

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Silver(I) diaquamagnesium catena-borodiphosphate(V) monohydrate, $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$

 Hafid Zouihri,^{a,b*} Mohammed Saadi,^b Boujemaa Jaber^a and Lehcen El Ammari^b
^aCentre National pour la Recherche Scientifique et Technique, Division UATRS Angle Allal AlFassi et Avenue des FAR, Hay Ryad, BP 8027, Rabat, Morocco, and

^bLaboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Université Mohammed V-Agdal, Avenue Ibn Batouta, BP 1014, Rabat, Morocco
Correspondence e-mail: zouihri@cnrst.ma

Received 17 May 2011; accepted 23 May 2011

 Key indicators: single-crystal X-ray study; $T = 296\text{ K}$; mean $\sigma(\text{O}-\text{B}) = 0.004\text{ \AA}$; R factor = 0.037; wR factor = 0.101; data-to-parameter ratio = 20.4.

The title compound contains infinite one-dimensional $[\text{BP}_2\text{O}_8]^{3-}$ borophosphate helical ribbons, built up from alternate BO_4 and PO_4 tetrahedra arranged around the 6_5 screw axes. The vertex-sharing BO_4 and PO_4 tetrahedra form a spiral ribbon of four-membered rings in which BO_4 and PO_4 groups alternate. The ribbons are connected through slightly distorted $\text{MgO}_4(\text{H}_2\text{O})_2$ octahedra, in which the four O atoms belong to the phosphate groups. The free threads of the helices are occupied by silver ions, which are in an irregular environment surrounded by six O atoms. The central channels of the helices, running along the c axis, are filled with the water molecules. The structure is stabilized by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the water molecules and O atoms that are part of the helices. The crystal structure of the title compound is isotopic with other analogous borophosphates such as $A^I M^{II}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$, where $A^I = \text{Li}, \text{Na}, \text{K}$ or NH_4^+ and $M^{II} = \text{Mg}, \text{Mn}, \text{Fe}, \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}$ or Cd .

Related literature

For isotopic Mg analogues, see: Kniep *et al.* (1997); Lin *et al.* (2008). For other similar borophosphates, see: Kniep *et al.* (1998); Ewald *et al.* (2007); Menezes *et al.* (2008).

Experimental

Crystal data

 $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$
 $M_r = 386.98$
 Hexagonal, $P6_5$ 27

 $a = 9.4577(4)\text{ \AA}$
 $c = 15.8301(13)\text{ \AA}$
 $V = 1226.27(7)\text{ \AA}^3$
 $Z = 6$
 Mo $K\alpha$ radiation
 $\mu = 2.99\text{ mm}^{-1}$
 $T = 296\text{ K}$
 $0.17 \times 0.10 \times 0.10\text{ mm}$

Data collection

 Bruker APEXII CCD detector
 diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Sheldrick, 1999)
 $T_{\min} = 0.705$, $T_{\max} = 0.741$

 18120 measured reflections
 1553 independent reflections
 1517 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.101$
 $S = 1.10$
 1553 reflections
 76 parameters
 H-atom parameters constrained

 $\Delta\rho_{\max} = 1.59\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -1.56\text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 543 Friedel pairs
 Flack parameter: $-0.01(5)$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{O6}-\text{H6A}\cdots\text{O3}^{\text{i}}$ | 0.86 | 1.89 | 2.744 (3) | 175 |
| $\text{O5}-\text{H5A}\cdots\text{O5}^{\text{ii}}$ | 0.86 | 2.06 | 2.889 (3) | 162 |
| $\text{O6}-\text{H6B}\cdots\text{O2}$ | 0.86 | 1.93 | 2.781 (3) | 170 |

 Symmetry codes: (i) $-x + y, -x + 1, z + \frac{1}{3}$; (ii) $-y + 1, -x + 1, -z + \frac{1}{6}$.

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).

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2421).

