

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

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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 = 0.024; wR factor = 0.060; data-to-parameter ratio = 11.4.

The title compound, $\text{Ag}_2\text{Ni}_3(\text{HPO}_4)\text{(PO}_4)_2$, has been synthesized by the hydrothermal method. Its structure is formed by two types of chains running along the b axis. The first chain results from a linear and continuous succession of NiO_6 octahedra linked to PO_4 tetrahedra by a common vertex. The second chain is built up from two adjacent edge-sharing octahedra (dimers) whose ends are linked to two PO_4 tetrahedra by a common edge. Those two types of chains are linked together by the phosphate groups to form polyhedral sheets parallel to the (001) plane. The three-dimensional framework delimits two types of hexagonal tunnels parallel to the a -axis direction, at $(x, 1/2, 0)$ and $(x, 0, 1/2)$, where the Ag atoms are located. Each silver cation is surrounded by eight O atoms. The same Ag^+ coordination is found in other phosphates with the alluaudite structure, for example, $\text{AgMn}_3(\text{PO}_4)_2(\text{HPO}_4)_2$. Moreover, O–H···O hydrogen bonds link three PO_4 tetrahedra so as to build a three-dimensional network.

Related literature

For related applications, see: Viter & Nagornyi (2009); Gao & Gao (2005); Clearfield (1988); Trad *et al.* (2010). For compounds with the same structure, see: Assani *et al.* (2010, 2011); Leroux *et al.* (1995); Ben Smail & Jouini (2002).

Experimental

Crystal data

$\text{Ag}_2\text{Ni}_3(\text{HPO}_4)\text{(PO}_4)_2$
 $M_r = 677.79$
Orthorhombic, $Im\bar{a}2$

$a = 12.9233 (3)$ Å
 $b = 6.5678 (2)$ Å
 $c = 10.6629 (3)$ Å

$V = 905.04 (4)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 10.98$ mm⁻¹
 $T = 296$ K
 $0.25 \times 0.13 \times 0.08$ mm

Data collection

Bruker X8 APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*MULABS*; Blessing, 1995)
 $T_{\min} = 0.382$, $T_{\max} = 0.471$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.060$
 $S = 1.06$
1125 reflections
99 parameters
1 restraint

H-atom parameters constrained
 $\Delta\rho_{\max} = 1.81$ e Å⁻³
 $\Delta\rho_{\min} = -1.12$ e Å⁻³
Absolute structure: Flack (1983),
467 Friedel pairs
Flack parameter: 0.55 (3)

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

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|-------------------------|--------------|--------------------|-------------|----------------------|
| O7–H7···O6 ⁱ | 0.86 | 2.06 | 2.847 (6) | 151 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT*; 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: RU2005).

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supporting information

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

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S1. Comment

As the improvement has arisen in the synthesis of a variety of interesting porous materials and open-framework structures, extensive studies are devoted to the metal phosphates which exhibit a rich structural diversity and have been widely studied as catalysts (Viter & Nagornyi, 2009; Gao & Gao, 2005), ion-exchangers (Clearfield, 1988) and as positive electrode in the lithium and sodium batteries (Trad *et al.* (2010)).

Within this family of compounds, the resulting anionic frameworks, generally constructed from the alternation of PO_4 tetrahedra connected to metal cations in different coordinate geometry MO_n (with $n=4, 5$ and 6), generate pores and channels offering suitable environment to accommodate different other cations. In our search for new phosphates with microporous framework, our most attention has been paid to the hydrothermal investigation of the $\text{A}_2\text{O}-\text{MO}-\text{P}_2\text{O}_5$ systems, with A = monovalent cations and M = divalent cations. Accordingly, we have succeed, for instance, to isolate new form of silver zinc phosphate ($\gamma\text{-AgZnPO}_4$) related to the ABW zeolite structure (Assani *et al.* 2010) while the silver magnesium phosphate, namely $\text{AgMg}_3(\text{PO}_4)_2(\text{HPO}_4)_2$, represent a new member of the well known alluaudite-like structure family (Assani *et al.* 2011). The present paper aims to develop the hydrothermal synthesis and the structural characterization of a new silver nickel phosphate, namely, $\text{Ag}_2\text{Ni}_3(\text{HPO}_4)(\text{PO}_4)_2$.

