

## A new polymorph of Lu(PO<sub>3</sub>)<sub>3</sub>

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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{Lu}-\text{O}) = 0.005$  Å;  $R$  factor = 0.033;  $wR$  factor = 0.061; data-to-parameter ratio = 26.2.

A new polymorph of lutetium polyphosphate, Lu(PO<sub>3</sub>)<sub>3</sub>, was found to be isotopic with the trigonal form of Yb(PO<sub>3</sub>)<sub>3</sub>. Two of the three Lu atoms occupy special positions (Wyckoff positions *3a* and *3b*, site symmetry  $\bar{3}$ ). The atomic arrangement consists of infinite helical polyphosphate chains running along the *c* axis, with a repeat period of 12 PO<sub>4</sub> tetrahedra, joined with LuO<sub>6</sub> octahedra.

### Related literature

For syntheses and optical properties, see: Briche *et al.* (2006); Jouini, Férid, Gacon, Grosvalet *et al.* (2003); Jouini, Férid, Gacon & Trabelsi-Ayadi (2003); Ternane *et al.* (2005); Graia *et al.* (2003); Anisimova *et al.* (1992). For the monoclinic polymorph of Lu(PO<sub>3</sub>)<sub>3</sub>, see: Höppe & Sedlmaier (2007); Yuan *et al.* (2008).

### Experimental

#### Crystal data

Lu(PO <sub>3</sub> ) <sub>3</sub>	$Z = 24$
$M_r = 411.88$	Mo $K\alpha$ radiation
Trigonal, $R\bar{3}$	$\mu = 13.59$ mm <sup>-1</sup>
$a = 20.9106$ (6) Å	$T = 100$ (2) K
$c = 12.0859$ (7) Å	$0.18 \times 0.18 \times 0.17$ mm
$V = 4576.6$ (3) Å <sup>3</sup>	

#### Data collection

Bruker APEXII CCD area-detector diffractometer	25139 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	4170 independent reflections
$T_{\min} = 0.102$ , $T_{\max} = 0.104$	3609 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.054$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$	159 parameters
$wR(F^2) = 0.060$	$\Delta\rho_{\text{max}} = 2.34$ e Å <sup>-3</sup>
$S = 1.05$	$\Delta\rho_{\text{min}} = -2.07$ e Å <sup>-3</sup>
4170 reflections	

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: APEX2; 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: FI2065).

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**supplementary materials**

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## A new polymorph of Lu(PO<sub>3</sub>)<sub>3</sub>

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### Comment

There is considerable scientific and technological interest in the synthesis, structure, and properties of yttrium and rare earth polyphosphates of the formula Ln(PO<sub>3</sub>)<sub>3</sub>, because these compounds offer thermal stability and richness of formulations and structures (Briche *et al.*, 2006, Jouini, Férid, Gacon, Grosvalet *et al.*, 2003, Jouini, Férid, Gacon & Trabelsi-Ayadi, 2003, Ternane *et al.*, 2005, Graia *et al.*, 2003). In this paper, we report the preparation and crystal structure refinement of the polyphosphate Lu(PO<sub>3</sub>)<sub>3</sub>, crystallizing in space group *R*-3. The existence of the trigonal polymorph was originally reported by Anisimova for the Yb(PO<sub>3</sub>)<sub>3</sub> polyphosphate (Anisimova *et al.*, 1992). The monoclinic polymorph of Lu(PO<sub>3</sub>)<sub>3</sub> was recently reported by Höppe and Yuan (Höppe & Sedlmaier, 2007, Yuan *et al.*, 2008). The atomic arrangement of these structures is characterized by a three-dimensional framework built of (PO<sub>3</sub>)<sub>n</sub> chains that are formed by corner-sharing of PO<sub>4</sub> tetrahedra. These two polymorphs differ by the polyphosphate chains configuration. The chains that were observed in monoclinic Lu(PO<sub>3</sub>)<sub>3</sub> form infinite zigzag chains (PO<sub>3</sub>)<sub>n</sub> that extend along *c* with a period of six tetrahedra. In trigonal Lu(PO<sub>3</sub>)<sub>3</sub>, the (PO<sub>3</sub>)<sub>n</sub> chains are helical with a period of 12 tetrahedra (Fig. 1) and are arranged about the 3<sub>1</sub> helical axis. The chains are joined to each other by LuO<sub>6</sub> octahedra (Fig 2.), no oxygen atom is shared between adjacent LuO<sub>6</sub> octahedra. Figure 3 shows the projection of Lu(PO<sub>3</sub>)<sub>3</sub> with anisotropic displacement parameters drawn at the 50% probability level.