References

- Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Ewald, B., Huang, Y.-X. & Kniep, R. (2007). *Z. Anorg. Allg. Chem.* **633**, 1517–1540.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Kniep, R., Engelhardt, H. & Hauf, C. (1998). *Chem. Mater.* **10**, 2930–2934.
 Kniep, R., Will, H. G., Boy, I. & Rohr, C. (1997). *Angew. Chem. Int. Ed.* **36**, 1013–1014.
 Lin, J.-R., Huang, Y.-X., Wu, Y.-H. & Zhou, Y. (2008). *Acta Cryst.* **E64**, i39–i40.
 Menezes, P. W., Hoffmann, S., Prots, Y. & Kniep, R. (2008). *Z. Kristallogr.* **223**, 333–334.
 Sheldrick, G. M. (1999). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2011). E67, i39 [doi:10.1107/S1600536811019477]

Silver(I) diaquamagnesium *catena*-borodiphosphate(V) monohydrate, $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$

Hafid Zouihri, Mohammed Saadi, Boujemaa Jaber and Lehcen El Ammari

S1. Comment

The rich structural chemistry of the borophosphates system has generated considerable contemporary interest as a consequence of the interesting physical and chemical properties of the porous or tunnel structures generally adopted by the inorganic solids which are formed (Kniep *et al.*, 1998, Ewald *et al.*, 2007). Most of these compounds were synthesized with alkali (A^I) and transition metal cations (M^{II}), with the general formula $A^I M^{II}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$, under hydrothermal conditions at 443–463 K (Kniep *et al.* (1997) and Lin *et al.* (2008)).

The crystal structure of the new synthesized helical borophosphate-hydrate $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$ is isotopic with other analogues borophosphates like $A^I M^{II}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$ ($A^I = \text{Li, Na, K, NH}_4^+$ and $M^{II} = \text{Mg, Mn, Fe, Co, Ni, Cu, Zn, Cd}$) (Menezes *et al.*, (2008)). Fig. 1 represents the plot of the asymmetric unit showing the cationic environment and the connection between different polyhedra. The BO_4 and PO_4 tetrahedra are relatively regular with B—O and P—O bond lengths ranging from 1.455 (3) Å to 1.480 (3) Å and from 1.503 (2) Å to 1.569 (2) Å, respectively. Whereas, in the distorted $\text{MgO}_4(\text{H}_2\text{O})_2$ octahedron, the distances Mg—O vary between 2.053 (2) Å and 2.169 (3) Å. Moreover, the AgO_6 polyhedron is more irregular with Ag—O distances in the range of 2.462–2.725 (3) Å.

The structure consists of infinite one dimensional helical anionic ribbons $[\text{BP}_2\text{O}_8]^{3-}$ constructed by corner-sharing BO_4 and PO_4 tetrahedra, arranged around the 6_5 screw axes. The ribbons borders are connected with Mg^{2+} cations *via* the terminal oxygen atoms of the phosphate groups. A three dimensional network is formed by interconnection between the $(\text{AgO}_6)_n$ helices running along [001] and the tetrahedral ribbons *via* the slightly distorted $\text{MgO}_4(\text{H}_2\text{O})_2$ octahedra. The central channels of the helices, running along *c* axis, are filled up with the water molecule as shown in Fig 2. The structure is stabilized by O—H \cdots O hydrogen bonds between water molecules and O atoms that are part of the helices (Table 1).

S2. Experimental

The compound was hydrothermally synthesized at 453 °K for 7 days in a 25 ml Teflon-lined steel autoclave from the mixture of MgO, H_3BO_3 , H_3PO_4 (85%), AgNO_3 and 5 ml of distilled water in the molar ratio of 1:4:6:1:165. The brilliant colourless octahedral crystals were recovered and washed with hot water, then dried in air. Except for boron and hydrogen the presence of the elements were additionally confirmed by EDAX measurements.

