Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Synthesis and crystal structure of bis[*trans*-diaqua(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)nickel(II)] *trans*-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)bis[4,4′,4″-(1,3,5-trimethylbenzene-2,4,6-triyl)tris(hydrogen phenylphosphonato-κO)]nickel(II) decahydrate

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Received 13 June 2022
Accepted 26 June 2022

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

Keywords: crystal structure; cyclam; nickel; triphosphonic acid.

CCDC reference: 2178456

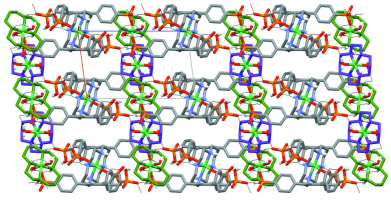
Supporting information: this article has supporting information at journals.iucr.org/e

The components of the title compound, $[Ni(C_{10}H_{24}N_4)(H_2O)_2]_2[Ni(C_{10}H_{24}N_4)(C_{27}H_{24}O_9P_3)_2]\cdot10H_2O$ are two centrosymmetric $[Ni(C_{10}H_{24}N_4)(H_2O)_2]^{2+}$ dications, a centrosymmetric $[Ni(C_{10}H_{24}N_4)(C_{27}H_{24}O_9P_3)_2]^{4-}$ tetra-anion and five crystallographically unique water molecules of crystallization. All of the nickel ions are coordinated by the four secondary N atoms of the macrocyclic cyclam ligands, which adopt the most energetically stable *trans*-III conformation, and the mutually *trans* O atoms of either water molecules in the cations or the phosphonate groups in the anion in a tetragonally distorted NiN_4O_2 octahedral coordination geometry. Strong O—H···O hydrogen bonds between the protonated and the non-protonated phosphonate O atoms of neighboring anions result in the formation of layers oriented parallel to the *bc* plane, which are linked into a three-dimensional network by virtue of numerous N—H···O and O—H···O hydrogen bonds arising from the *sec*-NH groups of the macrocycles, phosphonate O atoms and coordinated and non-coordinated water molecules.

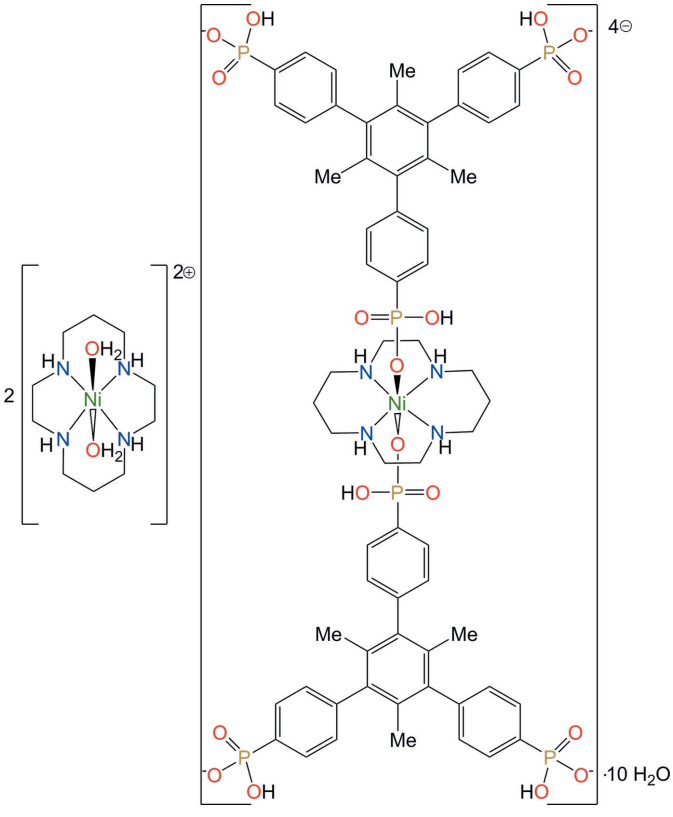
1. Chemical context

First-row transition-metal complexes of 14-membered cyclam-like tetraaza macrocyles (cyclam = 1,4,8,11-tetraazacyclotetradecane; $C_{10}H_{24}N_4$; *L*) are characterized by high thermodynamic stability and kinetic inertness (Yatsimirskii & Lampeka, 1985) and are popular metal-containing building units for the construction of MOFs (Lampeka & Tsymbal, 2004; Suh & Moon, 2007; Suh *et al.*, 2012; Stackhouse & Ma, 2018). These crystalline coordination polymers, in which oligocarboxylates are the most common bridging ligands (Rao *et al.*, 2004), possess permanent porosity and demonstrate many promising applications in different areas (MacGillivray & Lukehart, 2014; Kaskel, 2016).

The rigid trigonal aromatic linker 1,3,5-benzenetricarboxylate, $C_9H_3O_6^{3-}$, is widely used for the assembly of MOFs based on azamacrocyclic cations (Lampeka & Tsymbal, 2004). Its tris-monodentate coordination in the *trans*-axial coordination positions of the metal ions leads predominantly to the formation of two-dimensional coordination polymers with hexagonal nets of 6^3 topology (Alexandrov *et al.*, 2017).



Usually, the modification of this bridge through the substitution of the carboxylic groups by *para*-carboxybenzyl fragments (the ligand H_3BTB, 4,4′,4″-benzene-1,3,5-triyltribenzoic acid) does not affect the coordination properties of the carboxylate groups or the topological characteristics of polymeric nets but results in the enlargement of the hexagonal structural unit of the coordination polymers allowing interpenetration of the subnets (Lampeka *et al.*, 2012; Gong *et al.*, 2016). Compared to carboxylates, linkers with other coordinating functions, in particular oligophosphonates, have been studied to a much lesser extent (Gagnon *et al.*, 2012; Firmino *et al.*, 2018; Yücesan *et al.*, 2018), though one can expect that the substitution of a mono-anionic carboxylic group by a di-anionic phosphonate group with distinct acidity, number of donor atoms and spatial directivity of coordination bonds will strongly influence the composition and topology of the coordination nets. However, except for a very recent publication (Tsymbal *et al.*, 2022), no papers dealing with structural characterization of the complexes formed by metal azamacrocyclic cations and phosphonate ligands have been published to date.



We report here the synthesis and crystal structure of the product of the reaction of $[Ni(L)](ClO_4)_2$ with 4,4′,4″-(1,3,5-trimethylbenzene-2,4,6-triyl)triphosphonic acid, H_6Me_3BTP) – the structural analogue of H_3BTB, namely, bis[*trans*-diaqua-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)-nickel(II)] *trans*-{bis-[4,4′,4″-(1,3,5-trimethylbenzene-2,4,6-triyl)tris(hydrogen phenylphosphonato-κO)-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)-nickel(II)]} decahydrate, $[Ni(L)(H_2O)_2]_2[Ni(L)(H_3Me_3BTP)_2]\cdot10H_2O$, **I**.

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.067 (4)	Ni2—O1*W*	2.105 (4)
Ni1—N2	2.064 (4)	Ni3—N5	2.070 (4)
Ni1—O1	2.134 (3)	Ni3—N6	2.056 (5)
Ni2—N3	2.072 (4)	Ni3—O2*W*	2.136 (3)
Ni2—N4	2.076 (4)		
N1—Ni1—N2^{i}	85.31 (16)	N3—Ni2—N4	95.34 (16)
N1—Ni1—N2	94.69 (16)	N5—Ni3—N6^{iii}	85.2 (2)
N3—Ni2—N4^{ii}	84.66 (16)	N5—Ni3—N6	94.8 (2)

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

2. Structural commentary

The molecular structure of **I** is shown in Fig. 1. It represents a non-polymeric compound in which atom Ni1 is coordinated by two monodentate $H_3Me_3BTP^{3-}$ ligands *via* their phosphonate O atoms, resulting in the formation of an $[Ni(L)(H_3Me_3BTP)_2]^{4-}$ complex anion, which is charge-balanced by two structurally non-equivalent $[Ni(L)(H_2O)_2]^{2+}$ divalent cations formed by atoms Ni2 and Ni3. The coordination geometries of all the nickel ions in **I** have much in common: the Ni^{2+} ions (all with site symmetry $\overline{1}$) are coordinated by the four secondary N atoms of the macrocyclic ligands *L*, which adopt the most energetically stable *trans*-III (*R,R,S,S*) conformation (Bosnich *et al.*, 1965*a*; Barefield *et al.*, 1986) with the five-membered (N—Ni—N bite angles $\simeq 85°$) and six-

