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Tris(ethylenediamine-κ^2N,N')nickel(II) bis(dimethyl phosphate)

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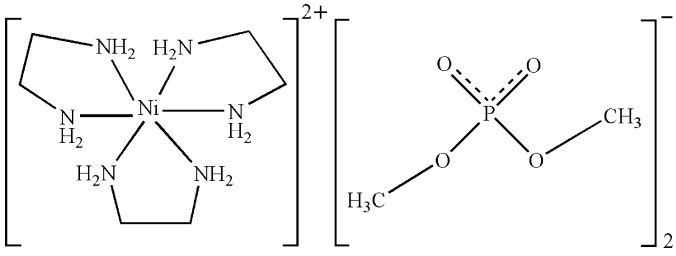
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.085; data-to-parameter ratio = 18.3.

In the title compound, $[Ni(C_2H_8N_2)_3][O_2P(OCH_3)_2]_2$, the Ni^{II} atom is six-coordinated in a distorted octahedral geometry by six N atoms from three ethylenediamine ligands. The P atoms of the anions adopt a distorted tetrahedral geometry. In the crystal, intermolecular N—H···O and C—H···O hydrogen bonds link the cations and anions into a three-dimensional network.

Related literature

For related structures, see: Amani *et al.* (2006); Jun & Zhang (2010); Rafizadeh & Amani (2006*a*,*b*, 2007); Rafizadeh, Amani & Aghayan (2006); Rafizadeh, Amani & Broushaky (2006); Rafizadeh, Hoseinzadeh & Amani (2006); Rafizadeh *et al.* (2005, 2007, 2009).



Experimental

Crystal data

$[Ni(C_2H_8N_2)_3](C_2H_6O_4P)_2$
M_r = 489.08
Monoclinic, $P2_1/n$
a = 9.2553 (5) Å
b = 12.4913 (5) Å
c = 18.190 (1) Å
β = 90.156 (4)°
V = 2102.95 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.12 mm^{-1}
T = 120 K
0.49 × 0.40 × 0.38 mm

Data collection

Stoe IPDS-2T diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED*; Stoe & Cie, 2002)
T_{min} = 0.590, T_{max} = 0.650
9338 measured reflections
5205 independent reflections
4555 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.085
S = 1.08
5205 reflections
284 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*C*···O2	0.90 (3)	2.17 (3)	3.057 (2)	171 (2)
N1—H1*D*···O5^{i}	0.93 (4)	2.36 (4)	3.263 (2)	165 (3)
N2—H2*C*···O6	0.87 (3)	2.35 (3)	3.111 (2)	146 (2)
N2—H2*D*···O6^{ii}	0.84 (3)	2.19 (3)	3.015 (2)	169 (2)
N3—H3*C*···O2^{iii}	0.92 (3)	2.11 (3)	2.971 (2)	157 (2)
N3—H3*D*···O6	0.90 (3)	2.13 (3)	3.002 (2)	163 (2)
N4—H4*C*···O1	0.92 (3)	2.00 (3)	2.910 (2)	176 (3)
N4—H4*D*···O8^{ii}	0.88 (3)	2.40 (3)	3.205 (2)	152 (2)
N5—H5*C*···O5^{i}	0.87 (3)	2.11 (3)	2.911 (2)	154 (2)
N6—H6*C*···O4	0.90	2.35	3.243 (2)	175
N6—H6*D*···O2^{iii}	0.90	2.20	3.064 (2)	160
C9—H9*C*···O1^{iv}	0.96	2.41	3.305 (3)	155

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: HY2566).

References

Amani, V., Rafizadeh, M., Yousefi, M. & Zargar, N. S. (2006). *Anal. Sci.* **22**, x303–x304.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Jun, Q. & Zhang, C. (2010). *Acta Cryst.* E**66**, m24–m25.
Rafizadeh, M. & Amani, V. (2006*a*). *Acta Cryst.* E**62**, m1776–m1777.
Rafizadeh, M. & Amani, V. (2006*b*). *Anal. Sci.* **22**, x211–x212.
Rafizadeh, M. & Amani, V. (2007). *Z. Anorg. Allg. Chem.* **633**, 2738–2741.
Rafizadeh, M., Amani, V. & Aghayan, H. (2006). *Acta Cryst.* E**62**, m2450–m2452.
Rafizadeh, M., Amani, V. & Broushaky, M. (2006). *Anal. Sci.* **22**, x213–x214.
Rafizadeh, M., Amani, V. & Farajian, H. (2007). *Z. Anorg. Allg. Chem.* **633**, 1143–1145.
Rafizadeh, M., Amani, V. & Mortazavi, N. S. (2009). *Bull. Korean Chem. Soc.* **30**, 489–492.
Rafizadeh, M., Hoseinzadeh, F. & Amani, V. (2006). *Anal. Sci.* **22**, x3–x4.
Rafizadeh, M., Tayebee, R., Amani, V. & Nasseh, M. (2005). *Bull. Korean Chem. Soc.* **26**, 594–598.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (2002). *X-AREA*, *X-RED* and *X-SHAPE*. Stoe & Cie, Darmstadt, Germany.

