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Poly[[μ_2-*trans*-1,2-di-4-pyridylethylene]hexa-μ_2-oxido-nickel(II)-divanadate(V)]

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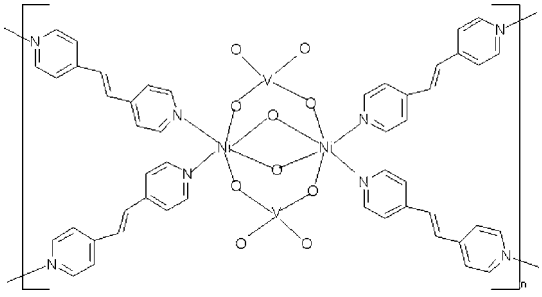
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(V–O) = 0.002 Å; disorder in main residue; R factor = 0.032; wR factor = 0.083; data-to-parameter ratio = 19.0.

The structure of the title compound, $[NiV_2O_6(C_{12}H_{10}N_2)]_n$, is composed of corner-sharing $[V_2O_6]^{2-}$ chains along the c axis, with the $[Ni(bpe)]^{2+}$ (bpe = *trans*-1,2-di-4-pyridylethylene) units covalently attached to every V site through O atoms. The Ni atom is octahedrally coordinated by two pyridyl N atoms from two different bpe ligands, and four O atoms from four different VO_4 tetrahedra. All the C and H atoms are disordered over two positions each; the site occupancy factors are *ca* 0.55 and 0.45.

Related literature

For related literature, see: DeBord *et al.* (1996); Khan (2000); Khan *et al.* (2006); Khan, Yohannes & Doedens (1999); Khan, Yohannes & Powell (1999); Li *et al.* (2003); Yang *et al.* (2001); Zhang *et al.* (1996, 1997, 2007).



Experimental

Crystal data

$[NiV_2O_6(C_{12}H_{10}N_2)]$
M_r = 438.81
Orthorhombic, *Pbcn*
a = 14.862 (3) Å
b = 7.6402 (15) Å
c = 26.947 (5) Å
V = 3059.8 (11) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.44 mm^{-1}
T = 293 (2) K
0.23 × 0.19 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.607, T_{max} = 0.789
27995 measured reflections
3500 independent reflections
3062 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.083
S = 1.03
3500 reflections
184 parameters
33 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.71 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN3050).

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supplementary materials

Acta Cryst. (2007). E**63**, m2233 [**doi:10.1107/S1600536807034277**]

Poly[[μ_2-*trans*-1,2-di-4-pyridylethylene]hexa-μ_2-oxido-nickel(II)divanadate(V)]

C.-C. Wang

Comment

Early transition metal oxide anion clusters (or POMs) are a rapidly growing class of compound. They have been of great interest because of their so-called 'Value-adding properties' and potential applications in such areas as catalysis, gas storage and chemical sensing (Khan, 2000; Khan, Yohannes & Doedens, 1999; Khan, Yohannes & Powell, 1999). Hydrothermal synthesis and structural characterization of vanadium oxide compounds containing Zn, Cu and Co -bipyridyl(or bipyridyl-like) complexes have been intensively studied because of their large structural diversity; for example, discrete zero-dimensional $\{Zn(2,2'\text{-bpy})_2\}_2V_4O_{12}$ (Zhang *et al.*, 1997); one dimensional $[Cu(2,2'\text{-bipy})V_2O_6]$ and $[Cu(2,2'\text{-bipy})_2V_2O_6]$ (De-Bord *et al.*, 1996); two-dimensional $[Zn(2,2'\text{-bipy})_2V_6O_{17}]$ (Zhang *et al.*, 1996); and three-dimensional $Co(bpy)V_2O_6$ (Li *et al.*, 2003), $[Ni_2(4,4'\text{-bipy})_3(H_2O)_2V_4O_{12}]$. $2.5H_2O$ (Yang *et al.*, 2001), $[\{Co_2(4,4'\text{-tmdp})_4\}V_4O_{12}]$ (Khan *et al.*, 2006) and $[Ni(bpp)_2]_2(V_4O_{12})$ (Zhang *et al.*, 2007). The present work reports a new three-dimensional compound, $Ni(bpe)V_2O_6$, which is built from zigzag vanadium oxide chains $[V_2O_6]^{2-}$, and complex nickel (II) cations, $[Ni(bpe)]^{2+}$, as shown in Fig. 1. The N1 atom is coordinated to Ni1, while the N2 atom in the same bpe ligand is coordinated to $Ni1^{vi}$ [Symmetry codes: (vi) *x*, *y* + 1, *z*] in the next layer. Thus, the $[Ni(bpe)]_n^{2n+}$ chains are interpenetrated with each other. It is interesting that the bpe ligands assume two conformation modes, resulting in coexisting of the two conformations in the title compound, 50% probability respectively.