The structure of this compound is formed by two types of chains running along the b axis. The first chain $(\text{Ni}_2\text{P}_2\text{HO}_9)_{\infty}$ is built up from Ni^{2+} and P^{4+} atoms in special Wyckoff position $4\ b\ (m)$ of the space group $\text{Im}\bar{a}2$. This chain results from linear and continuous succession of octahedron (Ni_2O_6) and $\text{P}_2\text{O}_5\text{OH}$ tetrahedron which share a vertex. The second chain $(\text{Ni}_2\text{P}_2\text{O}_{14})_n$ is built up from two adjacent edge sharing octahedra ($(\text{Ni}^{2+})_2\text{O}_{10}$ dimmers) whose ends are linked to two P_1O_4 tetrahedra by a common edge (Fig. 1). Those chains are linked together by the phosphate groups to form polyhedral sheets parallel to the $(0\ 0\ 1)$ plane as shown in Fig. 2.

The three dimensional framework delimits two types of hexagonal tunnels running along the a direction, at $x\ 1/2\ 0$ and $x\ 0\ 1/2$ (Fig. 3). The Ag^{2+} atom is located at centre of tunnels, this explains the high value of its anisotropic displacement U_{11} , whereas Ag^{1+} is slightly shifted from this center (Wyckoff positions: Ag^{2+} at $2a$: $0, 0, z$ and Ag^{1+} at $2b$: $1/4, y, z$). However, each Ag^{+} ion is surrounded by 8 O atoms with different $\text{Ag}-\text{O}$ distances. Indeed, the first coordination environment of Ag^{2+} is almost square planar with four short $\text{Ag}^{2+}-\text{O}$ distances between 2.373 (4) and 2.421 (4) Å and the other four larger distances are in the range of 2.869 (4) to 3.133 (4) Å. A similar coordination surrounding Ag^{1+} is observed with $\text{Ag}^{1+}-\text{O}$ bond lengths in the range of 2.537 (4)–2.616 (4) Å and the longest bonds are situated between 2.661 (4) and 2.963 (4) Å. The same coordination for this cation is found in other phosphate with alluaudite structure like $\text{AgMn}_3(\text{PO}_4)_2(\text{HPO}_4)_2$ (Leroux *et al.* (1995)) and $\text{AgNi}_3(\text{PO}_4)_2(\text{HPO}_4)_2$ (Ben Smail & Jouini (2002)).

Moreover, $\text{O}-\text{H}\cdots\text{O}$ hydrogen bondings link two adjacent P_2O_5 tetrahedra *via* a strong hydrogen bond $\text{O}7-\text{H}7\cdots\text{O}6$ to two P_1O_4 tetrahedra through weak bonds $\text{O}7-\text{H}7\cdots\text{O}4$ in the way to build an infinite three-dimensional network as shown in Table 1.

S2. Experimental

By means of hydrothermal synthesis, we have isolate a new silver nickel phosphate from the reaction mixture of silver nitrate (AgNO_3 ; 0.1699 g), metallic nickel (Ni; 0.0881 g), 85%wt phosphoric acid (H_3PO_4 ; 0,10 ml) and water (12 ml). The hydrothermal treatment was conducted in a 23 ml Teflon-lined autoclave under autogeneous pressure at 468 K for two days. After being filtered off, washed with deionized water and air dried, the reaction product consists of a monophasic green powder and some green parallelepipedic crystals corresponding to the title compound.

S3. Refinement

The structure is solved by direct method technique and refined by full-matrix least-squares using *SHELXS97* and *SHELXL97* program packages. The structure refinement in the centrosymmetric space group was unsuccessful. Infact the crystal is a racemic twinned with a refined ratio of 0.479 (26), which explains the ambiguity in the Flack parameter. The space group is not centro symmetric and the polar axis restraint is generated automatically by *SHELXL* program. Friedel opposites reflections are not merged. The O-bound H atom is initially located unambiguously in a difference map and refined with O—H distance restraints of 0.86 (1). In a the last cycle ther is refined in the riding model approximation with $U_{\text{iso}}(\text{H})$ set to $1.2U_{\text{eq}}(\text{O})$. The highest and deepest hole residual peak in the final difference Fourier map are located at 0.72 Å and 0.62 Å, from Ag1.