supporting information

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Tris(ethylenediamine-κ^2N,N')nickel(II) bis(dimethyl phosphate)

Masoud Rafizadeh, Hamid Reza Saadati Moshtaghin and Vahid Amani

S1. Comment

In recent years, we reported the synthesis and crystal structure of $[Ni(H_2O)_6](DMP)_2$ (Rafizadeh & Amani, 2006*a*) (DMP = dimethylphosphate anion, $[O_2P(OCH_3)_2]^-$). In this compound, DMP is not bonded to the metal but acts as a counterion. Also, we reported the syntheses and crystal structures of $[Cu_2(\mu\text{-DMP})_4(\mu\text{-DMSO})]_n$ (Rafizadeh *et al.*, 2005), $[UO_2(\mu\text{-DMP})_2(DMSO)]_n$ (Rafizadeh, Hoseinzadeh & Amani, 2006), $[La(\mu\text{-DMP})_2(\mu_3\text{-NO}_3)(DMSO)]_n$ (Rafizadeh, Amani & Broushaky, 2006), $[UO_2(\mu\text{-DEP})_2(DMSO)]_n$ (Rafizadeh & Amani, 2006*b*), $\{[Mn_2(\mu\text{-DMP})_3(\mu\text{-DMSO})_2(DMSO)(H_2O)]NO_3.H_2O\}_n$ (Rafizadeh, Amani, & Aghayan, 2006), $[Ce_2(\mu\text{-DEP})_6(TEP)]_n$ (Amani *et al.*, 2006), $[Mn_2(\mu_3\text{-DMP})_2(\mu\text{-DMP})_2]_n$ (Rafizadeh *et al.*, 2007), $[Nd(\mu\text{-DEP})_3]_n$ and $[Pr(\mu\text{-DMP})_2(\mu\text{-NO}_3)(DMSO)]_n$ (Rafizadeh *et al.*, 2009) (DMSO = dimethylsulfoxide, DEP = diethylphosphate and TEP = triethylphosphate). DMP and DEP act as O-donor ligands, thus forming coordination polymers in solid state. We have also reported the synthesis and crystal structure of $[Fe_{16}(\mu_3\text{-O})_8(\mu_3\text{-OH})_4(\mu\text{-OH})_4(\mu\text{-DMP})_{12}(\mu\text{-OAc})_{12}(DMSO)_4].2DMSO.1.5H_2O$ (Rafizadeh & Amani, 2007), which consists of sixteen iron ions connected by twelve bridging dimethylphosphates, twelve bridging acetates, eight μ_3-oxo, four μ_3-OH, four μ-OH groups and four DMSO ligands. We now report the synthesis and structure of the title compound, which was synthesized by the reaction of $[Ni(H_2O)_6](DMP)_2$ and ethylenediamine (en).

In the title compound (Fig. 1), the Ni^{II} atom is six-coordinated in a distorted octahedral geometry by six N atoms from three en ligands. The Ni—N bond lengths and angles are within normal range as observed in $[Ne(en)_3]_2[Mo(CN)_6].5H_2O$ (Jun & Zhang, 2010). Also, in the $[O_2P(OCH_3)_2]^-$ anions, the P atom is four-coordinated in a distorted tetrahedral geometry. The P—O bond lengths and angles are within normal range as observed in $[Ni(H_2O)_6](DMP)_2$ (Rafizadeh & Amani, 2006*a*). In the crystal, intermolecular N—H···O and C—H···O hydrogen bonds form a three-dimensional network (Table 1, Fig. 2).