The crystal structure of the title compound can also be described as the neutral two-dimensional $[NiV_2O_6]$ layers (Fig. 1) linked by neutral briding bpe ligand *via* Ni—O bonds to form 3-D framework. The Ni(II) center is octahedrally coordinated by two pyridyl N atoms from two different bpe ligands, and four O atoms from four VO_4 units, as depicted in Fig. 1, in which the N1, O5, $O5^{i}$ and $N2^{ii}$ form the equatorial plane, while the axial position are occupied by O1 and $O4^{i}$. The Ni-centered coordination octahedron is slightly distorted, with the bond lehgths, 2.0562 (19)- 2.0799 (17) Å for Ni—O bonds, 2.069 (2) Å and 2.081 (2) Å for Ni—N bonds, and the bond angles approximate to 90 ° or 180 °.

In the $[V_2O_6]$ units, O2 and O3 bridge two adjacent vanadium atoms; O1 and O4 are coordinated to two nickel atoms respectively; O6 acts as terminal oxygen, while O5 is coordinated to two adjacent Ni atoms. The distance between the adjacent Ni atoms is short, 2.9862 (8) Å, so an eight-membered ring of Ni1—O1—V2—O4—$Ni1^{i}$—$O1^{i}$—$V2^{i}$—$O4^{i}$ is built to form equatorial plane, while the O5 and $O5^{i}$ occupy the axial position.

Experimental

A mixture of $NiCl_2{\cdot}6H_2O$ (0.239 g, 1 mmol), *trans*-1,2-bis(4-pyridyl)ethylene (0.182 g, 1 mmol), NH_4VO_3 (0.117 g, 1 mmol), and H_2O (15 g, 833 mmol) was heated at 140 °C for 120 h. After cooling to room temperature, large green block-like crystals of $Ni(bpe)(V_2O_6)$ were filtered and collected in 86% yield based on Co.

Refinement

All H atoms were fixed geometrically and allowed to ride on their parent carbon atoms, with C—H distances of 0.93 Å and common isotropic displacement parameters (U_{iso} Å^2).

All the C atoms within the (4-pyridyl)ethylene ligand are disordered over two positions with occupancy ratio of 0.55/0.45. This disorder was treated using a constrained refinement using PART and SAME commands available in *SHELXL97* (Sheldrick, 1997). In the last stage of refinement, the C atomss of the disordered moieties were constrained to have the same anisotropic displacement parameters using the EADP instructions.

Figures

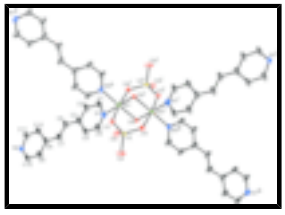


Fig. 1. A partial packing view showing the octahedral envoironnment of the nickel atoms. Ellipsoids are drawn at the 50% probability level. H atom have been omitted for clarity and only one component of the disordered ligand is shown for clarity. [Symmetry code: (i) −*x* + 2, −*y*, −*z* + 1; (ii) *x* − 1/2, *y* − 1/2, −*z* + 1/2; (iii) −*x* + 3/2, −*y* + 1/2, *z* − 1/2)

Poly[[μ$_2$-*trans*-1,2-di-4-pyridylethylene]hexa-μ$_2$– oxidonickel(II)divanadate(V)]

