

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

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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  $\text{BO}_4$  and  $\text{PO}_4$  tetrahedra arranged around the  $6_5$  screw axes. The vertex-sharing  $\text{BO}_4$  and  $\text{PO}_4$  tetrahedra form a spiral ribbon of four-membered rings in which  $\text{BO}_4$  and  $\text{PO}_4$  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  $A^I M^{\text{II}}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8]\cdot\text{H}_2\text{O}$ , where  $A^I = \text{Li}, \text{Na}, \text{K}$  or  $\text{NH}_4^+$  and  $M^{\text{II}} = \text{Mg}, \text{Mn}, \text{Fe}, \text{Co}, \text{Ni}, \text{Cu}, \text{Zn}$  or  $\text{Cd}$ .

## Related literature

For isotypic 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,  $P6_52$

$a = 9.4577(4)\text{ \AA}$   
 $c = 15.8301(13)\text{ \AA}$   
 $V = 1226.27(7)\text{ \AA}^3$

$Z = 6$   
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)  
 $T_{\text{min}} = 0.705$ ,  $T_{\text{max}} = 0.741$

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

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

**Table 1**  
Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ ).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O6}-\text{H6A}\cdots\text{O3}^{\text{i}}$	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).

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

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**supplementary materials**

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

H. Zouihri, M. Saadi, B. Jaber and L. El Ammari

### 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  $\text{BO}_4$  and  $\text{PO}_4$  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  $\text{AgO}_6$  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  $\text{BO}_4$  and  $\text{PO}_4$  tetrahedra, arranged around the  $6_5$  screw axes. The ribbons borders are connected with  $\text{Mg}^{2+}$  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).

### 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,  $\text{H}_3\text{BO}_3$ ,  $\text{H}_3\text{PO}_4$  (85%),  $\text{AgNO}_3$  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.

### 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.

## Figures

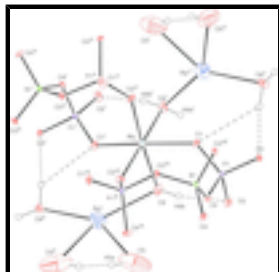


Fig. 1. Partial plot of  $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8] \cdot \text{H}_2\text{O}$  crystal structure showing polyhedra linkage. Displacement ellipsoids are drawn at the 50% probability level. Symmetry codes: (i)  $-y + 1, -x + 1, -z + 13/6$ ; (ii)  $y - 1, -x + y, z + 1/6$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

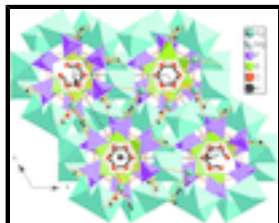


Fig. 2. Projection view of the  $\text{AgMg}(\text{H}_2\text{O})_2[\text{BP}_2\text{O}_8] \cdot \text{H}_2\text{O}$  framework structure showing tunnel running along  $c$  direction where water molecules are located.

## 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

$\omega$  and  $\varphi$  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  $F^2$

Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2 + 4.7811P]$
$wR(F^2) = 0.101$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{\max} < 0.001$
1553 reflections	$\Delta\rho_{\max} = 1.59 \text{ e } \text{\AA}^{-3}$
76 parameters	$\Delta\rho_{\min} = -1.56 \text{ e } \text{\AA}^{-3}$
0 restraints	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1+0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0049 (7)
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 543 Friedel pairs
	Flack 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 ( $\text{\AA}^2$ )

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O6—H6A $\cdots$ O3 <sup>ix</sup>	0.86	1.89	2.744 (3)	175
O5—H5A $\cdots$ O5 <sup>i</sup>	0.86	2.06	2.889 (3)	162
O6—H6B $\cdots$ O2	0.86	1.93	2.781 (3)	170

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

Fig. 1

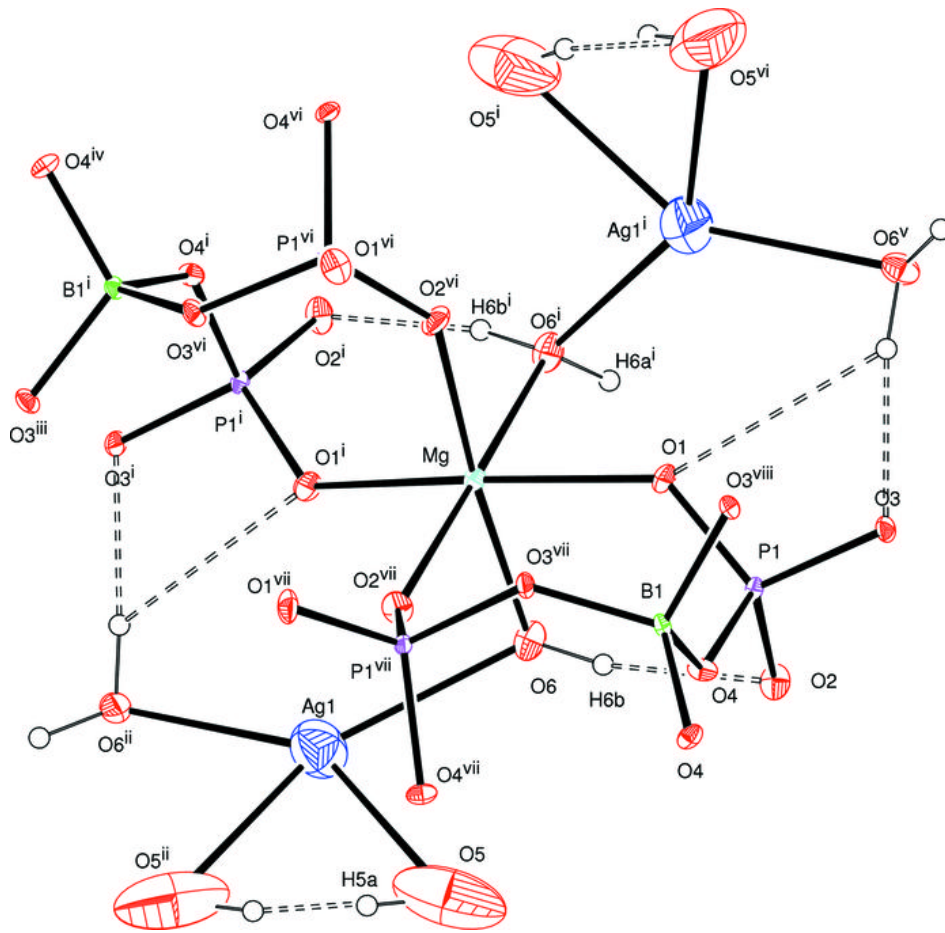




Fig. 2