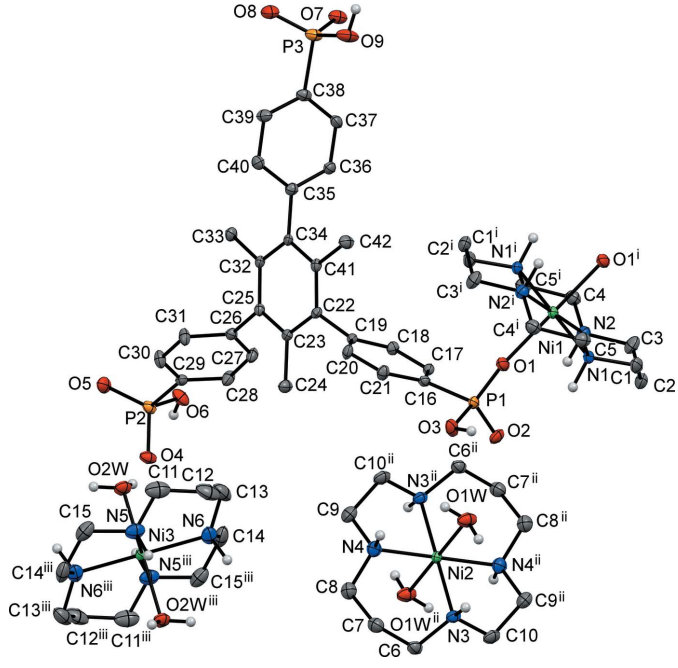


Figure 1
The extended asymmetric unit in **I** showing the coordination environment of the Ni atoms and the atom-labeling scheme (displacement ellipsoids are drawn at the 30% probability level). C-bound H atoms and uncoordinated water molecules are omitted for clarity. Symmetry codes: (i) $-x+2,-y+1,-z+2$; (ii) $-x+2,-y+2,-z+1$; (iii) $-x+1,-y+3,-z+1$.

membered (N—Ni—N bite angles $\simeq 95°$) chelate rings in *gauche* and chair conformations, respectively (Table 1). The coordination polyhedra of the metal ions can be described as tetragonally elongated *trans*-NiN_4O_2 octahedra with the Ni—N bond lengths [average value 2.068 (3) Å] slightly shorter than the Ni—O bonds which, in turn, do not display any dependence on the nature of the donor oxygen atoms. The location of the metal ions on crystallographic inversion centers enforces strict planarity of the $Ni(N_4)$ coordination moieties and the axial Ni—O bonds are nearly orthogonal to the NiN_4 planes (deviations of the angles N—Ni—O from 90° do not exceed 2°).

The pendant benzene rings of the $H_3Me_3BTP^{3-}$ tri-anion in **I** are substantially tilted relative to the central aromatic core [average angle between the mean planes = 76 (5)°] and this feature is caused by repulsive interactions between the hydrogen atoms of the pendant rings and those of the methyl substituents of the central ring. The P—OH bond lengths [average value 1.57 (3) Å] are larger than the other P—O bonds [average value 1.501 (5) Å], thus indicating the partially delocalized character of the phosphonate groups.

3. Supramolecular features

In the crystal of **I**, the $[Ni1(L)(H_3Me_3BTP)_2]^{4-}$ anions, $[Ni2/Ni3(L)(H_2O)_2]^{2+}$ cations and water molecules of crystallization are linked by numerous hydrogen bonds with participation of the phosphonate groups, the secondary amino groups of the macrocycles and both the coordinated and crystalline water molecules (Table 2). A distinct lamellar structure is inherent for this compound. In particular, strong hydrogen-bonding interactions between the protonated fragments of the P1 and P3 phosphonate groups of one molecule as the donors with the non-protonated O4 and O5 atoms of the P2 group of another molecule as the acceptors [P1—O3—H3*C*···O5($x, y-1, z$), P3—O9—H9*C*···O49($x, y-1, z+1$)], together with a weak N1—H1···O6 ($x, y-1, z$) hydrogen bond between the secondary amino group of the macrocyclic cation [Ni1(*L*)] and protonated P2—O6 phosphonate fragment result in the formation of anionic layers oriented parallel to the *bc* plane. The distance between the parallel mean planes of the staggered by 60° trimethylbenzene rings of neighboring $H_3Me_3BTP^{3-}$ anions is 5.248 (3) Å, thus allowing us to exclude the possibility of aromatic π–π stacking interactions between them. Additionally, the negative charge of the layers are partially compensated by the incorporation within the layers of the $[Ni2(L)(H_2O)_2]^{2+}$ cations *via* hydrogen bonding between the coordinated water molecules and the phosphonate O7 atom [O1*W*—H1W*B*···O7($x, y, z-1$)] (Fig. 2).

The second macrocyclic aqua cation $[Ni3(L)(H_2O)_2]^{2+}$, due to hydrogen bonding of the coordinated water molecule with the phosphonate O4 atom (O2*W*—H2*WB*···O4), serves as the bridge between the layers, arranging them into a three-dimensional network (Fig. 3), which is further stabilized by

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6^{iv}	1.00	2.32	3.196 (5)	146
N2—H2···O6*W*	1.00	2.18	3.039 (6)	143
N3—H3···O7^{v}	1.00	2.13	3.102 (6)	162
N4—H4···O4*W*	1.00	2.06	3.056 (6)	173
N5—H5···O9^{vi}	1.00	2.07	3.003 (6)	155
N6—H6···O7*W*^{ii}	1.00	1.98	2.956 (6)	166
O3—H3*C*···O5^{iv}	0.84	1.84	2.654 (5)	162
O6—H6*C*···O3*W*^{vii}	0.84	1.75	2.550 (5)	159
O9—H9*C*···O4^{viii}	0.84	1.74	2.517 (5)	154
O1*W*—H1*WB*···O7^{v}	0.87	1.81	2.679 (5)	173
O1*W*—H1*WA*···O4*W*	0.87	2.45	3.256 (6)	155
O2*W*—H2*WB*···O4	0.86	1.90	2.729 (5)	164
O2*W*—H2*WA*···O7*W*^{ix}	0.86	1.81	2.675 (6)	174
O3*W*—H3*WB*···O2	0.87	1.81	2.676 (4)	177
O3*W*—H3*WA*···O7^{v}	0.85	1.84	2.689 (5)	174
O4*W*—H4*WB*···O3	0.87	2.26	3.115 (6)	167
O4*W*—H4*WA*···O8^{v}	0.87	1.93	2.796 (6)	172
O5*W*—H5*WB*···O5^{x}	0.87	1.98	2.813 (5)	159
O5*W*—H5*WA*···O8^{xi}	0.87	1.87	2.725 (5)	168
O6*W*—H6*WB*···O2	0.87	2.02	2.799 (6)	149
O6*W*—H6*WA*···O5*W*	0.87	2.00	2.842 (5)	164
O7*W*—H7*WB*···O3*W*	0.85	2.02	2.731 (5)	140
O7*W*—H7*WA*···O5*W*	0.86	1.83	2.688 (5)	173

Symmetry codes: (ii) $-x+2, -y+2, -z+1$; (iv) $x, y-1, z$; (v) $x, y, z-1$; (vi) $-x+1, -y+2, -z+2$; (vii) $x, y+1, z$; (viii) $x, y-1, z+1$; (ix) $x-1, y+1, z$; (x) $x+1, y-1, z$; (xi) $x+1, y, z-1$.

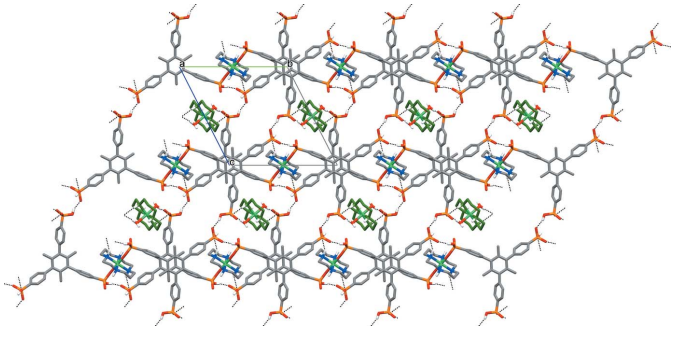


Figure 2
The hydrogen-bonded (dashed lines) layers in **I** viewed down the *a* axis. C-bound H atoms and macrocyclic cations formed by Ni3 have been omitted; C and N atoms of the macrocyclic cations formed by Ni2 are shown in green.

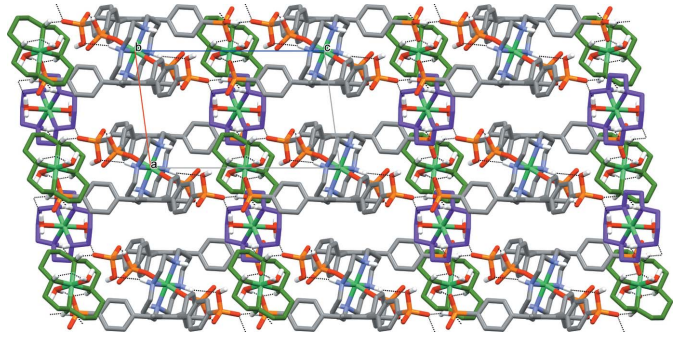


Figure 3
The structure of **I** viewed down the *b* axis. C-bound H atoms have been omitted; C and N atoms of the macrocyclic cation formed by Ni2 and Ni3 are shown in green and violet, respectively. Water molecules of crystallization are not shown; hydrogen bonds are depicted as dashed lines.

numerous O—H···O hydrogen bonds involving the water molecules of crystallization, O3*W*–O7*W* (Table 2).

