)	P3—O9	1.559 (2)
Lu1—O8	2.3213 (19)	P3—O10	1.5638 (19)
Lu1—O8 ⁱ	2.3213 (19)	P3—O7	1.566 (2)
Lu1—O12	2.3260 (19)	P4—O11	1.464 (2)
Lu1—O12 ⁱ	2.3260 (19)	P4—O12 ^{iv}	1.481 (2)
Lu2—O13 ⁱ	2.2326 (19)	P4—O4 ^v	1.6111 (19)
Lu2—O13	2.2326 (19)	P4—O10 ^{iv}	1.637 (2)
Lu2—O6	2.3016 (19)	P5—O13 ^{vi}	1.470 (2)
Lu2—O6 ⁱ	2.3016 (19)	P5—O14	1.470 (2)
Lu2—O1	2.356 (2)	P5—O2 ^{vii}	1.614 (2)
Lu2—O1 ⁱ	2.356 (2)	P5—O9 ^{viii}	1.623 (2)
Lu2—O5 ⁱⁱ	2.3604 (19)	O2—P5 ^{ix}	1.614 (2)
Lu2—O5 ⁱⁱⁱ	2.3604 (19)	O4—P4 ^x	1.6111 (19)
P1—O1	1.462 (2)	O5—Lu2 ⁱⁱⁱ	2.3604 (19)
P1—O2	1.555 (2)	O9—P5 ^{xi}	1.623 (2)
P1—O3	1.5659 (19)	O10—P4 ^{xii}	1.637 (2)
P1—O4	1.5665 (18)	O12—P4 ^{xii}	1.481 (2)
P2—O5	1.466 (2)	O13—P5 ^{xiii}	1.470 (2)
P2—O6	1.479 (2)		

