research communications

(μ-Methylenediphosphonato-$\kappa^4O,O′:O″,O‴$)bis-[(ethylenediamine-$\kappa^2N,N′$)palladium(II)] tetrahydrate

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The title compound, $[Pd_2(C_2H_8N_2)_2(CH_2O_6P_2)]\cdot 4H_2O$, comprises of a binuclear molecule (point group symmetry 2), with a twofold rotation axis running through the central C atom of the methylenediphosphonate (MDP) anion. The Pd[II] atom has a square-planar coordination environment defined by the N atoms of a bidentate ethylenediamine (en) ligand and two O atoms of the bridging MDP anion. In the crystal structure, metal complexes are arranged in layers parallel (001) and are sandwiched between layers containing disordered water molecules of crystallization. Extensive intralayer hydrogen bonds of the type $N-H\cdots O$ in the metal complex layer and $O-H\cdots O$ in the water layer, as well as $O-H\cdots O$ hydrogen bonds between the two types of layers, lead to the formation a three-dimensional network structure. The two lattice water molecules are each equally disordered over two positions.

1. Chemical context

Platinum drugs are some of the most important and clinically applied anti-cancer agents. Diphosphonic acids are therapeutic agents for treating osteoporosis and metastatic bone diseases. Therefore new complexes designed with a combination of platinum group metals with diphosphonic acid (or derivatives thereof) as bone-targeting groups can improve the chemotherapeutic efficacy in the treatment of bone cancer and can reduce adverse effects. Methylenediphosphonic acid (medronic acid, MDP, H_4L) is the smallest bisphosphonate, which accumulates on the sites of osteoid mineralization and can be used in combination with platinum metals to treat cancer and metastatic bone diseases. Platinum–bisphosphonate complexes, including bis{ethylenediamine)platinum(II)}-medronate, as novel Pt-prodrugs in the local treatment of bone tumors have been reported (Wani et al., 2016; Iafisco et al., 2009; Palazzo et al., 2007; Iafisco & Margiotta, 2012; Margiotta et al., 2009). The preparation and structure determination of [Pt₂(cis-diaminohexane)₂(methylenediphosphonate)] was reported by Bau et al. (1988).
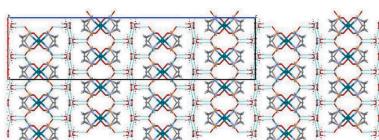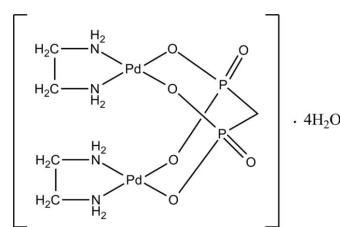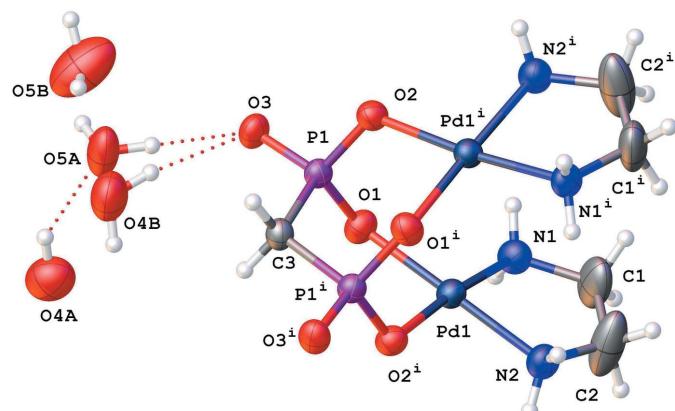
Figure 1
Molecular structure of the title complex and surrounding water molecules with displacement ellipsoids drawn at the 50% probability level. [Symmetry code: (i) $-x + \frac{3}{4}, -y + \frac{3}{4}, z$.]

