

# Poly[bis[ $\mu_2$ -(dimethylazaniumyl)-methylenediphosphonato]magnesium]

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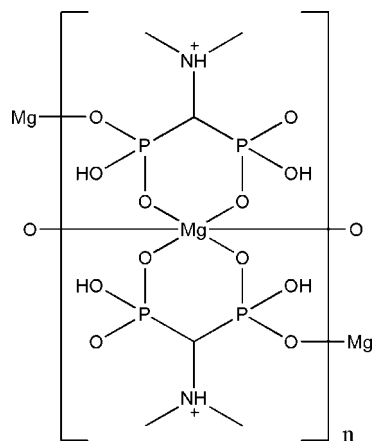
Received 31 January 2011; accepted 17 February 2011

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{N}-\text{C}) = 0.002$  Å;  $R$  factor = 0.024;  $wR$  factor = 0.066; data-to-parameter ratio = 13.0.

The title compound,  $[\text{Mg}(\text{C}_3\text{H}_{10}\text{NO}_6\text{P}_2)_2]_n$ , synthesized by a hydrothermal method, adopts a one-dimensional polymeric chain structure and is isotypic with the previously reported Cd complex based on the ligand  $N,N$ -dimethylaminomethane-1,1-diphosphonic acid ( $\text{H}_4\text{L}$ ). The asymmetric unit contains one half  $\text{Mg}^{2+}$  ion and one  $\text{H}_3\text{L}^-$  anion. The unique  $\text{Mg}^{2+}$  ion lies on an inversion center and is octahedrally coordinated by O atoms from six phosphonate groups of four different  $\text{H}_3\text{L}^-$  anions. Each  $\text{H}_3\text{L}^-$  anion, with one protonated N atom and two phosphonate OH groups, serves as a tridentate ligand. Two of its six phosphonate O atoms chelate to a  $\text{Mg}^{2+}$  cation in a bidentate fashion, while a third O atom bridges to a neighbouring  $\text{Mg}^{2+}$  ion. The interconnection of  $\text{Mg}^{2+}$  ions by the  $\text{H}_3\text{L}^-$  anions leads to the formation of a polymer chain along the  $a$  axis in which the adjacent  $\text{Mg}^{2+}$  ions are doubly bridged by two equivalent  $\text{H}_3\text{L}^-$  anions. These discrete chains are further assembled into a three-dimensional supra-molecular network *via* O—H...O and N—H...O hydrogen bonds involving the non-coordinated phosphonate O atoms and the protonated N atoms.

## Related literature

For other metal complexes based on the  $N,N$ -dimethylaminomethane-1,1-diphosphonate ligand, see: Du *et al.* (2009, 2010*a,b*). For bond-length data, see: Lutz & Muller (1995); Distler *et al.* (1999); Stock & Bein (2004).



## Experimental

### Crystal data

$[\text{Mg}(\text{C}_3\text{H}_{10}\text{NO}_6\text{P}_2)_2]$   
 $M_r = 460.43$   
 Monoclinic,  $P2_1/n$   
 $a = 5.4507$  (3) Å  
 $b = 11.2166$  (6) Å  
 $c = 12.5770$  (7) Å  
 $\beta = 94.984$  (1)°

$V = 766.03$  (7) Å<sup>3</sup>  
 $Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.61$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.40 \times 0.30 \times 0.24$  mm

### Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Bruker, 2008)  
 $T_{\min} = 0.675$ ,  $T_{\max} = 0.746$

4801 measured reflections  
 1492 independent reflections  
 1447 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.014$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$   
 $wR(F^2) = 0.066$   
 $S = 1.09$   
 1492 reflections

115 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.33$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{O3}$	0.91	2.57	3.0997 (18)	118
$\text{N1}-\text{H1B}\cdots\text{O4}^i$	0.91	2.31	3.1346 (18)	151
$\text{O3}-\text{H3D}\cdots\text{O6}^{ii}$	0.82	1.70	2.5011 (16)	166
$\text{O4}-\text{H4A}\cdots\text{O2}^{iii}$	0.82	1.81	2.6037 (16)	163

Symmetry codes: (i)  $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (ii)  $-x + \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (iii)  $-x + 1, -y + 1, -z + 1$ .

Data collection: SMART (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008) and DIAMOND (Brandenburg, 1999); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5102).

