

Silver europium(III) polyphosphate

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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{P}-\text{O}) = 0.005$ Å; R factor = 0.042; wR factor = 0.109; data-to-parameter ratio = 12.3.

Europium(III) silver polyphosphate, $\text{AgEu}(\text{PO}_3)_4$, was prepared by the flux method. The atomic arrangement is built up by infinite $(\text{PO}_3)_n$ chains (periodicity of 4) extending along the c axis. These chains are joined to each other by EuO_8 dodecahedra. The Ag^+ cations are located in the voids of this arrangement and are surrounded by five oxygen atoms in a distorted [4+1] coordination.

Related literature

For isotypic $\text{AgNd}(\text{PO}_3)_4$, see: Trunov *et al.* (1990). For related structures, see: Yamada *et al.* (1974); Hashimoto *et al.* (1991); Horchani *et al.* (2003); Durif (1995); Averbuch-Pouchot & Bagieu-Boucher (1987); Férid (2006).

Experimental

Crystal data

$\text{AgEu}(\text{PO}_3)_4$
 $M_r = 575.72$
 Monoclinic, $P2_1/n$
 $a = 9.9654$ (3) Å
 $b = 13.1445$ (7) Å

$c = 7.2321$ (3) Å
 $\beta = 90.42$ (1)°
 $V = 947.31$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 9.37$ mm⁻¹
 $T = 298$ (2) K

0.19 × 0.18 × 0.17 mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.167$, $T_{\max} = 0.201$

3371 measured reflections
 2019 independent reflections
 1704 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.109$
 $S = 1.03$
 2019 reflections

164 parameters
 $\Delta\rho_{\text{max}} = 2.43$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.06$ e Å⁻³

Data collection: COLLECT (Nonius, 2001); cell refinement: DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO/SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg, 1998); 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: BR2089).

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supplementary materials

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Comment

In the last decades, investigation of the synthesis and characterization of rare earth polyphosphates has gained much attention due to their potential applications in diverse areas such as phosphors and laser materials (Yamada *et al.*, 1974; Hashimoto *et al.*, 1991; Horchani *et al.*, 2003). In aim to study the condensed phosphates of rare earth and monovalent cations of general formula $MILn(PO_3)_4$ (with MI = monovalent cation)(Durif, 1995), ($Ln = Eu, Er, Yb$), we have synthesized single crystals of silver europium polyphosphate and investigated its crystalline structure. The atomic arrangement of this structure is characterized by a three-dimensional framework built of $(PO_3)_n$ chains that are formed by corner-sharing of PO_4 tetrahedra. Eu^{3+} and Ag^+ cations alternate in the middle of four such chains with $Eu-Ag$ distances of 3.64 (7) Å (figures 1,3). The EuO_8 dodecahedra are isolated from each other and the distances $Eu-O$ are arranged in interval 2.355 (5)- 2.508 (5)Å (figure 2). The polyphosphate chains display two types of distances, P—O terminal ranging from 1.479 (5) to 1.505 (5)Å and P—O bridging, ranging from 1.585 (5) to 1.609 (5)Å. These distances are comparable with those reported for other condensed phosphates (Durif, 1995; Averbuch-Pouchot & Bagieu Beucher, 1987; Férid (2006). The structural study reported for silver neodymium polyphosphate $AgNd(PO_3)_4$ (Trunov *et al.*, 1990) showed that the compound crystallize in the $P2_1/n$ space group and has similar unit cell parameters compared to $AgEu(PO_3)_4$.

Experimental

Single crystals of $AgEu(PO_3)_4$ were prepared by flux method. A mixture of Ag_2CO_3 (3 g), $EuCl_3 \cdot 6H_2O$ (0.5 g) and H_3PO_4 (85%, 17 ml), was progressively heated in a vitreous carbon crucible to 473 K for 12 h. The temperature was then raised and kept at 600 K for 16 days after that, the furnace was slowly cooled until the room temperature. The product was washed with boiling water to separate colorless single crystals from phosphoric acid.

Refinement

The highest peak and the deepest hole are located 1.22 Å and 0.73 Å, respectively, from Ag and Eu.