In this work, we synthesized a new platinum metal complex, *viz*. $[Pd_2(C_2H_8N_2)_2(CH_2O_6P_2)]\cdot4H_2O$ or $[Pd_2(en)_2(MDP)]\cdot4H_2O$, and report here its molecular and crystal structures.

2. Structural commentary

The binuclear $[Pd_2(en)_2(MDP)]$ complex molecule is uncharged and exhibits point group symmetry 2, with the twofold rotation axis passing through the central C atom of the MDP ligand (Fig. 1). The Pd^{II} atom has a square-planar environment defined by two nitrogen atoms (N1 and N2) of the chelating en ligand and two oxygen atoms (O2 and O3) of the bis-bidentately coordinating MDP ligand that bridges two symmetry-related Pd^{II} atoms into the binuclear complex. The deviation of the Pd1 site from the least-squares plane involving the ligand atoms N1, N2, O2 and O3 is 0.06 Å. The Pd—N and Pd—O bond lengths are in typical ranges whereby the Pd—N bonds are about 0.02 Å shorter than the Pd—O bonds (Table 1). The O—Pd—O, N—Pd—N and O—Pd—N bond

Table 1
Selected geometric parameters (Å, °).

|			
Pd1—O1	2.033 (5)	Pd1—N2	2.009 (6)
Pd1—O2^i	2.046 (5)	Pd1—N1	2.021 (5)
O1—Pd1—O2^i	93.32 (19)	N2—Pd1—N1	84.5 (2)
N2—Pd1—O2^i	91.2 (2)	N1—Pd1—O1	90.8 (2)

Symmetry code: (i) $-x + \frac{3}{4}, -y + \frac{3}{4}, z$.

angles vary within 84.5 (2)°–93.3 (2)° (Table 1) and indicate a slight distortion from a square-planar coordination. In general, the structural features of the $[Pd_2(en)_2(MDP)]$ complex are similar to those of the related complex $[Pd_2(en)_2EDP]$ where EDP is 1-hydroxyethane 1,1-diphosphonate or etidronate (Kozachkova *et al.*, 2018).

The Pd1—O1—P1—C3—P1^i—O1^i [symmetry code: (i) $\frac{3}{4} - x$, $\frac{3}{4} - y$, z] six-membered metallacycle adopts a chair conformation; the puckering parameters (Cremer & Pople, 1975; Zefirov *et al.*, 1990) are $S = 1.19$, $\psi = 16.52°$, $\theta = 3.02°$. The Pd1 and C3 atoms deviate by −0.95 and 0.82 Å, respectively, from the least-squares plane of the remaining atoms of this metallacycle with an s.u. of 0.01 Å. The Pd1—N1—C2—C1—N2 five-membered metallacycle involving the en ligand adopts an envelope conformation. The N1 atom deviates by 0.27 Å from the mean plane of the remaining atoms (the s.u. of this plane is 0.005 Å).