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## supporting information

*Acta Cryst.* (2011). E67, m362–m363 [doi:10.1107/S1600536811005976]

**Poly[bis[ $\mu_2$ -(dimethylazaniumyl)methylenediphosphonato]magnesium]****Qiao-Sheng Hu, Xiao-Yu Deng, Yu-Hui Sun and Zi-Yi Du****S1. Comment**

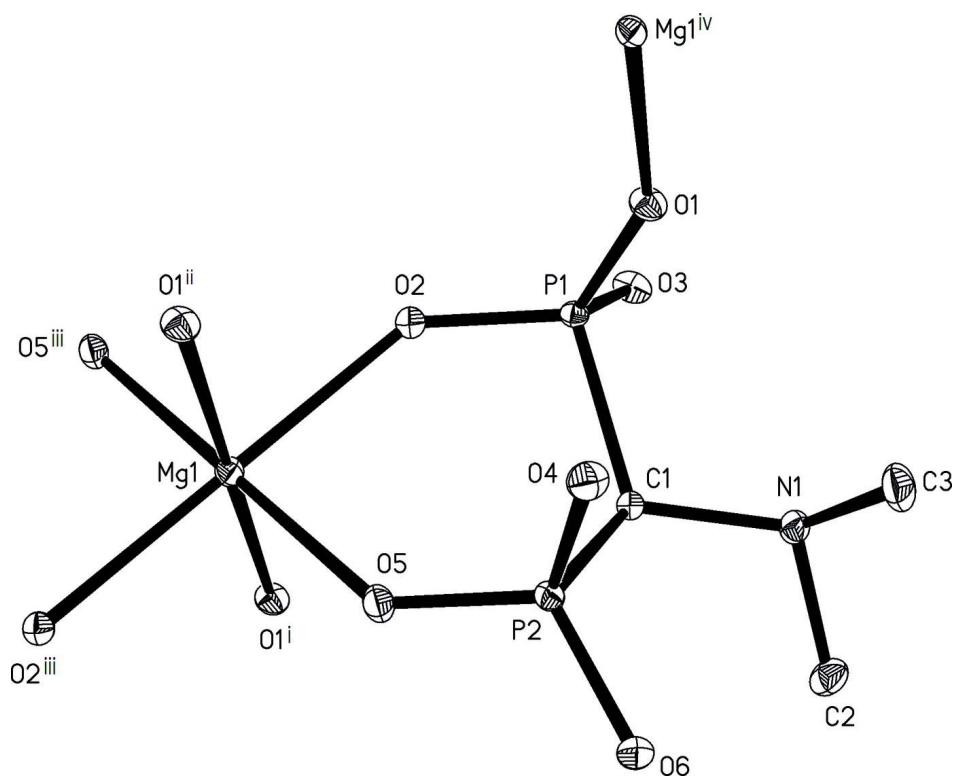
Among many of the phosphonate ligands studied so far, methylenediphosphonic acid and its derivatives are quite unique because they feature a close connection of two phosphonate moieties *via* one carbon atom, which facilitate their combined coordination ability to act as a [CP<sub>2</sub>O<sub>6</sub>] unit rather than two [CPO<sub>3</sub>] units. As a result, they show diversified coordination capabilities with metal ions and thus lead to the formation of new structural types. Recently, by using such ligand types, *i.e.* *N,N*-dimethylaminomethane-1,1-diphosphonate, we have isolated a series of diphosphonate complexes of metals such as Al<sup>III</sup>, Fe<sup>III</sup>, Cd<sup>II</sup>, Pb<sup>II</sup> and Ba<sup>II</sup>, which exhibit variable structures such as zero-dimensional, one-dimensional, double-1-dimensional, double-2-dimensional, and three-dimensional structures (Du *et al.*, 2009, 2010*a,b*). As an expansion of our previous work, we have also obtained a one-dimensional magnesium(II) diphosphonate, namely [Mg(C<sub>6</sub>H<sub>20</sub>N<sub>2</sub>O<sub>12</sub>P<sub>4</sub>)<sub>n</sub>]<sub>n</sub>, which is isostructural with the previously reported cadmium(II) complex based on the same ligand and shows a one-dimensional chain structure. The asymmetric unit contains a half Mg<sup>2+</sup> cation and one H<sub>3</sub>L<sup>-</sup> anion. The unique Mg<sup>2+</sup> cation lies on an inversion center and is octahedrally coordinated by the O atoms of six phosphonate groups from four H<sub>3</sub>L<sup>-</sup> anions. The Mg—O [2.0448 (11) – 2.1879 (11) Å] bond lengths are comparable to those reported for other Mg<sup>II</sup> phosphonate complexes (Lutz & Muller, 1995; Distler *et al.*, 1999; Stock & Bein, 2004). The unique H<sub>3</sub>L<sup>-</sup> anion, with one protonated N atom and two phosphonate OH groups, serves as a tridentate ligand. By using three of its six phosphonate O atoms, it chelates in a bidentate fashion with one Mg<sup>2+</sup> cation and also bridges to a second Mg<sup>2+</sup> ion. The interconnection of Mg<sup>2+</sup> cations by the H<sub>3</sub>L<sup>-</sup> anions leads to the formation of a one-dimensional chain along the *a*-axis, in which the adjacent Mg<sup>2+</sup> ions are doubly bridged by two equivalent H<sub>3</sub>L<sup>-</sup> anions. These discrete one-dimensional chains are further assembled into a three-dimensional supramolecular network *via* O—H $\cdots$ O and N—H $\cdots$ O hydrogen bonds involving the non-coordinated phosphonate O atoms and the protonated N atoms.

