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Disilver(I) tricobalt(II) hydrogenphosphate bis(phosphate), $Ag_2Co_3(HPO_4)(PO_4)_2$

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Key indicators: single-crystal X-ray study; T = 296 K; mean σ(P–O) = 0.004 Å; R factor = 0.028; wR factor = 0.064; data-to-parameter ratio = 14.0.

$Ag_2Co_3(HPO_4)(PO_4)_2$ contains CoO_6 octahedra and phosphate groups linked to form a three-dimensional network defining tunnels parallel to the a axis that are occupied by Ag^+ ions.

Related literature

Compounds prepared hydrothermally in the Ag_2O–MO–P_2O_5 (M = divalent cation) system include $AgMg_3(PO_4)(HPO_4)_2$ (Assani *et al.*, 2011*a*), $AgMn_3(PO_4)(HPO_4)_2$ (Leroux *et al.*, 1995), $AgCo_3(PO_4)(HPO_4)_2$ (Guesmi & Driss, 2002), $AgNi_3(PO_4)(HPO_4)_2$ (Ben Smail & Jouini, 2002), $Ag_2Ni_3(HPO_4)(PO_4)_2$ (Assani *et al.*, 2011*b*) and γ-$AgZnPO_4$ (Assani *et al.*, 2010).

Experimental

Crystal data

$Ag_2Co_3(HPO_4)(PO_4)_2$
M_r = 678.44
Orthorhombic, *Ima*2
a = 12.9814 (4) Å
b = 6.5948 (2) Å
c = 10.7062 (3) Å
V = 916.55 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 10.11 mm^{-1}
T = 296 K
0.26 × 0.12 × 0.09 mm

Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan (*MULABS*; Blessing, 1995)
T_{min} = 0.365, T_{max} = 0.424
3966 measured reflections
1388 independent reflections
1368 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.064
S = 1.05
1388 reflections
99 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.81 e Å^{-3}
$\Delta\rho_{min}$ = −1.54 e Å^{-3}
Absolute structure: Flack (1983), 653 Friedel pairs
Flack parameter: 0.55 (3)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O4^{i}	0.86	1.86	2.626 (7)	148

Symmetry code: (i) $-x-\frac{1}{2}, y, z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2119).

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supplementary materials

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Comment

Compounds prepared hydrothermally in the Ag_2O–MO–P_2O_5 (M = divalent cation) systems include $AgMg_3(PO_4)(HPO_4)_2$ (Assani *et al.*, 2011*a*), $AgMn_3(PO_4)(HPO_4)_2$ (Leroux *et al.*, 1995), $AgCo_3(PO_4)(HPO_4)_2$ (Guesmi & Driss, 2002), $AgNi_3(PO_4)(HPO_4)_2$ (Ben Smail & Jouini, 2002), $Ag_2Ni_3(HPO_4)(PO_4)_2$ (Assani *et al.*, 2011*b*), and γ-$AgZnPO_4$ (Assani *et al.*, 2010). $Ag_2Co_3(HPO_4)(PO_4)_2$, isostructural to the Ni analogue, contains CoO_6 octahedra and PO_4 and $PO_3(OH)$ tetrahedra which share corners and edges to form a three-dimensional framework (Fig. 1). Two types of tunnels aligned parallel to the *a*-direction accommodate Ag^+ cations (Fig. 2).

Experimental

A mixture of 0.0849 g $AgNO_3$, 0.0529 g $CoCO_3.Co(OH)_2$, 10 mL of 85 wt.% H_3PO_4, and 10 mL of distilled water was placed in a 23-mL Teflon-lined autoclave, which was heated at 468 K under autogeneous pressure for two days. Pink crystals of the title compound were obtained after the product was filtered, washed with deionized water, and dried in air.

Refinement

The O-bound H atom was initially located in a difference map and refined with O—H distance restraints of 0.86 (1) in a riding model approximation with U_{iso}(H) set to 1.2U_{eq}(O). The highest and deepest hole in the final difference Fourier map are located at 0.70 Å and 0.51 Å, respectively, from Ag1.