S2. Experimental

For the preparation of the title compound, en (0.40 ml, 6.0 mmol) was added to a solution of $[Ni(H_2O)_6](DMP)_2$ (0.83 g, 2.0 mmol) in DMSO (10 ml) and the resulting violet solution was stirred for 2 h at room temperature. This solution was left to evaporate slowly at room temperature. After 2 months, violet block crystals of the title compound were isolated (yield: 0.71 g, 72.6%).

S3. Refinement

H atoms on C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.97 (CH_2) and 0.96 (CH_3) Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. H atoms on N atoms were located from a difference Fourier map and refined isotropically, except those on N6. They were refined as riding atoms, with N—H = 0.90 Å and $U_{iso}(H) = 1.2U_{eq}(N)$.

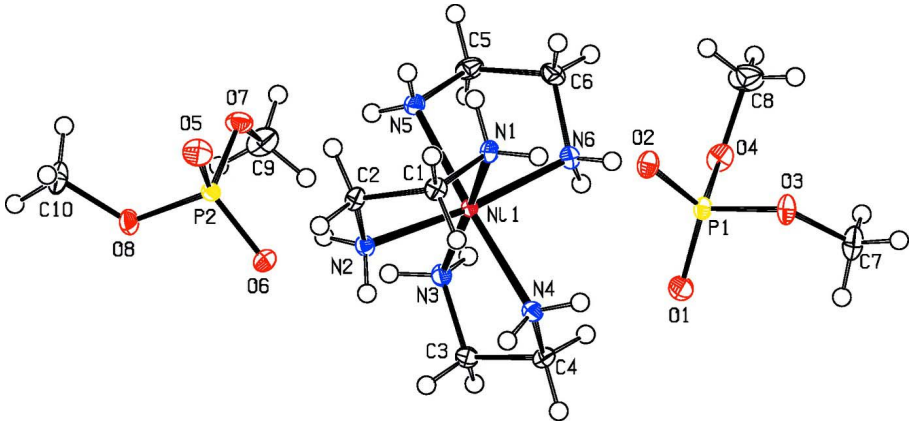

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

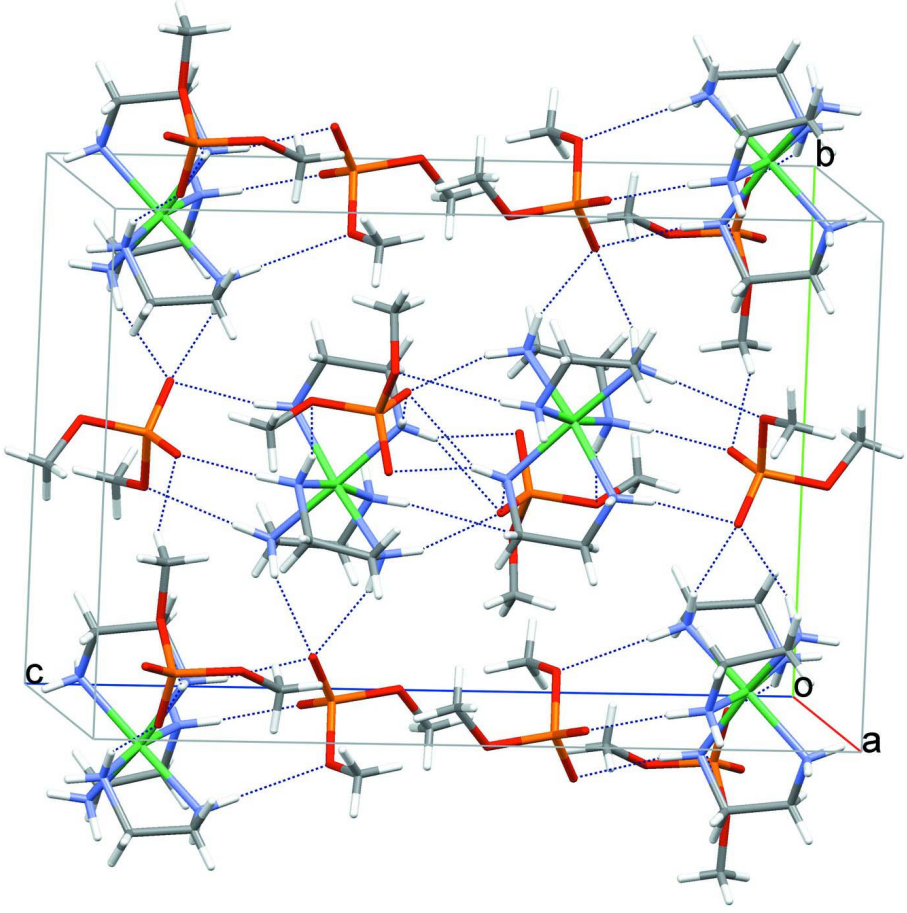

Figure 2

Crystal packing diagram for the title compound. Hydrogen bonds are shown as dashed lines.