Crystal data

$[NiV_2O_6(C_{12}H_{10}N_2)]$	F_{000} = 1744
M_r = 438.81	D_x = 1.905 Mg m^{-3}
Orthorhombic, *Pbcn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2n 2ab	Cell parameters from 21006 reflections
a = 14.862 (3) Å	θ = 3.0–27.5º
b = 7.6402 (15) Å	μ = 2.44 mm^{-1}
c = 26.947 (5) Å	*T* = 293 (2) K
V = 3059.8 (11) Å^3	Block, green
Z = 8	0.23 × 0.19 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	3500 independent reflections
Radiation source: rotating anode	3062 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 293(2) K	θ_{max} = 27.5º
oscillation scans	θ_{min} = 3.0º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −19→18
T_{min} = 0.607, T_{max} = 0.789	*k* = −9→9

27995 measured reflections

$l = -34 \rightarrow 33$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.083$

$S = 1.03$

3500 reflections

184 parameters

33 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

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 5.844P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.80$ e Å^{-3}

$\Delta\rho_{min} = -0.71$ e Å^{-3}

Extinction correction: none

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}	Occ. (<1)
Ni1	1.000877 (19)	0.03138 (4)	0.445312 (11)	0.01140 (9)	
O1	1.08060 (12)	−0.1892 (2)	0.44057 (6)	0.0187 (4)	
O2	1.24362 (13)	−0.2685 (3)	0.48670 (8)	0.0310 (5)	
O3	1.10701 (13)	−0.5327 (2)	0.47517 (7)	0.0230 (4)	
O4	1.08061 (12)	−0.2425 (2)	0.53989 (6)	0.0194 (4)	
O5	1.07726 (11)	0.1096 (2)	0.50603 (6)	0.0138 (3)	
O6	1.15705 (16)	0.3446 (3)	0.56686 (8)	0.0360 (5)	
V1	1.14782 (3)	0.28571 (5)	0.510151 (15)	0.01393 (11)	
V2	1.12514 (3)	−0.30379 (5)	0.486666 (15)	0.01319 (11)	
N1	1.08372 (17)	0.1648 (4)	0.39593 (8)	0.0304 (6)	
C1	1.0407 (4)	0.2985 (8)	0.3690 (2)	0.0322 (4)	0.55
H1	0.9873	0.3472	0.3806	0.039*	0.55
C2	1.0790 (5)	0.3575 (8)	0.32457 (19)	0.0322 (4)	0.55
H2	1.0485	0.4386	0.3051	0.039*	0.55
C3	1.1633 (5)	0.2942 (8)	0.3095 (2)	0.0322 (4)	0.55
C4	1.2067 (4)	0.1802 (8)	0.3419 (2)	0.0322 (4)	0.55