Figures

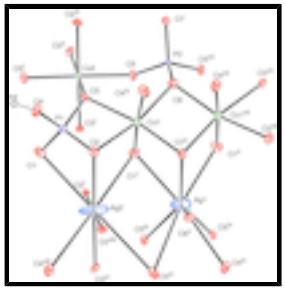

Fig. 1. Connectivity of metal-centred coordination polyhedra in $Ag_2Co_3(HPO_4)(PO_4)_2$. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) -*x*, -*y* + 1, *z*; (ii) *x* + 1/2, -*y*+ 1, *z*; (iii) *x*, -*y* + 3/2, *z* - 1/2; (iv) -*x* + 1/2, -*y* + 3/2, *z* - 1/2; (v) -*x* + 1/2, -*y* + 1/2, *z* - 1/2; (vi) -*x*, *y* + 1/2, *z* - 1/2; (vii) *x* + 1/2, *y* + 1/2, *z* - 1/2; (viii) *x*, -*y* + 1/2, *z* - 1/2; (ix) -*x*, -*y*, *z*; (*x*) -*x*, *y* + 1/2, *z* + 1/2; (xi) *x*, -*y* + 1/2, *z* + 1/2; (xii) -*x* + 1/2, *y*, *z*.

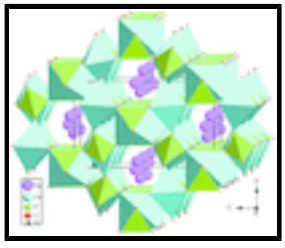

Fig. 2. Polyhedral representation of $Ag_2Co_3(HPO_4)(PO_4)_2$, showing tunnels running along the a direction at *x* 1/2 0 and *x* 0 1/2.

Disilver(I) tricobalt(II) hydrogenphosphate bis(phosphate)

Crystal data

$Ag_2Co_3(HPO_4)(PO_4)_2$	$F(000) = 1268$
$M_r = 678.44$	$D_x = 4.917$ Mg m^{-3}
Orthorhombic, *Ima*2	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: I 2 -2a	Cell parameters from 1388 reflections
$a = 12.9814$ (4) Å	$\theta = 3.1$–$30.0°$
$b = 6.5948$ (2) Å	$\mu = 10.11$ mm^{-1}
$c = 10.7062$ (3) Å	$T = 296$ K
$V = 916.55$ (5) Å^3	Prism, pink
$Z = 4$	0.26 × 0.12 × 0.09 mm