**S2. Experimental**

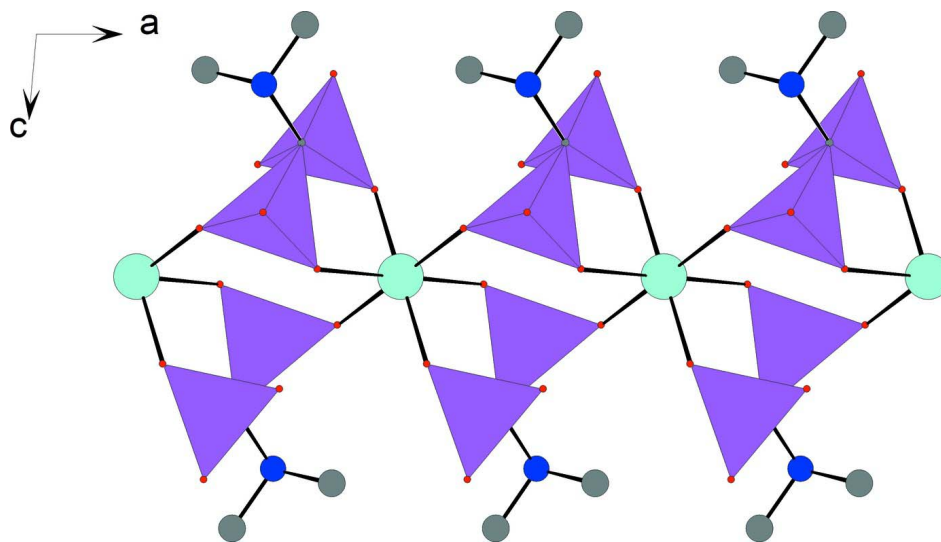
For the preparation of (I), a mixture of Mg(NO<sub>3</sub>)<sub>2</sub> (0.20 mmol) and H<sub>4</sub>L (0.50 mmol) and ethanol (3 ml) in 10 ml distilled water, was sealed into a Parr Teflon-lined autoclave (23 ml) and heated at 393 K for 3 d. Colorless block-shaped crystals were collected in *ca* 55% yield based on Mg. Analysis calculated for C<sub>6</sub>H<sub>20</sub>N<sub>2</sub>O<sub>12</sub>Mg<sub>1</sub>P<sub>4</sub>: C 15.65, H 4.38, N 6.08%; found: C 15.59, H 4.48, N 6.03%.