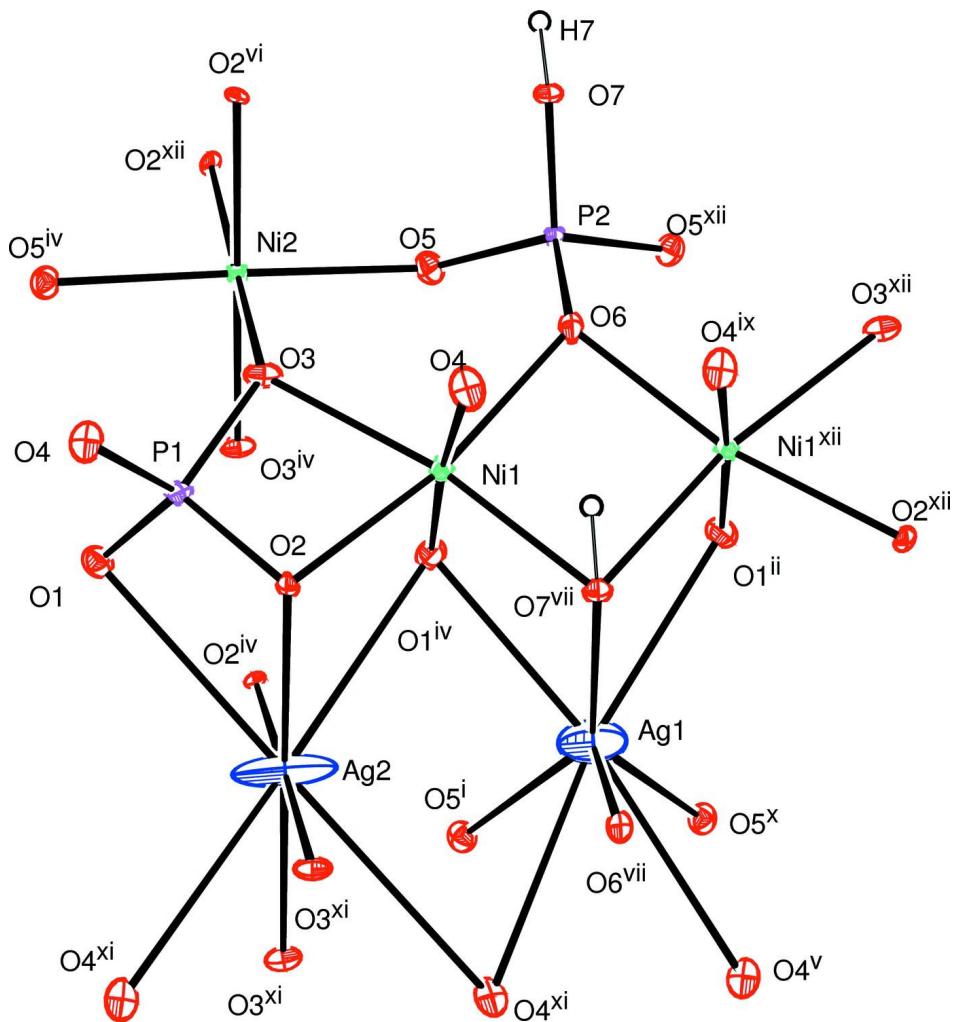


Figure 1

Partial plot of $\text{Ag}_2\text{Ni}_3(\text{HPO}_4)_2(\text{PO}_4)_2$ crystal structure. 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) $-x + 1/2, y, z$.