Data collection

Bruker X8 APEX diffractometer	1388 independent reflections
Radiation source: fine-focus sealed tube	1368 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.021$
φ and ω scans	$\theta_{max} = 30.0°$, $\theta_{min} = 3.1°$
Absorption correction: multi-scan (*MULABS*; Blessing, 1995)	$h = -17\rightarrow18$
$T_{min} = 0.365$, $T_{max} = 0.424$	$k = -3\rightarrow9$
3966 measured reflections	$l = -14\rightarrow15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.064$	$w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 2.5641P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
1388 reflections	$\Delta\rho_{max} = 1.81$ e Å^{-3}
99 parameters	$\Delta\rho_{min} = -1.54$ e Å^{-3}
1 restraint	Absolute structure: Flack (1983), 653 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.55 (3)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ag1	0.2500	0.61215 (8)	−0.01381 (7)	0.03097 (16)	
Ag2	0.0000	0.5000	−0.03770 (5)	0.0448 (2)	
Co1	0.13632 (3)	0.24907 (9)	0.20816 (6)	0.00759 (11)	
Co2	0.0000	0.5000	0.45678 (7)	0.00474 (13)	
P1	−0.07308 (7)	0.25700 (17)	0.20656 (12)	0.00728 (17)	
P2	0.2500	0.40742 (18)	0.45614 (14)	0.0051 (2)	
O1	−0.1344 (3)	0.4442 (5)	0.1740 (3)	0.0117 (6)	
O2	0.0039 (3)	0.2072 (5)	0.1002 (3)	0.0084 (6)	
O3	0.0017 (3)	0.2766 (5)	0.3204 (3)	0.0078 (6)	
O4	−0.1489 (3)	0.0787 (5)	0.2349 (3)	0.0116 (7)	
O5	0.15460 (18)	0.5409 (4)	0.4551 (3)	0.0102 (5)	
O6	0.2500	0.2616 (7)	0.3410 (4)	0.0109 (11)	
O7	0.2500	0.2663 (7)	0.5736 (4)	0.0083 (10)	
H4	−0.2103	0.0633	0.2635	0.010*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ag1	0.0498 (3)	0.0187 (2)	0.0244 (3)	0.000	0.000	0.0049 (2)
Ag2	0.1126 (6)	0.0094 (2)	0.0124 (3)	−0.0022 (2)	0.000	0.000
Co1	0.00546 (19)	0.0103 (2)	0.0070 (2)	0.0005 (2)	0.0001 (2)	−0.00120 (18)
Co2	0.0051 (2)	0.0051 (3)	0.0040 (3)	0.00074 (19)	0.000	0.000
P1	0.0069 (3)	0.0078 (4)	0.0071 (4)	0.0000 (4)	−0.0003 (5)	0.0005 (4)
P2	0.0043 (5)	0.0066 (5)	0.0044 (6)	0.000	0.000	−0.0005 (5)
O1	0.0133 (15)	0.0094 (14)	0.0124 (14)	0.0018 (11)	−0.0027 (10)	0.0002 (11)
O2	0.0096 (17)	0.0072 (12)	0.0083 (14)	−0.0004 (13)	−0.0014 (11)	−0.0031 (13)
O3	0.0080 (17)	0.0091 (14)	0.0063 (13)	0.0030 (12)	−0.0020 (10)	−0.0015 (12)
O4	0.0107 (17)	0.0083 (15)	0.0159 (18)	−0.0018 (11)	0.0039 (10)	0.0001 (10)
O5	0.0067 (9)	0.0115 (10)	0.0123 (13)	0.0014 (9)	0.0003 (12)	0.0000 (12)
O6	0.011 (3)	0.014 (2)	0.008 (2)	0.000	0.000	−0.0033 (15)
O7	0.007 (3)	0.010 (2)	0.0083 (19)	0.000	0.000	0.0021 (15)

Geometric parameters (Å, °)

Ag1—O1^{i}	2.537 (3)	Co2—O2^{xi}	2.056 (3)
Ag1—O1^{ii}	2.537 (3)	Co2—O3^{i}	2.074 (3)
Ag1—O5^{iii}	2.623 (3)	Co2—O3	2.074 (3)