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.43, last update March 2022; Groom *et al.*, 2016) gave no hits related to H_6Me_3BTP or its complexes with metal ions, so the present work is the first structural characterization of a complex of this ligand. At the same time, several works dealing with the structures of the non-methylated analogue of the phosphonate under consideration, namely, 4,4′,4″-benzene-1,3,5-triyl-triphosphonic acid (H_6BTP), have been published. They include a methanol solvate of the free acid (CSD refcode AKEPEO; Vilela *et al.*, 2021) and its pyridinium salt (YOLGEM; Beckmann *et al.*, 2008), molecular complexes with solvated Co^{II} and Ni^{II} ions (OQIZAR and OQIZEV; Pili *et al.*, 2016) and coordination polymers formed by Sr^{II} (SOTZOR; Vaidhyanathan *et al.*, 2009), Zn^{II} (ISELAV02; Hermer *et al.*, 2016), Y^{III} (AKEPOY; Vilela *et al.*, 2021), Zr^{IV} (COCLIR; Taddei *et al.*, 2014) and $V^{IV/V}$ (COQNAY; Ouellette *et al.*, 2009). Interestingly, as in **I**, in all the metal complexes except COCLIR and ISELAV02, the ligand acts as a H_3BTP^{3-} tri-anion with three monodeprotonated phosphonate groups. On the other hand, because of the absence of methyl substituents, the molecules of the anions $H_nBTP^{(6-n)-}$ as a whole are flatter than $H_3Me_3BTP^{3-}$ in **I** with a maximal tilting angle of pendant *versus* central benzene rings of *ca* 49° observed in ISELAV02. In addition, in the majority of complexes formed by $H_nBTP^{(6-n)-}$ ligands (except AKEPOY and ISELAV02), aromatic π–π stacking interactions of different strengths are observed with centroid-to-centroid distances between the central aromatic rings ranging from 3.4 to 3.9 Å.

The Cambridge Structural Database contains also 18 hits describing the structure of the $[Ni(L)(H_2O)_2]^{2+}$ complex cation in salts of different inorganic and organic anions as well as the charge-compensating part in anionic coordination polymers. In general, the structure of this cation in **I** is similar to other compounds, both from the point of view of the conformation of the macrocycle and the bond distances and angles characterizing the coordination polyhedron of the metal.

5. Synthesis and crystallization

All chemicals and solvents used in this work were purchased from Sigma–Aldrich and used without further purification. The acid H_6Me_3BTP was synthesized according to a procedure described previously for the preparation of H_6BTP (Vaidhyanathan *et al.*, 2009), starting from 1,3,5-trimethyl-2,4,6-tris(4′-bromophenyl)benzene instead of 1,3,5-tris(4′-bromophenyl)benzene. The complex $[Ni(L)](ClO_4)_2$ was prepared from ethanol solutions as described in the literature (Bosnich *et al.*, 1965*b*).

The title compound **$[Ni(L)(H_2O)_2]_2[Ni(L)(H_3Me_3BTP)_2]\cdot 10H_2O$**, **I**, was prepared as follows. A solution of $[Ni(L)](ClO_4)_2$ (46 mg, 0.1 mmol) in 5 ml of water was added to 5 ml of an aqueous solution of H_6Me_3BTP (18 mg, 0.03 mmol) containing 2 ml of pyridine. The pink precipitate, which formed in a week, was filtered off, washed with small amounts of water, methanol and diethyl ether, and dried in air. Yield: 7 mg (10% based on acid). Analysis calculated for $C_{84}H_{148}N_{12}Ni_3O_{32}P_6$: C 45.85, H 6.78, N 7.64%. Found: C 45.73, H 6.87, N 7.51%. Single crystals of **I** suitable for X-ray diffraction analysis were selected from the sample resulting from the synthesis.

Caution! Perchlorate salts of metal complexes are potentially explosive and should be handled with care.

Table 3
Experimental details.

Crystal data	
Chemical formula	$[Ni(C_{10}H_{24}N_4)(H_2O)_2]_2$-$[Ni(C_{10}H_{24}N_4)(C_{27}H_{24}O_9P_3)_2]\cdot$-$10H_2O$
M_r	2200.09
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	160
a, b, c (Å)	9.8779 (5), 17.2467 (11), 17.6707 (11)
α, β, γ (°)	61.409 (6), 77.515 (5), 77.713 (5)
V (Å^3)	2559.7 (3)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.72
Crystal size (mm)	0.40 × 0.10 × 0.10
Data collection	
Diffractometer	Rigaku Xcalibur Eos
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2020)
T_{min}, T_{max}	0.701, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23737, 9657, 6598
R_{int}	0.063
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.610
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.067, 0.161, 1.02
No. of reflections	9657
No. of parameters	629
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.67, −0.46

Computer programs: *CrysAlis PRO* (Rigaku OD, 2020), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms in **I** were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.95 Å (ring H atoms), 0.98 Å (methyl H atoms), 0.99 Å (methylene H atoms), N—H distances of 1.00 Å, O—H distances of 0.84 Å (protonated phosphonate groups) and 0.87 Å (water molecules) with U_{iso}(H) values of 1.2U_{eq} or 1.5U_{eq} times those of the corresponding parent atoms.

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supporting information

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Synthesis and crystal structure of bis[*trans*-diaqua(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)nickel(II)] *trans*-(1,4,8,11-tetraazacyclotetradecane-$\kappa^4N^1,N^4,N^8,N^{11}$)bis[4,4′,4′′-(1,3,5-trimethylbenzene-2,4,6-triyl)tris(hydrogen phenylphosphonato-κO)]nickel(II) decahydrate

Liudmyla V. Tsymbal, Rodinel Ardeleanu, Sergiu Shova and Yaroslaw D. Lampeka

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2020); cell refinement: *CrysAlis PRO* (Rigaku OD, 2020); data reduction: *CrysAlis PRO* (Rigaku OD, 2020); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

\ Bis[*trans*-diaqua(1,4,8,11-tetraazacyclotetradecane-κ^4N^1,\ N^4,N^8,N^{11})nickel(II)] *trans*-(1,4,8,11-tetraazacyclotetradecane-κ^4N^1,N^4,\ N^8,N^{11})bis[4,4′,4′′-(1,3,5-trimethylbenzene-2,4,6-triyl)\ tris(hydrogen phenylphosphonato-κO)]nickel(II) decahydrate

Crystal data

$[Ni(C_{10}H_{24}N_4)(H_2O)_2]_2[Ni(C_{10}H_{24}N_4)(C_{27}H_{24}O_9P_3)_2]\cdot 10H_2O$
$M_r = 2200.09$
Triclinic, $P\overline{1}$
$a = 9.8779$ (5) Å
$b = 17.2467$ (11) Å
$c = 17.6707$ (11) Å
$\alpha = 61.409$ (6)°
$\beta = 77.515$ (5)°
$\gamma = 77.713$ (5)°
$V = 2559.7$ (3) Å^3
$Z = 1$
$F(000) = 1166$
$D_x = 1.427$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5403 reflections
$\theta = 2.1$–$26.3°$
$\mu = 0.72$ mm^{-1}
$T = 160$ K
Prism, clear light colourless
0.40 × 0.10 × 0.10 mm

Data collection

Rigaku Xcalibur Eos diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1593 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2020)
$T_{min} = 0.701$, $T_{max} = 1.000$
23737 measured reflections
9657 independent reflections
6598 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 25.7°$, $\theta_{min} = 2.1°$
$h = -11\rightarrow12$
$k = -21\rightarrow20$
$l = -21\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.161$
$S = 1.02$
9657 reflections
629 parameters
1 restraint

Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 4.2781P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.67$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

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}
Ni1	1.000000	0.500000	1.000000	0.0228 (2)
P2	0.77348 (14)	1.51832 (8)	0.73137 (8)	0.0273 (3)
P1	0.86291 (13)	0.70402 (8)	0.82874 (8)	0.0243 (3)
P3	0.75955 (15)	0.78200 (9)	1.49060 (8)	0.0332 (3)
O5	0.6838 (4)	1.5710 (2)	0.7750 (2)	0.0380 (9)
C29	0.7561 (5)	1.4015 (3)	0.7965 (3)	0.0241 (10)
O4	0.7456 (4)	1.5452 (2)	0.6413 (2)	0.0333 (8)
O3W	1.0380 (3)	0.6623 (2)	0.6411 (2)	0.0311 (8)
H3WA	0.998703	0.703601	0.598132	0.047*
H3WB	1.016063	0.687031	0.675752	0.047*
O4W	0.6258 (4)	0.8895 (3)	0.6511 (3)	0.0607 (12)
H4WA	0.622702	0.869018	0.615193	0.091*
H4WB	0.655562	0.843018	0.696393	0.091*
C22	0.7292 (4)	0.9758 (3)	0.9717 (3)	0.0193 (10)
C38	0.7501 (5)	0.8505 (3)	1.3753 (3)	0.0274 (11)
C26	0.7310 (5)	1.2190 (3)	0.9041 (3)	0.0230 (10)
N1	0.9914 (4)	0.4354 (3)	0.9286 (2)	0.0275 (9)
H1	0.956271	0.481640	0.873064	0.033*
O3	0.7242 (4)	0.7049 (2)	0.7985 (2)	0.0387 (9)
H3C	0.724711	0.656089	0.798510	0.058*
C34	0.7272 (4)	0.9926 (3)	1.1008 (3)	0.0211 (10)
N2	1.1919 (4)	0.5419 (3)	0.9365 (3)	0.0330 (10)
H2	1.173656	0.596679	0.881233	0.040*
C3	1.2935 (5)	0.4794 (4)	0.9119 (4)	0.0421 (14)
H3A	1.320107	0.425875	0.965248	0.051*
H3B	1.378830	0.507709	0.878884	0.051*
O2	0.9773 (4)	0.7334 (2)	0.7517 (2)	0.0349 (8)
O9	0.7490 (5)	0.6880 (2)	1.5017 (2)	0.0490 (11)
H9C	0.757613	0.649777	1.553173	0.074*
C35	0.7357 (5)	0.9486 (3)	1.1963 (3)	0.0245 (10)