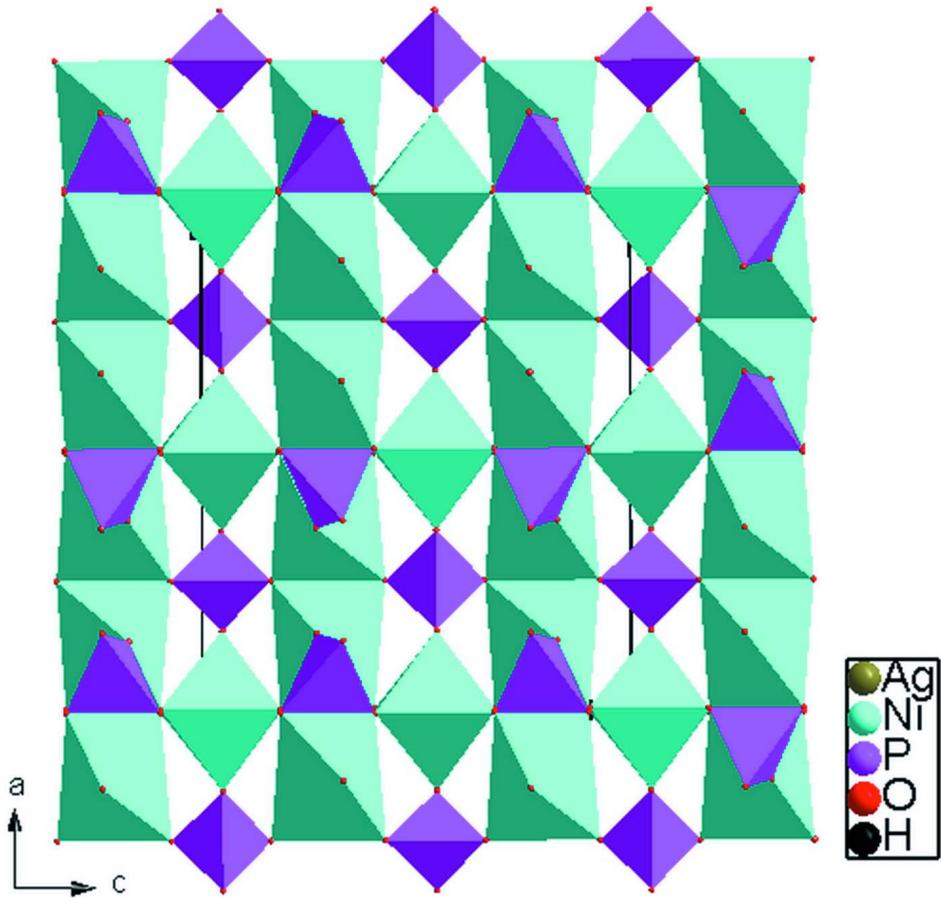
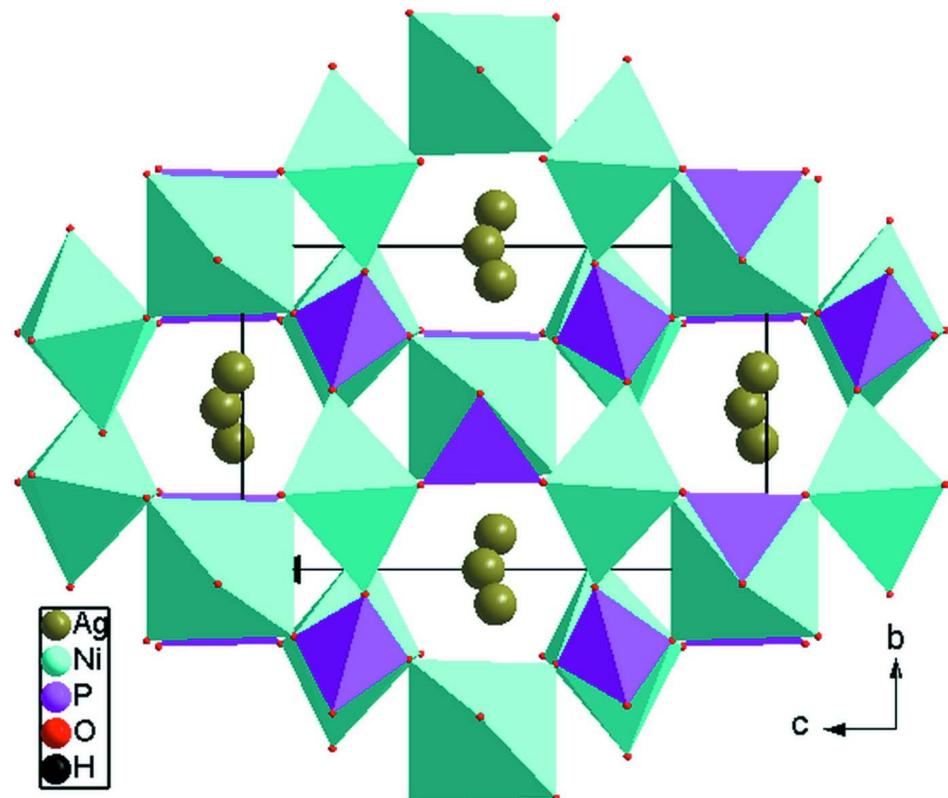


Figure 2

View along the b axis of polyhedral sheets parallel to the $(0\ 0\ 1)$ plane.