Ag1—O5^{iv}	2.623 (3)	P1—O1	1.510 (3)
Ag1—O7^{v}	2.666 (4)	P1—O2	1.550 (4)
Ag1—O6^{v}	2.914 (5)	P1—O4	1.563 (3)
Ag1—O4^{vi}	3.001 (3)	P1—O3	1.563 (4)
Ag1—O4^{vii}	3.001 (3)	P2—O5	1.519 (3)
Ag1—Ag2	3.33837 (16)	P2—O5^{xii}	1.520 (3)
Ag2—O3^{viii}	2.374 (3)	P2—O6	1.563 (5)
Ag2—O3^{vi}	2.374 (3)	P2—O7	1.564 (5)
Ag2—O2	2.431 (4)	O1—Co1^{i}	2.055 (3)
Ag2—O2^{i}	2.431 (4)	O1—O4	2.504 (4)
Ag2—O1	2.884 (3)	O1—O2	2.509 (5)
Ag2—O1^{i}	2.884 (3)	O1—Ag1^{i}	2.536 (3)
Ag2—O4^{viii}	3.151 (3)	O2—Co2^{xiii}	2.056 (3)
Ag2—O4^{vi}	3.151 (3)	O3—Ag2^{xiv}	2.374 (3)
Ag2—Ag1^{i}	3.33837 (16)	O4—Ag1^{xv}	3.001 (3)
Co1—O6	2.051 (3)	O4—Ag2^{xiv}	3.151 (3)
Co1—O1^{i}	2.055 (3)	O4—H4	0.8598
Co1—O7^{v}	2.065 (3)	O5—Ag1^{xvi}	2.623 (3)
Co1—O2	2.090 (3)	O6—Co1^{xii}	2.051 (3)
Co1—O3	2.128 (4)	O6—Ag1^{xvii}	2.914 (5)
Co1—O4^{ix}	2.187 (3)	O7—Co1^{xi}	2.065 (3)
Co2—O5^{i}	2.025 (2)	O7—Co1^{xvii}	2.065 (3)
Co2—O5	2.025 (2)	O7—Ag1^{xvii}	2.666 (4)
Co2—O2^{x}	2.056 (3)		
O1^{i}—Ag1—O1^{ii}	72.52 (15)	O2—Ag2—O4^{vi}	125.96 (10)
O1^{i}—Ag1—O5^{iii}	87.09 (10)	O2^{i}—Ag2—O4^{vi}	110.56 (10)
O1^{ii}—Ag1—O5^{iii}	120.52 (10)	O1—Ag2—O4^{vi}	177.68 (9)
O1^{i}—Ag1—O5^{iv}	120.52 (10)	O1^{i}—Ag2—O4^{vi}	102.42 (8)
O1^{ii}—Ag1—O5^{iv}	87.09 (10)	O4^{viii}—Ag2—O4^{vi}	78.84 (11)
O5^{iii}—Ag1—O5^{iv}	56.35 (11)	Ag1^{i}—Ag2—Ag1	171.21 (3)
O1^{i}—Ag1—O7^{v}	65.44 (10)	O6—Co1—O1^{i}	95.28 (16)
O1^{ii}—Ag1—O7^{v}	65.44 (10)	O6—Co1—O7^{v}	88.38 (11)
O5^{iii}—Ag1—O7^{v}	149.47 (7)	O1^{i}—Co1—O7^{v}	86.15 (16)
O5^{iv}—Ag1—O7^{v}	149.47 (7)	O6—Co1—O2	168.69 (14)
O1^{i}—Ag1—O6^{v}	107.39 (11)	O1^{i}—Co1—O2	91.26 (14)
O1^{ii}—Ag1—O6^{v}	107.39 (11)	O7^{v}—Co1—O2	101.28 (12)
O5^{iii}—Ag1—O6^{v}	132.08 (10)	O6—Co1—O3	101.28 (13)
O5^{iv}—Ag1—O6^{v}	132.08 (10)	O1^{i}—Co1—O3	90.38 (13)
O7^{v}—Ag1—O6^{v}	52.78 (11)	O7^{v}—Co1—O3	170.01 (13)
O1^{i}—Ag1—O4^{vi}	116.18 (10)	O2—Co1—O3	69.40 (10)
O1^{ii}—Ag1—O4^{vi}	163.45 (9)	O6—Co1—O4^{ix}	84.00 (15)