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Crystal data

$[Ni(C_2H_8N_2)_3](C_2H_6O_4P)_2$
$M_r = 489.08$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.2553$ (5) Å
$b = 12.4913$ (5) Å
$c = 18.190$ (1) Å
$\beta = 90.156$ (4)°
$V = 2102.95$ (18) Å^3
$Z = 4$
$F(000) = 1040$
$D_x = 1.545$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9338 reflections
$\theta = 2.7$–$29.2°$
$\mu = 1.12$ mm^{-1}
$T = 120$ K
Block, violet
0.49 × 0.40 × 0.38 mm

Data collection

Stoe IPDS-2T diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.15 pixels mm^{-1}
ω scans
Absorption correction: numerical (*X-SHAPE* and *X-RED*; Stoe & Cie, 2002)
$T_{min} = 0.590$, $T_{max} = 0.650$
9338 measured reflections
5205 independent reflections
4555 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 29.2°$, $\theta_{min} = 2.7°$
$h = -12 \rightarrow 10$
$k = -17 \rightarrow 14$
$l = -21 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.085$
$S = 1.08$
5205 reflections
284 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 1.1522P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.014$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.7792 (2)	0.35678 (14)	0.35218 (10)	0.0138 (3)
H1A	0.8421	0.2961	0.3434	0.017*
H1B	0.6870	0.3428	0.3286	0.017*

C2	0.7579 (2)	0.37245 (14)	0.43420 (10)	0.0142 (4)
H2A	0.7108	0.3102	0.4552	0.017*
H2B	0.8506	0.3818	0.4583	0.017*
C3	0.4548 (2)	0.69465 (15)	0.39362 (11)	0.0158 (4)
H3A	0.4030	0.7614	0.4005	0.019*
H3B	0.4050	0.6391	0.4209	0.019*
C4	0.4576 (2)	0.66600 (16)	0.31238 (10)	0.0162 (4)
H4A	0.3602	0.6530	0.2948	0.019*
H4B	0.4984	0.7246	0.2843	0.019*
C5	0.9829 (2)	0.72847 (15)	0.40027 (11)	0.0178 (4)
H5A	1.0827	0.7361	0.4159	0.021*
H5B	0.9296	0.7907	0.4169	0.021*
C6	0.9750 (2)	0.72044 (16)	0.31718 (11)	0.0186 (4)
H6A	1.0093	0.7863	0.2951	0.022*
H6B	1.0355	0.6621	0.3001	0.022*
C7	0.6815 (3)	0.55279 (17)	−0.01227 (11)	0.0242 (4)
H7A	0.7315	0.6196	−0.0064	0.029*
H7B	0.5815	0.5621	0.0001	0.029*
H7C	0.6891	0.5295	−0.0624	0.029*
C8	0.9788 (3)	0.61156 (19)	0.10517 (15)	0.0301 (5)
H8A	0.9829	0.5941	0.0538	0.036*
H8B	1.0351	0.5608	0.1326	0.036*
H8C	1.0169	0.6822	0.1128	0.036*
C9	0.8392 (3)	0.79999 (16)	0.61676 (13)	0.0242 (5)
H9A	0.7931	0.8165	0.6626	0.029*
H9B	0.7737	0.8144	0.5770	0.029*
H9C	0.9242	0.8434	0.6114	0.029*
C10	0.7784 (3)	0.56998 (19)	0.76650 (11)	0.0250 (5)
H10A	0.8726	0.6021	0.7677	0.030*
H10B	0.7877	0.4939	0.7607	0.030*
H10C	0.7290	0.5852	0.8116	0.030*
N1	0.84434 (19)	0.45511 (12)	0.32143 (9)	0.0126 (3)
H1C	0.837 (3)	0.449 (2)	0.2723 (16)	0.022 (6)*
H1D	0.940 (4)	0.456 (3)	0.3362 (18)	0.042 (9)*
N2	0.66754 (19)	0.46838 (12)	0.44496 (9)	0.0118 (3)
H2C	0.682 (3)	0.491 (2)	0.4897 (16)	0.024 (7)*
H2D	0.581 (3)	0.451 (2)	0.4390 (16)	0.026 (7)*
N3	0.60386 (18)	0.70561 (12)	0.42125 (9)	0.0130 (3)
H3C	0.636 (3)	0.773 (2)	0.4098 (14)	0.019 (6)*
H3D	0.606 (3)	0.696 (2)	0.4703 (15)	0.019 (6)*
N4	0.54660 (18)	0.56862 (13)	0.30284 (9)	0.0136 (3)
H4C	0.565 (3)	0.557 (2)	0.2541 (17)	0.029 (7)*
H4D	0.495 (3)	0.512 (2)	0.3150 (15)	0.025 (7)*
N5	0.92017 (18)	0.63031 (13)	0.43245 (9)	0.0133 (3)
H5C	0.981 (3)	0.578 (2)	0.4283 (15)	0.021 (7)*
H5D	0.903 (3)	0.638 (2)	0.4756 (15)	0.020 (6)*
N6	0.82331 (18)	0.70111 (12)	0.29583 (9)	0.0135 (3)
H6C	0.8192	0.6762	0.2495	0.016*

