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Disilver(I) tricobalt(II) hydrogenphosphate bis(phosphate), $\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$

Abderrazzak Assani,* Lahcen El Ammari, Mohammed Zriouil and Mohamed Saadi

 Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Battouta, BP 1014, Rabat, Morocco
 Correspondence e-mail: abder_assani@yahoo.fr

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

$\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$ 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 $\text{Ag}_2\text{O}-\text{MO}-\text{P}_2\text{O}_5$ ($M =$ divalent cation) system include $\text{AgMg}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Assani *et al.*, 2011a), $\text{AgMn}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Leroux *et al.*, 1995), $\text{AgCo}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Guesmi & Driss, 2002), $\text{AgNi}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Ben Smail & Jouini, 2002), $\text{Ag}_2\text{Ni}_3(\text{HPO}_4)(\text{PO}_4)_2$ (Assani *et al.*, 2011b) and $\gamma\text{-AgZnPO}_4$ (Assani *et al.*, 2010).

Experimental

Crystal data

$\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$	$V = 916.55(5)$ Å ³
$M_r = 678.44$	$Z = 4$
Orthorhombic, <i>Ima2</i>	Mo $K\alpha$ radiation
$a = 12.9814(4)$ Å	$\mu = 10.11$ mm ⁻¹
$b = 6.5948(2)$ Å	$T = 296$ K
$c = 10.7062(3)$ Å	$0.26 \times 0.12 \times 0.09$ mm

Data collection

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

Refinement

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

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4}\cdots\text{O4}^i$	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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Disilver(I) tricobalt(II) hydrogenphosphate bis(phosphate), $\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$ **Abderrazzak Assani, Lahcen El Ammari, Mohammed Zriouil and Mohamed Saadi****S1. Comment**

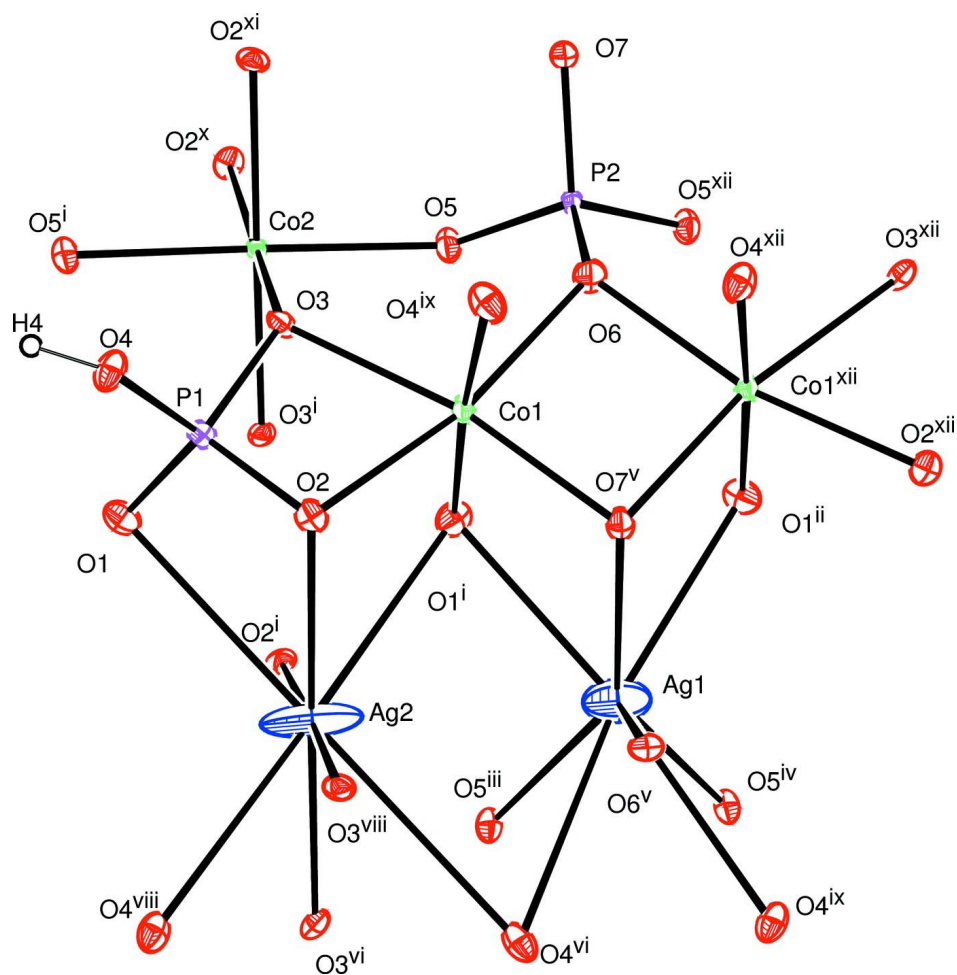
Compounds prepared hydrothermally in the $\text{Ag}_2\text{O}-\text{MO}-\text{P}_2\text{O}_5$ (M = divalent cation) systems include $\text{AgMg}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Assani *et al.*, 2011a), $\text{AgMn}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Leroux *et al.*, 1995), $\text{AgCo}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Guesmi & Driss, 2002), $\text{AgNi}_3(\text{PO}_4)(\text{HPO}_4)_2$ (Ben Smail & Jouini, 2002), $\text{Ag}_2\text{Ni}_3(\text{HPO}_4)(\text{PO}_4)_2$ (Assani *et al.*, 2011b), and $\gamma\text{-AgZnPO}_4$ (Assani *et al.*, 2010). $\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$, isostructural to the Ni analogue, contains CoO_6 octahedra and PO_4 and $\text{PO}_3(\text{OH})$ 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).