**Figure 3**

A three-dimensional polyhedral view of the crystal structure of the $\text{Ag}_2\text{Ni}_3(\text{HPO}_4)(\text{PO}_4)_2$, showing tunnels running along the a direction, at x , $1/2$, 0 and x , 0 , $1/2$.

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

Crystal data



$M_r = 677.79$

Orthorhombic, $Im\bar{a}2$

Hall symbol: I 2 -2a

$a = 12.9233$ (3) Å

$b = 6.5678$ (2) Å

$c = 10.6629$ (3) Å

$V = 905.04$ (4) Å³

$Z = 4$

$F(000) = 1280$

$D_x = 4.974 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1125 reflections

$\theta = 3.2\text{--}29.0^\circ$

$\mu = 10.98 \text{ mm}^{-1}$

$T = 296$ K

Prism, green

$0.25 \times 0.13 \times 0.08$ mm

Data collection

Bruker X8 APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan
(*MULABS*; Blessing, 1995)

$T_{\min} = 0.382$, $T_{\max} = 0.471$

3762 measured reflections

1125 independent reflections

1103 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 29.0^\circ$, $\theta_{\min} = 3.2^\circ$

$h = -13 \rightarrow 14$

$k = -16 \rightarrow 17$

$l = -8 \rightarrow 8$

*Refinement*Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.024$$

$$wR(F^2) = 0.060$$

$$S = 1.06$$

1125 reflections

99 parameters

1 restraint

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2 + 1.7344P]$$

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

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

$$\Delta\rho_{\max} = 1.81 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.12 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Flack (1983), 467 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)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Ag1 | 0.2500 | 0.60793 (8) | -0.01513 (7) | 0.02914 (18) |
| Ag2 | 0.0000 | 0.5000 | -0.03769 (5) | 0.0443 (2) |
| Ni1 | 0.13623 (4) | 0.24801 (10) | 0.20871 (7) | 0.00699 (13) |
| Ni2 | 0.0000 | 0.5000 | 0.45735 (7) | 0.00462 (15) |
| P1 | -0.07279 (7) | 0.25722 (19) | 0.20677 (13) | 0.00587 (19) |
| P2 | 0.2500 | 0.4102 (2) | 0.45653 (15) | 0.0042 (3) |
| O1 | -0.1343 (3) | 0.4456 (5) | 0.1739 (3) | 0.0091 (7) |
| O2 | 0.0044 (3) | 0.2070 (6) | 0.1000 (3) | 0.0056 (6) |
| O3 | 0.0036 (3) | 0.2785 (5) | 0.3204 (3) | 0.0072 (8) |
| O4 | -0.1494 (3) | 0.0786 (5) | 0.2360 (3) | 0.0096 (9) |
| O5 | 0.1543 (2) | 0.5443 (4) | 0.4552 (3) | 0.0085 (5) |
| O6 | 0.2500 | 0.2617 (8) | 0.3420 (5) | 0.0090 (12) |
| O7 | 0.2500 | 0.2692 (7) | 0.5756 (5) | 0.0064 (12) |
| H7 | 0.2500 | 0.3065 | 0.6529 | 0.008* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|------------|---------------|
| Ag1 | 0.0491 (4) | 0.0158 (3) | 0.0225 (3) | 0.000 | 0.000 | 0.0048 (3) |
| Ag2 | 0.1145 (7) | 0.0083 (2) | 0.0101 (3) | -0.0020 (3) | 0.000 | 0.000 |
| Ni1 | 0.0058 (2) | 0.0092 (3) | 0.0060 (3) | 0.0005 (2) | 0.0005 (3) | -0.00135 (19) |
| Ni2 | 0.0052 (3) | 0.0049 (3) | 0.0037 (3) | 0.0006 (2) | 0.000 | 0.000 |
| P1 | 0.0064 (4) | 0.0064 (5) | 0.0047 (4) | -0.0001 (5) | 0.0008 (6) | 0.0004 (4) |