O5^{iii}—Ag1—O4^{vi}	75.15 (9)	O1^{i}—Co1—O4^{ix}	175.52 (12)
O5^{iv}—Ag1—O4^{vi}	99.02 (9)	O7^{v}—Co1—O4^{ix}	89.40 (15)
O7^{v}—Ag1—O4^{vi}	104.24 (11)	O2—Co1—O4^{ix}	90.18 (13)
O6^{v}—Ag1—O4^{vi}	57.31 (10)	O3—Co1—O4^{ix}	94.10 (13)
O1^{i}—Ag1—O4^{vii}	163.45 (9)	O5^{i}—Co2—O5	178.97 (18)
O1^{ii}—Ag1—O4^{vii}	116.18 (10)	O5^{i}—Co2—O2^{x}	94.06 (13)
O5^{iii}—Ag1—O4^{vii}	99.02 (9)	O5—Co2—O2^{x}	86.71 (13)
O5^{iv}—Ag1—O4^{vii}	75.15 (9)	O5^{i}—Co2—O2^{xi}	86.71 (13)
O7^{v}—Ag1—O4^{vii}	104.24 (11)	O5—Co2—O2^{xi}	94.06 (13)
O6^{v}—Ag1—O4^{vii}	57.31 (10)	O2^{x}—Co2—O2^{xi}	83.4 (2)
O4^{vi}—Ag1—O4^{vii}	51.87 (13)	O5^{i}—Co2—O3^{i}	94.45 (13)
O3^{viii}—Ag2—O3^{vi}	100.42 (17)	O5—Co2—O3^{i}	84.82 (13)
O3^{viii}—Ag2—O2	77.19 (9)	O2^{x}—Co2—O3^{i}	93.07 (11)
O3^{vi}—Ag2—O2	177.52 (14)	O2^{xi}—Co2—O3^{i}	176.32 (16)
O3^{viii}—Ag2—O2^{i}	177.52 (14)	O5^{i}—Co2—O3	84.82 (13)
O3^{vi}—Ag2—O2^{i}	77.19 (9)	O5—Co2—O3	94.45 (13)
O2—Ag2—O2^{i}	105.21 (15)	O2^{x}—Co2—O3	176.32 (16)
O3^{viii}—Ag2—O1	114.25 (11)	O2^{xi}—Co2—O3	93.07 (11)
O3^{vi}—Ag2—O1	126.53 (11)	O3^{i}—Co2—O3	90.51 (18)
O2—Ag2—O1	55.53 (11)	O1—P1—O2	110.1 (2)
O2^{i}—Ag2—O1	67.13 (10)	O1—P1—O4	109.14 (18)
O3^{viii}—Ag2—O1^{i}	126.53 (11)	O2—P1—O4	112.90 (19)
O3^{vi}—Ag2—O1^{i}	114.25 (11)	O1—P1—O3	116.08 (18)
O2—Ag2—O1^{i}	67.13 (10)	O2—P1—O3	100.93 (14)
O2^{i}—Ag2—O1^{i}	55.53 (11)	O4—P1—O3	107.57 (19)
O1—Ag2—O1^{i}	76.38 (13)	O1—P1—Co1	122.99 (14)
O3^{viii}—Ag2—O4^{viii}	52.04 (11)	O5—P2—O5^{xii}	109.2 (2)
O3^{vi}—Ag2—O4^{viii}	68.06 (10)	O5—P2—O6	110.52 (15)
O2—Ag2—O4^{viii}	110.56 (10)	O5^{xii}—P2—O6	110.52 (15)
O2^{i}—Ag2—O4^{viii}	125.96 (10)	O5—P2—O7	110.52 (15)
O1—Ag2—O4^{viii}	102.42 (8)	O5^{xii}—P2—O7	110.53 (15)
O1^{i}—Ag2—O4^{viii}	177.68 (9)	O6—P2—O7	105.5 (2)
O3^{viii}—Ag2—O4^{vi}	68.06 (10)	P1—O4—H4	137.9
O3^{vi}—Ag2—O4^{vi}	52.04 (11)		