C41	0.7258 (5)	0.9377 (3)	1.0620 (3)	0.0229 (10)
C23	0.7243 (5)	1.0683 (3)	0.9212 (3)	0.0229 (10)
C30	0.6330 (5)	1.3737 (3)	0.8509 (3)	0.0335 (12)
H30	0.555907	1.416974	0.852158	0.040*
C25	0.7243 (5)	1.1221 (3)	0.9602 (3)	0.0216 (10)
C5	0.8806 (6)	0.3769 (4)	0.9791 (3)	0.0381 (13)
H5A	0.916444	0.324112	1.029994	0.046*
H5B	0.851180	0.356262	0.942372	0.046*
C33	0.7319 (5)	1.1422 (3)	1.0923 (3)	0.0280 (11)
H33A	0.769682	1.105369	1.147602	0.042*
H33B	0.637231	1.170079	1.102996	0.042*
H33C	0.791865	1.188533	1.053584	0.042*
C39	0.6285 (5)	0.9036 (3)	1.3453 (3)	0.0321 (12)
H39	0.549541	0.906986	1.385777	0.039*
C42	0.7241 (6)	0.8387 (3)	1.1158 (3)	0.0333 (12)
H42A	0.818887	0.807974	1.111379	0.050*
H42B	0.661713	0.818665	1.094399	0.050*
H42C	0.690678	0.825159	1.176739	0.050*
O7	0.9019 (4)	0.7838 (2)	1.5071 (2)	0.0399 (9)
C36	0.8576 (5)	0.8993 (3)	1.2269 (3)	0.0283 (11)
H36	0.938419	0.898840	1.186477	0.034*
C19	0.7533 (4)	0.9154 (3)	0.9299 (3)	0.0194 (10)
C21	0.6772 (5)	0.8176 (3)	0.8889 (3)	0.0282 (11)
H21	0.603252	0.793241	0.884480	0.034*
O8	0.6378 (4)	0.8126 (3)	1.5414 (2)	0.0476 (10)
C20	0.6477 (5)	0.8787 (3)	0.9222 (3)	0.0287 (11)
H20	0.553187	0.895677	0.940187	0.034*
C2	1.2358 (6)	0.4512 (4)	0.8569 (3)	0.0407 (14)
H2A	1.196217	0.505379	0.808660	0.049*
H2B	1.314402	0.421415	0.830904	0.049*
C37	0.8665 (5)	0.8500 (3)	1.3151 (3)	0.0306 (12)
H37	0.952338	0.815768	1.334400	0.037*
C32	0.7271 (4)	1.0848 (3)	1.0504 (3)	0.0211 (10)
C24	0.7140 (6)	1.1079 (3)	0.8254 (3)	0.0314 (12)
H24A	0.660985	1.072081	0.815897	0.047*
H24B	0.808141	1.108441	0.793050	0.047*
H24C	0.666278	1.168989	0.805139	0.047*
Ni2	1.000000	1.000000	0.500000	0.0233 (2)
O1W	0.9604 (4)	0.8687 (2)	0.5872 (2)	0.0452 (10)
H1WA	0.881040	0.872384	0.618973	0.068*
H1WB	0.944450	0.844264	0.557414	0.068*
N3	0.9605 (4)	0.9803 (3)	0.4004 (2)	0.0297 (10)
H3	0.924717	0.921388	0.427638	0.036*
C10	1.0986 (5)	0.9720 (4)	0.3496 (3)	0.0380 (13)
H10A	1.128892	1.031639	0.311897	0.046*
H10B	1.091906	0.945314	0.312116	0.046*
N4	0.7940 (5)	1.0447 (3)	0.5321 (3)	0.0377 (11)
H4	0.744740	0.990779	0.568716	0.045*

C9	0.7973 (6)	1.0856 (4)	0.5889 (3)	0.0408 (14)
H9A	0.703665	1.090138	0.621780	0.049*
H9B	0.824723	1.146279	0.553175	0.049*
C7	0.7183 (6)	1.0577 (4)	0.4022 (3)	0.0418 (14)
H7A	0.689758	0.997677	0.440380	0.050*
H7B	0.646435	1.092349	0.362786	0.050*
C6	0.8543 (5)	1.0479 (3)	0.3477 (3)	0.0350 (12)
H6A	0.837243	1.030970	0.304733	0.042*
H6B	0.891090	1.106025	0.315290	0.042*
C8	0.7168 (6)	1.1019 (4)	0.4586 (4)	0.0434 (14)
H8A	0.758644	1.158069	0.423021	0.052*
H8B	0.618830	1.116899	0.480465	0.052*
Ni3	0.500000	1.500000	0.500000	0.0280 (2)
O2W	0.5104 (4)	1.5055 (2)	0.6168 (2)	0.0374 (9)
H2WA	0.451405	1.548647	0.620771	0.056*
H2WB	0.589347	1.520291	0.613928	0.056*
N5	0.3476 (5)	1.4161 (3)	0.5683 (3)	0.0436 (12)
H5	0.333782	1.392139	0.529340	0.052*
O7W	1.3235 (4)	0.6425 (3)	0.6174 (3)	0.0660 (14)
H7WA	1.366682	0.665639	0.637536	0.099*
H7WB	1.244232	0.671069	0.624896	0.099*
N6	0.6644 (5)	1.3992 (3)	0.5280 (3)	0.0464 (12)
H6	0.671366	1.374659	0.486100	0.056*
C14	0.7918 (6)	1.4389 (5)	0.5068 (4)	0.0558 (18)
H14A	0.873557	1.397569	0.499126	0.067*
H14B	0.802863	1.450247	0.554899	0.067*
O6W	1.2369 (4)	0.7283 (3)	0.7946 (2)	0.0541 (11)
H6WA	1.317921	0.726218	0.763661	0.081*
H6WB	1.177461	0.729258	0.764271	0.081*
O5W	1.4763 (3)	0.7022 (2)	0.6828 (2)	0.0395 (9)
H5WA	1.538341	0.732237	0.641517	0.059*
H5WB	1.524801	0.660758	0.722817	0.059*
O1	0.9002 (4)	0.6178 (2)	0.9039 (2)	0.0326 (8)
O6	0.9335 (4)	1.5164 (2)	0.7321 (2)	0.0402 (9)
H6C	0.960853	1.564111	0.692096	0.060*
C17	0.9212 (5)	0.8304 (3)	0.8664 (3)	0.0229 (10)
H17	1.015334	0.815323	0.846193	0.027*
C16	0.8155 (5)	0.7918 (3)	0.8618 (3)	0.0216 (10)
C18	0.8904 (5)	0.8913 (3)	0.9005 (3)	0.0238 (10)
H18	0.964113	0.916614	0.903815	0.029*
C40	0.6201 (5)	0.9525 (3)	1.2561 (3)	0.0302 (12)
H40	0.535465	0.988328	1.236326	0.036*
C1	1.1247 (6)	0.3895 (3)	0.9048 (3)	0.0375 (13)
H1A	1.108999	0.364727	0.867554	0.045*
H1B	1.157958	0.339182	0.958205	0.045*
C28	0.8659 (5)	1.3371 (3)	0.7957 (3)	0.0323 (12)
H28	0.950712	1.353974	0.758385	0.039*
C31	0.6206 (5)	1.2844 (3)	0.9032 (3)	0.0319 (12)