H6D	0.7729	0.7627	0.2979	0.016*
O1	0.59564 (17)	0.52152 (13)	0.14823 (8)	0.0218 (3)
O2	0.82725 (16)	0.40819 (10)	0.15670 (7)	0.0163 (3)
O3	0.74502 (17)	0.47356 (11)	0.03552 (7)	0.0173 (3)
O4	0.83165 (17)	0.60793 (11)	0.12947 (8)	0.0185 (3)
O5	0.83257 (17)	0.49601 (11)	0.61469 (8)	0.0210 (3)
O6	0.62989 (16)	0.62537 (11)	0.57626 (8)	0.0171 (3)
O7	0.87916 (16)	0.68872 (11)	0.61572 (8)	0.0182 (3)
O8	0.69760 (16)	0.61289 (11)	0.70610 (7)	0.0176 (3)
P1	0.74375 (5)	0.49734 (4)	0.12224 (2)	0.01149 (10)
P2	0.75573 (5)	0.59976 (4)	0.62379 (2)	0.01087 (10)
Ni1	0.73359 (2)	0.587559 (17)	0.369019 (12)	0.00877 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0156 (9)	0.0121 (8)	0.0136 (8)	0.0000 (6)	−0.0006 (7)	−0.0019 (6)
C2	0.0162 (9)	0.0140 (8)	0.0124 (8)	0.0002 (7)	−0.0028 (7)	0.0014 (6)
C3	0.0129 (9)	0.0195 (9)	0.0149 (8)	0.0036 (7)	0.0019 (8)	0.0002 (7)
C4	0.0121 (9)	0.0218 (9)	0.0147 (8)	0.0028 (7)	−0.0018 (8)	0.0016 (7)
C5	0.0148 (9)	0.0152 (8)	0.0235 (10)	−0.0036 (7)	−0.0054 (8)	−0.0002 (7)
C6	0.0146 (9)	0.0205 (9)	0.0209 (9)	−0.0031 (7)	0.0030 (8)	0.0054 (7)
C7	0.0346 (13)	0.0236 (10)	0.0144 (9)	−0.0012 (9)	−0.0034 (9)	0.0051 (7)
C8	0.0210 (11)	0.0258 (11)	0.0435 (14)	−0.0081 (9)	−0.0027 (11)	0.0073 (10)
C9	0.0250 (11)	0.0149 (9)	0.0328 (11)	−0.0060 (8)	−0.0085 (10)	0.0026 (8)
C10	0.0312 (13)	0.0319 (11)	0.0118 (9)	0.0004 (9)	−0.0040 (9)	0.0039 (8)
N1	0.0143 (8)	0.0147 (7)	0.0088 (7)	−0.0001 (6)	−0.0005 (7)	−0.0010 (5)
N2	0.0132 (8)	0.0122 (7)	0.0101 (7)	−0.0021 (6)	0.0008 (7)	−0.0006 (5)
N3	0.0140 (8)	0.0135 (7)	0.0115 (7)	0.0008 (6)	0.0008 (6)	0.0006 (5)
N4	0.0118 (7)	0.0172 (7)	0.0120 (7)	−0.0008 (6)	0.0009 (6)	−0.0006 (6)
N5	0.0122 (8)	0.0167 (7)	0.0109 (7)	0.0002 (6)	−0.0007 (6)	−0.0017 (6)
N6	0.0149 (8)	0.0135 (7)	0.0121 (7)	−0.0005 (5)	0.0008 (7)	0.0012 (5)
O1	0.0172 (7)	0.0314 (8)	0.0168 (7)	0.0059 (6)	0.0028 (6)	0.0006 (6)
O2	0.0215 (7)	0.0128 (6)	0.0145 (6)	0.0022 (5)	−0.0014 (6)	0.0010 (5)
O3	0.0269 (8)	0.0160 (6)	0.0089 (6)	0.0010 (5)	−0.0006 (6)	−0.0001 (5)
O4	0.0225 (8)	0.0123 (6)	0.0206 (7)	−0.0010 (5)	−0.0036 (6)	−0.0013 (5)
O5	0.0211 (7)	0.0174 (7)	0.0244 (7)	0.0039 (6)	0.0023 (7)	−0.0039 (5)
O6	0.0147 (7)	0.0219 (7)	0.0147 (6)	−0.0020 (5)	−0.0037 (6)	0.0006 (5)
O7	0.0129 (7)	0.0181 (6)	0.0236 (7)	−0.0030 (5)	−0.0005 (6)	0.0022 (5)
O8	0.0187 (7)	0.0240 (7)	0.0101 (6)	0.0038 (5)	0.0024 (6)	0.0011 (5)
P1	0.0146 (2)	0.0111 (2)	0.00880 (19)	0.00133 (16)	−0.00036 (18)	−0.00062 (15)
P2	0.0095 (2)	0.0136 (2)	0.0095 (2)	−0.00065 (15)	0.00064 (18)	−0.00094 (15)
Ni1	0.00858 (12)	0.00985 (11)	0.00788 (11)	−0.00036 (8)	0.00025 (9)	0.00034 (7)