H4	1.2648	0.1426	0.3349	0.039*	0.55
C5	1.1639 (5)	0.1224 (10)	0.3842 (2)	0.0322 (4)	0.55
H5	1.1954	0.0484	0.4054	0.039*	0.55
C6	1.2037 (4)	0.3516 (8)	0.2623 (2)	0.0322 (4)	0.55
H6	1.1675	0.4143	0.2405	0.039*	0.55
C7	1.2868 (4)	0.3206 (8)	0.2489 (2)	0.0322 (4)	0.55
H7	1.3222	0.2615	0.2718	0.039*	0.55
C8	1.3312 (4)	0.3678 (9)	0.2018 (2)	0.0322 (4)	0.55
C9	1.4152 (4)	0.3013 (8)	0.1912 (2)	0.0322 (4)	0.55
H9	1.4426	0.2244	0.2133	0.039*	0.55
C10	1.4589 (5)	0.3481 (9)	0.1482 (2)	0.0322 (4)	0.55
H10	1.5153	0.3004	0.1416	0.039*	0.55
N2	1.42232 (15)	0.4621 (3)	0.11494 (8)	0.0228 (5)	
C11	1.3373 (5)	0.5090 (15)	0.1216 (3)	0.0322 (4)	0.55
H11	1.3080	0.5702	0.0965	0.039*	0.55
C12	1.2906 (4)	0.4698 (9)	0.1648 (2)	0.0322 (4)	0.55
H12	1.2323	0.5112	0.1692	0.039*	0.55
C1A	1.0590 (6)	0.2575 (13)	0.3583 (3)	0.0416 (7)	0.45
H1A	0.9990	0.2917	0.3564	0.050*	0.45
C2A	1.1169 (6)	0.3095 (13)	0.3203 (3)	0.0416 (7)	0.45
H2A	1.0962	0.3824	0.2951	0.050*	0.45
C3A	1.2059 (6)	0.2521 (13)	0.3201 (3)	0.0416 (7)	0.45
C4A	1.2356 (5)	0.1556 (12)	0.3599 (3)	0.0416 (7)	0.45
H4A	1.2962	0.1278	0.3635	0.050*	0.45
C5A	1.1726 (6)	0.1008 (16)	0.3948 (3)	0.0416 (7)	0.45
H5A	1.1899	0.0184	0.4183	0.050*	0.45
C6A	1.2666 (5)	0.3011 (11)	0.2792 (2)	0.0416 (7)	0.45
H6A	1.3279	0.2905	0.2855	0.050*	0.45
C7A	1.2445 (6)	0.3571 (12)	0.2358 (2)	0.0416 (7)	0.45
H7A	1.1834	0.3723	0.2297	0.050*	0.45
C8A	1.3068 (5)	0.3995 (14)	0.1947 (3)	0.0416 (7)	0.45
C9A	1.3985 (5)	0.3595 (13)	0.1970 (3)	0.0416 (7)	0.45
H9A	1.4230	0.3118	0.2257	0.050*	0.45
C10A	1.4533 (6)	0.3920 (13)	0.1556 (3)	0.0416 (7)	0.45
H10A	1.5139	0.3624	0.1573	0.050*	0.45
C11A	1.3369 (7)	0.514 (2)	0.1146 (4)	0.0416 (7)	0.45
H11A	1.3176	0.5816	0.0879	0.050*	0.45
C12A	1.2752 (6)	0.4754 (14)	0.1509 (3)	0.0416 (7)	0.45
H12A	1.2143	0.4989	0.1465	0.050*	0.45

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01027 (15)	0.01366 (17)	0.01028 (16)	−0.00039 (11)	0.00018 (10)	0.00108 (11)
O1	0.0176 (9)	0.0193 (10)	0.0193 (8)	0.0044 (7)	0.0003 (7)	−0.0010 (7)
O2	0.0108 (9)	0.0283 (11)	0.0540 (13)	−0.0018 (8)	−0.0035 (9)	0.0026 (10)
O3	0.0249 (10)	0.0123 (9)	0.0317 (10)	−0.0005 (7)	−0.0049 (8)	−0.0013 (7)
O4	0.0206 (9)	0.0167 (9)	0.0210 (9)	0.0048 (7)	−0.0020 (7)	0.0011 (7)

O5	0.0104 (8)	0.0149 (9)	0.0162 (8)	−0.0033 (7)	−0.0002 (6)	0.0000 (6)
O6	0.0485 (14)	0.0337 (13)	0.0260 (10)	−0.0179 (11)	−0.0051 (10)	−0.0060 (9)
V1	0.0098 (2)	0.0119 (2)	0.0201 (2)	−0.00288 (15)	−0.00073 (15)	−0.00112 (15)
V2	0.00889 (19)	0.0101 (2)	0.0206 (2)	0.00108 (14)	−0.00144 (15)	−0.00086 (15)
N1	0.0322 (14)	0.0399 (15)	0.0189 (11)	−0.0144 (12)	0.0053 (10)	0.0043 (10)
C1	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C2	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C3	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C4	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C5	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C6	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C7	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C8	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C9	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C10	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
N2	0.0208 (11)	0.0324 (14)	0.0152 (10)	−0.0036 (10)	0.0025 (9)	0.0019 (9)
C11	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C12	0.0380 (10)	0.0327 (10)	0.0257 (9)	0.0049 (8)	0.0186 (8)	0.0072 (7)
C1A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C2A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C3A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C4A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C5A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C6A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C7A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C8A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C9A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C10A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C11A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)
C12A	0.0366 (13)	0.0581 (18)	0.0303 (12)	−0.0040 (12)	0.0104 (10)	0.0100 (12)