### Experimental

Single crystals of Lu(PO<sub>3</sub>)<sub>3</sub> were grown by a flux method. Lutetium oxide was dissolved in an excess of phosphoric acid using the molar ratio Lu:P = 1:20. The resulting solution was heated in a vitreous graphite crucible at 573 K for 5 days. The obtained colourless crystals were then isolated from the acid solution using hot water.

### Refinement

The highest peak and the deepest hole are located 0.75 Å and 0.57 Å, respectively from O10 and Lu3.

### Figures

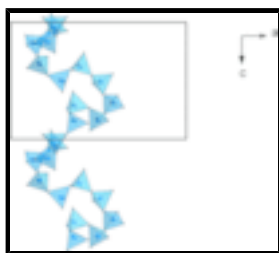


Fig. 1. A projection of the helical (PO<sub>3</sub>)<sub>n</sub> chains along *b*.

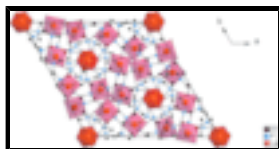


Fig. 2. A projection of  $\text{Lu}(\text{PO}_3)_3$  along  $c$ , showing the arrangement of the  $\text{LuO}_6$  octahedra and  $\text{PO}_4$  tetrahedra.

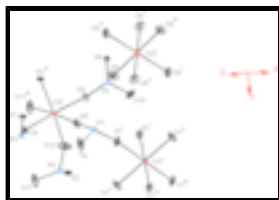


Fig. 3. Projection of the  $\text{Lu}(\text{PO}_3)_3$  polyphosphate, showing the lutetium coordination with displacement ellipsoids drawn at the 50% probability level. [Symmetry codes : (i)  $-y, x-y, z$ ; (ii)  $-x+y, -x, z$ ; (iii)  $-x+1/3, -y+2/3, -z+2/3$ ; (iv)  $y+1/3, -x+y+2/3, -z+2/3$ ; (v)  $x-y+1/3, x+2/3, -z+2/3$ ; (vi)  $-x, -y, -z$ ; (vii)  $-x+y+1/3, -x+2/3, z+2/3$ .]

## lutetium polyphosphate

### Crystal data

$\text{Lu}(\text{PO}_3)_3$	$Z = 24$
$M_r = 411.88$	$F_{000} = 4512$
Trigonal, $R\bar{3}$	$D_x = 3.587 \text{ Mg m}^{-3}$
Hall symbol: $-R\ 3$	Mo $K\alpha$ radiation
$a = 20.9106 (6) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 20.9106 (6) \text{ \AA}$	Cell parameters from 25 reflections
$c = 12.0859 (7) \text{ \AA}$	$\theta = 2.8\text{--}34.1^\circ$
$\alpha = 90^\circ$	$\mu = 13.59 \text{ mm}^{-1}$
$\beta = 90^\circ$	$T = 100 (2) \text{ K}$
$\gamma = 120^\circ$	Cube, colourless
$V = 4576.6 (3) \text{ \AA}^3$	$0.18 \times 0.18 \times 0.17 \text{ mm}$

### Data collection

Bruker APEXII CCD area-detector diffractometer	3609 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.054$
$T = 100(2) \text{ K}$	$\theta_{\text{max}} = 34.2^\circ$
$\omega$ scans	$\theta_{\text{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -32 \rightarrow 32$
$T_{\text{min}} = 0.102, T_{\text{max}} = 0.104$	$k = -32 \rightarrow 32$
25139 measured reflections	$l = -18 \rightarrow 18$
4170 independent reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0212P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.032$	where $P = (F_o^2 + 2F_c^2)/3$
	$(\Delta/\sigma)_{\text{max}} = 0.001$