Geometric parameters (Å, º)

C1—N1	1.479 (2)	C9—H9C	0.9600
C1—C2	1.518 (2)	C10—O8	1.431 (3)

C1—H1A	0.9700	C10—H10A	0.9600
C1—H1B	0.9700	C10—H10B	0.9600
C2—N2	1.475 (2)	C10—H10C	0.9600
C2—H2A	0.9700	N1—Ni1	2.1313 (15)
C2—H2B	0.9700	N1—H1C	0.90 (3)
C3—N3	1.474 (3)	N1—H1D	0.93 (4)
C3—C4	1.521 (3)	N2—Ni1	2.1220 (15)
C3—H3A	0.9700	N2—H2C	0.87 (3)
C3—H3B	0.9700	N2—H2D	0.83 (3)
C4—N4	1.479 (2)	N3—Ni1	2.1272 (15)
C4—H4A	0.9700	N3—H3C	0.92 (3)
C4—H4B	0.9700	N3—H3D	0.90 (3)
C5—N5	1.478 (2)	N4—Ni1	2.1187 (18)
C5—C6	1.516 (3)	N4—H4C	0.91 (3)
C5—H5A	0.9700	N4—H4D	0.89 (3)
C5—H5B	0.9700	N5—Ni1	2.1418 (18)
C6—N6	1.476 (3)	N5—H5C	0.86 (3)
C6—H6A	0.9700	N5—H5D	0.81 (3)
C6—H6B	0.9700	N6—Ni1	2.1166 (15)
C7—O3	1.442 (2)	N6—H6C	0.9000
C7—H7A	0.9600	N6—H6D	0.9000
C7—H7B	0.9600	O1—P1	1.4824 (15)
C7—H7C	0.9600	O2—P1	1.4925 (14)
C8—O4	1.434 (3)	O3—P1	1.6052 (14)
C8—H8A	0.9600	O4—P1	1.6084 (14)
C8—H8B	0.9600	O5—P2	1.4878 (14)
C8—H8C	0.9600	O6—P2	1.4835 (16)
C9—O7	1.438 (2)	O7—P2	1.6008 (14)
C9—H9A	0.9600	O8—P2	1.6008 (13)
C9—H9B	0.9600		