S3. Refinement

The highest peak in the difference map is at 0.76 Å from Ag1 atom, and the minimum peak is at 0.52 Å from Ag1 atom.

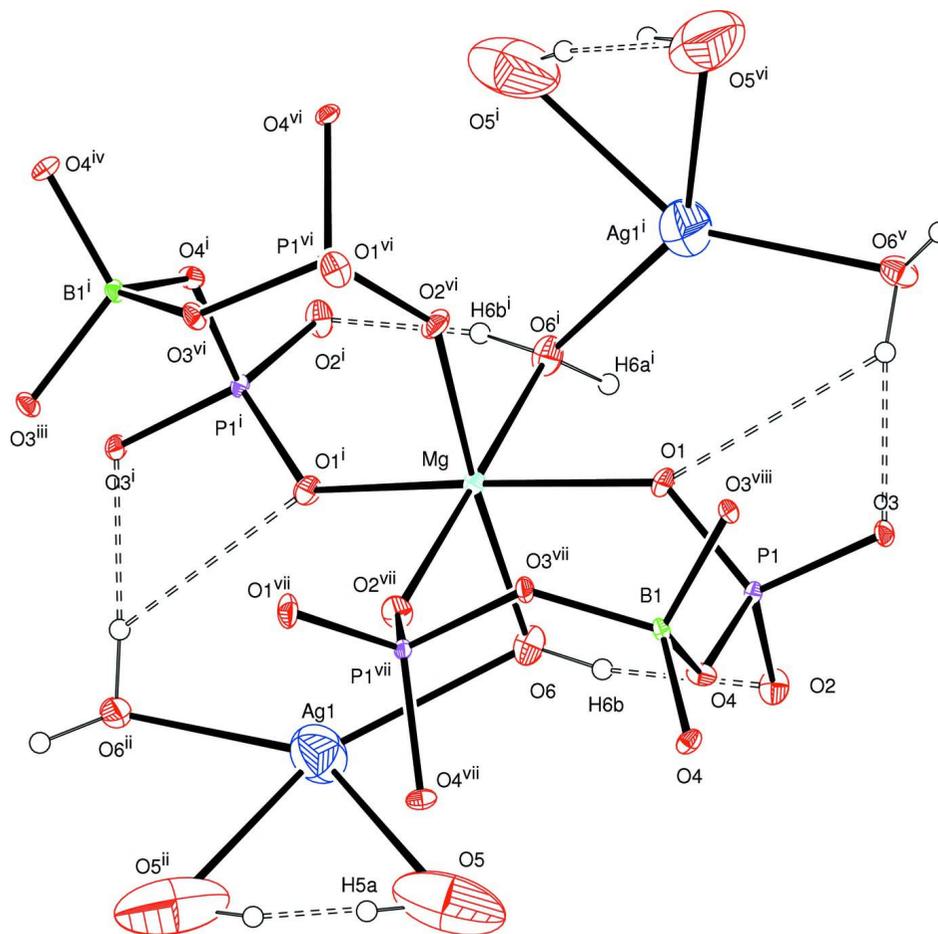


Figure 1

Partial plot of $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8] \cdot \text{H}_2\text{O}$ crystal structure showing polyhedra linkage. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i) $-y + 1, -x + 1, -z + 13/6$; (ii) $y - 1, -x + y, z + 1/6$; (iii) $y - 1, x, -z + 5/3$; (iv) $x, x - y + 1, -z + 11/6$; (v) $-x + y - 1, y, -z + 3/2$; (vi) $-x, -x + y, -z + 4/3$; (vii) $y, x + 1, -z + 5/3$; (viii) $x - y + 1, -y + 2, -z + 2$.