3. Supramolecular features

In the crystal, $[Pd_2(en)_2(MDP)]$ molecules are linked via $_{(en)}$N1—H1A⋯O1^{ii}$_{(MDP)}$ hydrogen bonds (Fig. 2, Table 2), forming chains parallel to [010]. Neighboring chains are connected to each other through $_{(en)}$N2—H2A⋯O3^{iii}$_{(MDP)}$ and $_{(en)}$N1—H1B⋯O3^{iv}$_{(MDP)}$ hydrogen bonds, forming layers parallel to (001). The disordered water molecules O4 and O5 are located between these layers and are connected to each other by O—H⋯O hydrogen bonds (Table 2), forming a
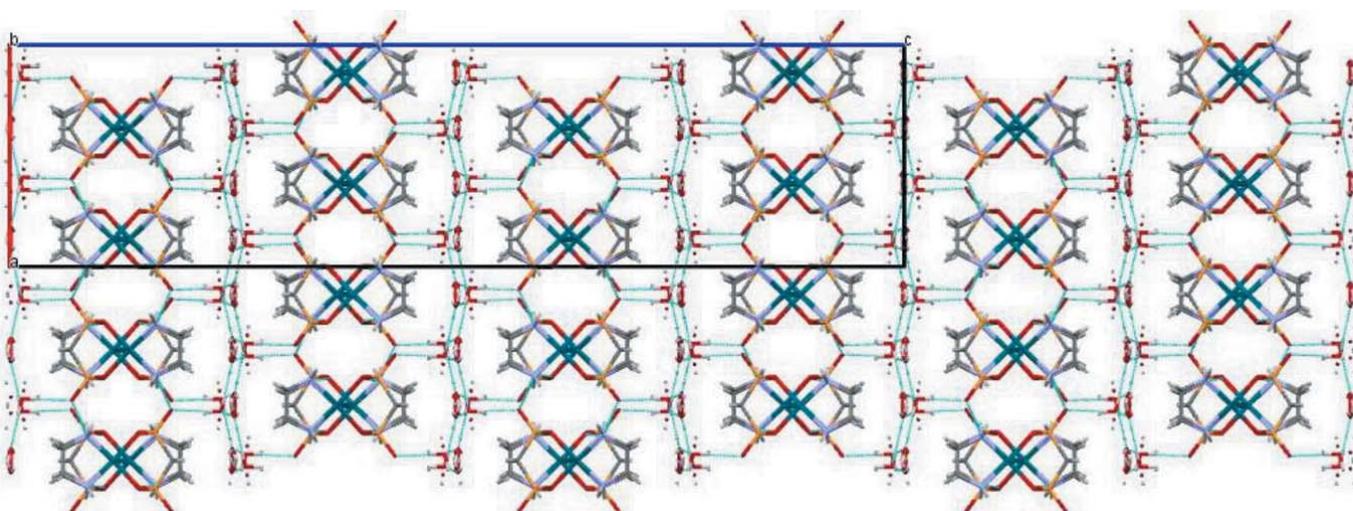
Figure 2
Crystal packing of the title compound in a view along [010] with hydrogen bonds shown as dashed lines.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$A\cdots$O3[ii]	0.89	2.07	2.932 (8)	163
N1—H1$A\cdots$O1[iii]	0.89	2.21	3.006 (8)	149
N1—H1$B\cdots$O3[iv]	0.89	2.06	2.906 (8)	159
O4A—H4$AA\cdots$O5A	0.86	2.05	2.79 (3)	145
O5B—H5$BA\cdots$O5B[v]	0.85	1.56	2.17 (4)	127
O5B—H5$BA\cdots$O4B[v]	0.85	2.29	2.97 (3)	136
O5B—H5$BB\cdots$O5B[vi]	0.85	1.97	2.73 (4)	148
O5B—H5$BB\cdots$O4B[vii]	0.85	2.24	2.78 (2)	121
O5A—H5$AA\cdots$O4A[vii]	0.85	1.98	2.786 (19)	158
O5A—H5$AB\cdots$O3	0.85	1.83	2.675 (16)	176
O4B—H4$BB\cdots$O3	0.85	1.93	2.761 (18)	165

Symmetry codes: (ii) $x + \frac{1}{2}, -y + \frac{3}{4}, -z + \frac{5}{4}$; (iii) $x, -y + \frac{1}{4}, -z + \frac{5}{4}$; (iv) $-x + \frac{1}{4}, y, -z + \frac{5}{4}$; (v) $-x + \frac{1}{2}, -y + 1, -z + \frac{3}{2}$; (vi) $-x + \frac{1}{4}, -y + \frac{5}{4}, z$; (vii) $x - \frac{1}{4}, y + \frac{1}{4}, -z + \frac{3}{2}$.

sandwich-type structure. The structural disorder of the water molecules is probably caused by different possibilities of possible positions favourable for hydrogen bonding with neighbouring molecules. Alternating layers containing [Pd₂(en)₂(MDP)] complexes and water molecules are stacked along [001] and are linked to each other by a series of O—H$\cdots$O hydrogen bonds (O5A—H5$AB\cdots$O3 and O4B—H4$BB\cdots$O3; Table 2).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.39, update November 2017; Groom *et al.*, 2016) for complexes containing the Pd(en) moiety yielded 226 hits with a mean Pd—N bond lengths of 2.028 Å. A search for Pd complexes with MPD as a ligand revealed only one entry (Kutsenko *et al.*, 2014) with Pd—O bond lengths of 1.999 and 2.004 Å.