O14—Lu1—O14 ⁱ	82.98 (10)	O13—Lu2—O5 ⁱⁱⁱ	76.40 (7)
O14—Lu1—O11	140.00 (7)	O6—Lu2—O5 ⁱⁱⁱ	73.85 (7)
O14 ⁱ —Lu1—O11	73.88 (7)	O6 ⁱ —Lu2—O5 ⁱⁱⁱ	142.33 (7)
O14—Lu1—O11 ⁱ	73.88 (7)	O1—Lu2—O5 ⁱⁱⁱ	73.25 (7)
O14 ⁱ —Lu1—O11 ⁱ	140.00 (7)	O1 ⁱ —Lu2—O5 ⁱⁱⁱ	126.65 (7)
O11—Lu1—O11 ⁱ	141.95 (10)	O5 ⁱⁱ —Lu2—O5 ⁱⁱⁱ	136.94 (9)
O14—Lu1—O8	150.35 (7)	O1—P1—O2	115.98 (12)
O14 ⁱ —Lu1—O8	109.89 (7)	O1—P1—O3	116.30 (11)
O11—Lu1—O8	69.49 (7)	O2—P1—O3	101.66 (11)
O11 ⁱ —Lu1—O8	79.86 (7)	O1—P1—O4	113.30 (12)
O14—Lu1—O8 ⁱ	109.89 (7)	O2—P1—O4	106.56 (12)
O14 ⁱ —Lu1—O8 ⁱ	150.35 (7)	O3—P1—O4	101.33 (10)
O11—Lu1—O8 ⁱ	79.86 (7)	O5—P2—O6	122.63 (12)
O11 ⁱ —Lu1—O8 ⁱ	69.49 (7)	O5—P2—O7	106.70 (11)
O8—Lu1—O8 ⁱ	72.36 (10)	O6—P2—O7	109.67 (11)
O14—Lu1—O12	85.80 (7)	O5—P2—O3	109.73 (11)
O14 ⁱ —Lu1—O12	72.29 (7)	O6—P2—O3	106.95 (11)
O11—Lu1—O12	116.25 (7)	O7—P2—O3	98.52 (11)
O11 ⁱ —Lu1—O12	73.85 (7)	O8—P3—O9	114.72 (12)
O8—Lu1—O12	73.73 (7)	O8—P3—O10	115.17 (11)
O8 ⁱ —Lu1—O12	133.38 (7)	O9—P3—O10	104.58 (12)
O14—Lu1—O12 ⁱ	72.29 (7)	O8—P3—O7	115.10 (12)
O14 ⁱ —Lu1—O12 ⁱ	85.80 (7)	O9—P3—O7	103.75 (11)
O11—Lu1—O12 ⁱ	73.85 (7)	O10—P3—O7	101.94 (11)
O11 ⁱ —Lu1—O12 ⁱ	116.25 (7)	O11—P4—O12 ^{iv}	121.99 (12)
O8—Lu1—O12 ⁱ	133.38 (7)	O11—P4—O4 ^v	105.88 (11)
O8 ⁱ —Lu1—O12 ⁱ	73.73 (7)	O12 ^{iv} —P4—O4 ^v	108.80 (11)
O12—Lu1—O12 ⁱ	150.82 (9)	O11—P4—O10 ^{iv}	109.37 (11)
O13 ⁱ —Lu2—O13	94.22 (10)	O12 ^{iv} —P4—O10 ^{iv}	108.74 (11)
O13 ⁱ —Lu2—O6	98.99 (7)	O4 ^v —P4—O10 ^{iv}	99.76 (11)
O13—Lu2—O6	142.75 (7)	O13 ^{vi} —P5—O14	121.90 (13)
O13 ⁱ —Lu2—O6 ⁱ	142.75 (7)	O13 ^{vi} —P5—O2 ^{vii}	104.38 (11)
O13—Lu2—O6 ⁱ	98.99 (7)	O14—P5—O2 ^{vii}	112.10 (11)
O6—Lu2—O6 ⁱ	91.22 (10)	O13 ^{vi} —P5—O9 ^{viii}	110.34 (11)
O13 ⁱ —Lu2—O1	147.63 (7)	O14—P5—O9 ^{viii}	104.96 (11)
O13—Lu2—O1	74.22 (7)	O2 ^{vii} —P5—O9 ^{viii}	101.37 (12)
O6—Lu2—O1	76.11 (7)	P1—O1—Lu2	138.32 (12)
O6 ⁱ —Lu2—O1	69.60 (7)	P1—O2—P5 ^{ix}	141.48 (14)
O13 ⁱ —Lu2—O1 ⁱ	74.22 (7)	P1—O3—P2	125.49 (12)
O13—Lu2—O1 ⁱ	147.63 (7)	P1—O4—P4 ^x	129.58 (12)
O6—Lu2—O1 ⁱ	69.60 (7)	P2—O5—Lu2 ⁱⁱⁱ	170.97 (13)
O6 ⁱ —Lu2—O1 ⁱ	76.11 (7)	P2—O6—Lu2	138.19 (12)

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O1—Lu2—O1 ⁱ	130.25 (10)	P3—O7—P2	133.72 (14)
O13 ⁱ —Lu2—O5 ⁱⁱ	76.40 (7)	P3—O8—Lu1	141.86 (12)
O13—Lu2—O5 ⁱⁱ	74.67 (7)	P3—O9—P5 ^{xi}	138.16 (14)
O6—Lu2—O5 ⁱⁱ	142.33 (7)	P3—O10—P4 ^{xii}	127.96 (13)
O6 ⁱ —Lu2—O5 ⁱⁱ	73.85 (7)	P4—O11—Lu1	148.50 (12)
O1—Lu2—O5 ⁱⁱ	126.65 (7)	P4 ^{xii} —O12—Lu1	147.46 (12)
O1 ⁱ —Lu2—O5 ⁱⁱ	73.25 (7)	P5 ^{xiii} —O13—Lu2	151.04 (13)
O13 ⁱ —Lu2—O5 ⁱⁱⁱ	74.67 (7)	P5—O14—Lu1	156.41 (13)