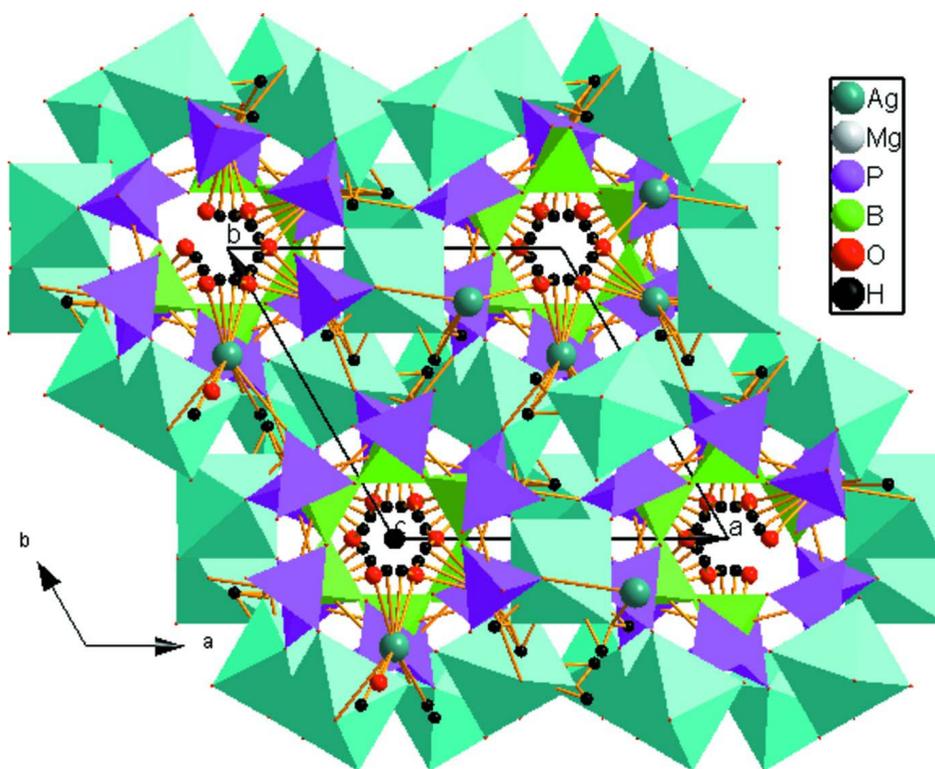


Figure 2

Projection view of the $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8] \cdot \text{H}_2\text{O}$ framework structure showing tunnel running along c direction where water molecules are located.

Silver(I) diaquamagnesium *catena*-borodiphosphate(V) monohydrate

Crystal data

$\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8] \cdot \text{H}_2\text{O}$

$M_r = 386.98$

Hexagonal, $P6_522$

Hall symbol: P 65 2 (0 0 1)

$a = 9.4577 (4) \text{ \AA}$

$c = 15.8301 (13) \text{ \AA}$

$V = 1226.27 (7) \text{ \AA}^3$

$Z = 6$

$F(000) = 1128$

$D_x = 3.144 \text{ Mg m}^{-3}$

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

Cell parameters from 1553 reflections

$\theta = 2.5\text{--}33.0^\circ$

$\mu = 2.99 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Prism, colourless

$0.17 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Bruker APEXII CCD detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1999)

$T_{\min} = 0.705$, $T_{\max} = 0.741$

18120 measured reflections

1553 independent reflections

1517 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 33.0^\circ$, $\theta_{\min} = 2.5^\circ$

$h = -14 \rightarrow 14$

$k = -14 \rightarrow 13$

$l = -24 \rightarrow 24$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.101$

$S = 1.10$

1553 reflections

76 parameters

0 restraints

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

$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 4.7811P]$

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

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

$\Delta\rho_{\max} = 1.59 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -1.56 \text{ e } \text{\AA}^{-3}$

Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0049 (7)

Absolute structure: Flack (1983), 543 Friedel
pairs

Absolute structure parameter: -0.01 (5)