| | | | | | | |
|----|-------------|-------------|-------------|--------------|--------------|--------------|
| P2 | 0.0043 (6) | 0.0054 (6) | 0.0030 (7) | 0.000 | 0.000 | -0.0012 (6) |
| O1 | 0.0086 (16) | 0.0100 (18) | 0.0088 (15) | 0.0003 (14) | -0.0017 (10) | -0.0010 (12) |
| O2 | 0.0044 (18) | 0.0088 (15) | 0.0035 (15) | -0.0002 (15) | -0.0010 (12) | -0.0029 (13) |
| O3 | 0.012 (2) | 0.0044 (17) | 0.0049 (15) | 0.0009 (15) | -0.0005 (12) | -0.0010 (12) |
| O4 | 0.010 (2) | 0.0023 (18) | 0.016 (2) | -0.0011 (13) | 0.0015 (11) | 0.0017 (11) |
| O5 | 0.0077 (12) | 0.0082 (12) | 0.0096 (15) | 0.0014 (10) | -0.0008 (13) | -0.0003 (12) |
| O6 | 0.005 (3) | 0.013 (3) | 0.009 (2) | 0.000 | 0.000 | -0.0007 (16) |
| O7 | 0.007 (3) | 0.007 (3) | 0.005 (2) | 0.000 | 0.000 | 0.0023 (16) |

Geometric parameters (\AA , ^\circ)

| | | | |
|---|-------------|--|-------------|
| Ag1—O1 ⁱ | 2.534 (3) | Ni2—O2 ^{xi} | 2.041 (3) |
| Ag1—O1 ⁱⁱ | 2.535 (3) | Ni2—O3 ⁱ | 2.062 (4) |
| Ag1—O5 ⁱⁱⁱ | 2.617 (3) | Ni2—O3 | 2.062 (4) |
| Ag1—O5 ^{iv} | 2.617 (3) | P1—O1 | 1.511 (4) |
| Ag1—O7 ^v | 2.659 (5) | P1—O2 | 1.549 (4) |
| Ag1—O6 ^v | 2.866 (5) | P1—O4 | 1.566 (4) |
| Ag1—O4 ^{vi} | 2.962 (3) | P1—O3 | 1.569 (4) |
| Ag1—O4 ^{vii} | 2.962 (3) | P2—O5 | 1.518 (3) |
| Ag1—Ag2 | 3.3164 (2) | P2—O5 ^{xii} | 1.518 (3) |
| Ag2—O3 ^{vi} | 2.375 (4) | P2—O6 | 1.563 (6) |
| Ag2—O3 ^{viii} | 2.375 (4) | P2—O7 | 1.572 (5) |
| Ag2—O2 | 2.421 (4) | O1—Ni1 ⁱ | 2.046 (4) |
| Ag2—O2 ⁱ | 2.421 (4) | O1—O4 | 2.507 (5) |
| Ag2—O1 | 2.869 (4) | O1—Ag1 ⁱ | 2.534 (3) |
| Ag2—O1 ⁱ | 2.869 (4) | O2—Ni2 ^{xiii} | 2.041 (3) |
| Ag2—O4 ^{viii} | 3.133 (4) | O3—Ag2 ^{xiv} | 2.375 (4) |
| Ag2—O4 ^{vi} | 3.133 (4) | O4—Ni1 ^{ix} | 2.172 (4) |
| Ni1—O1 ⁱ | 2.046 (4) | O4—Ag1 ^{xv} | 2.962 (3) |
| Ni1—O7 ^v | 2.047 (3) | O4—Ag2 ^{xiv} | 3.133 (4) |
| Ni1—O6 | 2.047 (4) | O5—Ag1 ^{xvi} | 2.617 (3) |
| Ni1—O2 | 2.079 (4) | O6—Ni1 ^{xii} | 2.047 (4) |
| Ni1—O3 | 2.097 (4) | O6—Ag1 ^{xvii} | 2.866 (5) |
| Ni1—O4 ^{ix} | 2.172 (4) | O7—Ni1 ^{xi} | 2.047 (3) |
| Ni2—O5 ⁱ | 2.015 (3) | O7—Ni1 ^{xvii} | 2.047 (3) |
| Ni2—O5 | 2.015 (3) | O7—Ag1 ^{xvii} | 2.659 (5) |
| Ni2—O2 ^x | 2.041 (3) | O7—H7 | 0.8600 |
| | | | |
| O1 ⁱ —Ag1—O1 ⁱⁱ | 72.33 (16) | O3 ^{viii} —Ag2—O4 ^{vi} | 67.92 (11) |
| O1 ⁱ —Ag1—O5 ⁱⁱⁱ | 86.46 (10) | O2—Ag2—O4 ^{vi} | 125.71 (11) |
| O1 ⁱⁱ —Ag1—O5 ⁱⁱⁱ | 119.74 (11) | O2 ⁱ —Ag2—O4 ^{vi} | 110.51 (11) |
| O1 ⁱ —Ag1—O5 ^{iv} | 119.74 (11) | O1—Ag2—O4 ^{vi} | 177.43 (10) |
| O1 ⁱⁱ —Ag1—O5 ^{iv} | 86.46 (10) | O1 ⁱ —Ag2—O4 ^{vi} | 102.27 (8) |
| O5 ⁱⁱⁱ —Ag1—O5 ^{iv} | 56.41 (12) | O4 ^{viii} —Ag2—O4 ^{vi} | 79.25 (12) |
| O1 ⁱ —Ag1—O7 ^v | 65.26 (11) | Ag1 ⁱ —Ag2—Ag1 | 171.68 (3) |
| O1 ⁱⁱ —Ag1—O7 ^v | 65.26 (11) | O1 ⁱ —Ni1—O7 ^v | 86.42 (17) |
| O5 ⁱⁱⁱ —Ag1—O7 ^v | 148.99 (7) | O1 ⁱ —Ni1—O6 | 95.26 (18) |
| O5 ^{iv} —Ag1—O7 ^v | 148.99 (7) | O7 ^v —Ni1—O6 | 88.15 (12) |