**S3. Refinement**

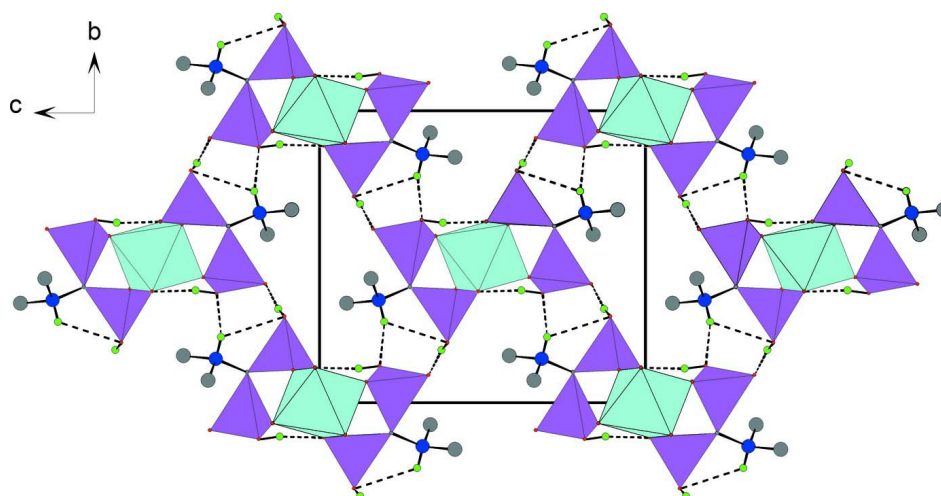
The N-bound and the tertiary C-bound H atoms were positioned geometrically and refined using a riding model: N—H = 0.91 and C—H = 0.98 Å, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N}, \text{C})$ ; while the O-bound and the primary C-bound H atoms were placed in idealized positions and constrained to ride on their parent atoms: O—H = 0.82 and C—H = 0.96 Å, with  $U_{\text{iso}}(\text{H}) = 1.5$  times  $U_{\text{eq}}(\text{O}, \text{C})$ .

**Figure 1**

A view of the selected unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen atoms have been omitted for clarity. [Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $-x + 1, -y + 1, -z + 1$ ; (iii)  $-x, -y + 1, -z + 1$ ; (iv)  $x + 1, y, z$ .]

**Figure 2**

A view of the chain structure of (I) along the  $a$ -axis. The  $\text{CPO}_3$  tetrahedra are shaded in purple. Mg, N and C atoms are drawn as cyan, blue and grey circles, respectively. Hydrogen atoms have been omitted for clarity.

**Figure 3**

A view of the three-dimensional supramolecular structure of (I) down the  $a$ -axis. The  $\text{MgO}_6$  octahedra and  $\text{CPO}_3$  tetrahedra are shaded in cyan and purple, respectively. N, C and H atoms are drawn as blue, grey and green circles, respectively. Hydrogen bonds are represented by dashed lines.

### Poly[bis[ $\mu_2$ -(dimethylazaniumyl)methylenediphosphonato]magnesium]

#### Crystal data

$[\text{Mg}(\text{C}_3\text{H}_{10}\text{NO}_6\text{P}_2)_2]$

$M_r = 460.43$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 5.4507(3)\ \text{\AA}$

$b = 11.2166(6)\ \text{\AA}$

$c = 12.5770(7)\ \text{\AA}$

$\beta = 94.984(1)^\circ$

$V = 766.03(7)\ \text{\AA}^3$

$Z = 2$

$F(000) = 476$

$D_x = 1.996\ \text{Mg m}^{-3}$

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

Cell parameters from 4647 reflections

$\theta = 2.4\text{--}29.4^\circ$

$\mu = 0.61\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colourless

$0.40 \times 0.30 \times 0.24\ \text{mm}$

#### Data collection

Bruker SMART APEX CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.675$ ,  $T_{\max} = 0.746$

4801 measured reflections

1492 independent reflections

1447 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 26.0^\circ$ ,  $\theta_{\min} = 2.4^\circ$

$h = -6 \rightarrow 6$

$k = -13 \rightarrow 13$

$l = -15 \rightarrow 15$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

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

$wR(F^2) = 0.066$

$S = 1.09$

1492 reflections

115 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

$$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2 + 0.6187P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.38 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$$

*Special details*

**Experimental.** IR data (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 3437 (*m*), 3137 (*s*), 3071 (*m*), 2986 (*m*), 2826 (*m*), 2280 (*m*), 1815 (*m*), 1473 (*m*), 1457 (*m*), 1421 (*m*), 1388 (*m*), 1256 (*s*), 1225 (*s*), 1200 (*versus*), 1155 (*s*), 1128 (*s*), 1088 (*s*), 1036 (*s*), 995 (*s*), 950 (*s*), 928 (*s*), 854 (*m*), 827 (*m*), 725 (*m*), 615 (*m*), 573 (*s*), 517 (*m*), 476 (*m*).