5. Synthesis and crystallization

[Pd₂(C₂H₈N₂)₂(CH₂O₆P₂)]·4H₂O was prepared as previously reported in the literature (Kozachkova *et al.*, 2018). A solution of AgNO₃ (0.4 mmol, 0.0679 g) in water (3 ml) was added to a suspension of [Pd(en)Cl₂] (0.2 mmol, 0.0474 g) in water (6 ml) under constant stirring and heating at 333 K for 30 min. The resulting suspension was refrigerated to facilitate the precipitation of AgCl. The solid material was removed by suction filtration. MDP (0.1 mmol, 0.0176 g) was added to the filtrate, and the pH was adjusted to 6 by addition of KOH (0.1 mol l^{-1}). The obtained slightly yellow solution was heated at 333 K for 20 min and then left to evaporate at room temperature. Yellow rectangular crystals of the title compound suitable for crystallographic studies were grown by slow evaporation of an aqueous solution at room temperature.

Compound [Pd₂(en)₂(MDP)]·4H₂O: yield 86%. Analysis found: C, 11.5; H, 3.6; N, 10.9; P, 11.8; Pd, 40.8. Calculated for C₅H₂₆N₄O₆P₂Pd₂: C, 11.9; H, 3.9; N, 11.1; P, 12.3; Pd, 41.9%. The ^{31}P-{H} NMR spectrum of an aqueous solutions of the synthesized compound exhibited a singlet with δ^P 35.0 ppm.

Table 3
Experimental details.

Crystal data	
Chemical formula	[Pd₂(C₂H₈N₂)₂(CH₂O₆P₂)]·4H₂O
M_r	577.04
Crystal system, space group	Orthorhombic, $Fddd$
Temperature (K)	294
a, b, c (Å)	11.8871 (6), 12.5405 (6), 48.052 (2)
V (Å³)	7163.2 (6)
Z	16
Radiation type	Mo $K\alpha$
μ (mm^{-1})	2.24
Crystal size (mm)	0.6 × 0.4 × 0.2
Data collection	
Diffractometer	Rigaku Oxford Diffraction Xcalibur, Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.418, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14597, 2065, 1746
R_{int}	0.075
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.175, 1.14
No. of reflections	2065
No. of parameters	126
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.69, −2.06

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Hydrogen atoms of the metal complex were located from difference-Fourier syntheses. The H atom of the central CH₂ bridge of the MDP ligand was refined freely, and the methylene and NH₂ hydrogen atoms of the en ligand were treated in the riding-model approximation with C—H = 0.97 Å (N—H = 0.89 Å) and U_{iso}(H) = $1.2U_{eq}$(C,N). The two lattice water molecules (O4 and O5) are each disordered over two positions and were refined with occupancies 0.5:0.5 each. Their hydrogen atoms were calculated, taking into account the direction of expected hydrogen bonds. The positions of these hydrogen atoms were fixed at the last steps of refinement with O—H = 0.85 Å, and with U_{iso}(H) = $1.5U_{eq}$(O).

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supporting information

(µ-Methylenediphosphonato-κ⁴*O,O′:O′′,O′′′*)bis[(ethylenediamine-κ²*N,N′*)palladium(II)] tetrahydrate

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Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

(µ-Methylenediphosphonato-κ⁴*O,O′:O′′,O′′′*)bis[(ethylenediamine-κ²*N,N′*)palladium(II)] tetrahydrate