Figures

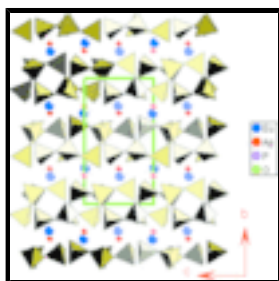


Fig. 1. : The structural arrangement of $AgEu(PO_3)_4$ along a axis.

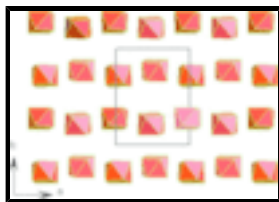


Fig. 2. : Projection of EuO_8 dodecahedra viewed along $[0\ 0\ 1]$ direction.

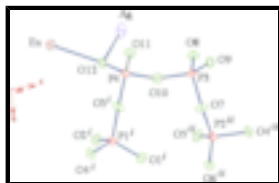


Fig. 3. : The sequence of PO_4 tetrahedra in $\text{AgEu}(\text{PO}_3)_4$, with displacement ellipsoids drawn at the 50% probability level. [Symmetry codes: (i) $1-x, 2-y, 2-z$; (ii) $1-x/2, 2-y, 3-z$]

Europium(III) silver polyphosphate

Crystal data

$\text{AgEu}(\text{PO}_3)_4$

$M_r = 575.72$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2yn$

$a = 9.9654\ (3)\ \text{\AA}$

$b = 13.1445\ (7)\ \text{\AA}$

$c = 7.2321\ (3)\ \text{\AA}$

$\beta = 90.42\ (1)^\circ$

$V = 947.31\ (7)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 1064$

$D_x = 4.037\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 25 reflections

$\theta = 2.4\text{--}30.1^\circ$

$\mu = 9.37\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Prism, colorless

$0.19 \times 0.18 \times 0.17\ \text{mm}$

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298\ \text{K}$

φ and ω scans

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

$T_{\min} = 0.167, T_{\max} = 0.201$

3371 measured reflections

2019 independent reflections

1704 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 3.8^\circ$

$h = -12 \rightarrow 12$

$k = -16 \rightarrow 15$

$l = -9 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

Secondary atom site location: difference Fourier map

$$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 1.0941P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$wR(F^2) = 0.109$	$\Delta\rho_{\max} = 2.43 \text{ e } \text{\AA}^{-3}$
$S = 1.03$	$\Delta\rho_{\min} = -2.06 \text{ e } \text{\AA}^{-3}$
2019 reflections	Extinction correction: SHELXL08 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
164 parameters	Extinction coefficient: 0.00014 (2)
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	$U_{\text{iso}}^*/U_{\text{eq}}$
Eu	0.52254 (4)	0.78212 (2)	0.51227 (4)	0.01862 (19)
Ag	0.43355 (7)	0.77670 (5)	1.00017 (8)	0.0322 (2)
P1	0.25190 (18)	0.90008 (12)	1.2536 (2)	0.0168 (4)
P2	0.19511 (18)	0.87338 (12)	1.6486 (2)	0.0167 (4)
P3	0.79921 (18)	0.90983 (12)	1.2635 (2)	0.0172 (4)
P4	0.73739 (18)	0.88594 (12)	0.8738 (2)	0.0162 (4)
O1	0.1995 (5)	0.8356 (3)	1.1018 (6)	0.0241 (11)
O2	0.3997 (5)	0.8951 (4)	1.2904 (7)	0.0243 (11)
O3	0.2039 (5)	1.0133 (3)	1.2168 (7)	0.0240 (11)
O4	0.1629 (5)	0.8769 (3)	1.4335 (6)	0.0198 (10)
O5	0.3444 (5)	0.8678 (4)	1.6767 (7)	0.0218 (10)
O6	0.1062 (6)	0.7906 (3)	1.7231 (6)	0.0228 (11)
O7	0.8644 (5)	1.0214 (3)	1.2777 (7)	0.0193 (10)
O8	0.9144 (5)	0.8395 (3)	1.2329 (6)	0.0236 (10)
O9	0.7055 (5)	0.8919 (3)	1.4217 (6)	0.0218 (10)
O10	0.7085 (5)	0.9202 (3)	1.0822 (6)	0.0203 (10)
O11	0.8483 (5)	0.8097 (3)	0.8671 (7)	0.0218 (10)
O12	0.6047 (5)	0.8557 (3)	0.7933 (6)	0.0198 (10)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Eu	0.0183 (3)	0.0189 (2)	0.0186 (2)	-0.00063 (12)	-0.00048 (15)	-0.00108 (12)
Ag	0.0262 (4)	0.0456 (4)	0.0249 (3)	0.0034 (2)	0.0008 (2)	-0.0088 (2)
P1	0.0201 (9)	0.0138 (7)	0.0165 (7)	-0.0001 (6)	0.0005 (6)	0.0001 (6)