$wR(F^2) = 0.061$	$\Delta\rho_{\max} = 2.34 \text{ e } \text{\AA}^{-3}$
$S = 1.05$	$\Delta\rho_{\min} = -2.07 \text{ e } \text{\AA}^{-3}$
4170 reflections	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
159 parameters	Extinction coefficient: 0.000061 (8)
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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
Lu1	0.6667	0.3333	0.3333	0.00741 (8)
Lu2	0.6667	0.3333	-0.1667	0.01207 (9)
Lu3	0.440661 (9)	0.365196 (10)	0.096806 (14)	0.00780 (5)
P1	0.63820 (6)	0.45920 (6)	0.16119 (9)	0.00821 (19)
P2	0.50313 (6)	0.54494 (6)	0.16810 (9)	0.0096 (2)
P3	0.39267 (6)	0.30556 (6)	0.37383 (10)	0.0111 (2)
P4	0.50120 (6)	0.25039 (6)	-0.01904 (10)	0.0107 (2)
O1	0.44368 (17)	0.46669 (17)	0.1552 (3)	0.0132 (6)
O2	0.34108 (18)	0.22020 (17)	0.3991 (3)	0.0132 (6)
O3	0.54706 (18)	0.58558 (18)	0.0709 (3)	0.0141 (6)
O4	0.45503 (18)	0.27392 (18)	0.0416 (3)	0.0168 (7)
O5	0.55847 (17)	0.42399 (18)	0.1374 (3)	0.0175 (7)
O6	0.45659 (17)	0.19780 (19)	-0.1185 (3)	0.0155 (7)
O7	0.66627 (18)	0.41823 (18)	0.2253 (3)	0.0190 (7)
O8	0.57293 (19)	0.3097 (2)	-0.0609 (3)	0.0247 (8)
O9	0.6655 (2)	0.5377 (2)	0.2137 (4)	0.0351 (11)
O10	0.5156 (2)	0.1948 (2)	0.0471 (3)	0.0315 (10)
O11	0.3569 (2)	0.3473 (2)	0.4088 (4)	0.0298 (9)
O12	0.4182 (3)	0.3127 (2)	0.2586 (3)	0.0364 (11)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Lu1	0.00684 (11)	0.00684 (11)	0.00854 (19)	0.00342 (6)	0.000	0.000
Lu2	0.01014 (13)	0.01014 (13)	0.0159 (2)	0.00507 (6)	0.000	0.000

## supplementary materials

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Lu3	0.00810 (8)	0.00796 (8)	0.00662 (8)	0.00347 (7)	0.00035 (6)	0.00000 (6)
P1	0.0086 (5)	0.0070 (5)	0.0084 (5)	0.0034 (4)	-0.0005 (4)	-0.0006 (4)
P2	0.0133 (5)	0.0095 (5)	0.0072 (5)	0.0065 (4)	-0.0008 (4)	-0.0007 (4)
P3	0.0150 (5)	0.0121 (5)	0.0095 (5)	0.0093 (4)	0.0006 (4)	0.0032 (4)
P4	0.0115 (5)	0.0132 (5)	0.0106 (5)	0.0084 (4)	-0.0009 (4)	0.0000 (4)
O1	0.0157 (15)	0.0117 (14)	0.0132 (15)	0.0076 (13)	0.0006 (12)	-0.0027 (12)
O2	0.0192 (16)	0.0124 (15)	0.0079 (14)	0.0077 (13)	0.0020 (12)	0.0008 (11)
O3	0.0216 (17)	0.0164 (16)	0.0055 (14)	0.0104 (14)	0.0015 (12)	-0.0001 (12)
O4	0.0141 (16)	0.0152 (16)	0.0215 (18)	0.0076 (13)	0.0028 (13)	-0.0036 (13)
O5	0.0089 (14)	0.0147 (16)	0.0269 (19)	0.0044 (13)	-0.0040 (13)	-0.0065 (14)
O6	0.0120 (15)	0.0265 (18)	0.0117 (15)	0.0125 (14)	-0.0042 (12)	-0.0051 (13)
O7	0.0154 (16)	0.0188 (17)	0.0264 (19)	0.0110 (14)	0.0048 (14)	0.0145 (14)
O8	0.0126 (16)	0.027 (2)	0.028 (2)	0.0050 (15)	0.0040 (14)	-0.0063 (16)
O9	0.023 (2)	0.030 (2)	0.060 (3)	0.0199 (18)	-0.019 (2)	-0.030 (2)
O10	0.063 (3)	0.025 (2)	0.021 (2)	0.033 (2)	-0.0207 (19)	-0.0072 (16)
O11	0.0202 (19)	0.0148 (17)	0.058 (3)	0.0114 (15)	0.0067 (18)	-0.0006 (18)
O12	0.063 (3)	0.022 (2)	0.0141 (19)	0.014 (2)	0.0127 (19)	0.0068 (15)