**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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mg1	0.0000	0.5000	0.5000	0.01237 (17)
P1	0.52519 (7)	0.34374 (3)	0.59421 (3)	0.01109 (12)
P2	0.34788 (7)	0.55738 (3)	0.72234 (3)	0.01225 (13)
N1	0.5807 (3)	0.35032 (12)	0.81729 (11)	0.0161 (3)
H1B	0.6307	0.2757	0.8007	0.019*
C1	0.4187 (3)	0.39596 (14)	0.72149 (12)	0.0126 (3)
H1A	0.2596	0.3565	0.7262	0.015*
C2	0.4447 (4)	0.33952 (16)	0.91574 (13)	0.0217 (4)
H2A	0.5545	0.3102	0.9737	0.032*
H2B	0.3096	0.2851	0.9024	0.032*
H2C	0.3831	0.4163	0.9341	0.032*
C3	0.8076 (3)	0.42374 (19)	0.84083 (15)	0.0264 (4)
H3A	0.9027	0.3918	0.9021	0.040*
H3B	0.7620	0.5045	0.8550	0.040*
H3C	0.9041	0.4221	0.7805	0.040*
O1	0.7774 (2)	0.38860 (10)	0.57970 (9)	0.0168 (3)
O2	0.3180 (2)	0.38023 (10)	0.51279 (9)	0.0160 (2)
O3	0.5417 (2)	0.20563 (10)	0.60652 (10)	0.0182 (3)
H3D	0.4114	0.1800	0.6253	0.027*
O4	0.5780 (2)	0.62560 (11)	0.68532 (9)	0.0177 (3)
H4A	0.5846	0.6161	0.6210	0.027*
O5	0.1277 (2)	0.57125 (10)	0.64393 (9)	0.0167 (3)
O6	0.3209 (2)	0.59441 (11)	0.83501 (9)	0.0187 (3)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Mg1	0.0099 (4)	0.0144 (4)	0.0127 (4)	-0.0013 (3)	0.0006 (3)	0.0007 (3)
P1	0.0111 (2)	0.0108 (2)	0.0117 (2)	-0.00042 (14)	0.00234 (15)	0.00033 (14)
P2	0.0116 (2)	0.0127 (2)	0.0124 (2)	0.00185 (14)	0.00060 (15)	-0.00118 (14)

N1	0.0179 (7)	0.0157 (7)	0.0144 (6)	0.0044 (5)	-0.0008 (5)	0.0006 (5)
C1	0.0116 (7)	0.0146 (7)	0.0114 (7)	0.0011 (6)	0.0004 (6)	0.0000 (6)
C2	0.0286 (10)	0.0216 (9)	0.0150 (8)	0.0014 (7)	0.0029 (7)	0.0032 (6)
C3	0.0153 (9)	0.0371 (10)	0.0257 (9)	-0.0014 (8)	-0.0047 (7)	0.0022 (8)
O1	0.0137 (6)	0.0172 (6)	0.0200 (6)	-0.0027 (4)	0.0041 (4)	0.0022 (5)
O2	0.0142 (6)	0.0197 (6)	0.0141 (5)	0.0016 (4)	0.0008 (4)	0.0009 (4)
O3	0.0178 (6)	0.0122 (6)	0.0253 (6)	-0.0009 (4)	0.0067 (5)	0.0010 (5)
O4	0.0171 (6)	0.0191 (6)	0.0171 (5)	-0.0036 (5)	0.0022 (5)	-0.0025 (5)
O5	0.0147 (6)	0.0173 (6)	0.0174 (6)	0.0022 (4)	-0.0018 (5)	-0.0004 (4)
O6	0.0196 (6)	0.0217 (6)	0.0148 (6)	0.0060 (5)	0.0015 (5)	-0.0033 (5)