| | | | |
|--|-------------|---------------------------------------|-------------|
| O1 ⁱ —Ag1—O6 ^v | 107.78 (12) | O1 ⁱ —Ni1—O2 | 90.92 (15) |
| O1 ⁱⁱ —Ag1—O6 ^v | 107.78 (12) | O7 ^v —Ni1—O2 | 101.25 (13) |
| O5 ⁱⁱⁱ —Ag1—O6 ^v | 132.46 (11) | O6—Ni1—O2 | 169.08 (15) |
| O5 ^{iv} —Ag1—O6 ^v | 132.46 (11) | O1 ⁱ —Ni1—O3 | 89.93 (14) |
| O7 ^v —Ag1—O6 ^v | 53.46 (12) | O7 ^v —Ni1—O3 | 170.53 (14) |
| O1 ⁱ —Ag1—O4 ^{vi} | 116.39 (11) | O6—Ni1—O3 | 100.89 (14) |
| O1 ⁱⁱ —Ag1—O4 ^{vi} | 164.32 (10) | O2—Ni1—O3 | 70.05 (11) |
| O5 ⁱⁱⁱ —Ag1—O4 ^{vi} | 74.98 (10) | O1 ⁱ —Ni1—O4 ^{ix} | 175.34 (13) |
| O5 ^{iv} —Ag1—O4 ^{vi} | 98.97 (10) | O7 ^v —Ni1—O4 ^{ix} | 88.97 (17) |
| O7 ^v —Ag1—O4 ^{vi} | 105.40 (12) | O6—Ni1—O4 ^{ix} | 83.92 (17) |
| O6 ^v —Ag1—O4 ^{vi} | 57.90 (11) | O2—Ni1—O4 ^{ix} | 90.62 (14) |
| O1 ⁱ —Ag1—O4 ^{vii} | 164.32 (10) | O3—Ni1—O4 ^{ix} | 94.73 (14) |
| O1 ⁱⁱ —Ag1—O4 ^{vii} | 116.39 (11) | O5 ⁱ —Ni2—O5 | 178.70 (19) |
| O5 ⁱⁱⁱ —Ag1—O4 ^{vii} | 98.97 (10) | O5 ⁱ —Ni2—O2 ^x | 94.43 (14) |
| O5 ^{iv} —Ag1—O4 ^{vii} | 74.98 (10) | O5—Ni2—O2 ^x | 86.54 (14) |
| O7 ^v —Ag1—O4 ^{vii} | 105.40 (12) | O5 ⁱ —Ni2—O2 ^{xi} | 86.55 (14) |
| O6 ^v —Ag1—O4 ^{vii} | 57.90 (11) | O5—Ni2—O2 ^{xi} | 94.42 (14) |
| O4 ^{vi} —Ag1—O4 ^{vii} | 52.10 (14) | O2 ^x —Ni2—O2 ^{xi} | 83.6 (2) |
| O3 ^{vi} —Ag2—O3 ^{viii} | 100.81 (18) | O5 ⁱ —Ni2—O3 ⁱ | 94.10 (14) |
| O3 ^{vi} —Ag2—O2 | 177.72 (14) | O5—Ni2—O3 ⁱ | 84.97 (15) |
| O3 ^{viii} —Ag2—O2 | 76.92 (10) | O2 ^x —Ni2—O3 ⁱ | 93.29 (11) |
| O3 ^{vi} —Ag2—O2 ⁱ | 76.92 (10) | O2 ^{xi} —Ni2—O3 ⁱ | 176.91 (18) |