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 $F^2 > 2\sigma(F^2)$ 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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| Ag1 | 0.18579 (4) | 0.81421 (4) | 1.0833 | 0.03836 (17) |
| Mg | 0.10244 (16) | 0.55122 (8) | 0.9167 | 0.0065 (2) |
| P1 | 0.16930 (8) | 0.78125 (8) | 0.75206 (4) | 0.00471 (13) |
| B1 | -0.1513 (2) | 0.6974 (5) | 0.7500 | 0.0052 (6) |
| O1 | 0.1362 (3) | 0.6230 (3) | 0.79151 (13) | 0.0098 (4) |
| O2 | 0.3176 (3) | 0.9319 (3) | 0.78429 (13) | 0.0103 (4) |
| O3 | 0.1801 (3) | 0.7639 (3) | 0.65405 (12) | 0.0070 (3) |
| O4 | 0.0211 (2) | 0.8082 (2) | 0.76691 (12) | 0.0067 (3) |
| O5 | 0.1244 (8) | 1.0000 | 1.0000 | 0.082 (3) |
| H5A | 0.0727 | 0.9715 | 1.0473 | 0.098* |
| O6 | 0.2931 (3) | 0.7990 (3) | 0.94403 (14) | 0.0129 (4) |
| H6A | 0.3869 | 0.8124 | 0.9579 | 0.016* |
| H6B | 0.3096 | 0.8516 | 0.8974 | 0.016* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|------------|---------------|---------------|
| Ag1 | 0.0451 (3) | 0.0451 (3) | 0.0305 (3) | 0.0267 (3) | -0.00288 (19) | -0.00288 (19) |
| Mg | 0.0067 (5) | 0.0063 (4) | 0.0066 (5) | 0.0034 (3) | 0.000 | 0.0011 (4) |
| P1 | 0.0044 (2) | 0.0053 (2) | 0.0044 (2) | 0.0023 (2) | 0.0003 (2) | 0.0011 (2) |
| B1 | 0.0057 (11) | 0.0073 (14) | 0.0030 (13) | 0.0036 (7) | 0.0004 (10) | 0.000 |
| O1 | 0.0145 (10) | 0.0083 (9) | 0.0072 (8) | 0.0062 (7) | 0.0010 (7) | 0.0031 (7) |

| | | | | | | |
|----|------------|-------------|------------|------------|-------------|-------------|
| O2 | 0.0054 (8) | 0.0108 (9) | 0.0102 (8) | 0.0008 (7) | -0.0015 (6) | -0.0002 (7) |
| O3 | 0.0103 (8) | 0.0089 (8) | 0.0038 (7) | 0.0062 (7) | 0.0009 (6) | 0.0011 (6) |
| O4 | 0.0039 (7) | 0.0065 (8) | 0.0095 (8) | 0.0023 (6) | -0.0015 (6) | -0.0025 (6) |
| O5 | 0.042 (2) | 0.072 (5) | 0.140 (8) | 0.036 (3) | -0.033 (3) | -0.066 (6) |
| O6 | 0.0099 (9) | 0.0136 (10) | 0.0130 (9) | 0.0041 (8) | -0.0003 (7) | 0.0031 (7) |

Geometric parameters (Å, °)