H31	0.534822	1.267424	0.939145	0.038*
C13	0.6486 (8)	1.3227 (4)	0.6163 (4)	0.0614 (19)
H13A	0.651043	1.342483	0.660143	0.074*
H13B	0.728211	1.275571	0.620745	0.074*
C15	0.2154 (6)	1.4748 (5)	0.5758 (4)	0.0502 (16)
H15A	0.212231	1.487228	0.625394	0.060*
H15B	0.133940	1.444182	0.586810	0.060*
C11	0.3777 (8)	1.3401 (5)	0.6484 (4)	0.0606 (19)
H11A	0.300674	1.302890	0.671624	0.073*
H11B	0.383358	1.360212	0.691424	0.073*
C27	0.8528 (5)	1.2485 (3)	0.8489 (3)	0.0315 (12)
H27	0.930342	1.205552	0.847868	0.038*
C12	0.5140 (8)	1.2849 (4)	0.6361 (4)	0.065 (2)
H12A	0.519087	1.227524	0.689642	0.078*
H12B	0.509541	1.271311	0.588269	0.078*
C4	1.2423 (5)	0.5705 (4)	0.9910 (3)	0.0360 (13)
H4A	1.318132	0.607996	0.956861	0.043*
H4B	1.279137	0.517994	1.041693	0.043*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0278 (5)	0.0183 (4)	0.0225 (4)	0.0031 (4)	−0.0059 (3)	−0.0108 (4)
P2	0.0380 (8)	0.0178 (6)	0.0273 (7)	−0.0020 (6)	−0.0117 (6)	−0.0089 (5)
P1	0.0314 (7)	0.0195 (6)	0.0258 (6)	0.0026 (6)	−0.0079 (5)	−0.0139 (5)
P3	0.0495 (9)	0.0290 (7)	0.0222 (7)	−0.0109 (7)	−0.0112 (6)	−0.0074 (6)
O5	0.056 (2)	0.0220 (18)	0.042 (2)	0.0005 (18)	−0.0129 (18)	−0.0184 (17)
C29	0.031 (3)	0.023 (2)	0.019 (2)	0.000 (2)	−0.0063 (19)	−0.010 (2)
O4	0.051 (2)	0.0229 (18)	0.0265 (18)	−0.0077 (17)	−0.0132 (16)	−0.0072 (15)
O3W	0.0317 (19)	0.0298 (19)	0.0354 (19)	0.0021 (16)	−0.0082 (15)	−0.0185 (16)
O4W	0.064 (3)	0.059 (3)	0.060 (3)	−0.013 (2)	−0.008 (2)	−0.026 (2)
C22	0.017 (2)	0.022 (2)	0.020 (2)	−0.002 (2)	0.0003 (18)	−0.012 (2)
C38	0.040 (3)	0.022 (2)	0.025 (3)	−0.007 (2)	−0.011 (2)	−0.010 (2)
C26	0.030 (3)	0.019 (2)	0.022 (2)	−0.002 (2)	−0.005 (2)	−0.011 (2)
N1	0.036 (2)	0.021 (2)	0.026 (2)	0.0064 (19)	−0.0090 (18)	−0.0131 (18)
O3	0.049 (2)	0.027 (2)	0.054 (2)	0.0082 (18)	−0.0240 (18)	−0.0272 (19)
C34	0.019 (2)	0.025 (2)	0.021 (2)	0.006 (2)	−0.0042 (18)	−0.014 (2)
N2	0.039 (3)	0.031 (2)	0.030 (2)	−0.003 (2)	−0.0034 (19)	−0.016 (2)
C3	0.033 (3)	0.052 (4)	0.046 (3)	−0.004 (3)	0.006 (2)	−0.032 (3)
O2	0.047 (2)	0.031 (2)	0.0312 (19)	−0.0057 (17)	0.0045 (16)	−0.0216 (17)
O9	0.090 (3)	0.034 (2)	0.028 (2)	−0.021 (2)	−0.022 (2)	−0.0065 (17)
C35	0.028 (3)	0.023 (2)	0.024 (2)	−0.004 (2)	−0.004 (2)	−0.012 (2)
C41	0.023 (2)	0.019 (2)	0.027 (2)	0.000 (2)	−0.0026 (19)	−0.012 (2)
C23	0.026 (3)	0.022 (2)	0.019 (2)	0.004 (2)	−0.0033 (19)	−0.010 (2)
C30	0.028 (3)	0.022 (3)	0.046 (3)	0.003 (2)	−0.006 (2)	−0.014 (2)
C25	0.021 (2)	0.018 (2)	0.025 (2)	0.001 (2)	−0.0017 (19)	−0.010 (2)
C5	0.053 (4)	0.030 (3)	0.034 (3)	−0.007 (3)	−0.010 (3)	−0.014 (2)
C33	0.035 (3)	0.022 (2)	0.031 (3)	0.000 (2)	−0.008 (2)	−0.016 (2)

C39	0.033 (3)	0.040 (3)	0.023 (3)	−0.007 (3)	−0.003 (2)	−0.013 (2)
C42	0.052 (3)	0.021 (3)	0.027 (3)	−0.004 (2)	−0.009 (2)	−0.010 (2)
O7	0.053 (2)	0.038 (2)	0.0282 (19)	−0.0107 (19)	−0.0119 (17)	−0.0096 (17)
C36	0.038 (3)	0.025 (3)	0.024 (3)	0.001 (2)	−0.006 (2)	−0.014 (2)
C19	0.023 (2)	0.018 (2)	0.015 (2)	0.001 (2)	−0.0015 (18)	−0.0077 (19)
C21	0.026 (3)	0.032 (3)	0.036 (3)	−0.002 (2)	−0.006 (2)	−0.023 (2)
O8	0.054 (2)	0.053 (3)	0.0258 (19)	−0.001 (2)	−0.0044 (17)	−0.0116 (19)
C20	0.023 (3)	0.033 (3)	0.036 (3)	0.004 (2)	−0.001 (2)	−0.024 (2)
C2	0.036 (3)	0.048 (4)	0.041 (3)	0.010 (3)	−0.005 (2)	−0.029 (3)
C37	0.038 (3)	0.025 (3)	0.030 (3)	0.007 (2)	−0.015 (2)	−0.013 (2)
C32	0.020 (2)	0.023 (2)	0.022 (2)	0.004 (2)	−0.0025 (18)	−0.013 (2)
C24	0.050 (3)	0.021 (2)	0.025 (3)	−0.003 (2)	−0.007 (2)	−0.012 (2)
Ni2	0.0292 (5)	0.0196 (4)	0.0220 (4)	0.0031 (4)	−0.0055 (4)	−0.0117 (4)
O1W	0.073 (3)	0.036 (2)	0.033 (2)	−0.014 (2)	−0.0077 (19)	−0.0169 (18)
N3	0.042 (3)	0.027 (2)	0.023 (2)	−0.007 (2)	−0.0027 (18)	−0.0130 (18)
C10	0.049 (3)	0.044 (3)	0.031 (3)	−0.007 (3)	−0.005 (2)	−0.024 (3)
N4	0.046 (3)	0.031 (2)	0.040 (3)	−0.002 (2)	−0.009 (2)	−0.019 (2)
C9	0.041 (3)	0.047 (3)	0.037 (3)	−0.002 (3)	0.001 (2)	−0.025 (3)
C7	0.046 (3)	0.041 (3)	0.037 (3)	0.001 (3)	−0.014 (3)	−0.016 (3)
C6	0.050 (3)	0.029 (3)	0.031 (3)	0.001 (3)	−0.015 (2)	−0.016 (2)
C8	0.047 (4)	0.039 (3)	0.043 (3)	0.002 (3)	−0.009 (3)	−0.020 (3)
Ni3	0.0357 (5)	0.0258 (5)	0.0275 (5)	−0.0032 (4)	−0.0080 (4)	−0.0148 (4)
O2W	0.046 (2)	0.043 (2)	0.034 (2)	−0.0071 (19)	−0.0093 (17)	−0.0240 (18)
N5	0.058 (3)	0.045 (3)	0.039 (3)	−0.017 (3)	−0.008 (2)	−0.023 (2)
O7W	0.037 (2)	0.102 (4)	0.104 (4)	0.006 (2)	−0.008 (2)	−0.089 (3)
N6	0.050 (3)	0.050 (3)	0.056 (3)	0.008 (3)	−0.023 (2)	−0.037 (3)
C14	0.042 (4)	0.071 (5)	0.079 (5)	0.013 (3)	−0.014 (3)	−0.059 (4)
O6W	0.052 (3)	0.059 (3)	0.042 (2)	0.004 (2)	−0.0005 (19)	−0.023 (2)
O5W	0.035 (2)	0.041 (2)	0.041 (2)	−0.0007 (18)	−0.0030 (16)	−0.0199 (18)
O1	0.045 (2)	0.0221 (18)	0.0296 (19)	0.0041 (16)	−0.0121 (16)	−0.0115 (15)
O6	0.042 (2)	0.026 (2)	0.044 (2)	−0.0058 (18)	−0.0156 (17)	−0.0044 (17)
C17	0.023 (2)	0.022 (2)	0.021 (2)	0.000 (2)	0.0004 (19)	−0.010 (2)
C16	0.028 (3)	0.020 (2)	0.019 (2)	0.000 (2)	−0.0066 (19)	−0.010 (2)
C18	0.025 (3)	0.022 (2)	0.028 (3)	−0.004 (2)	−0.005 (2)	−0.014 (2)
C40	0.025 (3)	0.034 (3)	0.031 (3)	0.001 (2)	−0.007 (2)	−0.015 (2)
C1	0.048 (3)	0.030 (3)	0.043 (3)	0.010 (3)	−0.016 (3)	−0.025 (3)
C28	0.036 (3)	0.027 (3)	0.032 (3)	−0.007 (2)	0.009 (2)	−0.017 (2)
C31	0.021 (3)	0.024 (3)	0.041 (3)	0.000 (2)	0.004 (2)	−0.011 (2)
C13	0.090 (5)	0.038 (4)	0.060 (4)	0.013 (4)	−0.044 (4)	−0.020 (3)
C15	0.041 (4)	0.070 (5)	0.058 (4)	−0.014 (3)	0.006 (3)	−0.046 (4)
C11	0.087 (5)	0.057 (4)	0.042 (4)	−0.035 (4)	−0.005 (3)	−0.017 (3)
C27	0.035 (3)	0.023 (3)	0.031 (3)	0.000 (2)	0.006 (2)	−0.013 (2)
C12	0.106 (6)	0.034 (4)	0.048 (4)	−0.015 (4)	−0.034 (4)	−0.002 (3)
C4	0.039 (3)	0.035 (3)	0.034 (3)	−0.003 (3)	−0.011 (2)	−0.014 (3)