*Geometric parameters (Å, °)*

Mg1—O5 <sup>i</sup>	2.0448 (11)	N1—C3	1.494 (2)
Mg1—O5	2.0448 (11)	N1—C2	1.502 (2)
Mg1—O1 <sup>ii</sup>	2.0615 (11)	N1—C1	1.5196 (19)
Mg1—O1 <sup>iii</sup>	2.0616 (11)	N1—H1B	0.9100
Mg1—O2	2.1879 (11)	C1—H1A	0.9800
Mg1—O2 <sup>i</sup>	2.1879 (11)	C2—H2A	0.9600
P1—O1	1.4898 (12)	C2—H2B	0.9600
P1—O2	1.5134 (12)	C2—H2C	0.9600
P1—O3	1.5587 (12)	C3—H3A	0.9600
P1—C1	1.8451 (15)	C3—H3B	0.9600
P2—O5	1.4938 (12)	C3—H3C	0.9600
P2—O6	1.4961 (12)	O1—Mg1 <sup>iv</sup>	2.0615 (11)
P2—O4	1.5737 (12)	O3—H3D	0.8200
P2—C1	1.8515 (16)	O4—H4A	0.8200
O5 <sup>i</sup> —Mg1—O5	179.999 (1)	C3—N1—C1	112.68 (13)
O5 <sup>i</sup> —Mg1—O1 <sup>ii</sup>	88.62 (5)	C2—N1—C1	112.71 (13)
O5—Mg1—O1 <sup>ii</sup>	91.38 (5)	C3—N1—H1B	107.1
O5 <sup>i</sup> —Mg1—O1 <sup>iii</sup>	91.38 (5)	C2—N1—H1B	107.1
O5—Mg1—O1 <sup>iii</sup>	88.62 (5)	C1—N1—H1B	107.1
O1 <sup>ii</sup> —Mg1—O1 <sup>iii</sup>	180.0	N1—C1—P1	112.08 (10)
O5 <sup>i</sup> —Mg1—O2	91.82 (4)	N1—C1—P2	115.59 (10)
O5—Mg1—O2	88.18 (4)	P1—C1—P2	113.39 (8)
O1 <sup>ii</sup> —Mg1—O2	84.94 (4)	N1—C1—H1A	104.8
O1 <sup>iii</sup> —Mg1—O2	95.06 (4)	P1—C1—H1A	104.8
O5 <sup>i</sup> —Mg1—O2 <sup>i</sup>	88.19 (4)	P2—C1—H1A	104.8
O5—Mg1—O2 <sup>i</sup>	91.82 (4)	N1—C2—H2A	109.5
O1 <sup>ii</sup> —Mg1—O2 <sup>i</sup>	95.06 (4)	N1—C2—H2B	109.5
O1 <sup>iii</sup> —Mg1—O2 <sup>i</sup>	84.94 (4)	H2A—C2—H2B	109.5
O2—Mg1—O2 <sup>i</sup>	180.00 (6)	N1—C2—H2C	109.5
O1—P1—O2	117.90 (7)	H2A—C2—H2C	109.5
O1—P1—O3	107.58 (7)	H2B—C2—H2C	109.5
O2—P1—O3	111.67 (7)	N1—C3—H3A	109.5
O1—P1—C1	111.24 (7)	N1—C3—H3B	109.5
O2—P1—C1	103.22 (7)	H3A—C3—H3B	109.5

O3—P1—C1	104.42 (7)	N1—C3—H3C	109.5
O5—P2—O6	117.19 (7)	H3A—C3—H3C	109.5
O5—P2—O4	111.67 (7)	H3B—C3—H3C	109.5
O6—P2—O4	106.96 (7)	P1—O1—Mg1 <sup>iv</sup>	148.50 (8)
O5—P2—C1	104.71 (7)	P1—O2—Mg1	139.08 (7)
O6—P2—C1	108.34 (7)	P1—O3—H3D	109.5
O4—P2—C1	107.57 (7)	P2—O4—H4A	109.5
C3—N1—C2	109.91 (14)	P2—O5—Mg1	137.38 (7)

Symmetry codes: (i)  $-x, -y+1, -z+1$ ; (ii)  $-x+1, -y+1, -z+1$ ; (iii)  $x-1, y, z$ ; (iv)  $x+1, y, z$ .

*Hydrogen-bond geometry (Å, °)*

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1B...O3	0.91	2.57	3.0997 (18)	118
N1—H1B...O4 <sup>v</sup>	0.91	2.31	3.1346 (18)	151
O3—H3D...O6 <sup>vi</sup>	0.82	1.70	2.5011 (16)	166
O4—H4A...O2 <sup>ii</sup>	0.82	1.81	2.6037 (16)	163

Symmetry codes: (ii)  $-x+1, -y+1, -z+1$ ; (v)  $-x+3/2, y-1/2, -z+3/2$ ; (vi)  $-x+1/2, y-1/2, -z+3/2$ .