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P2	0.0173 (9)	0.0155 (8)	0.0173 (8)	0.0007 (6)	-0.0001 (6)	-0.0002 (6)
P3	0.0206 (9)	0.0143 (7)	0.0168 (8)	0.0004 (6)	-0.0017 (6)	-0.0002 (6)
P4	0.0162 (9)	0.0160 (8)	0.0165 (8)	-0.0001 (6)	-0.0020 (6)	-0.0012 (6)
O1	0.034 (3)	0.019 (2)	0.019 (2)	0.004 (2)	-0.002 (2)	-0.0060 (19)
O2	0.023 (3)	0.026 (2)	0.023 (2)	0.000 (2)	-0.002 (2)	-0.002 (2)
O3	0.028 (3)	0.016 (2)	0.028 (3)	0.007 (2)	-0.004 (2)	0.0052 (19)
O4	0.019 (3)	0.025 (2)	0.016 (2)	0.0000 (19)	-0.0004 (18)	0.0039 (18)
O5	0.017 (3)	0.024 (2)	0.025 (2)	0.0021 (19)	-0.003 (2)	-0.007 (2)
O6	0.029 (3)	0.021 (2)	0.018 (2)	-0.005 (2)	0.000 (2)	0.0038 (18)
O7	0.013 (2)	0.014 (2)	0.031 (3)	-0.0005 (18)	0.0063 (19)	-0.003 (2)
O8	0.027 (3)	0.018 (2)	0.026 (2)	0.003 (2)	-0.001 (2)	-0.002 (2)
O9	0.027 (3)	0.023 (2)	0.016 (2)	-0.004 (2)	0.0040 (19)	-0.0026 (18)
O10	0.017 (2)	0.020 (2)	0.024 (2)	0.0009 (19)	-0.0016 (18)	-0.0023 (19)
O11	0.022 (3)	0.018 (2)	0.025 (2)	0.007 (2)	-0.001 (2)	0.002 (2)
O12	0.023 (3)	0.021 (2)	0.015 (2)	0.005 (2)	-0.0053 (19)	-0.0005 (18)

Geometric parameters (Å, °)

Eu—O11 ⁱ	2.355 (5)	P2—O4	1.587 (5)
Eu—O12	2.390 (4)	P2—O7 ^{vi}	1.598 (5)
Eu—O9 ⁱⁱ	2.420 (5)	P3—O8	1.492 (5)
Eu—O5 ⁱⁱ	2.422 (5)	P3—O9	1.500 (5)
Eu—O1 ⁱⁱⁱ	2.430 (5)	P3—O10	1.593 (5)
Eu—O6 ^{iv}	2.451 (5)	P3—O7	1.607 (5)
Eu—O2 ⁱⁱ	2.500 (5)	P4—O11	1.493 (5)
Eu—O8 ⁱ	2.508 (5)	P4—O12	1.495 (5)
Eu—Ag	3.6453 (7)	P4—O3 ^{vii}	1.592 (5)
Eu—Ag ⁱⁱ	3.8025 (7)	P4—O10	1.602 (5)
Ag—O8 ⁱ	2.470 (5)	O1—Eu ^{viii}	2.430 (5)
Ag—O12	2.503 (5)	O2—Eu ^v	2.500 (5)
Ag—O6 ⁱⁱⁱ	2.511 (5)	O3—P4 ^{vii}	1.592 (5)
Ag—O1	2.570 (5)	O5—Eu ^v	2.422 (5)
Ag—Eu ^v	3.8025 (7)	O6—Eu ^{ix}	2.451 (5)
P1—O1	1.479 (5)	O6—Ag ^{viii}	2.511 (5)
P1—O2	1.496 (6)	O7—P2 ^{vi}	1.598 (5)
P1—O3	1.585 (5)	O8—Ag ^x	2.470 (5)
P1—O4	1.609 (5)	O8—Eu ^x	2.508 (5)
P2—O5	1.502 (5)	O9—Eu ^v	2.420 (5)
P2—O6	1.505 (5)	O11—Eu ^x	2.355 (5)
O11 ⁱ —Eu—O12	146.43 (17)	O12—Ag—Eu	40.66 (10)
O11 ⁱ —Eu—O9 ⁱⁱ	137.70 (16)	O6 ⁱⁱⁱ —Ag—Eu	117.27 (12)
O12—Eu—O9 ⁱⁱ	74.62 (16)	O1—Ag—Eu	119.92 (10)
O11 ⁱ —Eu—O5 ⁱⁱ	85.21 (16)	O8 ⁱ —Ag—Eu ^v	142.17 (11)
O12—Eu—O5 ⁱⁱ	69.01 (16)	O12—Ag—Eu ^v	114.81 (10)