Geometric parameters (Å, º)

Ni1—N1^{i}	2.067 (4)	C24—H24A	0.9800
Ni1—N1	2.067 (4)	C24—H24B	0.9800
Ni1—N2	2.064 (4)	C24—H24C	0.9800
Ni1—N2^{i}	2.064 (4)	Ni2—N3^{ii}	2.072 (4)
Ni1—O1^{i}	2.134 (3)	Ni2—N3	2.072 (4)
Ni1—O1	2.134 (3)	Ni2—N4	2.076 (4)
P2—O5	1.495 (4)	Ni2—N4^{ii}	2.076 (4)
P2—C29	1.804 (5)	Ni2—O1W^{ii}	2.105 (4)
P2—O4	1.502 (3)	Ni2—O1W	2.105 (4)
P2—O6	1.576 (4)	O1W—H1WA	0.8701
P1—O3	1.570 (4)	O1W—H1WB	0.8691
P1—O2	1.518 (3)	N3—H3	1.0000
P1—O1	1.483 (3)	N3—C10	1.481 (6)
P1—C16	1.811 (5)	N3—C6	1.479 (6)
P3—C38	1.813 (5)	C10—H10A	0.9900
P3—O9	1.562 (4)	C10—H10B	0.9900
P3—O7	1.506 (4)	C10—C9^{ii}	1.496 (7)
P3—O8	1.499 (4)	N4—H4	1.0000
C29—C30	1.391 (7)	N4—C9	1.486 (6)
C29—C28	1.381 (7)	N4—C8	1.457 (7)
O3W—H3WA	0.8523	C9—H9A	0.9900
O3W—H3WB	0.8700	C9—H9B	0.9900
O4W—H4WA	0.8687	C7—H7A	0.9900
O4W—H4WB	0.8702	C7—H7B	0.9900
C22—C41	1.400 (6)	C7—C6	1.504 (7)
C22—C23	1.402 (6)	C7—C8	1.513 (7)
C22—C19	1.497 (6)	C6—H6A	0.9900
C38—C39	1.380 (7)	C6—H6B	0.9900
C38—C37	1.390 (7)	C8—H8A	0.9900
C26—C25	1.488 (6)	C8—H8B	0.9900
C26—C31	1.388 (6)	Ni3—N5	2.070 (4)
C26—C27	1.392 (6)	Ni3—N5^{iii}	2.070 (5)
N1—H1	1.0000	Ni3—N6	2.056 (5)
N1—C5	1.482 (6)	Ni3—N6^{iii}	2.056 (5)
N1—C1	1.474 (6)	Ni3—O2W^{iii}	2.137 (3)
O3—H3C	0.8400	Ni3—O2W	2.136 (3)
C34—C35	1.497 (6)	O2W—H2WA	0.8638
C34—C41	1.414 (6)	O2W—H2WB	0.8553
C34—C32	1.401 (6)	N5—H5	1.0000
N2—H2	1.0000	N5—C15	1.497 (7)
N2—C3	1.469 (6)	N5—C11	1.431 (8)
N2—C4	1.477 (6)	O7W—H7WA	0.8613
C3—H3A	0.9900	O7W—H7WB	0.8521
C3—H3B	0.9900	N6—H6	1.0000
C3—C2	1.521 (7)	N6—C14	1.452 (7)
O9—H9C	0.8400	N6—C13	1.487 (8)

C35—C36	1.368 (7)	C14—H14A	0.9900
C35—C40	1.392 (6)	C14—H14B	0.9900
C41—C42	1.507 (6)	C14—C15^{iii}	1.507 (9)
C23—C25	1.396 (6)	O6W—H6WA	0.8706
C23—C24	1.510 (6)	O6W—H6WB	0.8688
C30—H30	0.9500	O5W—H5WA	0.8696
C30—C31	1.382 (7)	O5W—H5WB	0.8704
C25—C32	1.409 (6)	O6—H6C	0.8400
C5—H5A	0.9900	C17—H17	0.9500
C5—H5B	0.9900	C17—C16	1.387 (6)
C5—C4^{i}	1.513 (7)	C17—C18	1.396 (6)
C33—H33A	0.9800	C18—H18	0.9500
C33—H33B	0.9800	C40—H40	0.9500
C33—H33C	0.9800	C1—H1A	0.9900
C33—C32	1.506 (6)	C1—H1B	0.9900
C39—H39	0.9500	C28—H28	0.9500
C39—C40	1.399 (6)	C28—C27	1.375 (7)
C42—H42A	0.9800	C31—H31	0.9500
C42—H42B	0.9800	C13—H13A	0.9900
C42—H42C	0.9800	C13—H13B	0.9900
C36—H36	0.9500	C13—C12	1.506 (9)
C36—C37	1.385 (6)	C15—H15A	0.9900
C19—C20	1.391 (6)	C15—H15B	0.9900
C19—C18	1.393 (6)	C11—H11A	0.9900
C21—H21	0.9500	C11—H11B	0.9900
C21—C20	1.389 (6)	C11—C12	1.514 (10)
C21—C16	1.398 (6)	C27—H27	0.9500
C20—H20	0.9500	C12—H12A	0.9900
C2—H2A	0.9900	C12—H12B	0.9900
C2—H2B	0.9900	C4—H4A	0.9900
C2—C1	1.513 (7)	C4—H4B	0.9900
C37—H37	0.9500		

N1—Ni1—N1^{i}	180.0	N4—Ni2—O1W	89.10 (17)
N1—Ni1—O1^{i}	91.79 (14)	N4^{ii}—Ni2—O1W	90.90 (17)
N1^{i}—Ni1—O1^{i}	88.21 (14)	N4^{ii}—Ni2—O1W^{ii}	89.10 (17)
N1^{i}—Ni1—O1	91.79 (14)	N4—Ni2—N4^{ii}	180.00 (13)
N1—Ni1—O1	88.21 (14)	Ni2—O1W—H1WA	106.8
N1—Ni1—N2^{i}	85.31 (16)	Ni2—O1W—H1WB	108.1
N1^{i}—Ni1—N2	85.31 (16)	H1WA—O1W—H1WB	104.5
N1^{i}—Ni1—N2^{i}	94.69 (16)	Ni2—N3—H3	107.3
N1—Ni1—N2	94.69 (16)	C10—N3—Ni2	105.7 (3)
N2^{i}—Ni1—N2	180.0	C10—N3—H3	107.3
N2^{i}—Ni1—O1	90.47 (15)	C6—N3—Ni2	114.6 (3)
N2—Ni1—O1	89.53 (15)	C6—N3—H3	107.3
N2^{i}—Ni1—O1^{i}	89.53 (15)	C6—N3—C10	114.3 (4)
N2—Ni1—O1^{i}	90.47 (15)	N3—C10—H10A	109.9
O1^{i}—Ni1—O1	180.0	N3—C10—H10B	109.9