### Geometric parameters (Å, °)

Lu1—O7 <sup>i</sup>	2.207 (3)	Lu3—O3 <sup>x</sup>	2.229 (3)
Lu1—O7 <sup>ii</sup>	2.207 (3)	P1—O5	1.475 (3)
Lu1—O7 <sup>iii</sup>	2.207 (3)	P1—O7	1.477 (3)
Lu1—O7 <sup>iv</sup>	2.207 (3)	P1—O10 <sup>iii</sup>	1.569 (4)
Lu1—O7	2.207 (3)	P1—O9	1.578 (4)
Lu1—O7 <sup>v</sup>	2.207 (3)	P2—O3	1.472 (3)
Lu2—O8 <sup>vi</sup>	2.180 (3)	P2—O1	1.488 (3)
Lu2—O8 <sup>iv</sup>	2.180 (3)	P2—O6 <sup>xi</sup>	1.585 (3)
Lu2—O8 <sup>iii</sup>	2.180 (3)	P2—O2 <sup>ii</sup>	1.593 (3)
Lu2—O8	2.180 (3)	P3—O11	1.467 (4)
Lu2—O8 <sup>vii</sup>	2.180 (3)	P3—O12	1.472 (4)
Lu2—O8 <sup>viii</sup>	2.180 (3)	P3—O9 <sup>i</sup>	1.573 (4)
Lu3—O11 <sup>ix</sup>	2.134 (3)	P3—O2	1.587 (3)
Lu3—O12	2.176 (4)	P4—O8	1.478 (4)
Lu3—O4	2.180 (3)	P4—O4	1.478 (3)
Lu3—O5	2.189 (3)	P4—O10	1.560 (4)
Lu3—O1	2.207 (3)	P4—O6	1.581 (3)
O7 <sup>i</sup> —Lu1—O7 <sup>ii</sup>	88.57 (14)	O4—Lu3—O5	87.21 (12)
O7 <sup>i</sup> —Lu1—O7 <sup>iii</sup>	180.0	O11 <sup>ix</sup> —Lu3—O1	91.99 (13)
O7 <sup>ii</sup> —Lu1—O7 <sup>iii</sup>	91.43 (14)	O12—Lu3—O1	95.32 (14)
O7 <sup>i</sup> —Lu1—O7 <sup>iv</sup>	91.43 (14)	O4—Lu3—O1	171.70 (12)
O7 <sup>ii</sup> —Lu1—O7 <sup>iv</sup>	180.0	O5—Lu3—O1	84.56 (12)
O7 <sup>iii</sup> —Lu1—O7 <sup>iv</sup>	88.57 (14)	O11 <sup>ix</sup> —Lu3—O3 <sup>x</sup>	88.69 (15)
O7 <sup>i</sup> —Lu1—O7	91.43 (14)	O12—Lu3—O3 <sup>x</sup>	174.96 (15)
O7 <sup>ii</sup> —Lu1—O7	91.43 (14)	O4—Lu3—O3 <sup>x</sup>	95.25 (12)