O9 ⁱⁱ —Eu—O5 ⁱⁱ	114.34 (16)	O6 ⁱⁱⁱ —Ag—Eu ^v	39.40 (11)
O11 ⁱ —Eu—O1 ⁱⁱⁱ	108.89 (17)	O1—Ag—Eu ^v	85.49 (10)
O12—Eu—O1 ⁱⁱⁱ	77.74 (15)	Eu—Ag—Eu ^v	152.34 (2)
O9 ⁱⁱ —Eu—O1 ⁱⁱⁱ	84.55 (16)	O1—P1—O2	116.6 (3)
O5 ⁱⁱ —Eu—O1 ⁱⁱⁱ	134.29 (16)	O1—P1—O3	108.0 (3)
O11 ⁱ —Eu—O6 ^{iv}	70.99 (18)	O2—P1—O3	111.5 (3)
O12—Eu—O6 ^{iv}	140.07 (18)	O1—P1—O4	107.3 (3)
O9 ⁱⁱ —Eu—O6 ^{iv}	74.93 (16)	O2—P1—O4	113.3 (3)
O5 ⁱⁱ —Eu—O6 ^{iv}	148.82 (17)	O3—P1—O4	98.4 (3)
O1 ⁱⁱⁱ —Eu—O6 ^{iv}	74.24 (16)	O5—P2—O6	120.1 (3)
O11 ⁱ —Eu—O2 ⁱⁱ	70.26 (16)	O5—P2—O4	109.1 (3)
O12—Eu—O2 ⁱⁱ	117.94 (15)	O6—P2—O4	104.9 (3)
O9 ⁱⁱ —Eu—O2 ⁱⁱ	80.70 (17)	O5—P2—O7 ^{vi}	111.5 (3)
O5 ⁱⁱ —Eu—O2 ⁱⁱ	71.45 (17)	O6—P2—O7 ^{vi}	106.6 (3)
O1 ⁱⁱⁱ —Eu—O2 ⁱⁱ	154.17 (16)	O4—P2—O7 ^{vi}	103.2 (3)
O6 ^{iv} —Eu—O2 ⁱⁱ	81.49 (16)	O8—P3—O9	120.0 (3)
O11 ⁱ —Eu—O8 ⁱ	68.78 (16)	O8—P3—O10	111.3 (3)
O12—Eu—O8 ⁱ	82.12 (15)	O9—P3—O10	106.8 (3)
O9 ⁱⁱ —Eu—O8 ⁱ	151.72 (16)	O8—P3—O7	105.3 (3)
O5 ⁱⁱ —Eu—O8 ⁱ	70.38 (16)	O9—P3—O7	110.4 (3)
O1 ⁱⁱⁱ —Eu—O8 ⁱ	74.87 (16)	O10—P3—O7	101.6 (3)
O6 ^{iv} —Eu—O8 ⁱ	116.40 (15)	O11—P4—O12	117.5 (3)
O2 ⁱⁱ —Eu—O8 ⁱ	125.20 (17)	O11—P4—O3 ^{vii}	105.7 (3)
O11 ⁱ —Eu—Ag	103.74 (12)	O12—P4—O3 ^{vii}	112.8 (3)
O12—Eu—Ag	43.03 (12)	O11—P4—O10	111.0 (3)
O9 ⁱⁱ —Eu—Ag	117.57 (11)	O12—P4—O10	106.1 (3)
O5 ⁱⁱ —Eu—Ag	49.41 (11)	O3 ^{vii} —P4—O10	102.9 (3)
O1 ⁱⁱⁱ —Eu—Ag	84.88 (11)	P1—O1—Eu ^{viii}	144.5 (3)
O6 ^{iv} —Eu—Ag	154.81 (10)	P1—O1—Ag	93.9 (3)
O2 ⁱⁱ —Eu—Ag	120.77 (12)	Eu ^{viii} —O1—Ag	112.98 (17)
O8 ⁱ —Eu—Ag	42.51 (10)	P1—O2—Eu ^v	128.1 (3)
O11 ⁱ —Eu—Ag ⁱⁱ	52.44 (12)	P1—O3—P4 ^{vii}	137.7 (4)
O12—Eu—Ag ⁱⁱ	155.75 (11)	P2—O4—P1	133.6 (3)
O9 ⁱⁱ —Eu—Ag ⁱⁱ	85.32 (11)	P2—O5—Eu ^v	133.2 (3)
O5 ⁱⁱ —Eu—Ag ⁱⁱ	108.69 (11)	P2—O6—Eu ^{ix}	142.4 (3)
O1 ⁱⁱⁱ —Eu—Ag ⁱⁱ	114.28 (11)	P2—O6—Ag ^{viii}	115.3 (2)
O6 ^{iv} —Eu—Ag ⁱⁱ	40.55 (12)	Eu ^{ix} —O6—Ag ^{viii}	100.06 (18)
O2 ⁱⁱ —Eu—Ag ⁱⁱ	43.64 (11)	P2 ^{vi} —O7—P3	131.3 (3)
O8 ⁱ —Eu—Ag ⁱⁱ	120.64 (11)	P3—O8—Ag ^x	108.8 (3)
Ag—Eu—Ag ⁱⁱ	152.34 (2)	P3—O8—Eu ^x	145.5 (3)
O8 ⁱ —Ag—O12	80.67 (15)	Ag ^x —O8—Eu ^x	94.16 (15)