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

Fig. 1

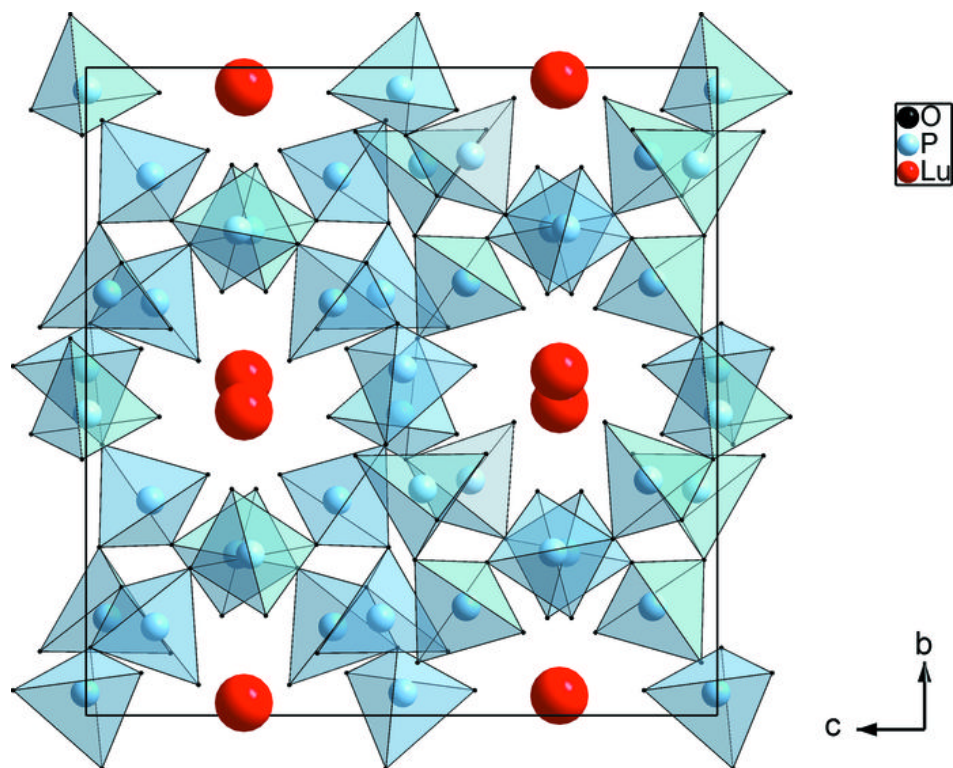


Fig. 2

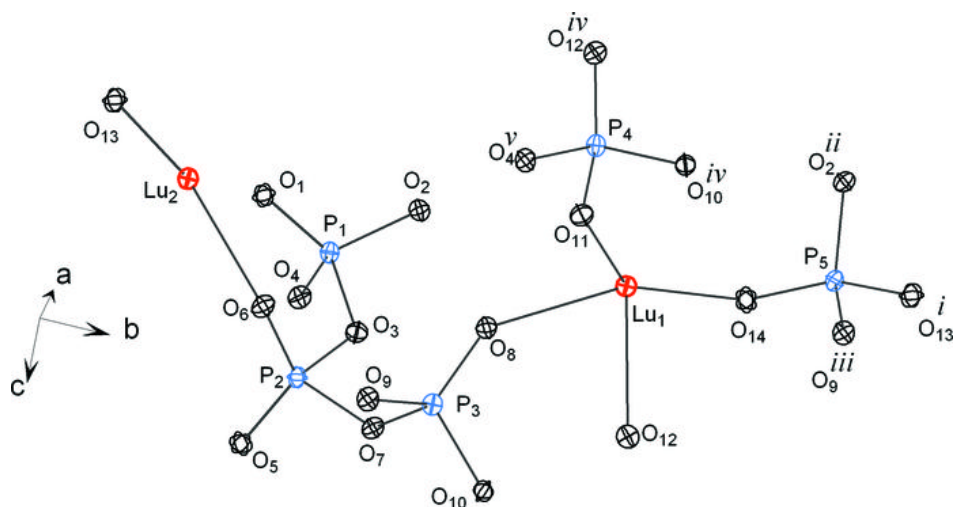


Fig. 3