Geometric parameters (Å, °)

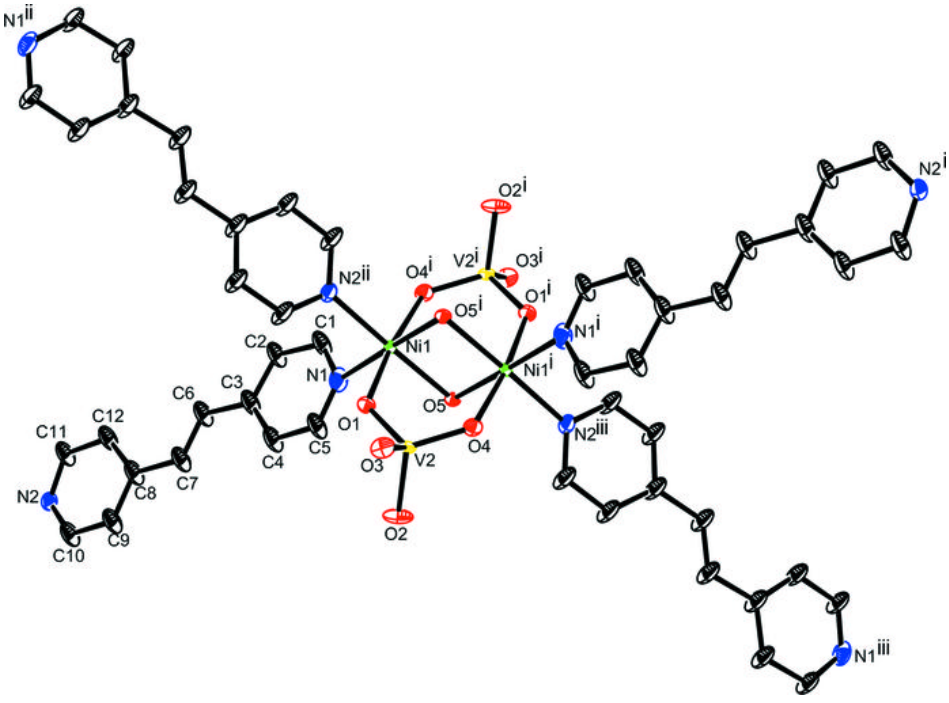
Ni1—O4^{i}	2.0558 (19)	C7—H7	0.9300
Ni1—O5^{i}	2.0561 (17)	C8—C9	1.378 (8)
Ni1—O1	2.0641 (19)	C8—C12	1.402 (8)
Ni1—N2^{ii}	2.069 (2)	C9—C10	1.377 (7)
Ni1—O5	2.0790 (16)	C9—H9	0.9300
Ni1—N1	2.080 (2)	C10—N2	1.362 (7)
Ni1—Ni1^{i}	2.9862 (8)	C10—H10	0.9300
O1—V2	1.6575 (18)	N2—C10A	1.304 (9)
O2—V2	1.781 (2)	N2—C11	1.326 (7)
O2—V1^{iii}	1.782 (2)	N2—C11A	1.331 (10)
O3—V1^{iv}	1.783 (2)	N2—Ni1^{vii}	2.069 (2)
O3—V2	1.7966 (19)	C11—C12	1.388 (7)
O4—V2	1.6476 (18)	C11—H11	0.9300
O4—Ni1^{i}	2.0558 (19)	C12—H12	0.9300
O5—V1	1.7098 (17)	C1A—C2A	1.397 (9)