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O8 ⁱ —Ag—O6 ⁱⁱⁱ	109.46 (15)	P3—O9—Eu ^v	140.7 (3)
O12—Ag—O6 ⁱⁱⁱ	93.64 (16)	P3—O10—P4	130.2 (3)
O8 ⁱ —Ag—O1	110.19 (16)	P4—O11—Eu ^x	150.8 (3)
O12—Ag—O1	132.06 (14)	P4—O12—Eu	137.3 (3)
O6 ⁱⁱⁱ —Ag—O1	122.79 (16)	P4—O12—Ag	118.8 (2)
O8 ⁱ —Ag—Eu	43.33 (11)	Eu—O12—Ag	96.31 (16)

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

Fig. 1

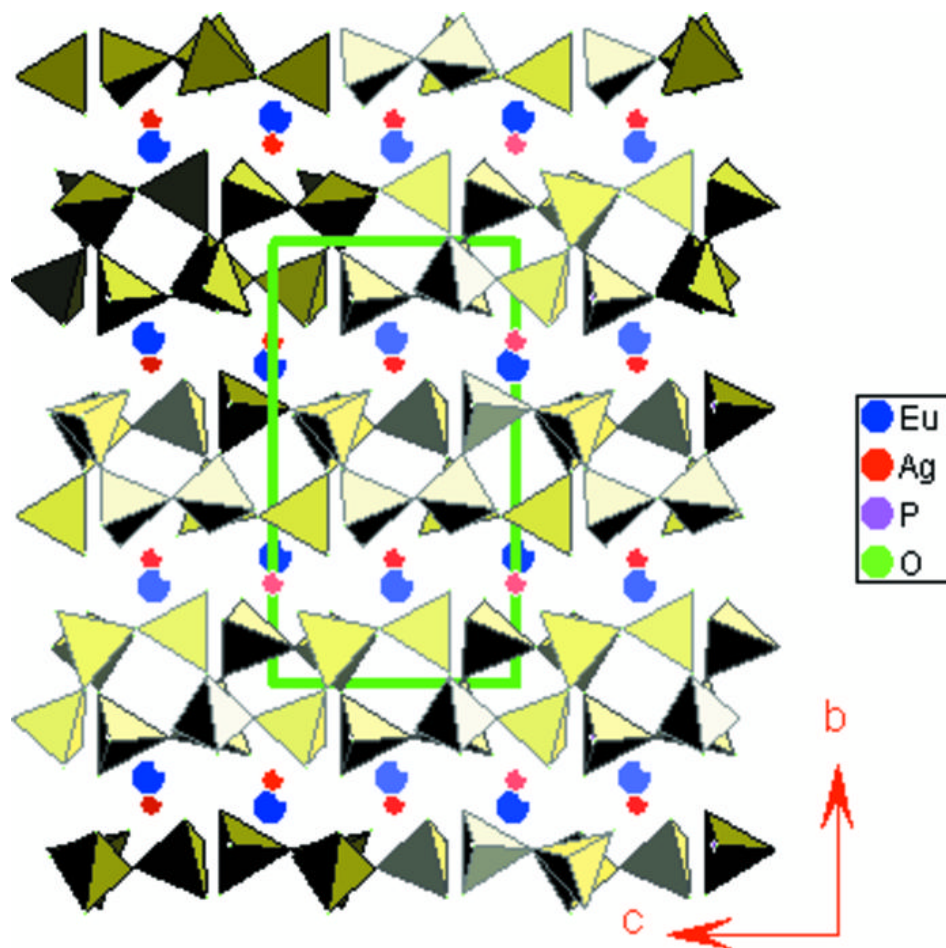


Fig. 2

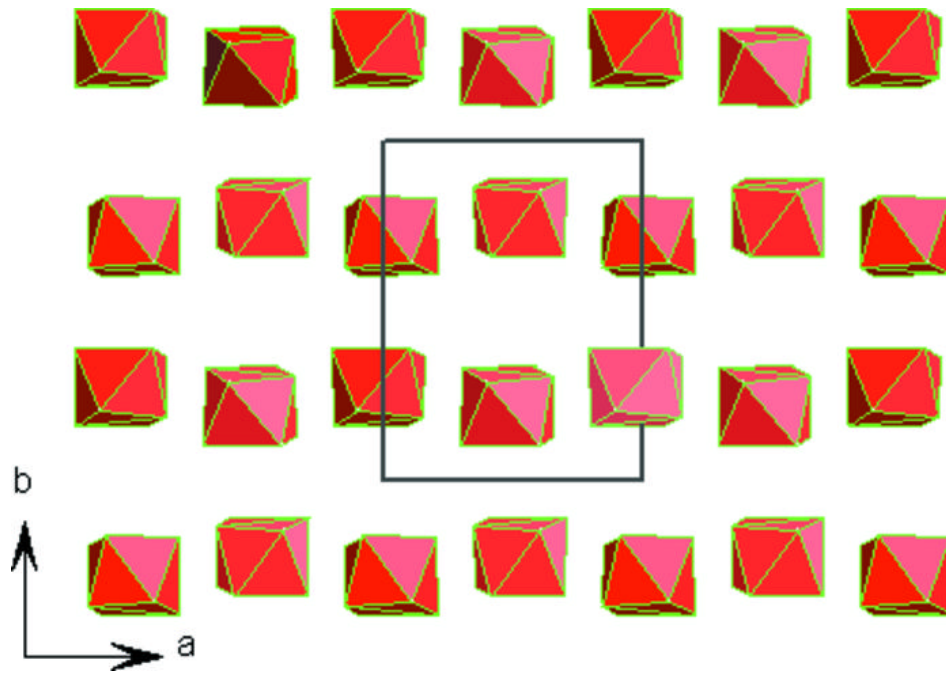


Fig. 3