Crystal data

[Pd$_2$(C$_2$H$_8$N$_2$)$_2$(CH$_2$O$_6$P$_2$)]·4H$_2$O	D_x = 2.140 Mg m^{-3}
M_r = 577.04	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, *Fddd*	Cell parameters from 2899 reflections
a = 11.8871 (6) Å	θ = 3.2–30.1°
b = 12.5405 (6) Å	μ = 2.24 mm^{-1}
c = 48.052 (2) Å	T = 294 K
V = 7163.2 (6) Å^3	Block, colourless
Z = 16	0.6 × 0.4 × 0.2 mm
$F(000)$ = 4576	

Data collection

Rigaku Oxford Diffraction Xcalibur, Sapphire3 diffractometer	T_{min} = 0.418, T_{max} = 1.000
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source	14597 measured reflections
	2065 independent reflections
Graphite monochromator	1746 reflections with $I > 2\sigma(I)$
Detector resolution: 16.1827 pixels mm^{-1}	R_{int} = 0.075
ω scans	θ_{max} = 27.5°, θ_{min} = 3.2°
Absorption correction: multi-scan (*CrysAlisPro*; Rigaku OD, 2018)	h = −15→8
	k = −16→16
	l = −58→62

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.063	$w = 1/[\sigma^2(F_o^2) + (0.0908P)^2 + 141.6934P]$
$wR(F^2)$ = 0.175	where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max}$ = 0.002
2065 reflections	$\Delta\rho_{max}$ = 1.69 e Å^{-3}
126 parameters	$\Delta\rho_{min}$ = −2.06 e Å^{-3}
0 restraints	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Pd1	0.37981 (4)	0.24678 (4)	0.61611 (2)	0.0284 (2)	
P1	0.24784 (12)	0.37098 (12)	0.66205 (3)	0.0273 (4)	
O1	0.2537 (4)	0.2699 (4)	0.64405 (11)	0.0346 (10)	
O2	0.2468 (4)	0.4739 (4)	0.64479 (10)	0.0322 (10)	
O3	0.1464 (4)	0.3667 (4)	0.68076 (9)	0.0362 (10)	
N2	0.4951 (5)	0.2116 (5)	0.58694 (13)	0.0387 (13)	
H2A	0.542935	0.265756	0.585250	0.046*	
H2B	0.533949	0.154405	0.592230	0.046*	
N1	0.2672 (5)	0.2073 (5)	0.58635 (12)	0.0355 (12)	
H1A	0.235915	0.144735	0.590388	0.043*	
H1B	0.212909	0.256180	0.585660	0.043*	
C3	0.375000	0.375000	0.68243 (19)	0.0299 (17)	
H3	0.373 (6)	0.437 (6)	0.6938 (17)	0.036*	
C1	0.3239 (8)	0.2011 (11)	0.55937 (18)	0.073 (3)	
H1C	0.293176	0.140753	0.549266	0.087*	
H1D	0.305487	0.264816	0.548883	0.087*	
C2	0.4418 (8)	0.1906 (13)	0.5599 (2)	0.089 (4)	
H2C	0.473610	0.239097	0.546319	0.107*	
H2D	0.461070	0.118672	0.554208	0.107*	
O4A	0.1779 (11)	0.1374 (12)	0.7512 (3)	0.072 (4)	0.5
H4AA	0.162933	0.203516	0.754198	0.108*	0.5
H4AB	0.115953	0.105016	0.747549	0.108*	0.5
O5B	0.1617 (16)	0.5219 (16)	0.7487 (5)	0.108 (7)	0.5
H5BA	0.226607	0.542097	0.753922	0.163*	0.5
H5BB	0.118927	0.574297	0.752362	0.163*	0.5
O5A	0.1535 (13)	0.3512 (16)	0.7363 (3)	0.064 (4)	0.5
H5AA	0.084422	0.344140	0.740362	0.096*	0.5
H5AB	0.154092	0.358020	0.718632	0.096*	0.5
O4B	0.1903 (15)	0.2992 (16)	0.7344 (4)	0.069 (5)	0.5
H4BA	0.228698	0.242206	0.733009	0.104*	0.5
H4BB	0.164928	0.316206	0.718439	0.104*	0.5