O5—Ni1^{i}	2.0561 (17)	C1A—H1A	0.9300
O6—V1	1.599 (2)	C2A—C3A	1.393 (11)
V1—O2^{v}	1.782 (2)	C2A—H2A	0.9300
V1—O3^{vi}	1.783 (2)	C3A—C4A	1.376 (10)
N1—C5	1.275 (7)	C3A—C6A	1.472 (9)
N1—C1A	1.290 (8)	C4A—C5A	1.391 (9)
N1—C1	1.407 (6)	C4A—H4A	0.9300
N1—C5A	1.409 (9)	C5A—H5A	0.9300
C1—C2	1.401 (7)	C6A—C7A	1.288 (9)
C1—H1	0.9300	C6A—H6A	0.9300
C2—C3	1.403 (8)	C7A—C8A	1.480 (9)
C2—H2	0.9300	C7A—H7A	0.9300
C3—C4	1.391 (8)	C8A—C12A	1.395 (9)
C3—C6	1.472 (6)	C8A—C9A	1.398 (10)
C4—C5	1.379 (7)	C9A—C10A	1.402 (9)
C4—H4	0.9300	C9A—H9A	0.9300
C5—H5	0.9300	C10A—H10A	0.9300
C6—C7	1.309 (7)	C11A—C12A	1.374 (9)
C6—H6	0.9300	C11A—H11A	0.9300
C7—C8	1.474 (6)	C12A—H12A	0.9300
O4^{i}—Ni1—O5^{i}	87.39 (7)	C7—C6—H6	117.6
O4^{i}—Ni1—O1	172.16 (7)	C3—C6—H6	117.6
O5^{i}—Ni1—O1	86.33 (7)	C6—C7—C8	128.1 (6)
O4^{i}—Ni1—N2^{ii}	91.18 (9)	C6—C7—H7	115.9
O5^{i}—Ni1—N2^{ii}	92.73 (8)	C8—C7—H7	115.9
O1—Ni1—N2^{ii}	93.80 (9)	C9—C8—C12	116.6 (5)
O4^{i}—Ni1—O5	86.77 (7)	C9—C8—C7	119.6 (5)
O5^{i}—Ni1—O5	87.53 (7)	C12—C8—C7	123.7 (5)
O1—Ni1—O5	88.27 (7)	C10—C9—C8	120.4 (6)
N2^{ii}—Ni1—O5	177.92 (9)	C10—C9—H9	119.8
O4^{i}—Ni1—N1	95.08 (10)	C8—C9—H9	119.8
O5^{i}—Ni1—N1	177.51 (10)	N2—C10—C9	122.1 (5)
O1—Ni1—N1	91.19 (10)	N2—C10—H10	118.9
N2^{ii}—Ni1—N1	87.56 (10)	C9—C10—H10	118.9
O5—Ni1—N1	92.27 (8)	C10A—N2—C11	109.5 (5)
O4^{i}—Ni1—Ni1^{i}	85.95 (5)	C10A—N2—C11A	117.8 (5)
O5^{i}—Ni1—Ni1^{i}	44.07 (5)	C11—N2—C11A	8.4 (6)
O1—Ni1—Ni1^{i}	86.27 (5)	C10A—N2—C10	17.0 (5)
N2^{ii}—Ni1—Ni1^{i}	136.77 (7)	C11—N2—C10	117.7 (4)
O5—Ni1—Ni1^{i}	43.46 (5)	C11A—N2—C10	125.3 (5)
N1—Ni1—Ni1^{i}	135.68 (7)	C10A—N2—Ni1^{vii}	124.5 (4)
V2—O1—Ni1	127.89 (10)	C11—N2—Ni1^{vii}	125.1 (3)
V2—O2—V1^{iii}	158.52 (14)	C11A—N2—Ni1^{vii}	117.1 (4)
V1^{iv}—O3—V2	127.96 (11)	C10—N2—Ni1^{vii}	117.0 (3)