N1—C1—C2	108.59 (14)	Ni1—N1—H1D	109 (2)
N1—C1—H1A	110.0	H1C—N1—H1D	111 (3)
C2—C1—H1A	110.0	C2—N2—Ni1	108.62 (11)
N1—C1—H1B	110.0	C2—N2—H2C	107.3 (19)
C2—C1—H1B	110.0	Ni1—N2—H2C	109.7 (18)
H1A—C1—H1B	108.4	C2—N2—H2D	108.2 (19)
N2—C2—C1	108.08 (15)	Ni1—N2—H2D	112 (2)
N2—C2—H2A	110.1	H2C—N2—H2D	111 (3)
C1—C2—H2A	110.1	C3—N3—Ni1	108.20 (11)
N2—C2—H2B	110.1	C3—N3—H3C	108.0 (17)
C1—C2—H2B	110.1	Ni1—N3—H3C	110.7 (16)
H2A—C2—H2B	108.4	C3—N3—H3D	109.8 (17)
N3—C3—C4	109.55 (15)	Ni1—N3—H3D	109.7 (16)
N3—C3—H3A	109.8	H3C—N3—H3D	110 (2)
C4—C3—H3A	109.8	C4—N4—Ni1	107.20 (12)
N3—C3—H3B	109.8	C4—N4—H4C	110.2 (19)
C4—C3—H3B	109.8	Ni1—N4—H4C	115 (2)

H3A—C3—H3B	108.2	C4—N4—H4D	109.3 (17)
N4—C4—C3	108.57 (16)	Ni1—N4—H4D	112.8 (19)
N4—C4—H4A	110.0	H4C—N4—H4D	103 (2)
C3—C4—H4A	110.0	C5—N5—Ni1	108.09 (12)
N4—C4—H4B	110.0	C5—N5—H5C	109.6 (18)
C3—C4—H4B	110.0	Ni1—N5—H5C	106.7 (19)
H4A—C4—H4B	108.4	C5—N5—H5D	111.3 (19)
N5—C5—C6	108.79 (16)	Ni1—N5—H5D	113 (2)
N5—C5—H5A	109.9	H5C—N5—H5D	108 (3)
C6—C5—H5A	109.9	C6—N6—Ni1	108.58 (12)
N5—C5—H5B	109.9	C6—N6—H6C	110.0
C6—C5—H5B	109.9	Ni1—N6—H6C	110.0
H5A—C5—H5B	108.3	C6—N6—H6D	110.0
N6—C6—C5	108.44 (15)	Ni1—N6—H6D	110.0
N6—C6—H6A	110.0	H6C—N6—H6D	108.4
C5—C6—H6A	110.0	C7—O3—P1	117.49 (12)
N6—C6—H6B	110.0	C8—O4—P1	118.86 (13)
C5—C6—H6B	110.0	C9—O7—P2	119.09 (13)
H6A—C6—H6B	108.4	C10—O8—P2	120.22 (13)
O3—C7—H7A	109.5	O1—P1—O2	119.76 (8)
O3—C7—H7B	109.5	O1—P1—O3	111.12 (9)
H7A—C7—H7B	109.5	O2—P1—O3	105.64 (8)
O3—C7—H7C	109.5	O1—P1—O4	105.48 (9)
H7A—C7—H7C	109.5	O2—P1—O4	110.20 (8)
H7B—C7—H7C	109.5	O3—P1—O4	103.54 (8)
O4—C8—H8A	109.5	O6—P2—O5	119.85 (9)
O4—C8—H8B	109.5	O6—P2—O7	110.87 (8)
H8A—C8—H8B	109.5	O5—P2—O7	104.66 (8)
O4—C8—H8C	109.5	O6—P2—O8	104.94 (8)
H8A—C8—H8C	109.5	O5—P2—O8	110.81 (8)
H8B—C8—H8C	109.5	O7—P2—O8	104.86 (8)
O7—C9—H9A	109.5	N6—Ni1—N4	92.22 (6)
O7—C9—H9B	109.5	N6—Ni1—N2	173.64 (7)
H9A—C9—H9B	109.5	N4—Ni1—N2	93.17 (7)
O7—C9—H9C	109.5	N6—Ni1—N3	92.25 (6)
H9A—C9—H9C	109.5	N4—Ni1—N3	82.53 (6)
H9B—C9—H9C	109.5	N2—Ni1—N3	91.81 (6)
O8—C10—H10A	109.5	N6—Ni1—N1	94.29 (6)
O8—C10—H10B	109.5	N4—Ni1—N1	94.33 (6)
H10A—C10—H10B	109.5	N2—Ni1—N1	81.93 (6)
O8—C10—H10C	109.5	N3—Ni1—N1	172.86 (6)
H10A—C10—H10C	109.5	N6—Ni1—N5	81.64 (6)
H10B—C10—H10C	109.5	N4—Ni1—N5	171.90 (6)
C1—N1—Ni1	107.09 (11)	N2—Ni1—N5	93.30 (7)
C1—N1—H1C	105.9 (17)	N3—Ni1—N5	92.40 (7)
Ni1—N1—H1C	116.1 (17)	N1—Ni1—N5	91.40 (7)
C1—N1—H1D	107 (2)		