S2. Experimental

A mixture of 0.0849 g AgNO_3 , 0.0529 g $\text{CoCO}_3\cdot\text{Co}(\text{OH})_2$, 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.

S3. 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 $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{O})$. The highest and deepest hole in the final difference Fourier map are located at 0.70 Å and 0.51 Å, respectively, from Ag1.


Figure 1

Connectivity of metal-centred coordination polyhedra in $\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$. 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) $-x + 1/2, y, z$.

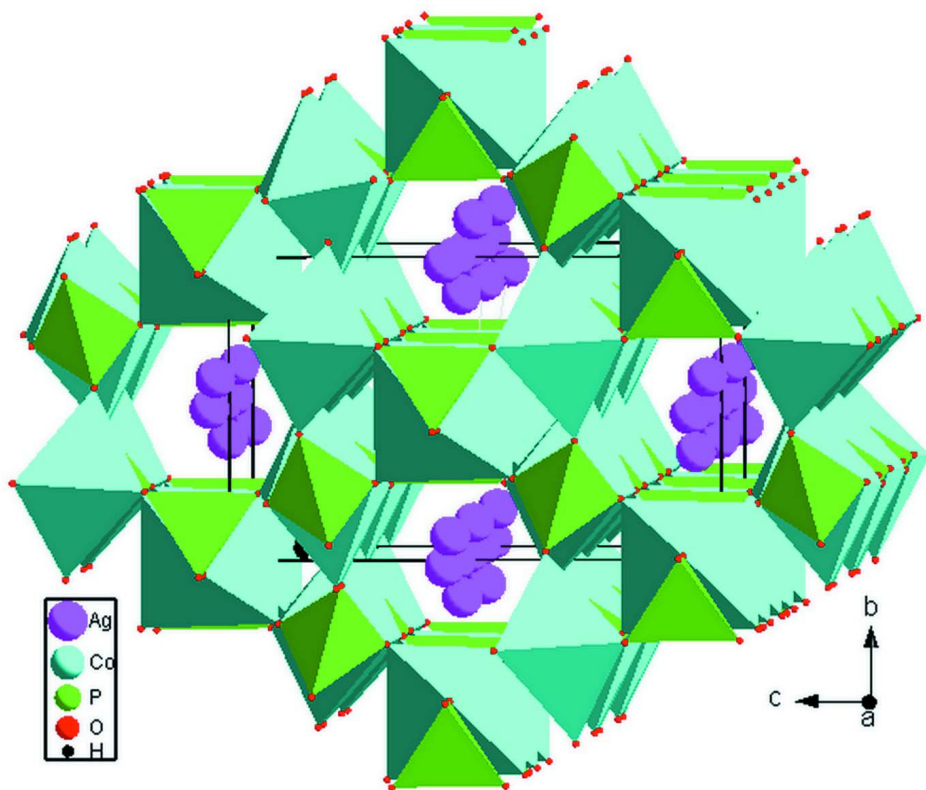


Figure 2

Polyhedral representation of $\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$, 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

$\text{Ag}_2\text{Co}_3(\text{HPO}_4)(\text{PO}_4)_2$

$M_r = 678.44$

Orthorhombic, *Ima2*

Hall symbol: I 2 -2a

$a = 12.9814 (4) \text{ \AA}$

$b = 6.5948 (2) \text{ \AA}$

$c = 10.7062 (3) \text{ \AA}$

$V = 916.55 (5) \text{ \AA}^3$

$Z = 4$

$F(000) = 1268$

$D_x = 4.917 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1388 reflections

$\theta = 3.1\text{--}30.0^\circ$

$\mu = 10.11 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Prism, pink

$0.26 \times 0.12 \times 0.09 \text{ mm}$

Data collection

Bruker X8 APEX

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*MULABS*; Blessing, 1995)

$T_{\min} = 0.365$, $T_{\max} = 0.424$

3966 measured reflections

1388 independent reflections

1368 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.1^\circ$

$h = -17 \rightarrow 18$

$k = -3 \rightarrow 9$

$l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.028$
 $wR(F^2) = 0.064$
 $S = 1.05$
 1388 reflections
 99 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.036P)^2 + 2.5641P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.81 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.54 \text{ e } \text{\AA}^{-3}$
 Absolute structure: Flack (1983), 653 Friedel
 pairs
 Absolute structure 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	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 (\AA^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	3.3384 (2)	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	3.3384 (2)	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, °)

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

Symmetry code: (xviii) $-x-1/2, y, z$.