V2—O4—Ni1^{i}	128.95 (10)	N2—C11—C12	122.1 (6)
V1—O5—Ni1^{i}	135.83 (9)	N2—C11—H11	118.9
V1—O5—Ni1	127.75 (9)	C12—C11—H11	118.9
Ni1^{i}—O5—Ni1	92.47 (7)	C11—C12—C8	120.1 (6)
O6—V1—O5	109.64 (10)	C11—C12—H12	120.0
O6—V1—O2^{v}	109.07 (12)	C8—C12—H12	120.0
O5—V1—O2^{v}	110.44 (9)	N1—C1A—C2A	123.8 (7)
O6—V1—O3^{vi}	108.39 (11)	N1—C1A—H1A	118.1
O5—V1—O3^{vi}	111.67 (9)	C2A—C1A—H1A	118.1
O2^{v}—V1—O3^{vi}	107.54 (10)	C3A—C2A—C1A	119.9 (7)
O4—V2—O1	109.98 (9)	C3A—C2A—H2A	120.0
O4—V2—O2	110.70 (10)	C1A—C2A—H2A	120.0
O1—V2—O2	108.38 (10)	C4A—C3A—C2A	118.1 (6)
O4—V2—O3	111.52 (10)	C4A—C3A—C6A	121.5 (8)
O1—V2—O3	108.97 (9)	C2A—C3A—C6A	120.3 (8)
O2—V2—O3	107.19 (10)	C3A—C4A—C5A	118.2 (7)
C5—N1—C1A	102.2 (5)	C3A—C4A—H4A	120.9
C5—N1—C1	118.8 (4)	C5A—C4A—H4A	120.9
C1A—N1—C1	21.0 (4)	C4A—C5A—N1	122.8 (7)
C5—N1—C5A	14.0 (4)	C4A—C5A—H5A	118.6
C1A—N1—C5A	116.1 (5)	N1—C5A—H5A	118.6
C1—N1—C5A	131.9 (5)	C7A—C6A—C3A	127.4 (8)
C5—N1—Ni1	126.0 (3)	C7A—C6A—H6A	116.3
C1A—N1—Ni1	127.1 (4)	C3A—C6A—H6A	116.3
C1—N1—Ni1	114.6 (3)	C6A—C7A—C8A	126.4 (8)
C5A—N1—Ni1	113.5 (4)	C6A—C7A—H7A	116.8
C2—C1—N1	119.3 (5)	C8A—C7A—H7A	116.8
C2—C1—H1	120.3	C12A—C8A—C9A	117.2 (7)
N1—C1—H1	120.3	C12A—C8A—C7A	120.9 (7)
C1—C2—C3	120.0 (6)	C9A—C8A—C7A	121.9 (7)
C1—C2—H2	120.0	C8A—C9A—C10A	119.5 (7)
C3—C2—H2	120.0	C8A—C9A—H9A	120.3
C4—C3—C2	116.7 (5)	C10A—C9A—H9A	120.3
C4—C3—C6	122.6 (6)	N2—C10A—C9A	122.4 (8)
C2—C3—C6	120.7 (6)	N2—C10A—H10A	118.8
C5—C4—C3	120.4 (6)	C9A—C10A—H10A	118.8
C5—C4—H4	119.8	N2—C11A—C12A	124.5 (8)
C3—C4—H4	119.8	N2—C11A—H11A	117.8
N1—C5—C4	123.7 (5)	C12A—C11A—H11A	117.8
N1—C5—H5	118.2	C11A—C12A—C8A	117.9 (8)
C4—C5—H5	118.2	C11A—C12A—H12A	121.1
C7—C6—C3	124.8 (6)	C8A—C12A—H12A	121.1

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1; (ii) *x*−1/2, *y*−1/2, −*z*+1/2; (iii) −*x*+5/2, *y*−1/2, *z*; (iv) *x*, *y*−1, *z*; (v) −*x*+5/2, *y*+1/2, *z*; (vi) *x*, *y*+1, *z*; (vii) *x*+1/2, *y*+1/2, −*z*+1/2.

Fig. 1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N2ii
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C2
C3
C4
C5
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C7
C8
C9
C10
C11
C12
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Ni1
O1
O2i
O4i
V2i
O3i
O5i
O1i
N1i
Ni1i
O5
O4
N2iii
V2
O3
O2
N2i
N1iii