N1—C1—C2—N2	56.9 (2)	C6—N6—Ni1—N1	−74.78 (13)
N3—C3—C4—N4	55.1 (2)	C6—N6—Ni1—N5	16.01 (12)
N5—C5—C6—N6	55.6 (2)	C4—N4—Ni1—N6	−73.62 (11)
C2—C1—N1—Ni1	−43.53 (17)	C4—N4—Ni1—N2	109.78 (11)
C1—C2—N2—Ni1	−40.33 (18)	C4—N4—Ni1—N3	18.36 (11)
C4—C3—N3—Ni1	−37.22 (17)	C4—N4—Ni1—N1	−168.09 (11)
C3—C4—N4—Ni1	−43.55 (17)	C2—N2—Ni1—N4	107.14 (13)
C6—C5—N5—Ni1	−39.95 (18)	C2—N2—Ni1—N3	−170.24 (13)
C5—C6—N6—Ni1	−42.24 (17)	C2—N2—Ni1—N1	13.21 (13)
C7—O3—P1—O1	53.70 (17)	C2—N2—Ni1—N5	−77.74 (13)
C7—O3—P1—O2	−174.96 (15)	C3—N3—Ni1—N6	102.42 (12)
C7—O3—P1—O4	−59.09 (16)	C3—N3—Ni1—N4	10.48 (12)
C8—O4—P1—O1	−177.29 (16)	C3—N3—Ni1—N2	−82.48 (12)
C8—O4—P1—O2	52.12 (18)	C3—N3—Ni1—N5	−175.86 (12)
C8—O4—P1—O3	−60.47 (18)	C1—N1—Ni1—N6	−168.38 (12)
C9—O7—P2—O6	45.21 (17)	C1—N1—Ni1—N4	−75.83 (12)
C9—O7—P2—O5	175.78 (15)	C1—N1—Ni1—N2	16.76 (12)
C9—O7—P2—O8	−67.52 (17)	C1—N1—Ni1—N5	109.90 (12)
C10—O8—P2—O6	164.87 (15)	C5—N5—Ni1—N6	13.37 (12)
C10—O8—P2—O5	34.15 (18)	C5—N5—Ni1—N2	−170.51 (12)
C10—O8—P2—O7	−78.24 (16)	C5—N5—Ni1—N3	−78.56 (12)
C6—N6—Ni1—N4	−169.29 (12)	C5—N5—Ni1—N1	107.49 (12)
C6—N6—Ni1—N3	108.10 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O2	0.90 (3)	2.17 (3)	3.057 (2)	171 (2)
N1—H1*D*···O5^{i}	0.93 (4)	2.36 (4)	3.263 (2)	165 (3)
N2—H2*C*···O6	0.87 (3)	2.35 (3)	3.111 (2)	146 (2)
N2—H2*D*···O6^{ii}	0.84 (3)	2.19 (3)	3.015 (2)	169 (2)
N3—H3*C*···O2^{iii}	0.92 (3)	2.11 (3)	2.971 (2)	157 (2)
N3—H3*D*···O6	0.90 (3)	2.13 (3)	3.002 (2)	163 (2)
N4—H4*C*···O1	0.92 (3)	2.00 (3)	2.910 (2)	176 (3)
N4—H4*D*···O8^{ii}	0.88 (3)	2.40 (3)	3.205 (2)	152 (2)
N5—H5*C*···O5^{i}	0.87 (3)	2.11 (3)	2.911 (2)	154 (2)
N6—H6*C*···O4	0.90	2.35	3.243 (2)	175
N6—H6*D*···O2^{iii}	0.90	2.20	3.064 (2)	160
C9—H9*C*···O1^{iv}	0.96	2.41	3.305 (3)	155

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+3/2, *y*+1/2, −*z*+1/2; (iv) *x*+1/2, −*y*+3/2, *z*+1/2.