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.0273 (3)	0.0295 (4)	0.0285 (4)	0.00014 (16)	−0.00047 (18)	−0.00197 (17)
P1	0.0229 (7)	0.0320 (8)	0.0270 (8)	−0.0004 (5)	0.0012 (6)	0.0012 (6)
O1	0.028 (2)	0.041 (3)	0.035 (3)	−0.0021 (18)	0.0028 (19)	−0.005 (2)
O2	0.028 (2)	0.035 (2)	0.034 (2)	0.0019 (17)	0.0010 (18)	0.0029 (19)

O3	0.026 (2)	0.051 (3)	0.031 (2)	0.0004 (18)	0.0045 (18)	−0.001 (2)
N2	0.032 (3)	0.047 (3)	0.036 (3)	0.000 (2)	0.002 (2)	0.000 (3)
N1	0.031 (3)	0.040 (3)	0.035 (3)	−0.003 (2)	−0.006 (2)	−0.005 (2)
C3	0.028 (4)	0.036 (5)	0.025 (4)	−0.001 (3)	0.000	0.000
C1	0.061 (6)	0.126 (10)	0.031 (4)	0.010 (6)	−0.010 (4)	−0.013 (5)
C2	0.047 (5)	0.176 (13)	0.045 (5)	−0.020 (7)	0.009 (4)	−0.028 (7)
O4A	0.058 (7)	0.081 (10)	0.077 (10)	0.006 (7)	−0.006 (7)	−0.007 (8)
O5B	0.074 (11)	0.117 (15)	0.134 (17)	−0.013 (10)	0.010 (11)	−0.061 (13)
O5A	0.046 (8)	0.114 (16)	0.032 (6)	0.000 (8)	0.002 (5)	−0.002 (8)
O4B	0.064 (11)	0.097 (14)	0.047 (8)	0.001 (9)	0.009 (7)	0.002 (9)

Geometric parameters (Å, °)

Pd1—Pd1^i	3.2180 (9)	C3—H3	0.95 (8)
Pd1—Pd1^{ii}	3.1715 (9)	C3—H3^i	0.95 (8)
Pd1—O1	2.033 (5)	C1—H1C	0.9700
Pd1—O2^i	2.046 (5)	C1—H1D	0.9700
Pd1—N2	2.009 (6)	C1—C2	1.408 (14)
Pd1—N1	2.021 (5)	C2—H2C	0.9700
P1—O1	1.536 (5)	C2—H2D	0.9700
P1—O2	1.534 (5)	O4A—H4AA	0.8600
P1—O3	1.506 (4)	O4A—H4AB	0.8590
P1—C3	1.802 (5)	O4A—H4AB^{iii}	0.56 (2)
N2—H2A	0.8900	O5B—H5BA	0.8503
N2—H2B	0.8900	O5B—H5BB	0.8504
N2—C2	1.468 (11)	O5A—H5AA	0.8495
N1—H1A	0.8900	O5A—H5AB	0.8515
N1—H1B	0.8900	O4B—H4BA	0.8502
N1—C1	1.464 (11)	O4B—H4BB	0.8500
Pd1^{ii}—Pd1—Pd1^i	164.246 (17)	Pd1—N1—H1B	109.8
O1—Pd1—Pd1^i	80.27 (14)	H1A—N1—H1B	108.2
O1—Pd1—Pd1^{ii}	87.69 (14)	C1—N1—Pd1	109.6 (5)
O1—Pd1—O2^i	93.32 (19)	C1—N1—H1A	109.8
O2^i—Pd1—Pd1^i	81.11 (13)	C1—N1—H1B	109.8
O2^i—Pd1—Pd1^{ii}	89.54 (13)	P1—C3—P1^i	114.2 (5)
N2—Pd1—Pd1^i	104.11 (19)	P1—C3—H3	108 (4)
N2—Pd1—Pd1^{ii}	88.64 (19)	P1—C3—H3^i	108 (4)
N2—Pd1—O1	174.2 (2)	P1^i—C3—H3^i	108 (4)
N2—Pd1—O2^i	91.2 (2)	P1^i—C3—H3	108 (4)
N2—Pd1—N1	84.5 (2)	H3—C3—H3^i	110 (10)
N1—Pd1—Pd1^{ii}	87.42 (18)	N1—C1—H1C	108.2
N1—Pd1—Pd1^i	102.77 (18)	N1—C1—H1D	108.2
N1—Pd1—O1	90.8 (2)	H1C—C1—H1D	107.3
N1—Pd1—O2^i	174.8 (2)	C2—C1—N1	116.5 (8)
O1—P1—C3	106.9 (2)	C2—C1—H1C	108.2
O2—P1—O1	113.0 (3)	C2—C1—H1D	108.2
O2—P1—C3	106.1 (2)	N2—C2—H2C	108.4