Symmetry codes: (i) −*x*, −*y*+1, *z*; (ii) *x*+1/2, −*y*+1, *z*; (iii) *x*, −*y*+3/2, *z*−1/2; (iv) −*x*+1/2, −*y*+3/2, *z*−1/2; (v) −*x*+1/2, −*y*+1/2, *z*−1/2; (vi) −*x*, *y*+1/2, *z*−1/2; (vii) *x*+1/2, *y*+1/2, *z*−1/2; (viii) *x*, −*y*+1/2, *z*−1/2; (ix) −*x*, −*y*, *z*; (x) −*x*, *y*+1/2, *z*+1/2; (xi) *x*, −*y*+1/2, *z*+1/2; (xii) −*x*+1/2, *y*, *z*; (xiii) −*x*, *y*−1/2, *z*−1/2; (xiv) −*x*, *y*−1/2, *z*+1/2; (xv) *x*−1/2, *y*−1/2, *z*+1/2; (xvi) −*x*+1/2, −*y*+3/2, *z*+1/2; (xvii) −*x*+1/2, −*y*+1/2, *z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O4^{xviii}	0.86	1.86	2.626 (7)	148.

Symmetry codes: (xviii) −*x*−1/2, *y*, *z*.

Fig. 1

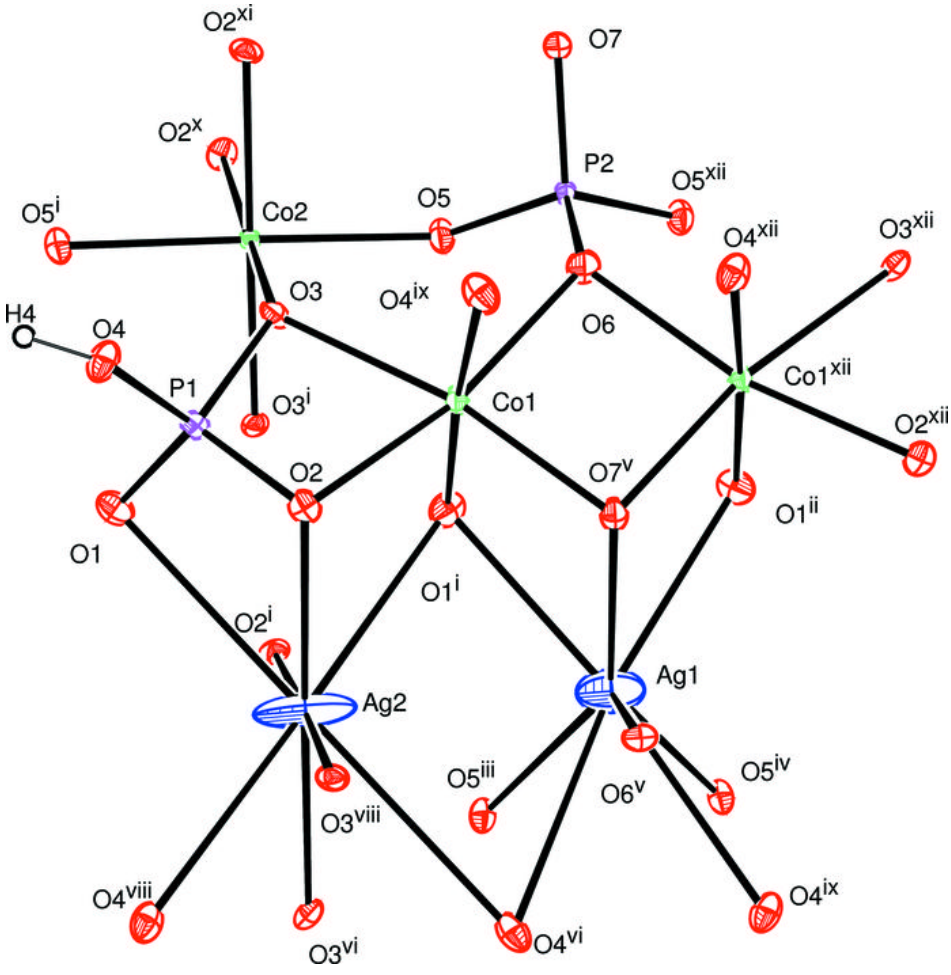

Fig. 2