O7 <sup>iii</sup> —Lu1—O7	88.57 (14)	O5—Lu3—O3 <sup>x</sup>	96.14 (13)
O7 <sup>iv</sup> —Lu1—O7	88.57 (14)	O1—Lu3—O3 <sup>x</sup>	84.56 (12)
O7 <sup>i</sup> —Lu1—O7 <sup>v</sup>	88.57 (14)	O5—P1—O7	119.4 (2)
O7 <sup>ii</sup> —Lu1—O7 <sup>v</sup>	88.57 (14)	O5—P1—O10 <sup>iii</sup>	106.7 (2)
O7 <sup>iii</sup> —Lu1—O7 <sup>v</sup>	91.43 (14)	O7—P1—O10 <sup>iii</sup>	110.3 (2)
O7 <sup>iv</sup> —Lu1—O7 <sup>v</sup>	91.43 (14)	O5—P1—O9	109.15 (19)
O7—Lu1—O7 <sup>v</sup>	180.0	O7—P1—O9	110.6 (2)
O8 <sup>vi</sup> —Lu2—O8 <sup>iv</sup>	180.0	O10 <sup>iii</sup> —P1—O9	98.7 (3)
O8 <sup>vi</sup> —Lu2—O8 <sup>iii</sup>	90.92 (15)	O3—P2—O1	119.23 (19)
O8 <sup>iv</sup> —Lu2—O8 <sup>iii</sup>	89.08 (15)	O3—P2—O6 <sup>xi</sup>	105.71 (18)
O8 <sup>vi</sup> —Lu2—O8	90.92 (15)	O1—P2—O6 <sup>xi</sup>	109.44 (19)
O8 <sup>iv</sup> —Lu2—O8	89.08 (15)	O3—P2—O2 <sup>ii</sup>	112.19 (18)
O8 <sup>iii</sup> —Lu2—O8	89.08 (15)	O1—P2—O2 <sup>ii</sup>	106.01 (18)
O8 <sup>vi</sup> —Lu2—O8 <sup>vii</sup>	89.08 (15)	O6 <sup>xi</sup> —P2—O2 <sup>ii</sup>	103.12 (18)
O8 <sup>iv</sup> —Lu2—O8 <sup>vii</sup>	90.92 (15)	O11—P3—O12	118.6 (3)
O8 <sup>iii</sup> —Lu2—O8 <sup>vii</sup>	180.0	O11—P3—O9 <sup>i</sup>	105.0 (2)
O8—Lu2—O8 <sup>vii</sup>	90.92 (15)	O12—P3—O9 <sup>i</sup>	108.6 (3)
O8 <sup>vi</sup> —Lu2—O8 <sup>viii</sup>	89.08 (15)	O11—P3—O2	110.6 (2)
O8 <sup>iv</sup> —Lu2—O8 <sup>viii</sup>	90.92 (15)	O12—P3—O2	107.7 (2)
O8 <sup>iii</sup> —Lu2—O8 <sup>viii</sup>	90.92 (15)	O9 <sup>i</sup> —P3—O2	105.49 (19)
O8—Lu2—O8 <sup>viii</sup>	180.0	O8—P4—O4	116.6 (2)
O8 <sup>vii</sup> —Lu2—O8 <sup>viii</sup>	89.08 (15)	O8—P4—O10	107.9 (2)
O11 <sup>ix</sup> —Lu3—O12	86.28 (18)	O4—P4—O10	113.3 (2)
O11 <sup>ix</sup> —Lu3—O4	96.31 (13)	O8—P4—O6	108.8 (2)
O12—Lu3—O4	85.59 (14)	O4—P4—O6	110.62 (18)
O11 <sup>ix</sup> —Lu3—O5	173.76 (15)	O10—P4—O6	97.91 (19)
O12—Lu3—O5	88.86 (16)		