O5—P2—C29	110.3 (2)	N3—C10—C9^{ii}	109.1 (4)
O5—P2—O4	115.4 (2)	H10A—C10—H10B	108.3
O5—P2—O6	111.3 (2)	C9^{ii}—C10—H10A	109.9
O4—P2—C29	108.0 (2)	C9^{ii}—C10—H10B	109.9
O4—P2—O6	110.5 (2)	Ni2—N4—H4	106.9
O6—P2—C29	100.2 (2)	C9—N4—Ni2	106.4 (3)
O3—P1—C16	102.1 (2)	C9—N4—H4	106.9
O2—P1—O3	110.2 (2)	C8—N4—Ni2	115.2 (3)
O2—P1—C16	107.1 (2)	C8—N4—H4	106.9
O1—P1—O3	111.4 (2)	C8—N4—C9	114.0 (4)
O1—P1—O2	115.2 (2)	C10^{ii}—C9—H9A	110.1
O1—P1—C16	110.06 (19)	C10^{ii}—C9—H9B	110.1
O9—P3—C38	101.5 (2)	N4—C9—C10^{ii}	108.1 (4)
O7—P3—C38	107.6 (2)	N4—C9—H9A	110.1
O7—P3—O9	109.8 (2)	N4—C9—H9B	110.1
O8—P3—C38	109.4 (2)	H9A—C9—H9B	108.4
O8—P3—O9	111.8 (2)	H7A—C7—H7B	107.3
O8—P3—O7	115.7 (2)	C6—C7—H7A	108.1
C30—C29—P2	120.9 (4)	C6—C7—H7B	108.1
C28—C29—P2	121.0 (4)	C6—C7—C8	116.9 (5)
C28—C29—C30	118.0 (4)	C8—C7—H7A	108.1
H3WA—O3W—H3WB	98.3	C8—C7—H7B	108.1
H4WA—O4W—H4WB	104.4	N3—C6—C7	112.6 (4)
C41—C22—C23	120.1 (4)	N3—C6—H6A	109.1
C41—C22—C19	118.6 (4)	N3—C6—H6B	109.1
C23—C22—C19	120.9 (4)	C7—C6—H6A	109.1
C39—C38—P3	121.3 (4)	C7—C6—H6B	109.1
C39—C38—C37	118.6 (4)	H6A—C6—H6B	107.8
C37—C38—P3	120.1 (4)	N4—C8—C7	111.9 (5)
C31—C26—C25	123.6 (4)	N4—C8—H8A	109.2
C31—C26—C27	116.3 (4)	N4—C8—H8B	109.2
C27—C26—C25	120.1 (4)	C7—C8—H8A	109.2
Ni1—N1—H1	106.9	C7—C8—H8B	109.2
C5—N1—Ni1	104.8 (3)	H8A—C8—H8B	107.9
C5—N1—H1	106.9	O2W—Ni3—O2W^{iii}	180.0
C1—N1—Ni1	116.4 (3)	N5^{iii}—Ni3—O2W	91.54 (15)
C1—N1—H1	106.9	N5—Ni3—O2W	88.46 (15)
C1—N1—C5	114.2 (4)	N5^{iii}—Ni3—O2W^{iii}	88.46 (15)
P1—O3—H3C	109.5	N5—Ni3—O2W^{iii}	91.54 (15)
C41—C34—C35	117.8 (4)	N5—Ni3—N5^{iii}	180.0
C32—C34—C35	121.4 (4)	N6—Ni3—O2W	89.19 (16)
C32—C34—C41	120.8 (4)	N6^{iii}—Ni3—O2W	90.81 (16)
Ni1—N2—H2	106.6	N6^{iii}—Ni3—O2W^{iii}	89.19 (16)
C3—N2—Ni1	116.1 (3)	N6—Ni3—O2W^{iii}	90.81 (16)
C3—N2—H2	106.6	N5—Ni3—N6^{iii}	85.2 (2)
C3—N2—C4	114.4 (4)	N5—Ni3—N6	94.8 (2)
C4—N2—Ni1	106.0 (3)	N5^{iii}—Ni3—N6	85.2 (2)
C4—N2—H2	106.6	N5^{iii}—Ni3—N6^{iii}	94.8 (2)

N2—C3—H3A	109.1	N6^{iii}—Ni3—N6	180.0
N2—C3—H3B	109.1	Ni3—O2W—H2WA	110.2
N2—C3—C2	112.3 (4)	Ni3—O2W—H2WB	109.7
H3A—C3—H3B	107.9	H2WA—O2W—H2WB	103.1
C2—C3—H3A	109.1	Ni3—N5—H5	106.1
C2—C3—H3B	109.1	C15—N5—Ni3	105.8 (4)
P3—O9—H9C	109.5	C15—N5—H5	106.1
C36—C35—C34	119.7 (4)	C11—N5—Ni3	117.4 (4)
C36—C35—C40	118.5 (4)	C11—N5—H5	106.1
C40—C35—C34	121.7 (4)	C11—N5—C15	114.5 (5)
C22—C41—C34	119.4 (4)	H7WA—O7W—H7WB	94.0
C22—C41—C42	119.5 (4)	Ni3—N6—H6	106.1
C34—C41—C42	121.1 (4)	C14—N6—Ni3	107.9 (4)
C22—C23—C24	118.7 (4)	C14—N6—H6	106.1
C25—C23—C22	120.0 (4)	C14—N6—C13	114.0 (5)
C25—C23—C24	121.2 (4)	C13—N6—Ni3	115.9 (4)
C29—C30—H30	119.4	C13—N6—H6	106.1
C31—C30—C29	121.2 (5)	N6—C14—H14A	109.8
C31—C30—H30	119.4	N6—C14—H14B	109.8
C23—C25—C26	118.9 (4)	N6—C14—C15^{iii}	109.4 (5)
C23—C25—C32	120.8 (4)	H14A—C14—H14B	108.2
C32—C25—C26	120.2 (4)	C15^{iii}—C14—H14A	109.8
N1—C5—H5A	110.0	C15^{iii}—C14—H14B	109.8
N1—C5—H5B	110.0	H6WA—O6W—H6WB	104.5
N1—C5—C4^{i}	108.4 (4)	H5WA—O5W—H5WB	104.5
H5A—C5—H5B	108.4	P1—O1—Ni1	167.3 (2)
C4^{i}—C5—H5A	110.0	P2—O6—H6C	109.5
C4^{i}—C5—H5B	110.0	C16—C17—H17	119.7
H33A—C33—H33B	109.5	C16—C17—C18	120.5 (4)
H33A—C33—H33C	109.5	C18—C17—H17	119.7
H33B—C33—H33C	109.5	C21—C16—P1	122.4 (4)
C32—C33—H33A	109.5	C17—C16—P1	118.7 (3)
C32—C33—H33B	109.5	C17—C16—C21	118.7 (4)
C32—C33—H33C	109.5	C19—C18—C17	121.1 (4)
C38—C39—H39	119.6	C19—C18—H18	119.5
C38—C39—C40	120.8 (5)	C17—C18—H18	119.5
C40—C39—H39	119.6	C35—C40—C39	120.0 (5)
C41—C42—H42A	109.5	C35—C40—H40	120.0
C41—C42—H42B	109.5	C39—C40—H40	120.0
C41—C42—H42C	109.5	N1—C1—C2	112.0 (4)
H42A—C42—H42B	109.5	N1—C1—H1A	109.2
H42A—C42—H42C	109.5	N1—C1—H1B	109.2
H42B—C42—H42C	109.5	C2—C1—H1A	109.2
C35—C36—H36	119.1	C2—C1—H1B	109.2
C35—C36—C37	121.8 (5)	H1A—C1—H1B	107.9
C37—C36—H36	119.1	C29—C28—H28	120.0
C20—C19—C22	124.0 (4)	C27—C28—C29	120.1 (5)
C20—C19—C18	117.9 (4)	C27—C28—H28	120.0

C18—C19—C22	118.0 (4)	C26—C31—H31	119.3
C20—C21—H21	119.9	C30—C31—C26	121.4 (4)
C20—C21—C16	120.2 (4)	C30—C31—H31	119.3
C16—C21—H21	119.9	N6—C13—H13A	109.2
C19—C20—H20	119.3	N6—C13—H13B	109.2
C21—C20—C19	121.5 (4)	N6—C13—C12	112.1 (5)
C21—C20—H20	119.3	H13A—C13—H13B	107.9
C3—C2—H2A	108.4	C12—C13—H13A	109.2
C3—C2—H2B	108.4	C12—C13—H13B	109.2
H2A—C2—H2B	107.5	N5—C15—C14^{iii}	110.1 (5)
C1—C2—C3	115.4 (4)	N5—C15—H15A	109.6
C1—C2—H2A	108.4	N5—C15—H15B	109.6
C1—C2—H2B	108.4	C14^{iii}—C15—H15A	109.6
C38—C37—H37	120.0	C14^{iii}—C15—H15B	109.6
C36—C37—C38	120.1 (5)	H15A—C15—H15B	108.2
C36—C37—H37	120.0	N5—C11—H11A	109.3
C34—C32—C25	118.8 (4)	N5—C11—H11B	109.3
C34—C32—C33	120.2 (4)	N5—C11—C12	111.5 (5)
C25—C32—C33	121.0 (4)	H11A—C11—H11B	108.0
C23—C24—H24A	109.5	C12—C11—H11A	109.3
C23—C24—H24B	109.5	C12—C11—H11B	109.3
C23—C24—H24C	109.5	C26—C27—H27	118.5
H24A—C24—H24B	109.5	C28—C27—C26	123.0 (5)
H24A—C24—H24C	109.5	C28—C27—H27	118.5
H24B—C24—H24C	109.5	C13—C12—C11	118.5 (6)
O1W^{ii}—Ni2—O1W	180.0	C13—C12—H12A	107.7
N3—Ni2—O1W	88.65 (15)	C13—C12—H12B	107.7
N3^{ii}—Ni2—O1W^{ii}	88.65 (15)	C11—C12—H12A	107.7
N3—Ni2—O1W^{ii}	91.35 (15)	C11—C12—H12B	107.7
N3^{ii}—Ni2—O1W	91.35 (15)	H12A—C12—H12B	107.1
N3—Ni2—N3^{ii}	180.0	N2—C4—C5^{i}	107.4 (4)
N3—Ni2—N4^{ii}	84.66 (16)	N2—C4—H4A	110.2
N3^{ii}—Ni2—N4^{ii}	95.34 (16)	N2—C4—H4B	110.2
N3^{ii}—Ni2—N4	84.66 (16)	C5^{i}—C4—H4A	110.2
N3—Ni2—N4	95.34 (16)	C5^{i}—C4—H4B	110.2
N4—Ni2—O1W^{ii}	90.90 (17)	H4A—C4—H4B	108.5