| O3 ^{viii} —Ag2—O2 ⁱ | 177.72 (14) | O5 ⁱ —Ni2—O3 | 84.97 (15) |
| O2—Ag2—O2 ⁱ | 105.35 (15) | O5—Ni2—O3 | 94.10 (14) |
| O3 ^{vi} —Ag2—O1 | 125.84 (12) | O2 ^x —Ni2—O3 | 176.91 (18) |
| O3 ^{viii} —Ag2—O1 | 114.65 (11) | O2 ^{xi} —Ni2—O3 | 93.29 (11) |
| O2—Ag2—O1 | 55.82 (11) | O3 ⁱ —Ni2—O3 | 89.8 (2) |
| O2 ⁱ —Ag2—O1 | 66.92 (11) | O1—P1—O2 | 110.0 (2) |
| O3 ^{vi} —Ag2—O1 ⁱ | 114.65 (11) | O1—P1—O4 | 109.1 (2) |
| O3 ^{viii} —Ag2—O1 ⁱ | 125.84 (12) | O2—P1—O4 | 113.2 (2) |
| O2—Ag2—O1 ⁱ | 66.92 (11) | O1—P1—O3 | 115.89 (19) |
| O2 ⁱ —Ag2—O1 ⁱ | 55.82 (11) | O2—P1—O3 | 100.46 (15) |
| O1—Ag2—O1 ⁱ | 76.28 (13) | O4—P1—O3 | 108.1 (2) |
| O3 ^{vi} —Ag2—O4 ^{viii} | 67.92 (11) | O5—P2—O5 ^{xii} | 109.1 (2) |
| O3 ^{viii} —Ag2—O4 ^{viii} | 52.70 (12) | O5—P2—O6 | 110.77 (16) |
| O2—Ag2—O4 ^{viii} | 110.51 (11) | O5 ^{xii} —P2—O6 | 110.77 (16) |
| O2 ⁱ —Ag2—O4 ^{viii} | 125.71 (11) | O5—P2—O7 | 110.44 (16) |
| O1—Ag2—O4 ^{viii} | 102.27 (8) | O5 ^{xii} —P2—O7 | 110.44 (16) |
| O1 ⁱ —Ag2—O4 ^{viii} | 177.43 (10) | O6—P2—O7 | 105.3 (2) |
| O3 ^{vi} —Ag2—O4 ^{vi} | 52.70 (12) | P2—O7—H7 | 127.4 |

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 (\AA , $^\circ$)

| $D\cdots H$ | $D\cdots A$ | $H\cdots A$ | $D\cdots A$ | $D\cdots H\cdots A$ |
|-------------|-------------|-------------|-------------|---------------------|
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|----------------------------|------|------|-----------|-----|
| O7—H7···O6 ^{xvii} | 0.86 | 2.06 | 2.847 (6) | 151 |
|----------------------------|------|------|-----------|-----|

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