| | | | |
|--|-------------|--|-------------|
| Ag1—O6 ⁱ | 2.461 (2) | P1—O2 | 1.503 (2) |
| Ag1—O6 | 2.462 (2) | P1—O4 | 1.563 (2) |
| Ag1—O5 | 2.487 (4) | P1—O3 | 1.569 (2) |
| Ag1—O5 ⁱ | 2.487 (4) | B1—O4 | 1.455 (3) |
| Ag1—Mg | 3.4363 (4) | B1—O4 ^v | 1.455 (3) |
| Ag1—Mg ⁱ | 3.4363 (4) | B1—O3 ^{vi} | 1.480 (3) |
| Mg—O2 ⁱⁱ | 2.053 (2) | B1—O3 ⁱⁱ | 1.480 (3) |
| Mg—O2 ⁱⁱⁱ | 2.053 (2) | O2—Mg ^{vii} | 2.053 (2) |
| Mg—O1 | 2.067 (2) | O3—B1 ^{vi} | 1.480 (3) |
| Mg—O1 ^{iv} | 2.067 (2) | O5—Ag1 ^{viii} | 2.487 (4) |
| Mg—O6 | 2.169 (3) | O5—H5A | 0.8600 |
| Mg—O6 ^{iv} | 2.169 (3) | O6—H6A | 0.8600 |
| P1—O1 | 1.503 (2) | O6—H6B | 0.8600 |
| O6 ⁱ —Ag1—O6 | 131.89 (11) | O2 ⁱⁱⁱ —Mg—O6 ^{iv} | 89.01 (10) |
| O6 ⁱ —Ag1—O5 | 148.09 (10) | O1—Mg—O6 ^{iv} | 83.08 (9) |
| O6—Ag1—O5 | 79.32 (11) | O1 ^{iv} —Mg—O6 ^{iv} | 85.88 (9) |
| O6 ⁱ —Ag1—O5 ⁱ | 79.32 (11) | O6—Mg—O6 ^{iv} | 87.91 (14) |
| O6—Ag1—O5 ⁱ | 148.09 (10) | O1—P1—O2 | 115.72 (13) |
| O5—Ag1—O5 ⁱ | 71.0 (2) | O1—P1—O4 | 110.01 (12) |
| O2 ⁱⁱ —Mg—O2 ⁱⁱⁱ | 94.25 (15) | O2—P1—O4 | 106.40 (12) |
| O2 ⁱⁱ —Mg—O1 | 99.94 (9) | O1—P1—O3 | 107.40 (12) |
| O2 ⁱⁱⁱ —Mg—O1 | 90.53 (9) | O2—P1—O3 | 110.86 (12) |
| O2 ⁱⁱ —Mg—O1 ^{iv} | 90.53 (9) | O4—P1—O3 | 106.05 (11) |
| O2 ⁱⁱⁱ —Mg—O1 ^{iv} | 99.94 (9) | O4—B1—O4 ^v | 102.9 (3) |
| O1—Mg—O1 ^{iv} | 164.64 (15) | O4—B1—O3 ^{vi} | 113.29 (11) |
| O2 ⁱⁱ —Mg—O6 | 89.01 (10) | O4 ^v —B1—O3 ^{vi} | 112.88 (11) |
| O2 ⁱⁱⁱ —Mg—O6 | 175.52 (10) | O4—B1—O3 ⁱⁱ | 112.88 (11) |
| O1—Mg—O6 | 85.88 (9) | O4 ^v —B1—O3 ⁱⁱ | 113.29 (11) |
| O1 ^{iv} —Mg—O6 | 83.08 (9) | O3 ^{vi} —B1—O3 ⁱⁱ | 102.0 (3) |
| O2 ⁱⁱ —Mg—O6 ^{iv} | 175.52 (10) | H6A—O6—H6B | 104.9 |

Symmetry codes: (i) $-y+1, -x+1, -z+13/6$; (ii) $y-1, -x+y, z+1/6$; (iii) $y-1, x, -z+5/3$; (iv) $x, x-y+1, -z+11/6$; (v) $-x+y-1, y, -z+3/2$; (vi) $-x, -x+y, -z+4/3$; (vii) $y, x+1, -z+5/3$; (viii) $x-y+1, -y+2, -z+2$.

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O6—H6A \cdots O3 ^{ix} | 0.86 | 1.89 | 2.744 (3) | 175 |

supporting information

| | | | | |
|--------------------------|------|------|-----------|-----|
| O5—H5A···O5 ⁱ | 0.86 | 2.06 | 2.889 (3) | 162 |
| O6—H6B···O2 | 0.86 | 1.93 | 2.781 (3) | 170 |

Symmetry codes: (i) $-y+1, -x+1, -z+13/6$; (ix) $-x+y, -x+1, z+1/3$.