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

Fig. 1

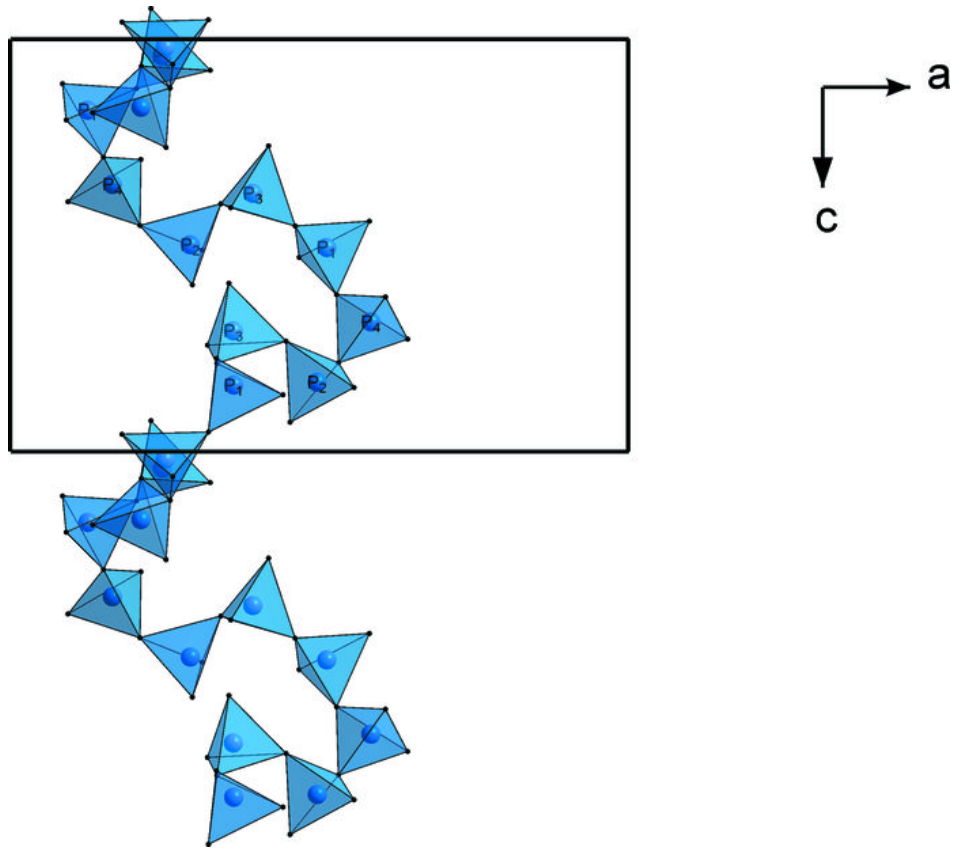




Fig. 2

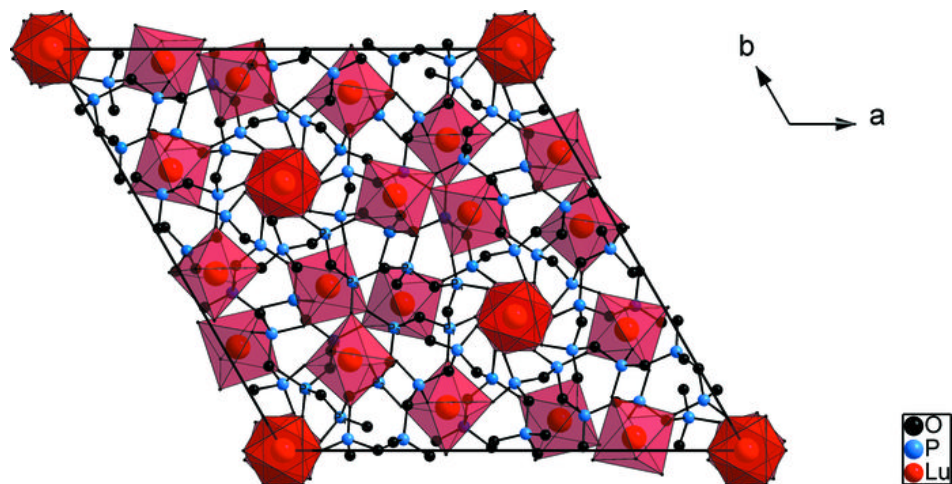


Fig. 3