Ni1—N1—C5—C4^{i}	44.0 (4)	O7—P3—C38—C37	38.0 (5)
Ni1—N1—C1—C2	−55.0 (5)	C36—C35—C40—C39	−2.7 (7)
Ni1—N2—C3—C2	55.3 (5)	C19—C22—C41—C34	169.3 (4)
Ni1—N2—C4—C5^{i}	−42.6 (4)	C19—C22—C41—C42	−9.3 (6)
P2—C29—C30—C31	−177.8 (4)	C19—C22—C23—C25	−169.7 (4)
P2—C29—C28—C27	177.0 (4)	C19—C22—C23—C24	12.3 (6)
P3—C38—C39—C40	−176.5 (4)	O8—P3—C38—C39	−15.9 (5)
P3—C38—C37—C36	177.2 (4)	O8—P3—C38—C37	164.4 (4)
O5—P2—C29—C30	29.0 (5)	C20—C19—C18—C17	−1.5 (7)
O5—P2—C29—C28	−149.2 (4)	C20—C21—C16—P1	173.3 (4)
C29—C30—C31—C26	0.7 (8)	C20—C21—C16—C17	−2.0 (7)

C29—C28—C27—C26	1.1 (8)	C37—C38—C39—C40	3.2 (7)
O4—P2—C29—C30	−98.0 (4)	C32—C34—C35—C36	−105.9 (5)
O4—P2—C29—C28	83.8 (4)	C32—C34—C35—C40	77.4 (6)
C22—C23—C25—C26	175.0 (4)	C32—C34—C41—C22	2.2 (7)
C22—C23—C25—C32	−1.0 (7)	C32—C34—C41—C42	−179.2 (4)
C22—C19—C20—C21	−174.9 (4)	C24—C23—C25—C26	−7.1 (7)
C22—C19—C18—C17	175.5 (4)	C24—C23—C25—C32	176.9 (4)
C38—C39—C40—C35	−0.6 (8)	Ni2—N3—C10—C9^{ii}	43.0 (5)
C26—C25—C32—C34	−177.1 (4)	Ni2—N3—C6—C7	−54.7 (5)
C26—C25—C32—C33	2.1 (7)	Ni2—N4—C9—C10^{ii}	−41.1 (5)
O3—P1—O1—Ni1	149.1 (9)	Ni2—N4—C8—C7	55.8 (5)
O3—P1—C16—C21	25.1 (4)	C10—N3—C6—C7	−176.9 (4)
O3—P1—C16—C17	−159.6 (3)	C9—N4—C8—C7	179.2 (5)
C34—C35—C36—C37	−173.4 (4)	C6—N3—C10—C9^{ii}	170.0 (4)
C34—C35—C40—C39	174.0 (4)	C6—C7—C8—N4	−71.7 (6)
N2—C3—C2—C1	−71.6 (6)	C8—N4—C9—C10^{ii}	−169.2 (4)
C3—N2—C4—C5^{i}	−171.9 (4)	C8—C7—C6—N3	71.3 (6)
C3—C2—C1—N1	71.1 (6)	Ni3—N5—C15—C14^{iii}	37.7 (5)
O2—P1—O1—Ni1	22.6 (10)	Ni3—N5—C11—C12	−53.9 (6)
O2—P1—C16—C21	141.0 (4)	Ni3—N6—C14—C15^{iii}	−39.2 (5)
O2—P1—C16—C17	−43.8 (4)	Ni3—N6—C13—C12	52.9 (6)
O9—P3—C38—C39	102.4 (4)	N5—C11—C12—C13	69.6 (7)
O9—P3—C38—C37	−77.3 (4)	N6—C13—C12—C11	−69.2 (7)
C35—C34—C41—C22	−175.0 (4)	C14—N6—C13—C12	178.9 (5)
C35—C34—C41—C42	3.6 (7)	O1—P1—C16—C21	−93.2 (4)
C35—C34—C32—C25	177.6 (4)	O1—P1—C16—C17	82.0 (4)
C35—C34—C32—C33	−1.6 (7)	O6—P2—C29—C30	146.4 (4)
C35—C36—C37—C38	−0.8 (8)	O6—P2—C29—C28	−31.8 (4)
C41—C22—C23—C25	3.8 (7)	C16—P1—O1—Ni1	−98.5 (10)
C41—C22—C23—C24	−174.2 (4)	C16—C21—C20—C19	−0.2 (7)
C41—C22—C19—C20	85.7 (6)	C16—C17—C18—C19	−0.7 (7)
C41—C22—C19—C18	−91.1 (5)	C18—C19—C20—C21	1.9 (7)
C41—C34—C35—C36	71.2 (6)	C18—C17—C16—P1	−173.0 (3)
C41—C34—C35—C40	−105.5 (5)	C18—C17—C16—C21	2.4 (7)
C41—C34—C32—C25	0.6 (7)	C40—C35—C36—C37	3.4 (7)
C41—C34—C32—C33	−178.6 (4)	C1—N1—C5—C4^{i}	172.6 (4)
C23—C22—C41—C34	−4.4 (7)	C28—C29—C30—C31	0.4 (8)
C23—C22—C41—C42	177.0 (4)	C31—C26—C25—C23	110.8 (5)
C23—C22—C19—C20	−100.7 (5)	C31—C26—C25—C32	−73.2 (6)
C23—C22—C19—C18	82.5 (5)	C31—C26—C27—C28	0.1 (7)
C23—C25—C32—C34	−1.2 (7)	C13—N6—C14—C15^{iii}	−169.4 (5)
C23—C25—C32—C33	178.0 (4)	C15—N5—C11—C12	−179.0 (5)
C30—C29—C28—C27	−1.3 (7)	C11—N5—C15—C14^{iii}	168.7 (5)
C25—C26—C31—C30	178.9 (5)	C27—C26—C25—C23	−69.4 (6)
C25—C26—C27—C28	−179.7 (5)	C27—C26—C25—C32	106.6 (5)
C5—N1—C1—C2	−177.5 (4)	C27—C26—C31—C30	−0.9 (7)

C39—C38—C37—C36	−2.5 (7)	C4—N2—C3—C2	179.3 (5)
O7—P3—C38—C39	−142.3 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O6^{iv}	1.00	2.32	3.196 (5)	146
N2—H2···O6*W*	1.00	2.18	3.039 (6)	143
N3—H3···O7^{v}	1.00	2.13	3.102 (6)	162
N4—H4···O4*W*	1.00	2.06	3.056 (6)	173
N5—H5···O9^{vi}	1.00	2.07	3.003 (6)	155
N6—H6···O7*W*^{ii}	1.00	1.98	2.956 (6)	166
O3—H3*C*···O5^{iv}	0.84	1.84	2.654 (5)	162
O6—H6*C*···O3*W*^{vii}	0.84	1.75	2.550 (5)	159
O9—H9*C*···O4^{viii}	0.84	1.74	2.517 (5)	154
O1*W*—H1*WB*···O7^{v}	0.87	1.81	2.679 (5)	173
O1*W*—H1*WA*···O4*W*	0.87	2.45	3.256 (6)	155
O2*W*—H2*WB*···O4	0.86	1.90	2.729 (5)	164
O2*W*—H2*WA*···O7*W*^{ix}	0.86	1.81	2.675 (6)	174
O3*W*—H3*WB*···O2	0.87	1.81	2.676 (4)	177
O3*W*—H3*WA*···O7^{v}	0.85	1.84	2.689 (5)	174
O4*W*—H4*WB*···O3	0.87	2.26	3.115 (6)	167
O4*W*—H4*WA*···O8^{v}	0.87	1.93	2.796 (6)	172
O5*W*—H5*WB*···O5^{x}	0.87	1.98	2.813 (5)	159
O5*W*—H5*WA*···O8^{xi}	0.87	1.87	2.725 (5)	168
O6*W*—H6*WB*···O2	0.87	2.02	2.799 (6)	149
O6*W*—H6*WA*···O5*W*	0.87	2.00	2.842 (5)	164
O7*W*—H7*WB*···O3*W*	0.85	2.02	2.731 (5)	140
O7*W*—H7*WA*···O5*W*	0.86	1.83	2.688 (5)	173

Symmetry codes: (ii) −*x*+2, −*y*+2, −*z*+1; (iv) *x*, *y*−1, *z*; (v) *x*, *y*, *z*−1; (vi) −*x*+1, −*y*+2, −*z*+2; (vii) *x*, *y*+1, *z*; (viii) *x*, *y*−1, *z*+1; (ix) *x*−1, *y*+1, *z*; (x) *x*+1, *y*−1, *z*; (xi) *x*+1, *y*, *z*−1.