O3—P1—O1	110.1 (3)	N2—C2—H2D	108.4
O3—P1—O2	110.3 (3)	C1—C2—N2	115.4 (8)
O3—P1—C3	110.4 (3)	C1—C2—H2C	108.4
P1—O1—Pd1	121.6 (3)	C1—C2—H2D	108.4
P1—O2—Pd1^i	120.7 (3)	H2C—C2—H2D	107.5
Pd1—N2—H2A	109.4	H4AA—O4A—H4AB	108.2
Pd1—N2—H2B	109.4	H4AA—O4A—H4AB^{iii}	72.3
H2A—N2—H2B	108.0	H4AB—O4A—H4AB^{iii}	38.4
C2—N2—Pd1	111.2 (5)	H5BA—O5B—H5BB	104.5
C2—N2—H2A	109.4	H5AA—O5A—H5AB	104.4
C2—N2—H2B	109.4	H4BA—O4B—H4BB	109.4
Pd1—N1—H1A	109.8		
Pd1—N2—C2—C1	1.3 (14)	O3—P1—O1—Pd1	−179.9 (3)
Pd1—N1—C1—C2	18.4 (13)	O3—P1—O2—Pd1^i	−177.9 (3)
O1—P1—O2—Pd1^i	−54.3 (4)	O3—P1—C3—P1^i	179.4 (2)
O1—P1—C3—P1^i	59.7 (2)	N1—C1—C2—N2	−13.4 (17)
O2—P1—O1—Pd1	56.3 (4)	C3—P1—O1—Pd1	−60.0 (4)
O2—P1—C3—P1^i	−61.1 (2)	C3—P1—O2—Pd1^i	62.5 (4)

Symmetry codes: (i) −x+3/4, −y+3/4, z; (ii) x, −y+1/4, −z+5/4; (iii) −x+1/4, −y+1/4, z.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O3^{iv}	0.89	2.07	2.932 (8)	163
N1—H1A⋯O1^{ii}	0.89	2.21	3.006 (8)	149
N1—H1B⋯O3^v	0.89	2.06	2.906 (8)	159
O4A—H4AA⋯O5A	0.86	2.05	2.79 (3)	145
O5B—H5BA⋯O5B^{vi}	0.85	1.56	2.17 (4)	127
O5B—H5BA⋯O4B^{vi}	0.85	2.29	2.97 (3)	136
O5B—H5BB⋯O5B^{vii}	0.85	1.97	2.73 (4)	148
O5B—H5BB⋯O4B^{viii}	0.85	2.24	2.78 (2)	121
O5A—H5AA⋯O4A^{viii}	0.85	1.98	2.786 (19)	158
O5A—H5AB⋯O3	0.85	1.83	2.675 (16)	176
O4B—H4BB⋯O3	0.85	1.93	2.761 (18)	165

Symmetry codes: (ii) x, −y+1/4, −z+5/4; (iv) x+1/2, −y+3/4, −z+5/4; (v) −x+1/4, y, −z+5/4; (vi) −x+1/2, −y+1, −z+3/2; (vii) −x+1/4, −y+5/4, z; (viii) x−1/4, y+1/4, −z+3/2.